

# ENVIRONMENTAL PROTECTION AGENCY

## 40 CFR Parts 136 and 141

[FRL-5800-2]

RIN 2040-AC93

### Guidelines Establishing Test Procedures for the Analysis of Pollutants and National Primary Drinking Water Regulations; Flexibility in Existing Test Procedures and Streamlined Proposal of New Test Procedures

**AGENCY:** Environmental Protection Agency.

**ACTION:** Proposed rule.

**SUMMARY:** The Environmental Protection Agency (EPA) proposes to streamline the process for EPA approval of analytical methods (and modifications thereof) under the Clean Water Act (CWA) and the Safe Drinking Water Act (SDWA). The current methods approval process applies to and is used by public and private laboratories, manufacturers of analytical equipment and analysts who modify analytical methods or who develop new methods for use in compliance monitoring under the CWA and SDWA. The proposed rule only affects states if they choose to adopt the proposed streamlined process as part of their laboratory auditing programs. Under the streamlined methods approval system, EPA would increase the analyst's flexibility to modify existing test procedures, expedite approval of new and modified test procedures, establish and require the use of standardized quality control (QC) and QC acceptance criteria in existing and new test procedures, and recommend use of standard data elements for reporting test results. Today's action responds to the Administration's Environmental Technology and Reinventing Government Initiatives and the National Technology Transfer and Advancement Act by promoting use of emerging technologies and encouraging participation of consensus standards organizations and other organizations in developing test procedures (analytical methods). The action proposed in today's rule would increase the options available to the regulated community in complying with EPA regulations under the CWA and SDWA. These actions are only an initial and interim step in the Agency's pursuit of a performance-based approach to environmental measurements, and are not meant to define or limit the Agency's ultimate implementation of a "pure"

performance-based measurement system. The increased flexibility provided by this proposed action should significantly reduce the need for Agency review of alternate test procedures and make it easier for the analyst to select analytical methods that are most suited to specific regulatory measurement needs.

**DATES:** Comments on this proposed rule will be accepted until June 26, 1997.

**ADDRESSES:** Send written comments to the Streamlining Methods Docket Clerk, Water Docket (MC-4101), USEPA, 401 M Street, SW., Washington, DC 20460. Please submit an original and three copies of your comments and enclosures (including references). To ensure that EPA can read, understand and therefore properly respond to comments, the Agency would prefer that commenters cite, where possible, the paragraph(s) or sections in the proposed regulation or in the supporting documents to which each comment refers. Commenters should use a separate paragraph for each issue discussed. Commenters who want EPA to acknowledge receipt of their comments should enclose a self-addressed, stamped envelope. No facsimiles (faxes) or electronic mail (email) will be accepted because EPA cannot ensure that they will be submitted to the Water Docket. A copy of the supporting documents cited in this proposal are available for review at EPA's Water Docket, 401 M Street, SW., Washington, DC 20460. For access to docket materials, call 202/260-3027 between 9:00 a.m. and 3:30 p.m. for an appointment.

**FOR FURTHER INFORMATION CONTACT:** Dr. Richard Reding, USEPA, Office of Ground Water and Drinking Water (MS-140), 26 W. Martin Luther King Drive, Cincinnati, OH 45268, 513/569-7961.

**SUPPLEMENTARY INFORMATION:** The supporting documents that are a part of the administrative record for this proposal may be obtained from the National Center for Environmental Publications and Information (NCEPI) (513/489-8190), from the National Technical Information Service (NTIS) (703/487-4650), from the Educational Resources Information Center (ERIC) (800/276-0462), and via the Internet on the EPA Office of Water home page at <http://www.epa.gov/watthome>. These documents are titled, Guide to Method Flexibility and Approval of EPA Water Methods, December 1996 Draft, EPA-821-D-96-004, NTIS PB97-117766, ERIC D-A43 or D-A46 (diskette) (Streamlining Guide, EPA 1996a), Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, December 1996, EPA-821-B-96-005,

NTIS PB97-125298, ERIC D-A44 or D-A47 (diskette) (Organic Methods, EPA 1996b), and Guidelines and Format for Methods to Be Proposed at 40 CFR Part 136 or Part 141, July 1996, EPA-821-B-96-003, NTIS PB96-210448, ERIC D-A42 or D-A45 (diskette) (Method Guidelines and Format, EPA 1996c).

### Regulated Entities

Entities potentially regulated by this action are those who seek EPA approval of analytical technologies for monitoring under the provisions of the CWA and SDWA. Entities potentially regulated by this action are listed in the table below. These entities potentially include consensus methods organizations that publish compendiums of analytical methods for water, and equipment manufacturers, instrument manufacturers and laboratories that modify compliance methods or seek approval of new methods for compliance monitoring.

Category	Examples of regulated entities
Public .....	Government laboratories that develop analytical methods for compliance with the CWA and the SDWA.
Private .....	Commercial laboratories, consensus methods organizations, instrument manufacturers, vendors, and other entities that develop or publish analytical methods for compliance with the CWA and the SDWA.

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 the 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 organization is likely to be regulated by this action, you should carefully read the applicability language of today's rule at §§ 136.4, 136.5 and 141.27. If you have questions regarding the applicability of this action to a particular entity, consult the individual listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

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## I. Authority

### A. Clean Water Act

The Clean Water Act (CWA) requires the U.S. Environmental Protection Agency (EPA) Administrator to promulgate effluent limitations guidelines for specified categories and classes of point sources. Section 301 of CWA prohibits the discharge of any pollutant into navigable waters unless the discharge complies with a National Pollutant Discharge Elimination System (NPDES) permit issued under CWA section 402. Section 307 requires the EPA Administrator to publish regulations establishing pretreatment

standards for introduction of pollutants into publicly owned treatment works (POTWs). Section 401 requires State and Tribal certification of a federal license that may result in any discharge into the navigable waters.

Section 304(h) of CWA requires the EPA Administrator to promulgate guidelines establishing test procedures for data gathering and for monitoring compliance with published guidelines. EPA's promulgation of analytical methods is authorized under this section of CWA, as well as the general rulemaking authority in CWA section 501(a). The section 304(h) test procedures (analytical methods) are published or incorporated by reference at 40 CFR part 136. They include Methods for Chemical Analysis of Water and Wastes (MCAWW); the EPA 200-, 600-, and 1600-series methods; methods published by consensus standards organizations such as ASTM, AOAC-International, and Standard Methods for the Examination of Water and Wastewater (Standard Methods) published jointly by the American Public Health Association (APHA), the American Water Works Association (AWWA), and the Water Environment Federation (WEF); methods used by the U.S. Geological Survey; methods developed by third parties; and other methods referenced in CWA regulations. These methods support development of effluent limitations guidelines and standards promulgated at 40 CFR parts 405-503, establish compliance with NPDES permits issued under CWA section 402, allow implementation of the pretreatment standards issued under CWA section 307, and apply to the certification of compliance with State water quality standards under CWA section 401.

### B. Safe Drinking Water Act

The Safe Drinking Water Act (SDWA) requires the EPA Administrator to promulgate national primary drinking water regulations (NPDWRs) that specify maximum contaminant levels (MCLs) or treatment techniques for listed drinking water contaminants (section 1412). Section 1445(a) authorizes the Administrator to establish regulations for monitoring to assist in determining whether persons comply with the requirements of SDWA. EPA's promulgation of analytical methods is authorized under these sections of SDWA, as well as the general rulemaking authority in SDWA section 1450(a).

SDWA section 1401(1)(D) specifies that NPDWRs contain criteria and procedures to ensure a supply of drinking water that dependably

complies with MCLs, including quality control (QC) and testing procedures to ensure compliance with such levels and to ensure proper operation and maintenance of drinking water supply and distribution systems. These test procedures are promulgated at 40 CFR part 141 and include three MCAWW methods, the 200-, 300-, and 500-series EPA methods, methods published by consensus standards organizations, and other methods referenced in SDWA regulations. EPA uses these test procedures to establish MCLs under SDWA section 1412 and to establish monitoring requirements under SDWA section 1445(a).

## II. Background and History

### A. Introduction

Within EPA, the Office of Water (OW) publishes analytical methods for use in data gathering and environmental monitoring under the Clean Water Act (CWA) and the Safe Drinking Water Act (SDWA). These methods have been developed by EPA, by consensus standards organizations, and by others. Many of these methods, especially those published before 1988, are prescriptive, with limited flexibility to change technologies to respond to specific situations or to incorporate advances in measurement technology. There has been a growing awareness, both within EPA and in the analytical community, that the requirement to use prescriptive measurement methods to comply with Agency regulations has imposed an unintended regulatory burden and potentially created a barrier to innovation in environmental monitoring.

To reduce this regulatory burden and to lower the barriers to innovation, the Agency in a future rulemaking may propose to adopt a completely performance-based approach to environmental measurements. As envisioned under such an approach, the Agency would specify the question(s) to be answered by the measurement, the decision(s) to be supported by the data, and the level of uncertainty that is acceptable. EPA would specify performance criteria for the measurement and data producers would be required to demonstrate that their proposed measurement system (i.e., methods, sample handling procedures) meets these specific performance criteria. Data producers would be required to document performance and certify that they have used appropriate quality assurance and QC procedures. The system would apply to physical, chemical, and biological measurements

conducted either in laboratories or in the field (EPA 1996d).

In a series of steps designed to adopt the performance-based approach, each program office in the Agency has developed (or will develop) an implementation plan that describes how the performance-based approach would be put into practice. The Agency's goal is to have these implementation plans as consistent as possible (i.e., "harmonized") from program to program (EPA 1996e). The streamlining initiative proposed in today's notice describes how EPA's Office of Water is taking immediate steps to remove some of the regulatory barriers to the use of new technologies for environmental measurements of chemical analytes under the CWA and SDWA. This initiative would use reference chemical methods that contain performance criteria and methods that are already approved at 40 CFR parts 136 and 141. Other implementation approaches to a performance-based measurement system, such as listing in the CFR only the required performance criteria for the measurement, are also possible; these approaches, which are not the subject of today's proposal, may be the subject of future rulemakings.

Today's rule proposes a process that would use standardized QC, QC acceptance criteria, and method validation procedures for stakeholders to gain approval of new and modified methods for compliance monitoring under the SDWA and CWA. Today's rule also proposes to designate certain approved drinking water and wastewater methods as reference methods. The approved reference methods either presently contain QC acceptance criteria, are supplemented with these criteria in today's proposal, or would be supplemented with these criteria in a future rulemaking. In subsequent rulemakings, EPA intends to extend the streamlined method approval process to physical and biological (including microbiological) measurements in the water programs.

Through public meetings, announcements, and technical presentations, EPA's Office of Water has coordinated this streamlining initiative with various EPA Headquarters offices, EPA Regions, the States, other governmental agencies, industry, consensus standards organizations, environmental laboratories, and other interested parties. With today's proposal, EPA attempts to define a comprehensive program to increase analytical choices in selection of compliance monitoring methods and to streamline the procedures for approval of water methods. In this initiative, EPA

seeks to promote rapid introduction of innovative technologies, to encourage non-EPA organizations to participate in the method development and approval process, and to implement procedures to expedite the review and approval of new and modified methods. Most importantly, EPA believes that this initiative also offers the opportunity to improve the quality of environmental monitoring.

The proposed streamlined procedures for approval of water methods would allow analysts to use professional judgement to modify and develop alternatives to established Agency methods and to take advantage of emerging technologies that reduce costs, overcome analytical difficulties, and enhance data quality. The proposal to increase the flexibility to modify reference methods would be governed by QC acceptance criteria designed to ensure that the quality of the environmental data would not be compromised. These criteria would be used to demonstrate that a modified method produces results equal or superior to results produced by the reference method. EPA also proposes to require that all new methods contain such QC acceptance criteria so that modifications could be made to new methods.

EPA believes that allowing reference method modifications and providing rapid approval of new methods would yield several benefits. On behalf of regulated entities, analysts could select the analytical method that yields the best performance in a specific situation. The QC acceptance criteria in the reference method would enable the analyst to document equivalent or superior performance to the satisfaction of reviewing authorities. New technologies could be utilized to overcome matrix interference problems, lower detection limits, improve laboratory productivity, or reduce the amount of hazardous materials used and hazardous wastes produced in the laboratory.

A more flexible method approval program is consistent with the Administration's Environmental Technology and Reinventing Government initiatives and the National Technology Transfer and Advancement Act of 1995 (NTTAA). The proposed program would empower stakeholders while decreasing demands on Agency resources and is intended to accelerate environmental technological innovation while enhancing and maintaining environmental protection. EPA believes that the incentives provided by a more flexible water test methods approval program would spur the development of

new technologies and, with them, new jobs. EPA also anticipates that the use of new technologies may lower the cost of environmental measurements, thereby reducing costs of environmental compliance for American industries and municipalities.

#### *B. Current Office of Water Methods Approval Programs*

Requirements for approval of alternate analytical techniques (methods) are specified at 40 CFR 136.4 and 136.5 for wastewater and at 40 CFR 141.27 for drinking water methods. These requirements are the basis for the Agency's alternative test procedures (ATP) program for water methods. Under the ATP program, persons may request approval to modify steps in a reference method or approval to use a new method. The person that submits the ATP application is responsible for validating the new or modified method. Agency staff review the ATP validation package and, if required, successful applications undergo formal rulemaking. Rulemaking is required when a new or revised method is to be added to the list of approved methods in the CFR. The ATP and rulemaking processes make heavy demands on stakeholder, contractor, EPA, and Office of Federal Register resources. The process can require one to two years to gain approval of a method. Because advances in analytical technology continue to outpace the capacity of OW's methods approval program, the program is slow to respond to emerging technologies and has been underutilized. Under the streamlining initiative described below, EPA proposes to increase method flexibility by amending the procedures at 40 CFR 136.4, 136.5 and 141.27 to specify a more rapid and less resource intensive process for approval of new technologies.

#### *C. Streamlining Initiative*

The proposed streamlining initiative is designed to improve overall resource use while making the method development process more efficient and accessible to non-EPA organizations. The goals of the initiative are to decrease the need for developers of modified methods to use the ATP program and to speed up the approval (or disapproval) of methods subject to ATP review. EPA believes the streamlining initiative would (1) encourage the use of emerging technologies by increasing the flexibility to modify approved methods without formal EPA approval, (2) provide a mechanism for non-EPA organizations to develop and submit new methods for

approval, and (3) expedite the approval of new and modified methods by improving the current ATP program. This initiative applies to approval of wastewater and drinking water methods. Because of current emphases on decreasing redundant activities, forming partnerships with stakeholders, and more quickly adopting advances in technology, EPA believes this is an appropriate time to look to organizations outside of EPA for assistance in developing new methods that take advantage of emerging technologies that reduce costs, overcome analytical difficulties, and enhance data quality. Once the streamlining initiative is in place, EPA expects to increase its reliance on outside organizations as the developers of many new methods. EPA would focus its method development activities on specialized or esoteric methods needed to support regulation development or compliance monitoring.

OW has coordinated the development of the streamlining initiative with various governmental entities, industry, consensus standards organizations, environmental laboratories, and other interested parties. These organizations include the National Environmental Laboratory Accreditation Committee (NELAC), and the Interagency Steering Committee for Quality Assurance for Environmental Measurements, which includes representatives from the Department of Energy, Department of Defense, EPA, Air Force, U.S. Army Corps of Engineers, U.S. Geological Survey (USGS), Bureau of Reclamation, and other organizations.

#### *D. Streamlining Objectives*

The purpose of the streamlining initiative is to implement a more performance-based approach to environmental measurements under the SDWA and CWA. The proposed streamlined methods approval procedures would revolutionize the water methods approval program to expand the flexibility to modify existing methods, provide a mechanism for non-EPA organizations to gain approval of new methods, and expedite the approval of new and modified methods. EPA has defined several specific streamlining objectives:

- Increase the current flexibility to modify approved chemical test procedures (methods) without formal EPA approval; this would allow laboratories to overcome matrix interferences and would facilitate early introduction of innovative technologies.
- Designate a reference method for each unique combination of analyte and determinative technique and establish

standardized QC tests for approved methods to ensure data quality.

- Develop and publish QC acceptance criteria for any reference method that does not have these criteria so that laboratories can demonstrate equivalent or superior performance of a modified method.
- Provide a standard method format and mechanism for validation and approval of new methods to expedite method approval and to increase confidence in the validity of the methods and resulting data.
- Encourage stakeholder participation in method development to keep pace with emerging technologies.
- Harmonize the wastewater and drinking water test procedures to eliminate unnecessary inconsistencies.
- Increase standardized data reporting by recommending use of standard data elements for reporting analytical results for environmental and QC samples.
- Identify and propose withdrawal of outdated or obsolete methods from 40 CFR parts 136 and 141 to modernize approved test methods and to eliminate methods that are no longer published by the issuing government agency, consensus methods organization, or vendor.
- Work with the Office of Federal Register to incorporate more methods by reference to reduce the volume of material published in the CFR while ensuring and improving access to those methods by all interested parties.

#### *E. Public Meetings and Stakeholder Participation in Streamlining Development*

EPA conducted four public meetings to develop a streamlined water test methods approval program. EPA held the meetings in Seattle, Washington, on September 28, 1995; in Boston, Massachusetts, on January 25, 1996; in Chicago, Illinois, on February 14, 1996; and in Denver, Colorado, on July 24, 1996. The purpose of the meetings was to present and discuss EPA's draft of the streamlining initiative and obtain stakeholder advice for refining the streamlining approach prior to proposal.

All meetings were announced in the **Federal Register** in advance. The first meeting, held in Seattle, was announced on September 12, 1995, in a **Federal Register** notice titled, "A Public Meeting and Availability of Documents on Streamlining Approval of Analytical Methods at 40 CFR part 136 and Flexibility in Existing Test Methods" (60 FR 47325). This **Federal Register** notice provided supplementary information regarding the streamlining effort and made available several supporting documents. Subsequent public meetings in Boston and Chicago were announced on December 18, 1995 (60 FR 65207), and the fourth public meeting in Denver was announced on July 10, 1996 (61 FR 36328). The supporting documents and summaries

of the four public meetings are in the rule docket.

In addition to the public meetings, EPA solicited support and expertise from each of the consensus standards organizations and government agencies that developed the methods already approved for use under the wastewater and drinking water programs. These groups include the American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF) as publishers of Standard Methods for the Examination of Water and Wastewater (Standard Methods); ASTM (formerly, American Society for Testing and Materials); AOAC-International (formerly, the Association of Official Analytical Chemists); and the USGS. EPA also provided the opportunity for individuals, the regulated industry, the States, local permitting authorities, vendors, laboratories, and laboratory organizations such as the International Association of Environmental Testing Laboratories (IAETL), to voice opinions at the meetings. The groups offered valuable insight concerning problems with the current program and recommended areas of improvement.

Through the public meeting process and through individual meetings with key stakeholder organizations, EPA received input from more than 400 stakeholders, including all major stakeholder organizations.

Following the first three public meetings, EPA compiled and reviewed preliminary stakeholder advice to assess the initial response to streamlining and revise the approach accordingly. In response to stakeholder suggestions, EPA made the following changes to the streamlining initiative:

- Included drinking water methods (40 CFR part 141);
- Expanded flexibility to allow changes to the determinative technique;
- Qualified flexibility to clarify that flexibility in front-end techniques does not apply to sample collection and preservation;
- Expanded Tier 1 validation to allow single-laboratory application of a method modification to multiple matrix types;
- Added an option to have EPA review Tier 2 and Tier 3 method modifications upon request;
- Added an option to have EPA formally approve, upon request, Tier 2 and Tier 3 method modifications through rulemaking; and
- Added an option to submit screening methods to EPA for approval.

The Streamlining Guide (EPA 1996a) and Method Guidelines and Format (EPA 1996c) served as the revised draft of the streamlining initiative that was discussed at the final public meeting on streamlining held in Denver. This

proposed rule incorporates suggestions received at the Denver public meeting, at previous public meetings, by mail, by electronic mail, and in informal discussions with and among EPA personnel, EPA contractors, and stakeholders.

Based upon the extensive involvement of internal and external parties, and the generally favorable response, EPA anticipates that the proposed regulations will be well received by regulatory authorities, the regulated community, the technology development community, and the laboratory service community.

#### *F. Preamble Structure*

Section III of this preamble outlines the key elements of streamlining. Section III.A describes EPA's proposal for increased flexibility within the method approval program and increased flexibility for modifications to existing methods. Section III.B describes the standardized QC requirements and QC acceptance criteria associated with implementation of flexibility. Section III.C describes the requirements for validating new methods and method modifications, using a system based on the intended application of the method or modification. Section III.D describes the expedited method approval process and includes procedures for submitting validated methods to EPA for approval. Section III.E describes other issues associated with the streamlining initiative. The descriptions in Section III delineate the framework of EPA's method flexibility and methods approval streamlining initiative. The Streamlining Guide (EPA 1996a) and other supporting documents cited in this notice contain specifics about the start-up and operation of the proposed streamlining initiative.

### **III. Summary of Proposed Rule**

#### *A. Method Flexibility*

In developing plans to improve the method approval program for drinking water and wastewater methods, EPA concluded that the program's success would depend largely on its ability to reflect the latest advances in technology. This required, in turn, that the program be efficient and flexible enough to encourage the development and use of new measurement techniques. To meet these objectives, EPA determined that the improved program would have two types of flexibility:

(1) Flexibility to modify reference methods without seeking formal approval through the regulatory process, and

(2) Flexibility to develop and submit for approval entirely new methods.

The first type of flexibility is primarily an expansion of the flexibility already provided in some approved water methods. Under the streamlining program, it would no longer be necessary to apply for ATP approval of a method modification, because an analyst would only need to demonstrate and document that the modified method produces results equal or superior to results produced by an EPA-designated reference method. A designated reference method that contains QC acceptance criteria against which performance of a method modification could be measured would be the primary control to ensure data quality. Other controls would include specific multi-laboratory and multi-matrix requirements for validating modified methods and checklists for documenting equivalency.

The second type of flexibility would expand the ATP concept by providing a mechanism whereby entirely new techniques would be submitted to the Agency for approval, even when these techniques would not serve as alternates to currently approved methods.

In designing a framework through which this flexibility could be implemented, EPA sought to balance the advantages of increased flexibility against the concern that results produced by modifications would be inferior to results produced by approved methods. To ensure that these competing objectives could be met, EPA has devised a framework that is based on:

(1) Use of a standardized QC program with elements that could be applied to all new and existing methods, and that is stringent enough to meet compliance monitoring objectives, extensive enough to be applied to a wide variety of analytical procedures, and yet simple enough to avoid unwieldy or unnecessary restrictions;

(2) Development and application of QC acceptance criteria for each QC element against which method modifications could be assessed and documented; and

(3) Designation of a single reference method for each unique combination of analyte and determinative technique. This reference method would contain the QC acceptance criteria used to assess each QC element for method equivalency.

In today's proposed revisions to 40 CFR parts 136 and 141, EPA would define the QC elements and associated acceptance criteria (e.g., calibration, sensitivity, accuracy, precision) necessary to demonstrate the

equivalency of a modified method to a reference method. These proposed QC requirements are based on the three components outlined above. Once equivalency was demonstrated, a modified method could be used immediately without review by EPA because EPA would have "preapproved" the modified method.

EPA believes that incorporating method flexibility into approved analytical methods would improve laboratory operations by allowing analysts to rely on professional judgement to ascertain the procedures and protocols necessary to obtain the best results. Analysts could employ new technologies to overcome matrix interferences, lower detection limits, improve the reliability of results, reduce the generation of hazardous wastes, improve laboratory productivity, and reduce analytical costs.

#### **1. Reference Method**

The foundation of the flexibility concept is the use of a reference method. For each unique combination of analyte and determinative technique, EPA has identified or would designate one approved method as the reference method. If the performance of the modified method is equal or superior to the performance of the reference method, the method modification would be allowed. EPA believes that the use of a reference method with defined QC acceptance criteria as the performance measure provides a means for implementing the streamlining initiative. This approach would clarify and reduce the effort required to demonstrate the equivalency of method modifications.

To implement the streamlining initiative, all reference methods would need to specify standardized QC and QC acceptance criteria. The QC and QC acceptance criteria would be necessary to demonstrate method equivalency. Some methods, such as those approved at 40 CFR part 136, Appendix A, already contain the necessary standardized QC and QC acceptance criteria. Some other methods do not specifically identify acceptance criteria for all of the standardized QC elements, but EPA has the data from which such criteria could be developed. For this proposed rule, selection of reference methods was based either on the existence of QC acceptance criteria in the method or the availability of data from which QC acceptance criteria could be developed. EPA is proposing QC acceptance criteria for some inorganic analytes and reference methods. These criteria are specified at 40 CFR 136.3 Table IF and at 141.27(d) in the proposed rule text.

The remaining criteria for other analytes and reference methods would be developed and proposed in subsequent rulemaking(s).

For some determinative techniques, no currently approved method contained either all of the QC acceptance criteria proposed in today's rule (e.g., Table ID in 40 CFR part 136) or sufficient data from which to develop such criteria. In these cases, no reference method has been proposed; therefore, all of those methods would be classified as other approved methods. Without a reference method, users would not be able to implement the method flexibility proposed in this streamlining initiative.

EPA plans to include standardized QC with QC acceptance criteria in all water methods under development and for all future water methods. However, for drinking water methods, some of the QC acceptance criteria (e.g., laboratory certification criteria) are currently (and may continue to be) specified in drinking water regulations because these criteria are an integral part of EPA's compliance monitoring requirements.

In the future, the selection of a new reference method would depend upon requirements imposed by the submitting organization, the availability of standardized QC and QC acceptance criteria in the method, and the timing of the selection. EPA intends to rely on outside organizations to develop the majority of the new methods. Therefore, it is anticipated that new reference methods for a particular determinative technique would be designated by being the first method approved for the given combination of analyte and determinative technique. To become a reference method, the new method would need to contain standardized QC and QC acceptance criteria, and be approved through an Agency rulemaking.

The purpose of specifying a single reference method for a specific combination of analyte and determinative technique is to avoid the possible confusion that could be created if two or more reference methods contained differing QC acceptance criteria. The QC acceptance criteria associated with the single reference method would be the sole criteria against which a method modification would be tested.

In today's action, EPA proposes to retain all methods approved for use at 40 CFR parts 136 or 141, but would re-categorize each of these methods as either a "reference method" or an "other approved method." Both types of methods would carry equal regulatory

status. The difference between the methods would be that the reference method would contain (or would be supplemented with) detailed QC acceptance criteria that would need to be used to assess the equivalency of a method modification.

## 2. Method Modifications

Currently, explicit flexibility to modify a method is provided in some of the approved 200-, 300-, 500-, 600-, and 1600-series methods published by EPA. The allowed flexibility is typically specified through use of the term "should" or the words "or equivalent." Substitution of a 500-mL beaker for a 250-mL beaker or use of an "equivalent" chromatographic column are examples of such explicit flexibility. The EPA 600- and 1600-series wastewater methods approved at 40 CFR part 136, Appendix A, also provide limited flexibility to improve separations and reduce the cost of measurements as long as method performance is not sacrificed. As specified in those methods, analysts who choose to exercise explicit flexibility are required to meet the QC acceptance criteria of the approved method and to maintain a record of the performance of the modified method for review at the request of an auditor. In the development of more recent methods (e.g., Method 1664 and Method 1613), EPA expanded its definition of "allowed flexibility" to further encourage use of new techniques that provide equal or better performance at lower costs. However, no approved methods provide unlimited flexibility and few provide the extensive flexibility that EPA proposes in this initiative.

The categories of method modifications considered in this proposal are: (1) Sample collection and holding procedures, (2) front-end techniques, (3) determinative techniques, and (4) analyte addition. These categories are defined below and described in terms of present and proposed flexibility to modify the procedures or techniques included in each category.

The first category, sample collection and holding procedures, includes procedures and reagents used in the field, in transit, and at the laboratory. This category includes sample containers, sample holding times, preservation reagents and procedures, and shipping and storage procedures and conditions. Currently, the Regional Administrator may approve modifications to these procedures for wastewater methods if the submitter so requests as specified at 40 CFR 136.3(c). In the drinking water program, except as explicitly allowed in the compliance

method, modifications of sample collection and holding procedures would be approved through the ATP specification at 40 CFR 141.27.

The flexibility proposed in today's rule would not extend to sample collection or holding procedures. Upon implementation of streamlining, modifications to sample collection and preservation conditions would continue to require EPA approval as specified at 40 CFR 136.3(c) and 141.27(b). The latter section, 141.27(b), is a proposed amendment of 40 CFR 141.27 that was written to conform more closely with the modification provisions at 40 CFR 136.3.

Front-end techniques, the second category of method modifications, are steps in the analytical process used at the laboratory that precede the determinative technique and include all procedures, equipment, solvents, etc., that are used to prepare a sample for analysis. The third category is the determinative technique, which is defined as the physical and/or chemical process by which an analyte is identified and its concentration measured. For most methods, the determinative technique consists of an instrumental measurement (e.g., a detector). The fourth category covers increasing the analytical scope of a reference method to include additional analytes.

Historically, the wastewater program has allowed some changes to front-end techniques, but only a few methods allow changes to the determinative step. The drinking water program has allowed similar changes provided the chemistry of the method is not changed. This means that some modifications, such as changing the extraction solvent, are not allowed in drinking water methods unless they receive formal EPA approval.

This proposed rule expands and more clearly defines proposed modifications to approved methods. EPA proposes to allow the laboratory analyst the flexibility to modify any and all front-end techniques, provided the modification is not explicitly prohibited in the reference method and provided the analyst demonstrates and documents that the modification produces results equal or superior to results produced by the reference method. The laboratory analyst would keep on file the documents that demonstrate equivalency. Readers are referred to the Streamlining Guide (EPA 1996a) for more guidance on this subject.

EPA considered restricting the flexibility to change front-end procedures, such as extraction solvents,

solvent-to-sample volumes, extraction media, and pH, because such changes require a deeper understanding of the measurement science than some users may have. However, EPA is not proposing to restrict front-end flexibility because EPA believes it is appropriate to allow the method development and auditing communities an opportunity to comment on a far-reaching change to the current system. The developer of a modified method always would have the option to ask EPA or another regulatory authority for a technical opinion on the acceptability of the validation data that supports the method. In the list of questions at the end of this preamble, EPA invites public comment on what, if any, additional QC would be needed to document the acceptability of front-end modifications to a reference method.

EPA proposes to allow use of an alternate determinative technique that is not explicitly prohibited in the reference method, provided that the analyst could demonstrate and document equivalency as outlined above, and provided that four conditions could be met: (1) The alternate determinative technique measures a property similar to the prescribed technique, (2) the alternate technique is demonstrated to be more specific (i.e., provides better separation of the analyte from interferences) and/or more sensitive (i.e., produces a lower detection limit) for the analyte of concern than the determinative technique in the reference method, (3) there is not another approved method that uses the alternate determinative technique for the determination of that analyte, and (4) use of the alternate determinative technique would not result in a nonsensical combination of analyte, front-end technique, and determinative technique.

Examples of allowed changes to a determinative technique would be substitution of a photoionization detector for a flame ionization detector for determination of polynuclear aromatic hydrocarbons, substitution of a nitrogen-phosphorous detector for an electron capture detector (ECD) for determination of analytes containing nitrogen or phosphorous, and substitution of a fluorescence detector for an ultraviolet or visible wavelength detector. Substitution of a mass spectrometer (MS) for an ECD would not be allowed if there is an approved MS method that measures the analyte of concern. Readers are referred to the Streamlining Guide (EPA 1996a) for more guidance on this subject.

EPA proposes to limit changes to a determinative technique by the four

conditions described above to preclude nonsensical combinations of analyte and determinative technique, to encourage a net benefit (increased sensitivity and/or specificity), and to preclude multiple reference methods with the same determinative technique but with different QC acceptance criteria for the same analyte(s) of concern. For example, if a mass spectrometer were substituted for the conventional detectors in EPA methods 601-612, all of these methods would become GC/MS methods, but all would contain different QC acceptance criteria. Further, they would all conflict with approved GC/MS Methods 625 and 1625. The proposed criteria for detector substitution also would be consistent with EPA's decision in the December 5, 1994, drinking water methods final rule (59 FR 62456) not to allow substitution of MS in methods that specify conventional GC detectors.

Another reason for proposing to limit changes to the determinative technique is that there are techniques, such as immunoassay, for which EPA has no reference method and therefore no history to ensure that the standardized QC proposed in today's rule would be germane to, or adequate for, assurance of the quality of data produced by the novel determinative technique. EPA would prefer that a new method be written and submitted for approval when a novel determinative technique is developed. EPA invites public comment on the suitability of the conditions EPA proposes to place on the flexibility to modify determinative techniques in EPA reference methods.

In today's proposed rule, EPA also has specified how the analyst would modify the analytical scope of a reference method to add additional analytes. This option is proposed in response to public comment on previous rules (59 FR 62456, December 5, 1994; 58 FR 65622, December 15, 1993) to extend the scope of a reference method to the determination of other analytes. Method developers seek this approval when they want to adapt an existing method rather than develop a new one to obtain occurrence data for a new analyte. EPA believes these requests would have merit when there is a potential for new regulatory requirements and historical monitoring data would be useful in making process, treatment, or regulatory decisions. Examples of monitoring for a new analyte would include industrial or POTW monitoring for ethers in a discharge, public water system (PWS) monitoring for unregulated pesticides or pesticide metabolites, and PWS monitoring for analytes on the drinking water priority list. EPA also believes

these requests would have merit when technological advances would make the measurement of additional analytes feasible (e.g., adding lead to the scope of EPA Method 200.7). Under the proposed flexibility procedures for modified and new methods, developers would obtain approval for the addition of analytes to a reference method as an allowed method modification if the conditions below would be met.

An analyst may add a new target analyte to a reference method provided (1) it could be demonstrated that the analyte would not interfere with determination of the analytes of concern in that method, (2) QC acceptance criteria were developed and employed for determination of the target analyte, (3) there would not be another approved method that uses the same determinative technique for that analyte, and (4) that the reason for adding the analyte would not be to avoid the sample preservation or sample (or extract) holding time conditions that are already required for that analyte in another approved method. The third and fourth criteria would preclude method shopping whereby an analyst might add analytes to a reference method with less rigid QC acceptance, sample collection or holding time criteria. Under the criteria proposed above, if a reference method for an analyte of concern required acidification of the sample, an analyst would not have the flexibility to modify a method that does not require sample acidification to include analysis of the analyte of concern. Modifications of this type would require EPA approval as a new method.

If QC acceptance criteria do not exist to allow addition of a new analyte, the guidelines specified at 40 CFR part 136 Appendix E, at 40 CFR 136.4, 136.5 and 141.27 would be followed to develop and obtain approval for these criteria. Alternatively, QC acceptance criteria for the new analyte could be transferred from the criteria for an analyte with similar chemical characteristics in the same method or from the criteria for the analyte in another approved method. EPA provides additional guidance on developing QC acceptance criteria in Chapter 3 of the Streamlining Guide (EPA 1996a).

#### *B. Quality Control*

In order to establish that method modifications do not degrade method performance, a standard would be required against which changes could be compared. This standard would consist of standardized QC elements and QC acceptance criteria that would be listed in the reference method and/



or in the regulations at 40 CFR parts 136 and 141. These criteria would serve as definitive test criteria for evaluating the performance of a method modification. As proposed, new methods would be required to include QC acceptance criteria that were developed from a method validation study according to procedures specified at 40 CFR 136.5, 141.27(c) and (e).

#### 1. Standardized Quality Control Elements

The standardized QC elements, described below, when paired with the relevant QC acceptance criteria for each element, would allow analysts to establish and document method performance. These elements would be specified at 40 CFR part 136 Appendix E and at 40 CFR 136.4, 136.5 and 141.27. Additional guidance on procedures and requirements for these QC elements are provided in the Streamlining Guide (EPA 1996a).

- Calibration—the process of establishing the relationship between the concentration or amount of material introduced into an instrument or measurement process and the output signal.

- Calibration Verification—the means of establishing that instrument performance remains within pre-established limits.

- Initial Precision and Recovery (IPR)—the mechanism to demonstrate that a laboratory would produce reliable results with the method prior to analysis of environmental samples. IPRs also would demonstrate that a method modification produces results equal or superior to those produced by a reference method.

- Ongoing Precision and Recovery—a process that demonstrates that a laboratory is able to produce reliable results continuously.

- Matrix Spike (MS)—a means to assess method performance (especially analyte recovery) on a sample by adding a known amount of the tested analyte.

- Matrix Spike Duplicate—a process to test the precision of an analysis by repeating the MS test.

- Method Blank—a test that checks for laboratory contamination.

- Method Detection Limit (MDL)—the MDL test, as specified at Appendix B of 40 CFR part 136, is used to confirm that a laboratory is capable of detecting an analyte of concern at the level specified in the method or at an acceptable level for regulatory compliance monitoring.

- Reference Sample—a test that serves as an external check on method accuracy.

- Retention Time and Relative Retention Time Precision—a means to assess the performance of a chromatographic separation system; used to aid in the identification of each target analyte in a complex mixture.

- Surrogate—a means to assess the performance of the method within the given sample matrix by adding a known amount of a different but chemically similar analyte. The results of these tests would be used to assess method and laboratory performance.

For each reference method, each QC test would have acceptance criteria that define data acceptability.

#### 2. Development of QC Acceptance Criteria

QC acceptance criteria would be used to ensure that a modified method produces results that are reliable, defensible and suitable for regulatory decisions. QC acceptance criteria would be specified as numeric limits. For example, the QC acceptance criteria for a MS/MSD test may be 75–125 percent recovery with a relative percent difference (RPD) of 20 or less. If these criteria were met for the MS/MSD test, and all other QC acceptance criteria were met, results produced using the modified method could be used for regulatory compliance purposes; if not, corrective action would need to be taken and the sample reanalyzed.

Some methods currently approved at 40 CFR parts 136 and 141 explicitly specify QC acceptance criteria for all of the standardized QC elements outlined in today's proposal, but many do not. In selecting reference methods for today's proposal, EPA chose those methods that contained QC acceptance criteria or data from which QC acceptance criteria could be developed. For those methods that did not contain QC acceptance criteria, QC acceptance criteria were developed from results of single-laboratory or interlaboratory study data contained in the method or from criteria contained in Appendix D of 40 CFR part 136. These criteria are provided at 40 CFR 141.27(d) and 136.3 Table IF for drinking water and wastewater reference methods, respectively. EPA would develop QC acceptance criteria for certain approved methods that do not presently contain these criteria. EPA would propose to designate these approved methods as reference methods in a future rulemaking.

#### C. Method Validation for Modified or New Methods

Method validation is the process by which an analyst or vendor would establish the performance of a new method or would substantiate the performance of a method modification to a reference method. Validation would be necessary to demonstrate and document that the new or modified method could yield reliable data for compliance monitoring and other purposes. The party who developed the method or method modification would be responsible for validating the method or method modification.

The requirements for validation would depend on the level of intended use for the method modification or new

method, and the characteristics of the sample to which the method modification or new method would be applied. Based on interactions with stakeholders, EPA proposes to establish three levels of validation:

- Tier 1 methods would be used in a single laboratory in a single matrix type from one industrial category or subcategory, or in additional matrix types from any industrial category or subcategory.

- Tier 2 methods would be used by all laboratories in one or more matrix types within one industrial category or subcategory.

- Tier 3 methods would be used by all laboratories in matrix types from all industrial categories or subcategories.

Proposed definitions of the terms laboratory, matrix type, medium, and tier are in the definitions sections at 40 CFR 136.2 and 141.2. In the streamlining initiative, the term matrix type would be defined and used to identify a sample medium with common characteristics across a given industrial category or subcategory. The terms facility or system would identify places where an industrial discharge activity occurs or where a water source is treated and distributed as drinking (potable) water. For example, all POTWs that comprise the municipal wastewater treatment industry would be considered to be in one industrial category. A typical municipal POTW has three matrix types: untreated wastewater, treated wastewater, and sludge. All PWSs that comprise the drinking water industry would be considered to be in one industrial category and to be one matrix type—potable water. Similar definitions would apply to matrix types in other industrial categories and subcategories. EPA invites public comment on these definitions and seeks suggestions on additional terms or concepts for which the public believes a regulatory definition would be useful in implementing and administering EPA's methods approval system.

Method validation would comprise three steps: (1) development of a validation study plan, (2) testing, and (3) preparation of a validation study report.

#### 1. Validation Study Plan

A validation study plan would be required for development of a new method at any tier or for modification of a reference method at Tiers 2 and 3. The organization responsible for conducting the study would prepare the validation study plan. Requirements for method validation would be specified at 40 CFR 136.4, 136.5 and 141.27 and at 40 CFR part 136 Appendix E. Additional guidance on suggested validation study



plans is available in the Streamlining Guide (EPA 1996a).

A validation study plan would not be required for Tier 1 method modifications, because EPA would expect that single-laboratory use modifications would be simple and straightforward, and that requiring a validation study plan for single-

laboratory modifications would impose an unnecessary regulatory burden on small laboratories.

## 2. Testing

The number of testing laboratories, matrices, and replicate QC tests for the method validation would depend on the tier at which the new or modified method would be validated, as

indicated in Table I below. The specific requirements and procedures for performing QC validation testing are specified at 40 CFR 136.4, 136.5 and at 141.27; additional guidance is available in the Streamlining Guide (EPA 1996a). Table I, which is taken from 40 CFR 136.5(d), summarizes validation requirements at each tier.

TABLE I.—SUMMARY OF VALIDATION REQUIREMENTS FOR NEW METHODS AND METHOD MODIFICATIONS <sup>1</sup>

Method application	Number of			Number of analyses required			
	Labs	Matrix types	Facilities/ PWSs	IPR-reagent water <sup>2</sup>	IPR-sample matrix <sup>3</sup>	MS/MSD	MDL <sup>4</sup>
Tier 1—Single-lab WW/DW—First matrix type or first PWS .....	1	1	1	4	4	<sup>5</sup> 2	7
WW—Each add'l matrix type (8 max.) from any industrial category .....	1	1	1	<sup>6</sup> 0	<sup>6</sup> 0	<sup>5</sup> 2	<sup>6</sup> 0
DW—Each add'l PWS (2 max.) .....	1	1	1	<sup>6</sup> 0	<sup>6</sup> 0	<sup>5</sup> 2 <sup>6</sup> 0	
Tier 2—Multi-lab, single matrix type WW/DW—Each matrix type in a single industrial category .....	3	1	3	12	0	<sup>7</sup> 6	21
Tier 3—Multi-lab, multiple matrix types WW only—All matrix types, all industrial categories .....	<sup>8</sup> 9	9	9	36	0	<sup>7</sup> 18	63

<sup>1</sup> Numbers of analyses in this table do not include background analyses or additional QC tests such as calibration, blanks, etc. Validation requirements are based on the intended application of the method. Method application would be designated by tier for wastewater (WW) and drinking water (DW) programs. Three would be the maximum number of public water systems (PWSs) that would be required to validate a new or modified drinking water method at Tier 1 or 2. Nine would be the maximum number of matrix types (or facilities) that would be required to validate a new or modified wastewater method at Tier 1 or 3; at Tier 2 the number would be three matrix types.

<sup>2</sup> IPR reagent water analyses would be used to validate a method modification and to establish QC acceptance criteria for initial precision and recovery (IPR) and ongoing precision and recovery (OPR) for a new method. The required number of IPR analyses, except as noted under footnote 7, would be four times the number of laboratories required to validate a method modification or new method because each laboratory would perform a 4-replicate IPR test.

<sup>3</sup> IPR sample matrix analyses would be used to establish QC acceptance criteria for matrix spike/matrix spike duplicate (MS/MSD) recovery and precision for a Tier 1 new method only. Would not be required for validation of Tier 2 or 3 new methods because this variability data would be obtained from MS/MSD tests. Would not be required for validation of a method modification because MS/MSD data from the reference method would be used.

<sup>4</sup> A method detection limit (MDL) test would be performed in each laboratory using the new or modified method. 40 CFR part 136 Appendix B requires a minimum of seven analyses per laboratory to determine an MDL. Each lab involved in validation of a wastewater modification would demonstrate that the modified method would achieve the detection limits specified in the regulations at 40 CFR parts 136 and 141 and/or in chapter 6 of the Streamlining Guide (EPA 1996a).

<sup>5</sup> MS/MSD analyses would be required only for a method modification because, for new methods, the MS/MSD QC acceptance criteria would be established by the 4-replicate sample matrix IPR test. For modified methods, the MS/MSD test would demonstrate that the reference method MS/MSD QC acceptance criteria have been met.

<sup>6</sup> The MDL, reagent water IPR, and sample matrix IPR tests would not have to be repeated after the first matrix type, facility, or PWS was validated.

<sup>7</sup> For validation of a new method, the MS/MSD analyses would establish QC acceptance criteria for MS/MSD recovery and precision. For validation of a method modification, the MS/MSD analyses would demonstrate that reference method MS/MSD recovery and precision have been met. The required number of MS/MSD analyses would be two times the number of facilities, PWSs or matrix types tested.

<sup>8</sup> The number of laboratories and samples would vary if a conventional interlaboratory study is used.

The tiered approach to validating new and modified methods would accommodate variability in the analytical performance of a method that can be attributed to the type of sample analyzed. This variability is termed a matrix effect and can be observed in samples taken at different locations in matrices of the same type (intramatrix) or in samples from different locations and in different matrix types (intermatrix). Under the streamlining initiative, each successive tier addresses matrix effects to a greater degree through increasing levels of sample matrix effect validation, broadly defined as a test of the extent to which differences, if any, in method

performance could be attributed to variability between samples obtained from different industrial matrices, facilities, or PWSs. Matrix effects would need to be tested by the IPR sample matrix and MS/MSD analyses listed in Table I. Intramatrix effects would need to be tested in water samples taken from different PWSs or from different waste streams. Intermatrix effects would need to be validated on a group of samples taken from discharge samples collected from several different industrial categories. In all cases, the laboratory would try to determine if the measurement result for the target analyte using a new or modified method differed from the result obtained in a

reagent water matrix or in a previously validated matrix type or PWS sample.

As indicated in Table I, a Tier 1 new or modified method would be validated in a single laboratory on one or more matrix types obtained from one or more facilities, or on samples obtained from one or more PWSs. Validation of additional facilities or PWSs would require analysis of MS/MSD samples for each additional facility or PWS. However, in response to stakeholder requests that there should be some maximum number of single-laboratory validations after which further validation would be unnecessary because sample matrix effects would have been sufficiently addressed, EPA

added a provision for a maximum number of matrix type, facility or PWS analyses for Tier 1 methods. For a wastewater method, the maximum number of matrix types or facilities tested under Tier 1 would be nine, each from a different industrial category or subcategory. For a drinking water method, the maximum number of PWS samples tested under Tier 1 would be three samples, each from a PWS with different water quality characteristics. EPA proposes to require validation in three rather than nine PWSs, because three is consistent with the validation data in many EPA drinking water methods and because the variability in drinking water samples (and therefore the probability of matrix effects) is usually less in drinking water samples than in wastewater samples.

Tier 2 validation would be applicable to one or more matrix types within a single industrial category or subcategory. Because Tier 2 new and modified methods would apply to each matrix across all laboratories, EPA developed Tier 2 validation requirements to incorporate intramatrix variability. Tier 2 would require validation of the method in drinking water samples obtained from three PWSs, or wastewater samples of one or more matrix types obtained from three or more facilities within a single industrial category or subcategory.

Tier 3 validation would be applicable to all matrix types in all industrial categories. Consequently, Tier 3 validation requirements would include provisions to account for both intramatrix and intermatrix variability. However, Tier 3 validation would not apply to the drinking water program because the program regulates only one matrix type, drinking (potable) water. The wastewater program regulates several industrial categories, each of which may contain more than one matrix type. Tier 3 would require validation of the method in wastewater samples of up to nine matrix types obtained from nine different facilities.

For all multi-matrix tiers, it would be extremely important to select suitable samples and matrix types for validation. The matrix types, facilities, or PWSs selected for matrix effect validation would need to have sufficiently different water quality characteristics so that the matrix effects, if any, could be observed. Proposed criteria for selecting matrix types, facilities, or PWSs from which to obtain these samples is specified at 40 CFR 136.4(a)(2)(i) and 141.27(b)(iii). Additional guidance on testing sample matrix effects is available in the Streamlining Guide (EPA 1996a).

EPA invites public comment on the number of tests, laboratories, matrix types, facilities, and PWSs that EPA is proposing for validation of Tier 1, 2, or 3 methods. EPA is specifically interested in suggestions for adding, deleting, or modifying the tests listed in Table I. Commenters should provide EPA with reasons for (and preferably data to support) any suggested changes.

### 3. Validation Study Report

A validation study report would be required for a new method or method modification at all tiers to document successful validation. The primary documents to be included in the report would be the Checklist for Initial Demonstration of Method Performance, the Checklist for Continuing Demonstration of Method Performance (collectively, the "Checklists"), and a Certification Statement. The Checklists would document that all requirements for establishing equivalency were met; the certification statement would commit the persons involved in the method development or modification effort to the statements made in the Checklists and the supporting information provided. The proposed Checklists would be specified at 40 CFR part 136 Appendix E. The checklists also would be published in the Streamlining Guide (EPA 1996a) with additional guidance on how to complete a checklist for a typical water method. This guidance would be provided to aid the method modifier or developer in understanding the information and test data to be provided. The Checklists and certification statement would be required as part of the validation study report. For Tier 1 method modifications, the Checklists and certification statement would comprise the data validation report. For all tiers, each laboratory involved in validation of a method modification would need to complete the Checklists and Certification Statement. More extensive documentation would be required for a modification at Tiers 2 and 3 and for all new methods.

The validation study report for Tiers 2 and 3 would need to specify the following information, as appropriate, for validation of a new or modified method:

- Narrative—includes (a) a description of the method being validated and the matrices, matrix types, and media to which the method is applicable; (b) an indication of whether the method is a modification of an approved reference method or a new method; (c) reason for and description of the modification, if applicable; and (d) information on the organization responsible for developing the new method or method modification.

- Analyte(s)—name and Chemical Abstracts Service (CAS) Registry Number or an EPA Environmental Monitoring Methods Index (EMMI) Number. If a CAS Registry Number has not been assigned, the submitter should attempt to obtain a number from the CAS Registry. If the CAS Registry will not assign a number, the submitter should contact the AMS Director for assignment of an EMMI Number. A report for a modified method should indicate whether the modification includes all forms of the analyte(s) in the scope of the reference method. The definition of AMS Director is at 40 CFR parts 136.2 and 141.2.

- Method or modified test procedure—prepared in a standard format; modified test procedures would be prepared in the format of the reference method.

- Methodology and procedures—indicates the tier level at which the new or modified method was tested, describes the approach used to implement the study, describes the procedures used to report and validate the data, and identifies the problems encountered during implementation of the study.

- Results—for modified methods, includes a summary of QC results required by the reference method and corresponding QC results obtained with the modified method.

- Conclusions—describes the conclusions and limitations of the study.

- Discussion—critically examines the study results.

The following items would need to be included in appendixes to the validation study report:

- Calculations;
- Raw data to allow an independent reviewer to verify each determination and calculation performed by the laboratory;
- For instruments involving data systems, raw data on magnetic tape or disk (upon request only);
- Names, titles, addresses, and phone numbers of analysts who performed analyses and QA Officer who verified analyses; and
- Completed Checklists and Certification Statement.

The validation study report for a new or modified method would need to be retained on file by the organization responsible for developing or applying the modification, and by regulated entities whose samples are tested with the method modification. The party responsible for developing and submitting the new method also would need to maintain on file the complete records of all validation study tests including the study plan, all laboratory results, the validation study report, completed Checklists and Certification Statement, and other information that supports the new method or method modification. All records would need to be made available for review upon request to an auditor, permitting authority, or other regulatory authority. These records would need to be submitted to EPA if the method

developer elected to request formal approval of a method modification at Tier 2 or 3.

#### 4. Further Validation of a New Method

After completing a Tier 1, 2, or 3 validation study of a new method, the organization responsible for developing the method would need to document the study results in accordance with requirements proposed at 40 CFR part 136 Appendixes E, F, and G and would need to submit the results and the method to EPA for review and approval. If, based on its review of the method, EPA concluded that the method was not sufficiently rugged or reliable for its intended use, EPA would require further method development and testing. The tests and studies that would be performed would need to be determined on a case-by-case basis as these situations arise and would depend on the analyte(s) and the analytical system.

#### 5. Approval of a Screening Method as a New Method

Methods currently approved for compliance monitoring at 40 CFR parts 136 and 141 are considered to be confirmatory methods if the method is sufficiently selective and quantitative so that most positive results do not have to be verified by analysis with another method. The term "confirmatory" is used to distinguish these methods from screening methods. When using a screening method, all positive results should be verified by re-analysis with a confirmatory method because screening methods can be less selective and/or quantitative and, therefore, more subject to false positives or imprecise results than confirmatory methods. Characteristics of screening methods are described in more detail in Chapter 2 of the Streamlining Guide (EPA 1996a).

EPA has been asked by many stakeholders to allow use of screening methods for wastewater and drinking water analyses. Although screening methods may be less selective and quantitative than confirmatory methods, they also could be designed to serve meaningful uses under those statutes. Screening methods could be especially useful when measuring trends in the contamination of a water source or when knowledge of the performance of a waste treatment process would be more important than an exact knowledge of the absolute amount and identity of the contaminant or pollutant.

Historically, EPA has not considered screening methods for approval at 40 CFR part 136 or part 141. Under the streamlining initiative, EPA proposes to consider the approval of screening methods for compliance monitoring under the Safe Drinking Water Act provided that: (1) the method would meet all the requirements specified in the regulations at 40 CFR 141.27, (2) all positive sample results obtained with the method would be confirmed and reported using an approved confirmatory method, and (3) the probability of the method producing a false negative result at concentrations of regulatory interest would be no more than one percent (1%). EPA has not yet specified how it intends to implement the use of screening methods under the SDWA; the term was only recently added in the 1996 SDWA amendments. Under the Clean Water Act, EPA is considering the appropriateness of screening methods for use in NPDES permit applications and ambient water quality monitoring by States. EPA proposes to publish a separate table at 40 CFR parts 136 and 141 to list approved screening methods. The Agency invites comment on the approval criteria for screening methods for the uses described in the SDWA, as

well as for NPDES permit applications and ambient water quality monitoring.

#### D. Method Review and Approval

Under this proposed rule, EPA expects to significantly reduce the number of methods that would pass through the ATP review and rulemaking processes. EPA has this expectation because, once implemented, the streamlining initiative would make it easier for method modifications to be judged as being "within the flexibility allowed by the streamlining initiative." Method modifications demonstrated and documented to be within the flexibility allowed by the streamlining initiative would be preapproved by EPA for use at the tier for which the modification was validated. Stakeholder remarks suggest that most laboratories and method development organizations would welcome and use this allowed flexibility.

Stakeholders also have asked EPA to approve more quickly revised versions of approved methods that are periodically published by EPA, consensus standards organizations, and other government agencies. In the past, EPA approved these revisions through a formal proposal and public comment process. Using the flexibility provisions of today's rule, users would be able to use a revised version of a reference method as soon as it is published, provided that the results produced were demonstrated to meet the QC acceptance criteria of the reference method. This benefit alone would relieve much stakeholder frustration, decrease the Agency's rulemaking burden, and improve EPA's partnership with other government agencies and consensus standards organizations.

Table II summarizes EPA's review and rulemaking responsibilities for new and modified methods by tier.

TABLE II.—EPA REVIEW AND ACTION FOR NEW AND MODIFIED METHODS

	New Method	Modified Method
Tier 1, Single-lab .....	EPA review required; EPA issues a letter of approval.	No EPA review.
Tier 2, Multi-lab, single matrix type	EPA review required; approved through rulemaking	If requested, EPA reviews and —issues letter of approval, or —conducts rulemaking.
Tier 3, Multi-lab, multiple matrix types.	EPA review required; approved through rulemaking	If requested, EPA reviews and —issues letter of approval, or —conducts rulemaking.

#### 1. Review and Approval of New Methods

Currently, all new methods must be approved by EPA through "formal" EPA approval including rulemaking and

publication at 40 CFR part 136 or 141 before use. In today's rule, EPA proposes to grant letter approvals of new methods that would be submitted under Tier 1 (i.e., single-laboratory,

limited-use methods). New methods developed for use under Tiers 2 or 3 would still require rulemaking. The purpose for not requiring formal rulemaking at Tier 1 would be to

provide the means by which (1) a new technology could be introduced, (2) confidentiality of a new technology could be maintained if desired by the user of the new method, and (3) specific matrix interference problems could be overcome. Allowing use of Tier 1 new methods would enable multiple single laboratories to use a new technology until a sufficient number of devices were available for interlaboratory validation as a Tier 2 or 3 new method.

EPA recognizes that allowing single-laboratory use of a new technology for regulatory compliance carries with it the risk that results produced with the new technology may not agree with results produced by a reference method. However, EPA believes that sufficient controls would be included in the streamlining program to ensure data quality. EPA also believes that there would be a net benefit to the regulated community by allowing new technologies that overcome matrix interference problems. EPA solicits comment on this aspect of streamlining, and is particularly interested in alternative ways EPA might allow introduction of new technologies without rulemaking.

## 2. Review and Approval of Modified Methods

Under the streamlining initiative proposed in today's rule, method modifications would not require formal EPA approval; they would be preapproved provided the analyst demonstrates and documents equivalency with or superiority to the reference method QC criteria. Although formal approval of a modification would not be required under the streamlining initiative, several stakeholders have commented that, in practice, use of a method modification would require the consent of the regulated entity and responsible regulatory authority. These stakeholders also expressed concern that without formal EPA approval, obtaining consent from the regulated entity and/or regulatory authority would be difficult. In response to these comments, EPA proposes to allow, but not require, laboratories, industry associations, consensus standards organizations, instrument manufacturers, and others to submit Tier 2 or Tier 3 method modifications for EPA review with the anticipation of a letter from EPA documenting approval. Also, for those seeking public recognition that their Tier 2 or 3 method modifications have been demonstrated to be acceptable for use, EPA proposes to work with the organization to approve the method at 40 CFR part 136 or 141. EPA would not review, provide

letters of approval, or conduct formal rulemaking for Tier 1 method modifications.

EPA recognizes that preapproving method modifications poses additional burdens for regulatory authorities, who may need to assess the reasonableness and effectiveness of each modification. EPA believes, however, that the Checklists, certification statement, and accompanying instructions, which are proposed at 40 CFR part 136 Appendix E, and the validation report for the method modification, which is proposed at 40 CFR part 136 Appendixes F and G, would provide a regulatory authority the information necessary to make equivalency assessments, and that this information would be presented in a standardized and readily understandable format. To further assist regulatory authorities in implementing this initiative, EPA has included detailed guidance on assessing method modifications for equivalency. This guidance is provided in Chapter 6 and in the appendixes of the Streamlining Guide (EPA 1996a).

## 3. Submission Package

The items to be submitted to EPA for proposal of a new method at Tier 2 or 3 would include the method validation study report, which would include the method prepared in a standard format. If the submitter requested formal rulemaking to propose the method for publication in the CFR, information in a format suitable for inclusion in a draft preamble would also be required. Additionally, the submission packet would need to include all relevant supporting documents.

To preclude a proliferation of potentially confusing formats, a method should be submitted in a standard format. EPA recommends and specifies the format that would be specified at 40 CFR part 136 Appendix F. This format is also detailed in Method Guidelines and Format (EPA 1996c). Appendix F describes all elements of the format prescribed by EPA's Environmental Monitoring Management Council (EMMC). An objective of the EMMC format is to standardize all Agency analytical methods. A standardized format used by a government agency such as the U.S. Geological Survey or from a consensus standards organization such as Standard Methods, ASTM, or AOAC-International could be used, but EPA recommends that these formats be reserved for those organizations to avoid the possible confusion over authorship. EPA would not accept methods in non-standard formats because of the confusion that could be created by a proliferation of method formats.

A new method would need to include the standardized QC elements and QC acceptance criteria. The QC acceptance criteria would need to be developed from data gathered in the method validation study. Chapter 3 of the Streamlining Guide (EPA 1996a) provides guidance on the detailed technical requirements for developing criteria that meet the requirements that would be specified at 40 CFR 136.4, 136.5 and 141.27 and at 40 CFR part 136 Appendix E.

## 4. Regulatory Assistance Provided by Submitter

Using procedures that would be specified at 40 CFR part 136 Appendix G, EPA would ask method submitters to assist EPA by providing, as part of the submission package for methods to be proposed in the **Federal Register**, information that would facilitate EPA's drafting of a proposed rule. EPA would also ask submitters to provide technical assistance, when necessary, in responding to public comments on the submitter's method. Other assistance could be requested by EPA. The information should be submitted in a format corresponding to the preamble drafting conventions specified by the Office of the Federal Register. Citations of examples for preambles are given in 40 CFR part 136 Appendix G and in the Streamlining Guide (EPA 1996a). Instructions for drafting documents for the Office of the Federal Register are given in the Document Drafting Handbook, for sale by the Superintendent of Documents, Mail Stop: SSOP, Washington, DC 20402-9328 (Document 1993 O-351-677 QL3).

## 5. EPA Review of Submission Package

Upon receipt of a request for approval, EPA would first check the submission packet for completeness. If all of the documentation was in order, EPA would use an internal workgroup to assess the scientific merit of the method or modification and to evaluate the validation study for consistency and appropriateness. Should any problems be identified, the workgroup would contact the submitter to resolve the outstanding issues. If these issues could not be resolved, EPA would take no further action on the submission. If all validation requirements were met and the submission passed internal review, EPA would either issue a letter of approval or begin the rulemaking process. All method modifications are preapproved, but a submitter would have the option to request an EPA letter of approval or to request a formal rulemaking for Tier 2 and 3 method

modifications. All new methods would be subject to EPA review. For Tier 1 new methods, EPA would issue letter approvals; Tier 2 and 3 new methods would require formal Agency rulemaking.

#### 6. Proposal of Methods

For rulemaking, EPA would prepare the proposed rule based on the draft preamble provided by the submitter. EPA would add the appropriate updates to CFR tables or language and submit the proposed rule to the Office of the Federal Register for publication. The proposed rule would request public comment and allow a specified comment period (typically 60 days after publication in the **Federal Register**). At the end of the comment period, EPA would forward significant public comments, if any, to the method submitter. The submitter would need to provide technical assistance to EPA in drafting responses to the comments. If the comments could not be adequately addressed, EPA would not take final action to approve the method. If all comments are addressed, EPA (with assistance from the submitter) would need to complete a response-to-comments document and prepare a final rule to approve the proposed method. The final rule would state the date that the rule becomes effective, typically 30 days after rule publication. As of this effective date, the method would be approved (promulgated) and the appropriate tables in the CFR would be updated.

To expedite approval of noncontroversial updates to methods, such as revisions to the methods published by EPA, other government organizations, and consensus standards organizations, EPA intends to use "direct final" rulemaking. Direct final rules would be warranted when the action would not be expected to elicit public comment to which the Agency would normally respond (i.e., no adverse comment). In this process, the final rule and the companion proposal would be published simultaneously as a "direct final rule" in the **Federal Register**. In a direct final rule, the proposed rule has a specific comment period and the final rule has a later effective date. If no adverse public comments are received during the comment period for the proposed rule, the actions become effective on the effective date of the final rule. If adverse comment is received, the companion final rule is withdrawn and a second final rule that responds to the public comments is prepared and published with a new effective date.

#### E. Other Issues

##### 1. Legal Impacts

Stakeholders expressed concern regarding potential conflicts between regulators and regulated entities when using modified methods. For example, there was widespread concern over a situation in which a discharger used a modified method and demonstrated compliance with a regulatory concentration limit while a regulatory authority used the unmodified reference method and obtained results suggesting that the discharger was out of compliance.

Based on internal EPA discussions, it became apparent that the streamlining initiative would work only if the modified method, once demonstrated to be equivalent to the reference method, carried the same legal force and effect as the reference method. Therefore, the difference in results produced by the modified and unmodified methods would be attributable not to the modification, but to differences in results produced by two laboratories. This situation is no different than the existing situation where two laboratories can produce different results, one set of which is above and the other below, a regulatory compliance limit. The legal resolution would therefore remain the same as today—a decision would be made based on examination of all the relevant data.

##### 2. Method-Defined Analytes

The method flexibility introduced in today's proposal does not extend to methods in which some part of the method "defines" the analyte of concern. This type of analyte is termed a method-defined analyte. Because method-defined analytes do not have a specific, known composition, the result of the analytical measurement depends totally on how the measurement is made. Examples of method-defined analytes include adsorbable organic halides, biochemical oxygen demand, total organic carbon, and whole effluent toxicity. Changes to the front-end steps or the determinative techniques in these methods have the potential of changing the result produced. EPA believes, however, that certain parts of procedures for method-defined analytes could be modified without adversely affecting method performance.

##### 3. Biological Methods

EPA intends to expand method flexibility to include biological methods, but not in today's proposal. Biological methods include both the testing of an environmental sample for the presence of microbiological material

(e.g., bacteria, protozoa and viruses) and the use of biological organisms to measure whole effluent toxicity (WET) of an environmental sample. EPA believes that flexibility in testing for biological material would be similar to the flexibility allowed in the modification to chemical analytical methods. Both the front-end and determinative techniques should be able to be modified when the modifications produce equivalent or superior results. EPA has protocols for some microbiological methods that are currently used in the ATP program (EPA 1995a, b). In a future rulemaking, EPA may revise the microbiology protocols to conform with streamlining and method flexibility procedures. In keeping with Agency goals for a more performance-based approach to all environmental measurements, EPA also may develop and propose method flexibility and new method approval procedures for biological methods and for microbiological parameters not covered under current EPA protocols.

For WET methods, both new and modified methods are possible. New methods may involve the use of a different taxonomic category other than those currently listed at 40 CFR part 136. Method modifications may be defined as the variation of one of the established summary test conditions of the method, such as temperature or salinity. Method modifications to the summary test conditions would not change the acceptance criteria (e.g., control survival) which serve to identify the standards of comparison of the "reference method." EPA has not sufficiently explored this issue to propose the specific requirements to allow flexibility in all approved biological methods. Until EPA can clarify the extent of acceptable flexibility, requests for changes in biological methods would be reviewed and approved on an individual basis.

##### 4. Proprietary Reagents, Instruments, and Methods

Stakeholders expressed concern over the role of proprietary components in the streamlined water method approval process. EPA separates proprietary components into three categories: proprietary reagents, proprietary instruments, and proprietary methods. EPA intends to attempt to accommodate the inclusion of proprietary reagents and instruments in the approval of analytic methods for compliance purposes to the extent that such inclusion still provides an adequate opportunity for public review and comment under the Administrative Procedure Act. EPA does not anticipate,

however, that it could approve the use of proprietary methods for determining compliance with regulatory requirements where the entire method is claimed as "confidential business information" because the opportunity for public review and comment might be restricted too severely. If a proprietary method is patented, the method would be considered for approval as a compliance method because the public would be able to comment on the patented method. EPA believes the restriction on approval of proprietary methods is not serious because reagents or instruments, not complete methods, will continue to be the most common proprietary components used in compliance methods.

Proprietary reagents and instruments are currently included for use in approved methods and would continue to be allowed in approved methods. The details of the proprietary elements would need to be disclosed to EPA, but would be withheld from the public if the person requesting protection for the confidential business information (CBI) demonstrates that the information is entitled to confidential treatment under 40 CFR part 2. Examples of proprietary components may include immunoassay reagents and antibodies and liquid phases in GC columns; e.g., DB-1<sup>®</sup>, SPB-octyl, Dextsil<sup>®</sup>, etc. A new or modified method submitted for EPA approval would need to include language stating that the proprietary reagent or instrument could be replaced by an equivalent. Changes made to the method after EPA approval would require the manufacturer to demonstrate, through supporting documentation, that the new proprietary equipment, substance, or reagent would produce results equal or superior to results produced with the material originally tested and on which the method approval is based. Additionally, EPA would not propose a method containing a proprietary reagent without accurate, specific instructions for handling the reagent and for safe disposal of each spent proprietary reagent and/or reaction product. When a material safety data sheet (MSDS) would need to accompany the proprietary material, the MSDS would be the appropriate vehicle to provide these instructions. Submission of a complete MSDS with a new method would satisfy EPA's need for instructions for safe handling and disposal of the reagent.

#### 5. Restrictions by Consensus Standards Organizations

As envisioned, this initiative allows modification to a reference method, provided that the QC acceptance criteria are met. Many of the methods approved at 40 CFR parts 136 and 141 were developed by consensus standard organizations such as Standard Methods, ASTM, and AOAC-International. EPA expects to rely on these and other consensus standards organizations for future methods, as required by the National Technology Transfer and Advancement Act of 1995 (NTTAA) and because of limited Agency resources for method development.

Consensus standards organizations have expressed concern that a modification to their methods would constitute a violation of the method being considered a "standard." Standard Methods, ASTM, and AOAC-International have declined to allow unlimited modification of their approved methods and, therefore, their methods could not serve as reference methods nor be modified under the procedures outlined in this initiative, as can be seen in the proposed CFR tables. This restriction would not greatly affect the streamlining initiative because an EPA method exists that would be used as a reference method for nearly all analytes, and because most methods from consensus standards organizations have sufficient internal flexibility to meet the objectives of streamlining or are updated frequently to reflect recent advances in technologies.

#### 6. Standard Data Format

For this proposed rule, EPA would not establish a standard format for the submission of analytical data because of the large variety of formats currently in use. However, EPA strongly recommends the Department of Energy's Environmental Management Electronic Data Deliverable Master Specification (DEEMS) because it is comprehensive and it would expedite processing of a submitter's request. DEEMS is a list of data elements that laboratories should submit to document the method modification process. A DEEMS data element dictionary is provided in the Streamlining Guide (EPA 1996a).

#### 7. Withdrawal of Outdated Methods

EPA also is considering withdrawal of methods that the Agency believes are obsolete or are no longer used. For example, 40 CFR part 136, Table ID, footnote 3, references methods published in 1978 that include thin-layer chromatography (TLC) methods.

Because gas chromatography and high performance liquid chromatography methods provide better monitoring data and are more cost effective, most, if not all, laboratories no longer use TLC methods. The TLC methods were proposed for withdrawal in a previous notice (60 FR 53988, October 18, 1995), and EPA believes there may be similar outdated methods. EPA is conducting a careful examination of Tables IA through IE of 40 CFR part 136 and of the tables at 40 CFR part 141, for obsolete or outdated methods, and intends to propose withdrawal of those methods for which newer methods are available.

#### 8. Administrative Record: Organic Methods, Streamlining Guide, and Method Guidelines and Format

EPA specifies several 600- and 1600-series analytical methods at 40 CFR part 136 Appendix A for analysis of organic chemicals. If the Office of the **Federal Register** approves incorporation by reference of the Appendix A methods, EPA will withdraw Appendix A and publish all of these methods in the document Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, December 1996, EPA-821-B-96-005, NTIS PB97-125298, ERIC D-A44/D-A47 (Organic Methods, EPA 1996b). This document is part of the administrative record for this proposed rule; copies can be inspected or obtained from NTIS or other sources as described in the ADDRESSES section above.

EPA also has drafted two guidance documents that are an integral part of the administrative record for this proposed rule. The first document, Guide to Method Flexibility and Approval of EPA Water Methods, December 1996 Draft, EPA-821-D-96-004, PB97-117766 (Streamlining Guide, EPA 1996a), provides detailed guidance on the overall streamlining initiative. The second document, Guidelines and Format for Methods to Be Proposed at 40 CFR Part 136 or Part 141, EPA-821-B-96-003, PB96-210448, July 1996 (Method Guidelines and Format, EPA 1996c), specifies the content and format required for new methods developed by outside organizations. These documents are readily and widely available to the public through NTIS, online, and other sources listed in the ADDRESSES section above.

The Streamlining Guide (EPA 1996a) in particular was drafted to help method developers use the procedures proposed in today's rule to validate and obtain approval of new or modified methods. The guidance was written for use by laboratory auditors, permittees, water utilities, regulatory authorities,

purveyors of new technology, and analytical laboratory personnel. The document is organized into seven chapters, some of which are procedural and others are descriptive, as appropriate to the topic. Chapter 1 summarizes the proposed streamlining initiative. Chapter 2 describes the proposed expanded method flexibility. Chapter 3 describes the proposed standard quality control tests and useful statistical procedures for developing QC acceptance criteria for new methods. Chapter 4 describes the proposed tiered system for validating a new method or a method modification. Chapter 5 describes the proposed method approval process, a standard method format, and procedures for submitting validated methods to EPA for approval. Chapter 6 provides guidance for assessing the method equivalency. Chapter 7 describes possible future plans to extend method flexibility to microbiological and macrobiological methods.

The Streamlining Guide (EPA 1996a) also includes eight appendixes. Appendix A provides a list of acronyms and abbreviations. Appendix B provides a glossary of terms used in the streamlining initiative. Appendix C provides examples of currently allowed method modifications. Appendix D contains a DEEMS data element dictionary, which is a Department of Defense reporting format that EPA suggests would speed review of method validation data. Appendix E provides the EMMC method equivalency checklists and certification statement. Appendix F provides an example of a completed Appendix E checklist. Appendix G contains bibliographic references. Appendix H describes EPA derived the proposed QC acceptance criteria for inorganic chemicals, which are proposed at 40 CFR 136.3 Table IF and 141.27(d), were calculated.

EPA proposes to make some of the information in the Streamlining Guide (EPA 1996a) and Method Guidelines and Format (EPA 1996c) a regulatory requirement. Specifically, EPA proposes to include much of the information in Chapter 2 (Method Flexibility), Chapter 6 (Assessing Method Equivalency), Chapter 5 (Method Approval Process) and Appendix E (Equivalency Checklists) as a requirement for approval of drinking and wastewater methods. EPA proposes to accomplish this by designating the excerpts from Chapters 2, 5 and 6 as 40 CFR part 136 Appendix G and the equivalency checklists in Appendix E as 40 CFR part 136 Appendix E. Other provisions of the Streamlining Guide (EPA 1996a), including, but not limited to, Table 4–

2, definitions of standardized QC elements, QC acceptance criteria for inorganic chemicals, would also be included at 40 CFR 136.2, 136.3 Table IF, 136.4, 136.5, 141.2, and 141.27. EPA would also adopt most of the provisions in Method Guidelines and Format (EPA 1996c) as Appendix F at 40 CFR part 136. EPA invites public comment on these two guidance documents and solicits comments on whether additional guidance in these documents should be a regulatory requirement.

#### 9. Coordination with Other Federal Register Proposals

On October 18, 1995 (60 FR 53988), EPA proposed to amend the list of approved methods at 40 CFR part 136 by adding new or revised methods for certain metal and inorganic analytes and by adding method citations to Table IB and amending the incorporation by reference section accordingly. EPA also proposed to withdraw approval of certain outdated or rarely used analytical methods, as well as certain methods that require use of hazardous or toxic reagents. As of today, EPA has not promulgated a final rule implementing the proposed actions.

The methods proposed for withdrawal that relate to this streamlining initiative are primarily the EPA 200-series flame atomic absorption spectrophotometry (FLAA) methods. Although approval of the EPA FLAA methods is proposed to be withdrawn, FLAA methods published by ASTM, Standard Methods, AOAC-International, and USGS would remain approved and would remain listed in 40 CFR 136.3, Table IB. Withdrawal of approval of EPA FLAA methods would remove these methods as reference methods and would remove the QC acceptance criteria associated with these methods. The net impact would be that there would be no FLAA method against which modifications would be made. EPA does not consider this a serious limitation because four FLAA methods (ASTM, Standard Methods, AOAC-International, and USGS) would remain approved for nearly all metals and the flexibility afforded by these methods should adequately cover method modifications.

In 1997, EPA intends to amend the regulations at parts 136 and 141, as appropriate, to update outdated versions of methods to versions published in the 19th edition of Standard Methods (APHA 1995), the 1996 Annual Book of ASTM Standards, Vols. 11.01 and 11.02 (ASTM 1996), and in EPA's August 1995 manual titled, Methods for the Determination of Organic Compounds in Drinking Water—Supplement III (EPA 1995c). If and when the provisions

of today's rule are promulgated, EPA expects to be able to list these 1995 and 1996 versions of the compliance methods as approved methods in the tables listed at 40 CFR parts 136 and 141. If inclusion of these more recent versions would provide a basis to change any of the QC acceptance criteria for the reference methods, the public would be notified and provided with the opportunity to comment on the new criteria.

#### 10. Laboratory Certification and Laboratory Auditing

Broad requirements for States to have an approved laboratory certification program for analysis of drinking water samples are specified at 40 CFR 142.10(b)(3). EPA provides more specific help to State certification officers through written and verbal guidance. To improve the uniformity of these certification programs, some laboratory certification officers, method developers, and vendors have asked EPA to provide more specific regulatory requirements. Today's rule responds to these requests by proposing standardized QC elements for all water compliance methods at 40 CFR 136.2 and 141.2, and at Appendix G of 40 CFR part 136. To standardize and facilitate laboratory audits, EPA also would recommend use of several detailed checklists for auditing both modified and unmodified methods. These standardized checklists would be specified at Appendix E of 40 CFR part 136. EPA understands that increasing the analyst's current flexibility to modify steps in a compliance method could make the conduct of laboratory audits more difficult. However, EPA believes that the proposal to specify standardized QC elements for all methods and to require that laboratories use standardized checklists to document and check method performance will ameliorate these potential problems. EPA invites public comment and is especially interested in what additional action, if any, the Agency should take to facilitate the auditing of water laboratories.

#### IV. Regulatory Analysis

##### A. Executive Order 12866

Under Executive Order 12866 [58 FR 51,735 (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 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.

This regulation is not major because it is intended to reduce costs through flexibility and innovation. Therefore, this regulation would not result in a cost to the economy of \$100 million or more; would not result in a major increase in costs or prices for consumers or individual industries; and would not have significant adverse effects on competition, investment, innovation, or international trade.

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 OMB review.

#### *B. Unfunded Mandates*

Title II of the Unfunded Mandates Reform Act of 1995 (UMRA), Pub. L. 104-4, establishes requirements for Federal agencies to assess the effects of their regulatory actions on State, local, and tribal 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 notifying 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.

EPA has determined that this rule does not contain a Federal mandate that may result in expenditures of \$100 million or more for State, local, and tribal governments, in the aggregate, or the private sector in any one year. EPA has further determined that this rule contains no regulatory requirements that might significantly or uniquely affect small governments. This rulemaking should have minimal financial impact, if any, on the current regulatory burden imposed on regulated entities and regulators because the rulemaking does not establish any additional regulatory requirements. The proposed rule simply provides the option to modify approved methods or propose new methods, if desired. EPA believes that method modifications and new methods would not be used if not cost effective. Thus, today's rule is not subject to the requirements of sections 202, 203, and 205 of the UMRA.

#### *C. Regulatory Flexibility Act*

The Regulatory Flexibility Act, 5 U.S.C. 601 *et seq.*, requires EPA and other agencies to prepare a final regulatory flexibility analysis for regulations that have a significant impact on a substantial number of small entities. This regulatory action does not have any adverse impact on either small or large entities. Therefore, a regulatory flexibility analysis is not required. Pursuant to section 605(b) of the Regulatory Flexibility Act, 5 U.S.C. 605(b), the Administrator certifies that this rule will not have a significant economic impact on a substantial number of small entities.

#### *D. Paperwork Reduction Act*

The information collection requirements in this proposed rule will be submitted for approval to the Office of Management and Budget (OMB) under the Paperwork Reduction Act, 44 U.S.C. 3501 *et seq.* shortly. EPA is preparing an information collection request (ICR) document for this proposed rule and will solicit public comment on it prior to promulgating a final regulation. Comments on the

proposed rule, preamble, and ICR will all be considered before a final rule is promulgated. The information collection requirements in this proposal are described in Parts III.A (Method Flexibility), III.B (Quality Control), III.C (Method Validation), III.D (Method Review), and III.E.6 (Standard Data Format). The information collection requirements in this proposal are specified in Appendix E (Equivalency Checklists), Appendix F (Guidelines and Format for Methods) and Appendix G (Method Flexibility, Equivalency, and Approval) of 40 CFR part 136 and at 40 CFR 136.3(d); 136.4 (b) and (c); 136.5 (a), (b), (c), and (d); and at 40 CFR 141.27 (a), (b), and (c).

The information requirements are not effective until OMB approves them. 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 are listed in 40 CFR part 9 and 48 CFR chapter 15.

#### **V. Request for Comments**

##### *A. General*

EPA is interested in eliciting constructive comments that would allow the Agency to incorporate flexibility into existing methods and to streamline the proposal and promulgation of new methods at 40 CFR parts 136 and 141. On the other hand, EPA is interested in compelling reasons why such a program may not work, even with extensive built-in controls to ensure that the results produced by modified or new analytical methods are reliable. EPA looks forward to working with all interested and concerned parties to produce an improved system for methods approval under the water methods program.

##### *B. Specific*

EPA is soliciting public comment on the following specific questions and options that relate to technical and policy decisions that EPA may need to make to implement the streamlining initiative.

1. As described in this preamble and the Streamlining Guide (EPA 1996a), the streamlining initiative would use a performance-based approach in which a reference method that contains or is supplemented with QC acceptance criteria is the standard against which a method modification would be tested to demonstrate equivalency. In contrast to the proposed performance-based reference-method approach, another performance-based approach would be to specify only the QC acceptance

criteria without the need for a reference method. Should EPA retain the proposed reference method approach with QC acceptance criteria? Or should EPA change to a QC acceptance criteria approach only?

2. Regarding question number one above, for what analytes, methods or monitoring situations, if any, do you believe EPA should allow use of either the performance-based reference method approach or the QC acceptance criteria only approach?

3. It may not be appropriate to develop QC acceptance criteria to allow modification of methods for "method-defined parameters," such as biochemical oxygen demand or total suspended solids. What chemical, microbiological, or biological analytes or analytical procedures do you believe might not be amenable to streamlining or method flexibility procedures?

4. Should EPA implement streamlining and method flexibility procedures only for new regulatory actions? Should EPA apply these procedures to existing regulatory requirements but only when these requirements are updated for some other purpose? Or should EPA apply these proposed procedures to existing regulations now?

5. EPA has undertaken several pilot studies of new methods to test the streamlined method approval process, and expects the pilots to be completed prior to promulgation of a final rule. Should EPA conduct more extensive pilot studies, e.g., several pilots at each tier, or should the changeover take place as soon as possible? If a pilot or phase-in approach is adopted, should EPA phase-in by analyte group (e.g., VOCs, metals, pesticides)? Or by the technologies employed by the reference method (electron capture, mass spectrometry)?

6. Is the proposed flexibility to modify the front-end and determinative steps in a reference method broad enough to be of value to the methods development community? For what steps in a reference method, if any, would you increase or decrease the flexibility to modify a method? If method flexibility were broadened, what additional standardized QC elements or checklist items should be added to ensure and document acceptable performance of the modification?

7. If you believe that the proposed flexibility is too broad for some methods, would you prefer that EPA limit flexibility by revising approved methods to indicate the steps that could or could not be changed? If yes, for which steps in a method (e.g., extraction/digestion, concentration,

determinative) or for which types of method (e.g., those with method-defined analytes) should changes be allowed or prohibited? If possible, please cite methods listed in 40 CFR part 136 or 141 as examples.

8. If method flexibility were implemented as proposed, are the standardized QC elements (accuracy, precision, detection limit, calibration, reference sample, matrix spikes, etc.) described in part III.C of this proposal and in the Streamlining Guide (EPA 1996a) adequate to validate the acceptability of a modification to a reference method? If not, which QC elements should be added? On the other hand, are the QC elements too extensive? If yes, which QC elements should be deleted? And why?

9. There has been some concern about the effect that changes to the chemistry of a method may have on a laboratory or method developer's ability to validate the performance of a modified method using the Checklists and other requirements in the Streamlining Guide (EPA 1996a). For example, what effect, if any, might changing the extraction solvent have on extract holding times that would not be picked up by the Checklists' criteria? What effect, if any, might use of a different extraction technique or a different solvent-to-sample ratio have that would not be picked up by the standardized QC? What, if any, QC elements should be changed or added to mitigate these concerns?

10. Once EPA adopts streamlining and method flexibility procedures, should EPA continue to develop and publish new methods or should EPA rely on the private sector and consensus standards organizations? In addressing this question, please consider the effect on small laboratories, PWSs, and POTWs, if EPA discontinued providing EPA methods.

11. EPA has determined that, for wastewater programs, a modified method, once validated and documented in accordance with the details in this proposal, would carry the same force and legal effect as a reference method. Do stakeholders believe that a modified method should have equal status with a reference method? Or should EPA require different levels of documentation for data gathered with the modified method? If a modified method had a different level of documentation, would stakeholders accept that it has legal status equal to that of an unmodified method?

12. Should EPA change the QC acceptance criteria in a reference method when a significant technological advance or some other factor

demonstrates that the criteria could be made more rigorous? In your response, you may assume that changing the criteria would not adversely decrease the number of qualified laboratories needed to conduct compliance monitoring with the more rigorous method.

13. EPA plans to implement streamlining and method flexibility for water methods through informal gathering of public comment and through rulemaking (**Federal Register** proposal, public comment, and final rule), of which this proposal is a part. Are there additional measures needed to ensure that all stakeholders would be aware of the initiative and, if so, what additional steps should EPA take?

14. Given that a laboratory would be able to modify a method without prior EPA approval, how would current EPA and state laboratory auditing and certification programs continue to ensure that the regulated community is properly conducting monitoring activities and documenting monitoring system performance? Should documentation be retained at the testing laboratory? At the facility? Or should EPA require that the data be submitted to EPA or other regulatory authority with each data package that results from use of the modification?

15. Adoption of streamlining and method flexibility procedures would require a deeper understanding of the science behind measurement methods. Consequently, "first-line" compliance and enforcement efforts may require additional resources and training of auditors. What training would EPA, the Regions, the States, laboratories, and the regulated community need to employ to successfully implement streamlining or method flexibility procedures? What courses could be developed, and who should be responsible for their development?

16. Under the streamlining initiative, requests for approval of new methods (i.e., new technologies or determinative techniques) would be submitted to EPA under a streamlined ATP-type program. Should EPA process these requests in the order received or should EPA have the discretion to accelerate review of methods that provide the most benefit to the Agency's regulatory program and/or to the needs of the regulated community?

17. What additional steps, if any, should the Agency take to ensure that the use of method flexibility does not compromise enforceability of applicable statutes and regulatory requirements? Will additional training be sufficient or will inspectors need additional qualifications to be able to assess the

quality of CWA and SDWA compliance data produced by a modified or new reference method? What resources would be required to mitigate concerns about the need for appropriate training of inspectors?

18. EPA proposes to define several administrative (e.g., Assistant Administrator, AMS Director) and technical (e.g., screening method, standardized quality control) terms in the definitions at 40 CFR 136.2 and 141.2 and invites public comment on these definitions. Should EPA omit any of the proposed definitions to avoid unnecessary confusion or restrictions? Are there additional terms or concepts for which a regulatory definition would be useful in implementing and administering EPA's proposed methods approval system?

19. EPA invites public comment on the guidance contained in the Streamlining Guide (EPA 1996a) and in Method Guidelines and Format (EPA 1996c). These documents, which are part of the administrative record for this proposal, provide guidance on method flexibility and method validation procedures under the proposed streamlining initiative. The documents also provide examples of certification statements and checklists that would satisfy EPA's proposed requirements for documenting the performance and equivalency of a modified or new method. Portions of these documents are proposed to be regulatory requirements (for example, see the proposed Appendixes E, F, and G and other amendments to 40 CFR parts 136 and 141). Which, if any, of the proposed requirements should EPA remove from the regulations and only keep as guidance?

20. In future rulemakings, EPA may propose to make more of the information in the two documents described above regulatory requirements. EPA would accomplish this by amending the wastewater and drinking water regulations or, with the approval of the Office of the Federal Register, incorporate by reference all or parts of the Streamlining Guide (EPA 1996a) and Method Guidelines and Format (EPA 1996c) into the CFR. What, if any, additional guidance from these documents should EPA propose as a regulatory requirement?

## VI. References

- APHA. 1995. Nineteenth edition of Standard Methods for the Examination of Water and Wastewater, 1992, American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.
- ASTM. 1996. Annual Book of ASTM Methods, 1996, Vol. 11.01 and 11.02,

American Society for Testing and Materials, 101 Barr Harbor Drive, West Conshohocken, PA 19428.

- EPA. 1995a. Protocol for Alternate Test Procedures for Coliform Bacteria in Compliance with Drinking Water Regulations: Presence/Absence Liquid Culture Methods for Finished Waters, Ver. 1.2, December 1995, U.S. Environmental Protection Agency.
- EPA. 1995b. Protocol for Alternate Test Procedures for Coliform Bacteria in Compliance with Drinking Water Regulations: Presence/Absence Membrane Filter Methods for Finished Waters, Ver. 1.2, December 1995, U.S. Environmental Protection Agency.
- EPA. 1995c. Methods for the Determination of Organic Compounds in Drinking Water—Supplement III, EPA-600/R-95-131, August 1995, NTIS PB95-261616.
- EPA. 1996a. Guide to Method Flexibility and Approval of EPA Water Methods, December 1996 Draft, EPA-821-D-96-004, NTIS PB97-117766, ERIC D-A43/D-A46 (Streamlining Guide, EPA 1996a).
- EPA. 1996b. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, December 1996, EPA-821-B-96-005, NTIS PB97-125298, ERIC D-A44/D-A47 (Organic Methods, EPA 1996b).
- EPA. 1996c. Guidelines and Format for Methods to Be Proposed at 40 CFR Part 136 or Part 141, EPA-821-B-96-003, NTIS PB96-210448, ERIC D-A42/D-A45, July 1996 (Method Guidelines and Format, EPA 1996c).
- EPA. 1996d. Draft Memorandum from Assistant Administrators, "Implementation Plan for the Agency Performance-Based Measurement System," October 25, 1996, U.S. Environmental Protection Agency.
- EPA. 1996e. Memorandum from Robert Perciasepe, "Agency-wide Adoption of the Performance-Based Measurement System Approach," November 1, 1996, U.S. Environmental Protection Agency.

## List of Subjects

### 40 CFR Part 136

Environmental protection, Laboratories, Water pollution control, Reporting and recordkeeping requirements.

### 40 CFR Part 141

Environmental protection, Laboratories, Water supply, Reporting and recordkeeping requirements.

Dated: March 17, 1997.

**Carol M. Browner,**  
Administrator.

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

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

1. The authority for part 136 is proposed to be revised 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.).

\* \* \* \* \*

2. Section 136.2 is proposed to be revised to read as follows:

### § 136.2 Definitions.

As used in this part, the term:

**Accuracy** means the degree of agreement between an observed value and an accepted reference value. Accuracy includes random error (precision) and systematic error (bias) that are caused by sampling and analysis.

**Act** means the Clean Water Act.

**Administrator** means the Administrator of the U.S. Environmental Protection Agency (EPA).

**Analyte or Analyte of concern** means a substance or property that is to be measured by an analysis.

**Approved method** means a testing procedure or analytical method promulgated at this part or at 40 CFR parts 405 through 500.

**Assistant Administrator (AA)** means the EPA Assistant Administrator for Water.

**Calibration (CAL)** means the process of establishing the relationship between the concentration or amount of material introduced into an instrument or measurement process and the output signal.

**Calibration linearity** means the degree to which calibration points lie along a straight line.

**Calibration verification** means the means of establishing that instrument performance remains within pre-established limits.

**Determinative technique** means the process (physical or chemical or both) to measure the identity and concentration of an analyte. In test methods, the determinative technique follows the front-end techniques.

**Director** means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

**Front-end technique** means any technique in the analytical process that precedes the determinative technique, including all procedures, equipment, solvents, etc. that are used in the laboratory in the preparation and cleanup of a sample but this excludes conditions and/or procedures for the collection, preservation, shipment and storage of the sample.

**Initial precision and recovery test (IPR)** means analysis of a minimum of four spiked replicate reference matrix samples under the same conditions as

will be used for analysis of environmental samples. The IPR is used to demonstrate that a laboratory is able to produce reliable results with the method prior to analysis of environmental samples.

*Interference* means a positive or negative effect on a measurement caused by a substance other than the analyte being determined.

*Matrix* means the component or substrate that contains the target analyte.

*Matrix spike (MS)* means a sample prepared by adding a known quantity of target analyte to a specified amount of a sample matrix for which an independent estimate of target analyte concentration is available.

*Matrix spike duplicate (MSD)* means a duplicate of the matrix spike. The MS/MSD are used in combination to test the precision of an analysis.

*Matrix type* means a sample medium with common characteristics across a given industrial category or industrial subcategory. Examples include: C-stage effluents from chlorine bleach mills in the Pulp, Paper, and Paperboard industrial category; effluent from the continuous casting subcategory of the Iron and Steel industrial category; publicly owned treatment work (POTW) sludge; and in-process streams in the Atlantic and Gulf Coast Hand-shucked Oyster Processing subcategory.

*Medium* means the physical phase of a sample matrix. Air, water, soil, sediment, rock, and sludge are sample media.

*Method* means an orderly and systematic arrangement of procedures and techniques for performing an analysis.

*Method blank* (or *blank*) means a sample absent the analytes of interest and interferences, which is processed through all steps of a method simultaneously with and under the same conditions as samples that may contain an analyte of interest.

*Method detection limit (MDL)* means the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero as determined by the procedure set forth in appendix B of this part.

*Method Guidelines and Format* means the procedures set forth in appendix F of this part.

*Method modification* means a change to a reference method. The change may be to a front-end technique or to the determinative technique.

*Method validation* means a process by which a laboratory or vendor establishes the performance of a new method or

substantiates the performance of a method modification.

*Minimum level (ML)* means the lowest level at which an entire analytical system gives a recognizable signal and acceptable calibration point for an analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and clean-up procedures have been employed.

*National Pollutant Discharge Elimination System (NPDES)* means the national system for the issuance of permits under section 402 of the Clean Water Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Clean Water Act.

*New method* means a combination of analyte of concern and determinative technique that is different from those in the approved methods.

*Ongoing precision and recovery sample (OPR)* means a spiked reference matrix sample that is processed through all steps of a method simultaneously with and under the same conditions as samples that may contain an analyte of interest. Also called a laboratory control sample (LCS), the OPR/LCS is used to demonstrate that a laboratory is able to produce reliable results continuously.

*Organic Methods* means the document titled: Methods for the Determination of Organic Compounds in Drinking Water—Supplement III (available from the National Technical Information Service (NTIS), U.S. Department of Commerce, Springfield, Virginia, 22161, 703/487-4600, at NTIS publication PB97-125298).

*Other approved method* means a promulgated method that is not designated as a reference method.

*Percent recovery* means the recovery multiplied by one hundred.

*Person* means an individual; corporation; company; association; partnership; municipality; or State, Federal, or tribal agency.

*Precision* means the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms.

*Preparation* means processing performed on a sample prior to analysis, including extraction, concentration, and cleanup.

*Procedure* means a set of systematic instructions for performing an activity.

*Promulgated method* means a method that has been published or incorporated

by reference into 40 CFR parts 136 or 405 through 500.

*Quality assurance (QA)* means an integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

*Quality control (QC)* means the overall system of technical activities conducted to measure and control the quality of a product or service so that it meets the needs of a user. The purpose of QC is to provide quality that is satisfactory, adequate, dependable, and economical.

*Quality control acceptance criteria (QC acceptance criteria)* means performance specifications developed from validation data and used to control the limits within which an analytical method is operated.

*Recovery* means the total amount of analyte found divided by the amount of analyte added as a spike.

*Reference method* means an approved method that is designated as a standard to which a modified method can be compared. A reference method includes standardized QC and QC acceptance criteria as well as sample preparation, cleanup, and other procedures.

*Regional Administrator* means an EPA Regional Administrator.

*Screening method* means a method that employs a qualitative determinative technique for an analyte of interest that is different from the determinative techniques used in the approved methods for that analyte. The screening method should produce a false negative probability less than 1%.

*Selectivity* means the capability of a method or instrument to respond to an analyte in the presence of interferences.

*Sensitivity* means the capability of a method or instrument to differentiate between different amounts or concentrations of an analyte.

*Spike* means the process of adding a known amount of an analyte to a sample to determine the recovery.

*Spike amount* means a known quantity of analyte added to a sample and used to determine the recovery of a method.

*Standard deviation* means the measure of the dispersion of observed values expressed as the positive square root of the sum of the squares of the difference between the individual values of a set and the arithmetic mean of the set, divided by one less than the number of values in the set.

*Standardized quality control (standardized QC)* means a uniform set of performance testing procedures that ensure reliable results. Depending on

the method, standardized QC procedures include, but are not limited to, the following: calibration, calibration linearity, calibration verification, absolute retention time, absolute and relative retention time precision, initial precision and recovery, ongoing precision and recovery (laboratory control sample), surrogate or labeled compound recovery, analysis of blanks, matrix spike and matrix spike duplicate recovery and precision, demonstration of method detection limit(s), and analysis of a reference sample.

*Surrogate* means a substance with properties that mimic the behavior of an analyte, that is unlikely to be found in an environmental sample, and that is added to the sample for quality control purposes.

*Tier 1* means the application of a new or modified method in a single laboratory to one or more matrix types.

*Tier 2* means the application of a new or modified method by all laboratories to one or more matrix types within a single industrial category or subcategory.

*Tier 3* means the application of a new or modified method by all laboratories to all matrix types in all industrial categories and subcategories (nationwide use).

3. Section 136.3 is proposed to be amended by revising the last two sentences and Tables IB, IC, and ID in paragraph (a); by adding Table IF in paragraph (a); by revising paragraphs (c) and (d); and by removing paragraph (e) (Table II following paragraph (e) is unchanged) to read as follows:

**§ 136.3 Identification of test procedures.**

\* \* \* \* \*

(a) \* \* \*

The discharge parameter values for which reports are required must be determined by one of the standard

analytical test procedures incorporated by reference and described in Tables IA, IB, IC, ID, and IE, or by any alternate test procedure which has been approved by the Administrator or Assistant Administrator under the provisions of paragraph (d) of this section and §§ 136.4 and 136.5. Under paragraphs (b), (c) of this section and 40 CFR 401.13 alternate test procedures may be used when such other test procedures have been previously approved by the Administrator, Assistant Administrator, or 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. Standardized QC and QC acceptance criteria for modifications of the inorganic contaminant reference methods in Table IB are specified in Table IF.

\* \* \* \* \*

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter/methodology	Reference method <sup>1, 35</sup>	Other approved methods				
		Standard methods 18th Ed. <sup>39</sup>	ASTM <sup>39</sup>	USGS <sup>2, 39</sup>	AOAC—Intl. <sup>39</sup>	Other
1. Acidity, as CaCO <sub>3</sub> , mg/L: Electrometric endpoint or phenolphthalein endpoint.	305.1	2310 B(4a)	D1067–92			
2. Alkalinity, as CaCO <sub>3</sub> , mg/L: Electrometric or Colorimetric titration to pH 4.5, manual or automated.	310.1 310.2	2320 B	D1067–92	I–1030–85 I–2030–85	973.43 <sup>3</sup>	
3. Aluminum—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by: AA direct aspiration <sup>36</sup> ..... AA furnace ..... Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP/AES). <sup>36</sup> Direct Current Plasma (DCP) <sup>36</sup> ..... Colorimetric (Eriochrome cyanine R) ....	202.1 202.2 <sup>5</sup> 200.7	3111 D 3113 B 3120 B		I–3051–85		
			D4190–82(88)			AES0029 <sup>34</sup>
4. Ammonia (as N), mg/L: Manual, distillation (at pH 9.5) <sup>6</sup> followed by: Nesslerization ..... Titration ..... Electrode ..... Automated phenate ..... Automated electrode .....	350.2 350.2 350.2 350.3 350.1	4500–NH <sub>3</sub> B 4500–NH <sub>3</sub> C 4500–NH <sub>3</sub> E 4500–NH <sub>3</sub> F or G 4500–NH <sub>3</sub> H	D1426–93(A) D1426–93(B)	I–3520–85 I–4523–85	973.49 <sup>3</sup> 973.49 <sup>3</sup>	379–75WE <sup>7</sup>
5. Antimony—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by: AA direct aspiration <sup>36</sup> ..... AA furnace ..... ICP/AES <sup>36</sup> .....	204.1 204.2 <sup>5</sup> 200.7	3111 B 3113 B 3120 B				
6. Arsenic—Total, <sup>4</sup> mg/L: Digestion <sup>4</sup> followed by ..... AA gaseous hydride ..... AA furnace ..... ICP/AES <sup>36</sup> ..... Colorimetric (SDDC) .....	206.5 206.3 206.2 <sup>5</sup> 200.7 206.4	3114 B 4.d 3113 B 3120 B 3500–As C	D2972–93(B) D2972–93(C) D2972–93(A)	I–3062–85 I–3060–85		
7. Barium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by: AA direct aspiration <sup>36</sup> ..... AA furnace ..... ICP/AES <sup>36</sup> ..... DCP <sup>36</sup> .....	208.1 208.2 <sup>5</sup> 200.7	3111 D 3113 B 3120 B	D4382–91	I–3084–85		AES0029 <sup>34</sup>
8. Beryllium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter/methodology	Reference method <sup>1, 35</sup>	Other approved methods				
		Standard methods 18th Ed. <sup>39</sup>	ASTM <sup>39</sup>	USGS <sup>2, 39</sup>	AOAC—Intl. <sup>39</sup>	Other
AA direct aspiration .....	210.1	3111 D	D3645–93(88)(A)	I–3095–85		
AA furnace .....	210.2	3113 B	D3645–93(88)(B)			
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
DCP .....			D4190–82(88)			AES0029 <sup>34</sup>
Colorimetric (aluminon) .....		3500–Be D				
9. Biochemical oxygen demand (BOD <sub>5</sub> ), mg/L:						
Dissolved Oxygen Depletion .....	405.1	5210 B		I–1578–78 <sup>8</sup>	973.44 <sup>3</sup>	p. 17 <sup>9</sup>
10. Boron <sup>37</sup> —Total, mg/L:						
Colorimetric (curcumin) .....	212.3	4500–B B		I–3112–85		
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
DCP .....			D4190–82(88)			AES0029 <sup>34</sup>
11. Bromide, mg/L:						
Titrimetric .....	320.1		D1246–82(88)(C)	I–1125–85		p. S44 <sup>10</sup>
12. Cadmium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration <sup>36</sup> .....	213.1	3111 B or C	D3557–90 (A or B)	I–3135–85 or I–3136–85	974.27 <sup>3</sup>	p. 37 <sup>9</sup>
AA furnace .....	213.2	3113 B	D3557–90(C)			
ICP/AES <sup>36</sup> .....	<sup>5</sup> 200.7	3120 B		I–1472–85		
DCP <sup>36</sup> .....			D4190–82(88)			AES0029 <sup>34</sup>
Voltametry <sup>11</sup> .....			D3557–90(C)			
Colorimetric (Dithizone) .....		3500–Cd D				
13. Calcium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	215.1	3111B	511–93(B)	I–3152–85		
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
DCP .....						AES0029 <sup>34</sup>
Titrimetric (EDTA) .....	215.2	3500–Ca D	511–93(A)			
14. Carbonaceous biochemical oxygen demand (CBOD <sub>5</sub> ), mg/L <sup>12</sup> :						
Dissolved Oxygen Depletion with nitrification inhibitor .....		5210B				
15. Chemical oxygen demand (COD), mg/L; Titrimetric						
.....	410.1	5220 C	D1252–88(A)	I–3560–85	973.46 <sup>3</sup>	p. 17 <sup>9</sup>
.....	410.2			I–3562–85		
.....	410.3					
Spectrophotometric, manual or automated .....	410.4	5220 D	D1252–88(B)	I–3561–85		Notes 13 or 14
16. Chloride, mg/L:						
Titrimetric (silver nitrate) .....		4500–Cl–B	D512–89(B)	I–1183–85		
(Mercuric nitrate) .....	325.3	4500–Cl–C	D512–89(A)	I–1184–85	973.51 <sup>3</sup>	
Colorimetric, manual .....				I–1187–85		
Automated (Ferricyanide) .....	325.1 or 325.2	4500–Cl–E		I–2187–85		
17. Chlorine—Total residual, mg/L; Titrimetric:						
Amperometric direct .....	330.1	4500–Cl D	D1253–86(92)			
Iodometric direct .....	330.3	4500–Cl B				
Back titration ether end-point <sup>15</sup> .....	330.2	4500–Cl C				
DPD-FAS .....	330.4	4500–Cl F				
Spectrophotometric, DPD .....	330.5	4500–Cl G				
or Electrode .....						Note 16
18. Chromium VI dissolved, mg/L; 0.45 micron filtration followed by:						
AA chelation-extraction .....	218.4	3111 C		I–1232–85		
Colorimetric (Diphenylcarbazide) .....		3500–Cr D	D1687–92(A)	I–1230–85		
19. Chromium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration <sup>36</sup> .....	218.1	3111 B	D1687–92(B)	I–3236–85	974.27 <sup>3</sup>	
AA chelation-extraction .....	218.3	3111 C				
AA furnace .....	218.2	3113 B	D1687–92(C)			
ICP/AES <sup>36</sup> .....	<sup>5</sup> 200.7	3120 B				
DCP <sup>36</sup> .....			D4190–82(88)			AES0029 <sup>34</sup>
Colorimetric (Diphenylcarbazide) .....		3500–Cr D				

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter/methodology	Reference method <sup>1, 35</sup>	Other approved methods				
		Standard methods 18th Ed. <sup>39</sup>	ASTM <sup>39</sup>	USGS <sup>2, 39</sup>	AOAC—Intl. <sup>39</sup>	Other
20. Cobalt-Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	219.1	3111 B or C	D3558–90(A or B)	I–3239–85		p. 37 <sup>9</sup>
AA furnace .....	219.2	3113 B	D3558–90(C)			
ICP/AES .....	<sup>5</sup> 200.7	3120B				
DCP .....			D4190–82(88)			AES0029 <sup>34</sup>
21. Color platinum cobalt units or dominant wavelength, hue, luminance purity:						
Colorimetric (ADMI) .....	110.1	2120 E				Note 18
(Platinum cobalt) .....	110.2	2120 B		I–1250–85		
Spectrophotometric .....	110.3	2120 C				
22. Copper—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration <sup>36</sup> .....	<sup>5</sup> 220.1	3111 B or C	D1688–90(A or B)	I–3270–85 or I–3271–85	974.27 <sup>3</sup>	p. 37 <sup>9</sup>
AA furnace .....	220.2	3113 B	D1688–90(C)			
ICP/AES <sup>36</sup> .....	<sup>5</sup> 200.7	3120 B				
DCP <sup>36</sup> .....			D4190–82(88)			AES0029 <sup>34</sup>
Colorimetric (Neocuproine) .....		3500–Cu D				8506 <sup>19</sup>
(Bicinchoninate) .....		Or E				
23. Cyanide—Total, mg/L:						
Manual distillation with MgCl <sub>2</sub> followed by:		4500–CN C	D2036–91(A)			
Titrimetric .....		4500–CN D				p. 22 <sup>9</sup>
Spectrophotometric, manual .....	<sup>31</sup> 335.2	4500–CN E	D2036–91(A)	I–3300–85		
Automated <sup>20</sup> .....	<sup>31</sup> 335.3					
24. Cyanide amenable to chlorination, mg/L:						
Manual distillation with MgCl <sub>2</sub> followed by titrimetric or Spectrophotometric.	335.1	4500–CN G	D2036–91(B)			
25. Fluoride—Total, mg/L:						
Manual distillation <sup>6</sup> followed by .....		4500–F B				
Electrode, manual .....	340.2	4500–F C	D1179–93(B)			
Automated .....				I–4327–85		
Colorimetric (SPADNS) .....	340.1	4500–F D	D1179–93(A)			
Automated complexone .....	340.3	4500–F E				
26. Gold—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	231.1	3111 B				
AA furnace .....	231.2					
DCP .....						AES0029 <sup>34</sup>
27. Hardness—Total, as CaCO <sub>3</sub> , mg/L:						
Automated colorimetric .....	130.1					
Titrimetric (EDTA), or Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33)..	130.2	2340 B or C	D1126–86(92)	I–1338–85	973.52B <sup>3</sup>	
28. Hydrogen ion (pH), pH units:						
Electrometric measurement .....	150.1	4500–H <sup>+</sup> B	D1293–84(90) (A or B)	I–1586–85	973.41 <sup>3</sup>	
Automated electrode .....						378–75WA <sup>21</sup>
29. Iridium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	235.1	3111 B				
AA furnace .....	235.2					
30. Iron—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration <sup>36</sup> .....	236.1	3111 B or C	D1068–90 (A or B)	I–3381–85	974.27 <sup>3</sup>	
AA furnace .....	236.2	3113 B	D1068–90(C)			
ICP/AES <sup>36</sup> .....	<sup>5</sup> 200.7	3120 B				
DCP <sup>36</sup> .....			D4190–82(88)			AES0029 <sup>34</sup>
Colorimetric (Phenanthroline) .....		3500–Fe D	D1068–90(C)			8008 <sup>22</sup>
31. Kjeldahl Nitrogen—Total, (as N), mg/L:						
Digestion and distillation followed by ....	351.3	4500–NH <sub>3</sub> B or C	D3590–89(A)		973.48 <sup>3</sup>	
Titration .....	351.3	4500–NH <sub>3</sub> E	D3590–89(A)			
Nesslerization .....	351.3	4500–NH <sub>3</sub> C	D3590–89(A)			



TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter/methodology	Reference method <sup>1, 35</sup>	Other approved methods				
		Standard methods 18th Ed. <sup>39</sup>	ASTM <sup>39</sup>	USGS <sup>2, 39</sup>	AOAC—Intl. <sup>39</sup>	Other
Electrode .....	351.3	4500—NH <sub>3</sub> F or G				
Automated phenate colorimetric .....	351.1			I-4551-78 <sup>8</sup>		
Semi-automated block digester colorimetric .....	351.2		D3590-89(B)			
Manual or block digester Potentiometric .....	351.4		D3590-89(A)			
32. Lead—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration <sup>36</sup> .....	239.1	3111 B or C	D3559-90 (A or B)	I-3399-85	974.27 <sup>3</sup>	
AA furnace .....	239.2	3113 B	D3559-90(C)			
ICP/AES <sup>36</sup> .....	<sup>5</sup> 200.7	3120 B				
DCP <sup>36</sup> .....			D4190-82(88)			AES0029 <sup>34</sup>
Voltametry <sup>11</sup> .....			D3559-90(C)			
Colorimetric (Dithizone) .....		3500—Pb D				
33. Magnesium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	242.1	3111 B	D511-93(B)	I-3447-85	974.27 <sup>3</sup>	
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
DCP .....						AES0029 <sup>34</sup>
Gravimetric .....		3500—Mg D				
34. Manganese—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration <sup>36</sup> .....	243.1	3111 B	D858-90 (A or B)	I-3454-85	974.27 <sup>3</sup>	
AA furnace .....	243.2	3113 B	D858-90(C)			
ICP/AES <sup>36</sup> .....	200.7	3120 B				
DCP <sup>36</sup> .....	<sup>5</sup> 200.7	3120 B				AES0029 <sup>34</sup>
Colorimetric (Persulfate) .....		3500—Mn D			920.203 <sup>3</sup>	8034 <sup>23</sup>
(Periodate) .....						
35. Mercury—Total <sup>4</sup> , mg/L:						
Cold vapor, manual .....	245.1	3112 B	D3223-91	I-3462-85	977.22 <sup>3</sup>	
Automated .....	245.2					
36. Molybdenum—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	246.1	3111 D		I-3490-85		
AA furnace .....	246.2	3113 B				
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
DCP .....						AES0029 <sup>34</sup>
37. Nickel—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration <sup>36</sup> .....	249.1	3111 B or C	D1886-90 (A or B)	I-3499-85		
AA furnace .....	249.2	3113 B	D1886-90(C)			
ICP/AES <sup>36</sup> .....	<sup>5</sup> 200.7	3120 B				
DCP <sup>36</sup> .....			D4190-82(88)			AES0029 <sup>34</sup>
Colorimetric (heptoxime) .....		3500—Ni D				
38. Nitrate (as N), mg/L:						
Colorimetric (Brucine sulfate), or Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).	352.1				973.50 <sup>3</sup>	419 D <sup>17</sup> , p. 28 <sup>9</sup>
39. Nitrate-nitrite (as N), mg/L:						
Cadmium reduction, manual .....	353.3	4500—NO <sub>3</sub> <sup>-</sup> E	D3867-90(B)			
Automated .....	353.2	4500—NO <sub>3</sub> <sup>-</sup> F	D3867-90(A)	I-4545-85		
Automated hydrazine .....	353.1	4500—NO <sub>3</sub> <sup>-</sup> H				
40. Nitrite (as N), mg/L; Spectrophotometric:						
Manual .....	354.1	4500—NO <sub>2</sub> <sup>-</sup> B				8507 <sup>25</sup>
Automated (Diazotization) .....				I-4540-85		
41. Oil and grease—Total recoverable, mg/L:						
Gravimetric (extraction) .....	413.1	5520 B <sup>38</sup>				
42. Organic carbon—Total (TOC), mg/L:						
Combustion or oxidation .....	415.1	5310 B, C, or D	D2579-93 (A or B)		973.47 <sup>3</sup>	p. 14 <sup>24</sup>
43. Organic nitrogen (as N), mg/L:						
Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4) .....						
44. Orthophosphate (as P), mg/L Ascorbic acid method:						
Automated .....	365.1	4500—P F		I-4601-85	973.56 <sup>3</sup>	

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter/methodology	Reference method <sup>1, 35</sup>	Other approved methods				
		Standard methods 18th Ed. <sup>39</sup>	ASTM <sup>39</sup>	USGS <sup>2, 39</sup>	AOAC—Intl. <sup>39</sup>	Other
Manual single reagent .....	365.2	4500-P E	D515-88(A)		973.55 <sup>3</sup>	
Manual two reagent .....	365.3					
45. Osmium—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	252.1	3111 D				
AA furnace .....	252.2					
46. Oxygen, dissolved, mg/L:						
Winkler (Azide modification) .....	360.2	4500-O C	D888-92(A)	I-1575-78 <sup>8</sup>	973.45B <sup>3</sup>	
Electrode .....	360.1	4500-O G	D888-92(B)	I-1576-78 <sup>8</sup>		
47. Palladium—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	253.1	3111 B				p. S27 <sup>10</sup>
AA furnace .....	253.2					p. S28 <sup>10</sup>
DCP .....						AES0029 <sup>34</sup>
48. Phenols, mg/L:						
Manual distillation <sup>26</sup> .....	420.1					Note 27
Followed by:						
Colorimetric (4AAP) manual .....	420.1					Note 27
Automated <sup>19</sup> .....	420.2					
49. Phosphorus (elemental), mg/L:						
Gas-liquid chromatography .....						Note 28
50. Phosphorus—Total, mg/L:						
Persulfate digestion followed by .....	365.2	4500-P B,5			973.55 <sup>3</sup>	
Manual .....	365.2 or 365.3	4500-P E	D515-88(A)			
Automated ascorbic acid reduction .....	365.1	4500-P F		I-4600-85	973.56 <sup>3</sup>	
Semi-automated block digester .....	365.4		D515-88(B)			
51. Platinum—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	255.1	3111 B				
AA furnace .....	255.2					
DCP .....						AES0029 <sup>34</sup>
52. Potassium—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	258.1	3111 B		I-3630-85	973.53 <sup>3</sup>	
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
Flame photometric .....		3500-K D				317 B <sup>17</sup>
Colorimetric .....						
53. Residue—Total, mg/L:						
Gravimetric, 103°–105° .....	160.3	2540 B		I-3750-85		
54. Residue—filterable, mg/L:						
Gravimetric, 180° .....	160.1	2540 C		I-1750-85		
55. Residue—nonfilterable (TSS), mg/L:						
Gravimetric, 103°–105° post washing of residue .....	160.2	2540 D		I-3765-85		
56. Residue—settleable, mg/L:						
Volumetric, (Imhoff cone), or gravimetric .....	160.5	2540 F				
57. Residue—Volatile, mg/L:						
Gravimetric, 550° .....	160.4			I-3753-85		
58. Rhodium—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	265.1	3111 B				
AA furnace .....	265.2					
59. Ruthenium—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	267.1	3111 B				
AA furnace .....	267.2					
60. Selenium—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:						
AA furnace .....	270.2	3113 B	D3859-93(B)			
ICP/AES <sup>36</sup> .....	<sup>5</sup> 200.7	3120 B				
AA gaseous hydride .....		3114 B	D3859-93(A)	I-3667-85		
61. Silica <sup>37</sup> —Dissolved, mg/L; 0.45 micron filtration followed by:						
Colorimetric, manual .....	370.1	4500-Si D	D859-88	I-1700-85		
Automated (Molybdosilicate) .....				I-2700-85		
ICP .....	<sup>5</sup> 200.7	3120 B				
62. Silver—Total <sup>4</sup> , mg/L; Digestion <sup>4, 29</sup> followed by:						

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter/methodology	Reference method <sup>1, 35</sup>	Other approved methods				
		Standard methods 18th Ed. <sup>39</sup>	ASTM <sup>39</sup>	USGS <sup>2, 39</sup>	AOAC—Intl. <sup>39</sup>	Other
AA direct aspiration .....	272.1	3111 B or C		I-3720-85	974.27 <sup>3</sup>	p. 37 <sup>9</sup>
AA furnace .....	272.2	3113 B				
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
DCP .....						AES0029 <sup>34</sup>
63. Sodium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	273.1	3111 B		I-3735-85	973.54 <sup>3</sup>	
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
DCP .....						AES0029 <sup>34</sup>
Flame photometric .....		3500 Na D				
64. Specific conductance, micromhos/cm at 25 °C:						
Wheatstone bridge .....	120.1	2510 B	D1125-91(A)	I-1780-85	973.40 <sup>3</sup>	
65. Sulfate (as SO <sub>4</sub> ), mg/L:						
Automated colorimetric (barium chloranilate) .....	375.1					
Gravimetric .....	375.3	4500-SO <sub>4</sub> <sup>-2</sup> C or D			925.54 <sup>3</sup>	
Turbidimetric .....	375.4		D516-90			426C <sup>30</sup>
66. Sulfide (as S), mg/L:						
Titrimetric (iodine) .....	376.1	4500-S <sup>-2</sup> E		I-3840-85		
Colorimetric (methylene blue) .....	376.2	4500-S <sup>-2</sup> D				
67. Sulfite (as SO <sub>3</sub> ), mg/L:						
Titrimetric (iodine-iodate) .....		377.1	4500-SO <sub>3</sub> <sup>-2</sup> B			
68. Surfactants, mg/L:						
Colorimetric (methylene blue) .....	425.1	5540 C	D2330-88			
69. Temperature, °C:						
Thermometric .....	170.1	2550 B				Note 32
70. Thallium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	279.1	3111 B				
AA furnace .....	279.2					
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
71. Tin—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	282.1	3111 B		I-3850-78 <sup>8</sup>		
AA furnace .....	282.2	3113 B				
ICP/AES .....	<sup>5</sup> 200.7					
72. Titanium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	283.1	3111 D				
AA furnace .....	283.2					
DCP .....						AES0029 <sup>34</sup>
73. Turbidity, NTU:						
Nephelometric .....	180.1	2130 B	D1889-88(A)	I-3860-85		
74. Vanadium—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration .....	286.1	3111 D				
AA furnace .....	286.2		D3373-93			
ICP/AES .....	<sup>5</sup> 200.7	3120 B				
DCP .....			D4190-82(88)			AES0029 <sup>34</sup>
Colorimetric (Gallic acid) .....		3500-V D				
75. Zinc—Total, <sup>4</sup> mg/L; Digestion <sup>4</sup> followed by:						
AA direct aspiration <sup>36</sup> .....	289.1	3111 B or C	D1691-90(A or B)	I-3900-85	974.27 <sup>3</sup>	p. 37 <sup>9</sup>
AA furnace .....	289.2					
ICP/AES <sup>36</sup> .....	<sup>5</sup> 200.7	3120 B				
DCP <sup>36</sup> .....			D4190-82(88)			AES00290 <sup>34</sup>
Colorimetric (Dithizone) .....		3500-Zn E				
(Zincon) .....		3500-Zn F				8009 <sup>33</sup>

Table IB notes:

<sup>1</sup> "Methods for Chemical Analysis of Water and Wastes", Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-CI), EPA-600/4-79-020, Revised March 1983 and 1979 where applicable.

<sup>2</sup> 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.

<sup>3</sup> "Official Methods of Analysis of the Association of Official Analytical Chemists", methods manual, 15th ed. (1990).

<sup>4</sup>For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979 and 1983". One (Section 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (Section 4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all sample types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials may also benefit by this vigorous digestion, however, vigorous digestion with concentrated nitric acid will convert antimony and tin to insoluble oxides and render them unavailable for analysis. Use of ICP/AES as well as determinations for certain elements such as antimony, arsenic, the noble metals, mercury, selenium, silver, tin, and titanium require a modified sample digestion procedure and in all cases the method write-up should be consulted for specific instructions and/or cautions.

NOTE: If the digestion procedure for direct aspiration AA included in one of the other approved references is different than the above, the EPA procedure must be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion of the filtrate for dissolved metals (or digestion of the original sample solution for total metals) may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses, provided the sample solution to be analyzed meets the following criteria:

- a. has a low COD (<20),
- b. is visibly transparent with a turbidity measurement of 1 NTU or less,
- c. is colorless with no perceptible odor, and
- d. is of one liquid phase and free of particulate or suspended matter following acidification.

<sup>5</sup>The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes", is given at Appendix C of this Part 136.

<sup>6</sup>Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

<sup>7</sup>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, N.Y. 10523.

<sup>8</sup>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).

<sup>9</sup>American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

<sup>10</sup>"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).

<sup>11</sup>The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

<sup>12</sup>Carbonaceous biochemical oxygen demand (CBOD<sub>5</sub>) must not be confused with the traditional BOD<sub>5</sub> test which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD<sub>5</sub> parameter. A discharger whose permit requires reporting the traditional BOD<sub>5</sub> may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD<sub>5</sub> is required can the permittee report data using the nitrification inhibitor.

<sup>13</sup>OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.

<sup>14</sup>Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

<sup>15</sup>The back titration method will be used to resolve controversy.

<sup>16</sup>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.

<sup>17</sup>The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976.

<sup>18</sup>National Council of the Paper Industry for Air and Stream Improvement, (Inc.) Technical Bulletin 253, December 1971.

<sup>19</sup>Copper, Biocinchonate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

<sup>20</sup>After the manual distillation is completed, the autoanalyzer manifolds in EPA Methods 335.3 (cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

<sup>21</sup>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.

<sup>22</sup>Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

<sup>23</sup>Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, CO 80537.

<sup>24</sup>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.

<sup>25</sup>Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

<sup>26</sup>Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1+9 NaOH.

<sup>27</sup>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 spectrophotometric procedure.

<sup>28</sup>R.F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography", Journal of Chromatography, Vol. 47, No. 3, pp. 421-426, 1970.

<sup>29</sup>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<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

<sup>30</sup>The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition.

<sup>31</sup>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.

<sup>32</sup>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.

<sup>33</sup>Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333, Hach Chemical Company, Loveland, CO 80537.

<sup>34</sup>"Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029", 1986—Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923.

<sup>35</sup>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".

<sup>36</sup>"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.

<sup>37</sup>When determining boron and silica, only plastic, PTFE, or quartz sampling and laboratory ware may be used from time of collection until completion of analysis.

<sup>38</sup> Only the trichlorofluoromethane extraction solvent is approved.

<sup>39</sup> Methods published by this organization and approved for use under this part may not be modified beyond the modifications expressly allowed and defined in each method.

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

Parameter <sup>1</sup> /methodology	Reference method <sup>27</sup>	Other approved methods		
		Standard methods 18th Ed. <sup>8</sup>	ASTM <sup>8</sup>	Other
1. Acenaphthene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
2. Acenaphthylene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
3. Acrolein:				
GC/FID .....	603			
GC/MS .....	<sup>4</sup> 604			
GC/MS/Isotope .....	1624			
4. Acrylonitrile:				
GC/FID .....	603			
GC/MS .....	<sup>4</sup> 624			
GC/MS/Isotope .....	1624			
HPLC/UV .....	610			
5. Anthracene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
6. Benzene:				
GC/PID .....	602	7220 B		
GC/MS .....	624			
GC/MS/Isotope .....	1624			
HPLC/UV .....				
7. Benzidine:	Note 3,		p. 1.	
GC/MS .....	<sup>5</sup> 625			
GC/MS/Isotope .....	1625			
HPLC/ELCD .....	605			
8. Benzo(a)anthracene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6440 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
9. Benzo(a)pyrene:				
GC/FID .....	610	6410 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
10. Benzo(b)fluoranthene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
11. Benzo(g, h, i)perylene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
12. Benzo(k)fluoranthene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
13. Benzyl chloride .....				Note 3, p. 130: Note 6, p. S102.
14. Benzyl butyl phthalate:				
GC/ECD .....	606			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			

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

Parameter <sup>1</sup> /methodology	Reference method <sup>27</sup>	Other approved methods		
		Standard methods 18th Ed. <sup>8</sup>	ASTM <sup>8</sup>	Other
15. Bis(2-chloroethoxy) methane:				
GC/ELCD .....	611			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
16. Bis(2-chloroethyl) ether:				
GC/ELCD .....	611			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
17. Bis (2-ethylhexyl) phthalate:				
GC/ECD .....	606	6230 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
18. Bromodichloromethane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
19. Bromoform:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
20. Bromomethane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6410 B		
GC/MS/Isotope .....	1624			
21. 4-Bromophenylphenyl ether:				
GC/ELCD .....	611			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
22. Carbon tetrachloride:	Note 3,			p. 130.
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6410 B		
GC/MS/Isotope .....	1624			
23. 4-Chloro-3-methylphenol:				
GC/FID .....	604	6420 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
24. Chlorobenzene:	Note 3,			p. 130.
GC/ELCD .....	601	6230 B		
GC/PID .....	602	6220 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
25. Chloroethane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
26. 2-Chloroethylvinyl ether:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
27. Chloroform:				Note, p. 130.
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
28. Chloromethane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
29. 2-Chloronaphthalene:				
GC/ECD .....	612			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
30. 2-Chlorophenol:				
GC/FID .....	604	6420 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
31. 4-Chlorophenylphenyl ether:				
GC/ELCD .....	611			
GC/MS .....	625	6410 B		

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

Parameter <sup>1</sup> /methodology	Reference method <sup>27</sup>	Other approved methods		
		Standard methods 18th Ed. <sup>8</sup>	ASTM <sup>8</sup>	Other
GC/MS/Isotope .....	1625			
32. Chrysene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
33. Dibenzo(a,h)anthracene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
34. Dibromochloromethane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
35. 1,2-Dichlorobenzene:				
GC/ELCD .....	601	6230 B		
GC/PID .....	602	6220 B		
GC/ECD .....	612			
GC/MS .....	624, 625	6410 B		
GC/MS/Isotope .....	1625			
36. 1,3-Dichlorobenzene:				
GC/ELCD .....	601	6230 B		
GC/PID .....	602	6220 B		
GC/ECD .....	612			
GC/MS .....	624, 625	6410 B		
GC/MS/Isotope .....	1625			
37. 1,4-Dichlorobenzene:				
GC/ELCD .....	601	6230 B		
GC/PID .....	602	6220 B		
GC/ECD .....	612			
GC/MS .....	624, 625	6410 B		
GC/MS/Isotope .....	1625			
38. 3,3-Dichlorobenzidine:				
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/ELCD .....	605			
39. Dichlorodifluoromethane:				
GC/ELCD .....	601	6230 B		
40. 1,1-Dichloroethane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
41. 1,2-Dichloroethane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
42. 1,1-Dichloroethene:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
43. trans-1,2-Dichloroethene:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
44. 2,4-Dichlorophenol:				
GC/FID .....	604	6420 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
45. 1,2-Dichloropropane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
46. cis-1,3-Dichloropropene:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
47. trans-1,3-Dichloropropene:				



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

Parameter <sup>1</sup> /methodology	Reference method <sup>27</sup>	Other approved methods		
		Standard methods 18th Ed. <sup>8</sup>	ASTM <sup>8</sup>	Other
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
48. Diethyl phthalate:				
GC/ECD .....	606			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
49. 2,4-Dimethylphenol:				
GC/FID .....	604	6420 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
50. Dimethyl phthalate:				
GC/ECD .....	606			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
51. Di-n-butyl phthalate:				
GC/ECD .....	606			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
52. Di-n-octyl phthalate:				
GC/ECD .....	606			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
53. 2,3-Dinitrophenol:				
GC/FID .....	604	6420 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
54. 2,4-Dinitrotoluene:				
GC/ECD .....	609			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
55. 2,6-Dinitrotoluene				
GC/ECD .....	609			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
56. Epichlorohydrin .....	.....			Note 3, p. 130; Note 6, p. S102.
57. Ethylbenzene:				
GC/PID .....	602	6220 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
58. Fluoranthene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657-92	
59. Fluorene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440B	D4657-92	
60. Hexachlorobenzene:				
GC/ECD .....	612			
GC/MS .....	625	6410B		
GC/MS/Isotope .....	1625			
61. Hexachlorobutadiene:				
GC/ECD .....	612			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
62. Hexachlorocyclopentadiene:				
GC/ECD .....	612			
GC/MS .....	<sup>5</sup> 625	6410 B		
GC/MS/Isotope .....	1625			
63. Hexachloroethane:	.....			616
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
64. Ideno(1,2,3-cd) pyrene:				

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

Parameter <sup>1</sup> /methodology	Reference method <sup>27</sup>	Other approved methods		
		Standard methods 18th Ed. <sup>8</sup>	ASTM <sup>8</sup>	Other
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657–92	
65. Isophorone:				
GC/ECD .....	609			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
66. Methylene chloride:				Note 3, p. 130
GC/ELCD .....	601	6230 B		
GC/MS .....	624			
GC/MS/Isotope .....	1624			
67. 2-Methyl-4,6-dinitrophenol:				
GC/ECD .....		6420 B		
GC/FID .....	604	6420 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
68. Naphthalene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B		
69. Nitrobenzene:				
GC/ECD .....	609			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
70. 2-Nitrophenol:				
GC/ECD .....		6420 B		
GC/FID .....	604	6420 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
71. 4-Nitrophenol:				
GC/ECD .....		6420B		
GC/FID .....	604	6420B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
72. N-Nitrosodimethylamine:				
GC/NPD .....	607			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
73. N-Nitrosodi-n-propylamine:				
GC/NPD .....	607			
GC/MS .....	<sup>5</sup> 625	6410 B		
GC/MS/Isotope .....	1625			
74. N-Nitrosodiphenylamine:				
GC/NPD .....	607			
GC/MS .....	<sup>5</sup> 625	6410B		
GC/MS/Isotope .....	1625			
75. 2,2-Oxybis(1-chloropropane):				
GC/ELCD .....	611			
GC/MS .....	614	6410 B		
GC/MS/Isotope .....	1625			
76. PCB–1016:				Note 3, p. 43.
GC/ECD .....	608			
GC/MS .....	625	6410 B		
77. PCB–1221:				Note 3, p. 43.
GC/ECD .....	608			
GC/MS .....	625	6410 B		
78. PCB–1232:				Note 3, p. 43.
GC/ECD .....	608			
GC/MS .....	625	6410 B		
79. PCB–1242:				Note 3, p. 43.
GC/ECD .....	608			
GC/MS .....	625	6410 B		
80. PCB–1248:				
GC/ECD .....	608			
GC/MS .....	625			
81. PCB–1254:				Note 3, p. 43.

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

Parameter <sup>1</sup> /methodology	Reference method <sup>27</sup>	Other approved methods		
		Standard methods 18th Ed. <sup>8</sup>	ASTM <sup>8</sup>	Other
GC/ECD .....	608			
GC/MS .....	625	6410 B		
82. PCB-1260:				Note 3, p. 43.
GC/ECD .....	608	6630 B		
GC/MS .....	625	6410 B		
83. Pentachlorophenol:				Note 3, p. 140.
GC/ECD .....		6630 B		
GC/FID .....	604			
GC/MS .....	625			
GC/MS/Isotope .....	1625	6410 B		
84. Phenanthrene:				
GC/FID .....	610	6440 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440 B	D4657-92	
85. Phenol:				
GC/FID .....	604	6420 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
86. Pyrene:				
GC/FID .....	610	6440 B		
GC/MS .....	625			
GC/MS/Isotope .....	1625			
HPLC/UV .....	610	6440B	D4675-92	
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin:				Note 3, p. 130.
GC/MS .....	<sup>5a</sup> 613			
88. 1,1,2,2-Tetrachloroethane:				Note 3, p. 130.
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
89. Tetrachloroethene:				Note 3, p. 130.
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
90. Toluene:				
GC/PID .....	602	6220 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
91. 1,2,4-Trichlorobenzene:				Note 3, p. 130.
GC/ECD .....	612			
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
92. 1,1,1-Trichloroethane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
93. 1,1,2-Trichloroethane:				Note 3, p. 130.
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
94. Trichloroethene:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			
95. Trichlorofluoromethane:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
96. 2,4,6-Trichlorophenol:				
GC/FID .....	604	6240 B		
GC/MS .....	625	6410 B		
GC/MS/Isotope .....	1625			
97. Vinyl chloride:				
GC/ELCD .....	601	6230 B		
GC/MS .....	624	6210 B		
GC/MS/Isotope .....	1624			

Table IC notes:

<sup>1</sup> All parameters are expressed in micrograms per liter (µg/L).

<sup>2</sup>The full text of Methods 601–613, 624, 625, 1624, and 1625, 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.

<sup>3</sup>“Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater”, U.S. Environmental Protection Agency, September 1978.

<sup>4</sup>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 1624.

<sup>5</sup>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 1625, are preferred methods for these compounds.

<sup>5a</sup> 625, Screening only.

<sup>6</sup>“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).

<sup>7</sup>Each Analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 624, 625, 1624, and 1625 (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 1624 and 1625) 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 and cannot be reported to demonstrate regulatory compliance.

NOTE: These warning limits are promulgated as an “interim final action with a request for comments”.

<sup>8</sup>Methods published by this organization and approved for use under this part may not be modified beyond the modifications expressly allowed and defined in each method.

NOTE: The following acronyms are used in this table:

ECD Electron Capture Detector

ELCD Electrolytic Conductivity Detector/Electrochemical Detector

FID Flame Ionization Detector

GC Gas Chromatography

GC/MS Gas Chromatography/Mass Spectrometry

HPLC High Performance Liquid Chromatography

NPD Nitrogen Phosphorous Detector

PID Photoionization Detector

UV Ultraviolet Detector

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES <sup>1</sup>

Parameter/methodology	Method	Reference method <sup>2,7</sup>	Other approved methods		
			Standard methods 18th ed. <sup>8</sup>	ASTM <sup>8</sup>	Other
1. Aldrin .....	GC/ECD	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 30.
	GC/ELCD	.....		3086–90	
	GC/MS	625	6410 B		
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.
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/ECD	608	6630 B & C	3086–90	Note 3, p. 7.
	GC/ELCD	.....		D3086–90	
	GC/MS	<sup>5</sup> 625	6410 B		
9. β-BHC .....	GC/ECD	608	6630	3086–90	D3086–90
	GC/ELCD	.....		D3086–90	
	GC/MS	<sup>5</sup> 625	6410 B		
10. δ-BHC .....	GC/ECD	608	6630 B & C	D3086–90	D3086–90
	C/ELCD	.....		D3086–90	
	GC/MS	<sup>5</sup> 625	6410 B		
11. γ-BHC (Lindane) .....	GC/ECD	608	6630 B & C	3086–90	Note 3, p. 7; Note 4, p. 30.
	GC/ELCD	.....		D3086–90	
	GC/MS	625	6410 B		
12. Captan .....	GC/ECD	.....	6630 B	D3086–90	Note 3, p. 7.
	GC/ELCD	.....		D3086–90	
13. Carbaryl .....	TLC	.....			Note 3, p. 94; Note 6, p. S60.
14. Carbophenothion .....	GC	.....			Note 4, p. 30; Note 6, p. S73.
15. Chlordane .....	GC/ECD	608	6630 B & C	3086–90	Note 3, p. 7.
	GC/ELCD	.....		D3086–90	
	GC/MS	625	6410 B		
16. Chloropropham .....	TLC	.....			Note 3, p. 104; Note 6, p. S64.
17. 2,4-D .....	GC/ECD	.....	6640 B		Note 3, p. 115; Note 4, p. 35.
18. 4,4'-D-DDD .....	GC/ECD	608	6630 B & C	D3086–90	Note 3, p. 7; Note 4, p. 30.
	GC/ELCD	.....		D3086–90	
	GC/MS	625	6410 B		
19. 4,4'-DDE .....	GC/ECD	608	6630 B & C	3086–90	Note 3, p. 7; Note 4, p. 30.
	GC/ELCD	.....		D3086–90	
	GC/MS	625	6410 B		

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES <sup>1</sup>—Continued

Parameter/methodology	Method	Reference method <sup>2 7</sup>	Other approved methods		
			Standard methods 18th ed. <sup>8</sup>	ASTM <sup>8</sup>	Other
20. 4,4'-DDT .....	GC/ECD GC/ELCD GC/MS	608 ..... 625	6630 B & C 6410 B	D3086-90 D3086-90	Note 3, p. 7; Note 4, p. 30.
21. Demeton-O .....	GC	.....			Note 3, p. 25; Note 6, p. S51.
22. Dementon-S .....	GC	.....			Note 3, p. 25; Note 6, p. S51.
23. Diazinon .....	GC	.....			Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
24. Dicamba .....	GC	.....			Note 3, p. 115.
25. Dichlofenthion .....	GC	.....			Note 4, p. 30; Note 6, p. S73.
26. Dichloran .....	GC/ECD	.....	6630 B & C		Note 3, p. 7.
27. Dicofol .....	GC/ECD GC/ELCD	..... .....		D3086-90 D3086-90	
28. Dieldrin .....	GC/ECD GC/MS	608 625	6630 B & C 6410 B		Note 3, p. 7; Note 4, p. 30.
29. Dioxathion .....	GC	.....			Note 4, p. 30; 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/ECD GC/ELCD GC/MS	608 ..... <sup>5</sup> 625	6630 B & C 6410 B	D3086-90 D3086-90	Note 3, p. 7.
33. Endosulfan II .....	GC/ECD GC/ELCD GC/MS	608 ..... <sup>5</sup> 625	6630 B & C 6410 B	D3086-90 D3086-90	Note 3, p. 7.
34. Endosulfan Sulfate .....	GC GC/MS	608 625	6630 C 6410 B		
35. Endrin .....	GC/ECD GC/ELCD GC/MS	608 ..... <sup>5</sup> 625	6630 B & C 6410 B	D3086-90 D3086-90	Note 3, p. 7; Note 4, p. 30.
36. Endrin aldehyde .....	GC/ECD	608			
37. Ethion .....	GC	.....			Note 4, p. 30; 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/ECD GC/ELCD GC/MS	608 ..... 625	6630 B & C 6410 B	D3086-90 D3086-90	Note 3, p. 7; Note 4, p. 30.
41. Heptachlor epoxide .....	GC/ECD GC/ELCD GC/MS	608 ..... 625	6630 B 6410 B	D3086-90 D3086-90	Note 3, p. 7; Note 4, p. 30.
42. Isodrin .....	GC	.....			Note 6, p. S73.
43. Linuron .....	GC	.....			Note 4, p. 30; Note 6, p. S73.
44. Malathion .....	GC/ECD	.....	6630 C		Note 3, p. 104; Note 6, p. S64.
45. Methiocarb .....	TLC	.....			Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
46. Methoxychlor .....	GC/ECD GC/ELCD	..... .....	6630 B & C	D3086-90 D3086-90	Note 3, p. 94; Note 6, p. S60.
47. Mexacarbate .....	TLC	.....			Note 3, p. 7; Note 4, p. 30.
48. Mirex .....	GC/ECD	.....	6630 B & C		Note 3, p. 94; Note 6, p. S60.
49. Monuron .....	TLC	.....			Note 3, p. 7.
50. Monuron .....	TLC	.....			Note 3, p. 104; Note 6, p. S64.
51. Nuburon .....	TLC	.....			Note 3, p. 104; Note 6, p. S64.
52. Parathion methyl .....	GC/ECD	.....	6630 C		Note 3, p. 104; Note 6, p. S64.
53. Parathion ethyl .....	GC/ECD	.....	6630 C		Note 3, p. 25; Note 4, p. 30.
54. PCNB .....	GC/ECD	.....	6630 B & C		Note 3, p. 25.
55. Perthane .....	GC/ECD GC/ELCD	..... .....		D3086-90 D3086-90	Note 3, p. 7.
56. Prometron .....	GC	.....			Note 3, p. 83; Note 6, p. S68.
57. Prometryn .....	GC	.....			Note 3, p. 83; Note 6, p. S68.
58. Propazine .....	GC	.....			Note 3, p. 83; Note 6, p. S68.
59. Propham .....	TLC	.....			Note 3, p. 104; Note 6, p. S64.
60. Propoxur .....	TLC	.....			Note 3, p. 94; Note 6, p. S60.
61. Sebumeton .....	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.
64. Strobane .....	GC/ECD	.....	6630 B & C		Note 3, p. 7.
65. Sweb .....	TLC	.....			Note 3, p. 104; Note 6, p. S64.
66. 2,4,5-T .....	GC/ECD	.....	6640 B		Note 3, p. 115; Note 4, p. 35.
67. 2,4,5-TP (Silvex) .....	GC/ECD	.....	6640 B		Note 3, p. 115.
68. Terbutylazine .....	GC	.....			Note 3, p. 83; Note 6, p. S68.
69. Toxaphene .....	GC/ECD GC/ELCD	608 .....	6630 B & C	3086-90	Note 3, p. 7; Note 4, p. 30.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES<sup>1</sup>—Continued

Parameter/methodology	Method	Reference method <sup>2,7</sup>	Other approved methods		
			Standard methods 18th ed. <sup>8</sup>	ASTM <sup>8</sup>	Other
70. Trifluralin .....	GC/MS GC	625 .....	6410 B 6630 B	D3086–90	Note 3, p. 7.

## Table ID notes:

<sup>1</sup> 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.

<sup>2</sup> 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.

<sup>3</sup> 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.

<sup>4</sup> "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).

<sup>5</sup> The method may be extended to include  $\alpha$ -BHC,  $\gamma$ -BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

<sup>6</sup> "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).

<sup>7</sup> 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 and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other Methods cited.

**Note:** These warning limits are promulgated as an "Interim final action with a request for comments."

<sup>8</sup> Methods published by this organization and approved for use under this part may not be modified beyond the modifications expressly allowed and defined in each method.

**Note:** The following acronyms are used in this table:

ECD: Electron Capture Detector.

ELCD: Electrolytic Conductivity Detector/Electrochemical Detector.

FID: Flame Ionization Detector.

GC: Gas Chromatography.

GC/MS: Gas Chromatography/Mass Spectrometry.

\* \* \* \* \*

BILLING CODE 6560–50–P

Table IF- Standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB

No. Analyte	Data			Specs													
	Reference Method	Prec-Recovery	Labs	Source	CAL points	CAL lin	Spike conc	IPR		Prec-ision		OPR		MSMSD		ML Value	ML Calc
								Low	High	Low	High	Low	High	Low	High		
1. Acidity (CaCO3)	305.1	---	1.00	Multi	MCAWW	2	---	---	---	3.6	---	---	---	---	---	10 mg/L	Range
2. Alkalinity*	310.1	---	2.00	Multi	*	2	---	---	---	7.2	---	---	---	---	---	10 mg/L	310.2
* *	310.2	99.50	0.50	Single	*	2	---	100 mg/L	97.0	102.0	1.8	97.0	102.0	97.0	102.0	10 mg/L	Range
3.. Aluminum - Flame	202.1	99.13	31.60	Multi	Apex D	5	25 %	100 ug/L	35.0	163.0	64.0	29.0	169.0	29.0	169.0	300 ug/L	3.18 x DL
* - Furnace	202.2	103.69	42.74	Multi	Apex D	5	25 %	100 ug/L	18.0	190.0	86.0	9.0	198.0	9.0	198.0	20 ug/L	Range
* - ICP	200.7	96.33	24.19	Multi	Apex C	5	25 %	100 ug/L	47.0	145.0	49.0	43.0	150.0	43.0	150.0	50 ug/L	3.18 x MDL
* - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
* - Color	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
4. Ammonia - distill	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
* - Nessler	350.2	100.46	14.27	Multi	MCAWW	3	10 %	2.0 mg/L	71.0	129.0	29.0	69.0	132.0	69.0	132.0	50 ug/L	Range
* - Titir	350.2	100.46	14.27	Multi	MCAWW	3	10 %	2.0 mg/L	71.0	129.0	29.0	69.0	132.0	69.0	132.0	1.0 mg/L	Range
* - ISE	350.3	91.00	2.31	Single	MCAWW	3	10 %	130 ug/L	82.0	100.0	8.4	81.0	101.0	81.0	101.0	30 ug/L	Range
* - Phenate	350.1	103.00	1.16	Single	MCAWW	1	---	0.5 mg/L	98.0	108.0	4.2	98.0	108.0	98.0	108.0	10 ug/L	Range
* - Auto elec	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
5. Antimony - Flame	204.1	96.50	1.13	Single	MCAWW	1	---	10 mg/L	92.0	101.0	4.1	91.0	102.0	91.0	102.0	1.0 mg/L	Range
Antimony - Furnace	204.2	71.20	38.17	Multi	Apex D	5	25 %	100 ug/L	d	148.0	77.0	d	156.0	d	156.0	20 ug/L	Range
Antimony - ICP	200.7	76.00	15.44	Multi	Apex C	3	10 %	500 ug/L	45.0	107.0	31.0	42.0	110.0	42.0	110.0	8 ug/L	3.18 x MDL
6. Arsenic	206.5	Digestion - no specs	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
* - Hydride	206.3	98.38	8.19	Single	3114 B	3	10 %	200 ug/L	68.0	128.0	30.0	65.0	132.0	65.0	132.0	2.0 ug/L	Range
* - Furnace	206.2	98.63	15.98	Multi	Apex D	3	10 %	100 ug/L	66.0	131.0	32.0	63.0	134.0	63.0	134.0	5.0 ug/L	Range
* - ICP	200.7	92.17	14.79	Multi	Apex C	3	10 %	100 ug/L	62.0	122.0	30.0	59.0	125.0	59.0	125.0	20 ug/L	3.18 x MDL
* - Color (SDDC)	206.4	100.00	13.80	Multi	MCAWW	3	10 %	40 ug/L	72.0	128.0	28.0	69.0	131.0	69.0	131.0	10 ug/L	Method
Barium - Flame	208.1	103.50	8.63	Single	MCAWW	3	10 %	1 mg/L	72.0	135.0	32.0	69.0	138.0	69.0	138.0	1.0 mg/L	Range
* - Furnace	208.2	142.14	31.10	Multi	Apex D	5	25 %	100 ug/L	79.0	205.0	63.0	73.0	211.0	73.0	211.0	10 ug/L	Range
* - ICP	200.7	77.30	20.97	Multi	Apex C	3	10 %	100 ug/L	35.0	120.0	42.0	31.0	124.0	31.0	124.0	2 ug/L	3.18 x MDL
* - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
8. Beryllium - Flame	210.1	98.33	4.27	Single	MCAWW	3	10 %	50 ug/L	82.0	114.0	16.0	81.0	116.0	81.0	116.0	50 ug/L	Range
* - Furnace	210.2	106.66	21.76	Multi	Apex D	5	25 %	100 ug/L	63.0	151.0	44.0	58.0	155.0	58.0	155.0	1.0 mg/L	Range
* - ICP	200.7	96.34	2.31	Multi	Apex C	3	10 %	100 ug/L	91.0	101.0	4.7	91.0	102.0	91.0	102.0	1.0 ug/L	3.18 x MDL
* - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
* - Color	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
9. BOD	405.1	---	24.10	Multi	MCAWW	---	---	100 mg/L	---	---	49.0	---	---	---	---	N/A	Range
10. Boron - Color	212.3	100.00	22.80	Multi	MCAWW	5	25 %	240 ug/L	54.0	146.0	46.0	49.0	151.0	49.0	151.0	100 ug/L	Range

Table IF- Standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB

No. Analyte	Data				Specs											
	Reference Method	Recovery	Precision	Labs	Source	CAL points	CAL lin conc	Spike conc	IPR				OPR			
									Low	High	Recovery	Precision	Low	High	Recovery	MS/MSD
	Method	Recovery	Precision													
11. Bromide	200.7	97.07	25.60	Multi	Apx C	5	25 %	100 ug/L	45.0	149.0	52.0	40.0	154.0	52.0	3 ug/L	3.18 x MDL
12. Cadmium - Flame	320.1	93.75	7.17	Single	MCAWW	3	10 %	5 mg/L	67.0	120.0	26.0	65.0	123.0	26.0	2 mg/L	Range
Cadmium - Furnace	213.1	94.87	15.88	Multi	Apx D	3	10 %	100 ug/L	63.0	127.0	32.0	59.0	130.0	32.0	50 ug/L	Range
Cadmium - ICP	213.2	98.43	23.05	Multi	Apx D	5	25 %	100 ug/L	52.0	145.0	47.0	47.0	150.0	47.0	0.5 ug/L	Range
Cadmium - DCP	200.7	98.56	7.59	Multi	Apx C	3	10 %	100 ug/L	83.0	114.0	16.0	81.0	116.0	16.0	1 ug/L	3.18 x MDL
Cadmium - Volt	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Cadmium - Color	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
13. Calcium - Flame	215.1	99.00	3.33	Single	MCAWW	3	10 %	10 ug/L	87.0	111.0	12.0	85.0	113.0	12.0	200 ug/L	Range
Calcium - ICP	200.7	89.22	22.38	Multi	Apx C	5	25 %	100 ug/L	44.0	134.0	45.0	39.0	139.0	45.0	10 ug/L	3.18 x MDL
Calcium - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Calcium - Titr	215.2	98.10	9.20	Single	MCAWW	3	10 %	100 ug/L	64.0	132.0	34.0	61.0	135.0	34.0	2 mg/L	3.18 x LDL
14. CBOD5	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
15. COD - Titr	410.1	95.30	17.76	Multi	MCAWW	3	10 %	250 mg/L	59.0	131.0	36.0	56.0	135.0	36.0	50 mg/L	Method
COD - Titr	410.2	100.30	4.15	Multi	MCAWW	3	10 %	10 mg/L	92.0	109.0	8.3	91.0	110.0	8.3	5 mg/L	Range
COD - Titr	410.3	100.00	10.00	No data	Default	3	10 %	10 mg/L	64.0	136.0	36.0	60.0	140.0	36.0	---	---
COD - Spectro	410.4	100.00	10.00	No data	Default	3	10 %	10 mg/L	64.0	136.0	36.0	60.0	140.0	36.0	---	---
16. Chloride - Titr/Ag	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Chloride - Titr/Hg	325.3	97.10	3.30	Multi	MCAWW	3	10 %	250 mg/L	90.0	104.0	6.6	89.0	105.0	6.6	---	---
Chloride - Color	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Chloride - Auto	325.1	100.50	3.00	Single	MCAWW	3	10 %	10 mg/L	89.0	112.0	11.0	88.0	113.0	11.0	1 mg/L	Range
Chloride - Auto	325.2	100.00	10.00	No data	Default	3	10 %	10 mg/L	64.0	136.0	36.0	60.0	140.0	36.0	1 mg/L	Range
Chlorine - Ampere	330.1	91.20	12.50	Multi	MCAWW	3	10 %	250 mg/L	66.0	117.0	25.0	63.0	119.0	25.0	---	---
Chlorine - Iodo	330.3	81.50	32.40	Multi	MCAWW	5	25 %	1.0 mg/L	16.0	147.0	65.0	10.0	153.0	65.0	0.1 mg/L	Method
Chlorine - Back titr	330.2	98.80	4.30	Single	MCAWW	3	10 %	1.0 mg/L	83.0	115.0	16.0	81.0	116.0	16.0	---	---
Chlorine - DPD-FAS	330.4	91.90	19.20	Multi	MCAWW	3	10 %	1.0 mg/L	53.0	131.0	39.0	49.0	135.0	39.0	0.1 mg/L	Method
Chlorine - Spectro	330.5	84.40	27.60	Multi	MCAWW	5	25 %	1.0 mg/L	29.0	140.0	56.0	23.0	146.0	56.0	0.2 mg/L	Method
Chlorine - Electrode	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
18. Chromium VI - AA	218.4	98.49	6.96	Multi	MCAWW	3	10 %	100 ug/L	84.0	113.0	14.0	83.0	114.0	14.0	10 ug/L	Range
Chromium VI - Color	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
19. Chromium - Flame	218.1	101.54	17.36	Multi	Apx D	3	10 %	100 ug/L	66.0	137.0	35.0	63.0	140.0	35.0	250 ug/L	Method
Chromium - Chelate	218.3	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	36.0	1 ug/L	Method



Table IF- Standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB

No. Analyte	Data				Specs											
	Reference		Prec- Recovery	Labs	Source	CAL points	CAL lin	Spike conc	IPR		Prec- Recovery		OPR		MSMSD Recovery	
	Method	Recovery							Low	High	Low	High	Low	High	Low	High
Chromium - Furnace	218.2	91.43	17.69	Multi	Apex D	3	10 %	100 ug/L	56.0	127.0	36.0	52.0	131.0	52.0	131.0	36.0
Chromium - ICP	200.7	98.54	9.39	Multi	Apex C	3	10 %	100 ug/L	79.0	118.0	19.0	77.0	120.0	77.0	120.0	19.0
Chromium - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Chromium - Color	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
20. Cobalt - Flame	219.1	98.00	1.00	Single	MCAWW	3	10 %	1.0 mg/L	94.0	102.0	3.6	94.0	102.0	94.0	102.0	3.6
Cobalt - Furnace	219.2	89.38	22.27	Multi	Apex D	5	25 %	100 ug/L	44.0	134.0	45.0	40.0	139.0	40.0	139.0	45.0
Cobalt - ICP	200.7	87.59	8.16	Multi	Apex C	3	10 %	100 ug/L	71.0	104.0	17.0	69.0	106.0	69.0	106.0	17.0
Cobalt - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
21. Color - ADML	110.1	100.00	10.00	No data	Default	1	---	100 C.U.	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0
Color - P/Co	110.2	100.00	10.00	No data	Default	3	---	100 C.U.	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0
Color - Spectro	110.3	100.00	10.00	No data	Default	3	---	100 C.U.	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0
22. Copper - Flame	220.1	99.79	17.00	Multi	MCAWW	3	10 %	100 ug/L	65.0	134.0	34.0	62.0	138.0	62.0	138.0	34.0
Copper - Furnace	220.2	92.54	27.29	Multi	Apex D	5	25 %	100 ug/L	37.0	148.0	55.0	32.0	153.0	32.0	153.0	55.0
Copper - ICP	200.7	95.82	7.07	Multi	Apex C	3	10 %	100 ug/L	81.0	110.0	15.0	80.0	112.0	80.0	112.0	15.0
Copper - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Copper - Color/Neo	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Copper - Color/Bicin	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
23. Cyanide - Distill	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Cyanide - Titr	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Cyanide - Spectro	335.2	85.00	11.07	Single	MCAWW	3	10 %	250 ug/L	45.0	125.0	40.0	40.0	130.0	40.0	130.0	40.0
Cyanide - Auto	335.3	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0
CATC - Titr	335.1	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0
CATC - Spectro	335.1	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0
Fluoride - Distill	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Fluoride - Elec/man	340.2	98.82	3.53	Multi	MCAWW	3	10 %	1.0 mg/L	91.0	106.0	7.1	91.0	107.0	91.0	107.0	7.1
Fluoride - Elec/auto	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Fluoride - SPADNS	340.1	97.59	10.72	Multi	MCAWW	3	10 %	1.0 mg/L	76.0	120.0	22.0	74.0	122.0	74.0	122.0	22.0
Fluoride - Auto	340.3	89.00	12.00	Single	MCAWW	3	10 %	150 ug/L	45.0	133.0	44.0	41.0	137.0	41.0	137.0	44.0
Gold - Flame	231.1	100.00	10.00	No data	Default	3	10 %	1.0 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0
Gold - Furnace	231.2	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0
Gold - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Hardness - Color/auto	130.1	89.00	7.89	Single	MCAWW	3	10 %	50 mg/L	60.0	118.0	29.0	57.0	121.0	57.0	121.0	29.0
Hardness - Titr/EDTA	130.2	99.13	9.26	Multi	MCAWW	3	10 %	30 mg/L	80.0	118.0	19.0	78.0	120.0	78.0	120.0	19.0

Table IF- Standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB

No. Analyte	Data				Specs													
	Reference		Prec- Recovery	Labs	Source	CAL points	Spike CAL lin conc	IPR		OPR		MS/MSD		MDL	ML Value	ML Calc		
	Method	Recovery						islon	Low	High	Recovery	Prec- ision	Low				High	Recovery
28. pH - Electrode	150.1	N/A	1.30	Multi	MCAWW	2	--	N/A				2.6						
pH - Auto	--																	
29. Iridium - Flame	235.1	100.00	10.00	No data	Default	3	10 %	100 mg/L	64.0	136.0	36.0	36.0	60.0	140.0	60.0	140.0	36.0	Range
Iridium - Furnace	235.2	100.00	10.00	No data	Default	3	10 %	200 ug/L	64.0	136.0	36.0	36.0	60.0	140.0	60.0	140.0	36.0	Range
30. Iron - Flame	236.1	97.69	17.00	Multi	Apex D	3	10 %	100 ug/L	63.0	132.0	34.0	34.0	60.0	136.0	60.0	136.0	34.0	Range
Iron - Furnace	236.2	144.71	36.03	Multi	Apex D	5	25 %	100 ug/L	72.0	217.0	73.0	73.0	65.0	224.0	65.0	224.0	73.0	Range
Iron - ICP	200.7	95.29	18.33	Multi	Apex C	3	10 %	100 ug/L	58.0	132.0	37.0	37.0	54.0	136.0	54.0	136.0	37.0	3.18 x MDL
Iron - DCP	--																	
Iron - Color	--																	
31. TKN - Digest	351.3	101.03	25.76	Multi	MCAWW	5	25 %	2 mg/L	49.0	153.0	52.0	52.0	44.0	158.0	44.0	158.0	52.0	Range
TKN - Titr	351.3	101.03	25.76	Multi	MCAWW	5	25 %	2 mg/L	49.0	153.0	52.0	52.0	44.0	158.0	44.0	158.0	52.0	Range
TKN - Nessler	351.3	101.03	25.76	Multi	MCAWW	5	25 %	2 mg/L	49.0	153.0	52.0	52.0	44.0	158.0	44.0	158.0	52.0	Range
TKN - Electrode	351.3	101.03	25.76	Multi	MCAWW	5	25 %	2 mg/L	49.0	153.0	52.0	52.0	44.0	158.0	44.0	158.0	52.0	Range
TKN - Phenate	351.1	71.70	27.98	Multi	MCAWW	5	25 %	2 mg/L	15.0	128.0	56.0	56.0	10.0	134.0	10.0	134.0	56.0	Range
TKN - Block/color	351.2	99.00	8.82	Single	MCAWW	3	10 %	2 mg/L	67.0	131.0	32.0	32.0	63.0	135.0	63.0	135.0	32.0	Range
TKN - Potentilo	351.4	100.00	10.00	No data	Default	3	10 %	10 ug/L	64.0	136.0	36.0	36.0	60.0	140.0	60.0	140.0	36.0	Range
32. Lead - Flame	239.1	109.90	36.70	Multi	Apex D	5	25 %	100 ug/L	36.0	184.0	74.0	74.0	29.0	191.0	29.0	191.0	74.0	Data
Lead - Furnace	239.2	93.80	22.75	Multi	Apex D	5	25 %	100 ug/L	48.0	140.0	46.0	46.0	43.0	144.0	43.0	144.0	46.0	Range
Lead - ICP	200.7	94.79	12.58	Multi	Apex C	3	10 %	100 ug/L	69.0	120.0	26.0	26.0	67.0	123.0	67.0	123.0	26.0	3.18 x MDL
Lead - DCP	--																	
Lead - Volt	--																	
Lead - Color	--																	
33. Magnesium - Flame	242.1	97.90	29.81	Multi	MCAWW	5	25 %	100 ug/L	38.0	158.0	60.0	60.0	32.0	164.0	32.0	164.0	60.0	Range
Magnesium - ICP	200.7	97.71	17.67	Multi	Apex C	3	10 %	100 ug/L	62.0	134.0	36.0	36.0	58.0	137.0	58.0	137.0	36.0	3.18 x MDL
Magnesium - DCP	--																	
Magnesium - Grav	--																	
34. Manganese - Flame	243.1	95.43	13.15	Multi	Apex D	3	25 %	100 ug/L	69.0	122.0	27.0	27.0	66.0	125.0	66.0	125.0	27.0	Range
Manganese - Furnace	243.2	106.20	21.05	Multi	Apex D	5	25 %	100 ug/L	64.0	149.0	43.0	43.0	59.0	153.0	59.0	153.0	43.0	Range
Manganese - ICP	200.7	94.30	4.12	Multi	Apex C	3	10 %	100 ug/L	86.0	103.0	8.3	8.3	85.0	104.0	85.0	104.0	8.3	3.18 x MDL
Manganese - DCP	--																	
Manganese - Persulf	--																	
Manganese - Perioda	--																	
35. Mercury - CV/Man	245.1	92.90	29.40	Multi	MCAWW	5	25 %	4 ug/L	34.0	152.0	59.0	59.0	28.0	158.0	28.0	158.0	59.0	DL

Table IF- Standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB

Data			Specs															
No. Analyte	Reference		Prec- ision	Labs	Source	CAL points	CAL lin	Spike conc	IPR		Pre- ision		Recovery		MS/MSD		ML Value	ML Calc
	Method	Recovery							Low	High	Low	High	Low	High	Low	High		
36. Mercury - CV/Auto Molybdenum - Flame Molybdenum - Furnace Molybdenum - ICP Molybdenum - DCP	245.2	102.00	2.00	Single	MCAWW	3	10 %	10 ug/L	94.0	110.0	7.2	94.0	110.0	94.0	110.0	7.2	0.2 ug/L	DL
	246.1	97.00	2.33	Single	MCAWW	3	10 %	300 ug/L	88.0	106.0	8.4	87.0	107.0	87.0	107.0	8.4	300 ug/L	Data
	246.2	100.00	10.00	No data	Default	3	10 %	10 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	3 ug/L	Range
	200.7	96.92	7.78	Multi	Apx C	3	10 %	100 ug/L	81.0	113.0	16.0	79.0	115.0	79.0	115.0	16.0	4 ug/L	3.18 x MDL
37. Nickel - Flame Nickel - Furnace Nickel - ICP Nickel - DCP Nickel - Color	249.1	96.67	2.00	Single	MCAWW	3	10 %	1 ug/L	89.0	104.0	7.2	88.0	105.0	88.0	105.0	7.2	0.2 ug/L	Data
	249.2	90.37	26.65	Multi	Apx D	5	25 %	100 ug/L	37.0	144.0	54.0	31.0	149.0	31.0	149.0	54.0	5 ug/L	Range
	200.7	95.48	10.44	Multi	Apx C	3	10 %	100 ug/L	74.0	117.0	21.0	72.0	119.0	72.0	119.0	21.0	20 ug/L	3.18 x MDL
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38. Nitrate 39. NO2-NO3 - Cd/Man NO2-NO3 - Cd/Auto NO2-NO3 - Cd/Hydra 40. Nitrite - Spec/Man Nitrite - Spec/Auto	352.1	104.12	22.69	Multi	MCAWW	5	25 %	1 mg/L	58.0	150.0	46.0	54.0	155.0	54.0	155.0	46.0	0.1 mg/L	Range
	353.3	100.00	12.50	Single	MCAWW	3	10 %	40 ug/L	55.0	145.0	45.0	50.0	150.0	50.0	150.0	45.0	10 ug/L	Range
	353.2	105.75	4.14	Single	MCAWW	3	10 %	290 ug/L	90.0	121.0	15.0	89.0	123.0	89.0	123.0	15.0	50 ug/L	Range
	353.1	99.00	5.13	Single	MCAWW	3	10 %	400 ug/L	80.0	118.0	19.0	78.0	120.0	78.0	120.0	19.0	10 ug/L	Range
41. Oil & Grease 42. TOC 43. Organic nitrogen 44. O-phosphate - Auto O-phosphate - Man 1 O-phosphate - Man 2	354.1	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	10 ug/L	Range
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	413.1	93.00	6.43	Single	MCAWW	1	10 %	15 mg/L	69.0	117.0	24.0	67.0	119.0	67.0	119.0	24.0	5 mg/L	Range
	415.1	101.01	7.78	Multi	MCAWW	3	10 %	100 mg/L	85.0	117.0	16.0	83.0	119.0	83.0	119.0	16.0	1 mg/L	Method
45. Osmium - Flame Osmium - Furnace 46. DO - Winkler DO - Electrode	365.1	87.20	22.00	Multi	MCAWW	5	25 %	300 ug/L	43.0	132.0	45.0	38.0	136.0	38.0	136.0	45.0	10 ug/L	Range
	365.2	97.25	5.37	Multi	MCAWW	3	10 %	300 ug/L	86.0	108.0	11.0	85.0	110.0	85.0	110.0	11.0	10 ug/L	Range
	365.3	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	10 ug/L	Range
	252.1	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	1 mg/L	Method
47. Palladium - Flame Palladium - Furnace Palladium - DCP	252.2	100.00	10.00	No data	Default	3	10 %	10 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	50 ug/L	Range
	360.2	100.00	1.00	Single	MCAWW	3	10 %	1 mg/L	96.0	104.0	3.6	96.0	104.0	96.0	104.0	3.6	50 ug/L	Range
	360.1	100.00	1.00	Single	MCAWW	3	10 %	1 mg/L	96.0	104.0	3.6	96.0	104.0	96.0	104.0	3.6	50 ug/L	Range
	253.1	100.00	10.00	No data	Default	3	10 %	1 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	500 ug/L	Range
48. Phenol - Color/Man Phenol - Color/Auto 49. Phosphorus - GC 50. Phosphorus - Aso/Man	253.2	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	20 ug/L	Range
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	420.1	100.00	10.31	Multi	MCAWW	3	10 %	300 ug/L	79.0	121.0	21.0	77.0	123.0	77.0	123.0	21.0	5 ug/L	Method
	420.2	98.00	1.12	Single	MCAWW	3	10 %	1 mg/L	93.0	103.0	4.1	93.0	103.0	93.0	103.0	4.1	2 ug/L	Range
50. Phosphorus - Aso/Man	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	365.2	103.09	30.00	Multi	MCAWW	5	25 %	300 ug/L	43.0	164.0	60.0	37.0	170.0	37.0	170.0	60.0	10 ug/L	Range

## Data

Data				Specs															
No.	Analyte	Reference		Prec- Recovery	Labs	Source	CAL points	CAL lin conc	Spike	IPR		Pre- Recovery		MS/MSD		RPD	MDL	ML Value	ML Calc
		Method	Recovery							Low	High	Low	High	Low	High				
51.	Phosphorus - Asc/Man	365.3	99.00	22.00	Multi	MCAWW	5	25 %	300 ug/L	55.0	143.0	44.0	50.0	148.0	50.0	148.0	44.0	10 ug/L	Range
	Phosphorus - Asc/Auto	365.1	87.20	22.00	Multi	MCAWW	5	25 %	300 ug/L	43.0	132.0	45.0	38.0	136.0	38.0	136.0	45.0	10 ug/L	Range
	Phosphorus - Block	365.4	98.00	3.00	Single	MCAWW	3	10 %	2 mg/L	87.0	109.0	11.0	86.0	110.0	86.0	110.0	11.0	10 ug/L	Range
	Platinum - Flame	255.1	100.00	10.00	No data	Default	3	10 %	10 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	5 mg/L	Range
	Platinum - Furnace	255.2	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	100 ug/L	Range
52.	Platinum - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Potassium - Flame	258.1	103.00	12.50	Single	MCAWW	3	10 %	2 mg/L	58.0	148.0	45.0	53.0	153.0	53.0	153.0	45.0	100 ug/L	Range
	Potassium - ICP	200.7	83.05	17.12	Multi	Apx C	3	10 %	1 mg/L	48.0	118.0	35.0	45.0	121.0	45.0	121.0	35.0	300 ug/L	3.18 x MDL
	Potassium - FPD	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Potassium - Color	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
53.	Total Solids	160.3	100.00	10.00	No data	Default	1	---	100 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	10 mg/L	Range
	TDS	160.1	100.00	10.00	No data	Default	1	---	100 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	10 mg/L	Range
	TSS	160.2	100.00	10.00	No data	Default	1	---	100 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	4 mg/L	Range
	Settleable Solids	160.5	100.00	10.00	No data	Default	1	---	100 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	0.2 mL/L/h	Method
	Volatile Residue	160.4	100.00	6.47	Multi	MCAWW	3	10 %	300 ug/L	87.0	113.0	13.0	85.0	115.0	85.0	115.0	13.0	10 mg/L	Range
54.	Rhodium - Flame	285.1	100.00	10.00	No data	Default	3	10 %	1 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	1 mg/L	Range
	Rhodium - Furnace	285.2	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	20 ug/L	Range
	Ruthenium - Flame	267.1	100.00	10.00	No data	Default	3	10 %	1 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	1 mg/L	Range
	Ruthenium - Furnace	267.2	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0	100 ug/L	Range
	Selenium - Furnace	270.2	96.12	16.72	Multi	Apx D	3	10 %	100 ug/L	62.0	130.0	34.0	59.0	133.0	59.0	133.0	34.0	5 ug/L	Range
55.	Selenium - ICP	200.7	91.13	26.35	Multi	Apx C	5	25 %	1 mg/L	38.0	144.0	53.0	33.0	150.0	33.0	150.0	53.0	20 ug/L	3.18 x MDL
	Selenium - Hydride	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Selenium - Color/Man	370.1	85.70	7.80	Multi	MCAWW	3	10 %	5 mg/L	70.0	102.0	16.0	68.0	103.0	68.0	103.0	16.0	2 mg/L	Range
	Silica - Color/Auto	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Silica - ICP	200.7	53.86	45.38	Multi	Apx C	5	25 %	1 mg/L	d	145.0	91.0	d	154.0	d	154.0	91.0	20 ug/L	3.18 x MDL
56.	Silver - Flame	272.1	89.40	17.60	Multi	MCAWW	3	10 %	50 ug/L	54.0	125.0	36.0	50.0	129.0	50.0	129.0	36.0	100 ug/L	Range
	Silver - Furnace	272.2	94.88	18.20	Multi	Apx D	3	10 %	100 ug/L	58.0	132.0	37.0	54.0	135.0	54.0	135.0	37.0	1 ug/L	Range
	Silver - ICP	200.7	49.73	47.50	Multi	Apx C	5	25 %	100 ug/L	d	145.0	95.0	d	155.0	d	155.0	95.0	2 ug/L	3.18 x MDL
	Silver - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Sodium - Flame	273.1	100.00	1.54	Multi	MCAWW	3	10 %	5 mg/L	96.0	104.0	3.1	96.0	104.0	96.0	104.0	3.1	30 ug/L	Range
57.	Sodium - ICP	200.7	99.77	24.27	Multi	Apx C	5	25 %	1 mg/L	51.0	149.0	49.0	46.0	154.0	46.0	154.0	49.0	30 ug/L	3.18 x MDL
	Sodium - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Sodium - FPD	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---

Table IF- Standardized QC and QC Acceptance Criteria for Methods in 40 CFR Part 136, Table IB

Data										Specs												
No.	Analyte	Reference			Labs	Source	CAL points	CAL lin conc	Spike	IPR		Pre- cision		Recovery		MS/MSD		RPD	MDL	Value	ML	Calc
		Method	Recovery	ision						Low	High	Low	High	Low	High							
64.	Specific conductance	120.1	97.98	7.55	Multi	MCAWW	3	10 %	5 mg/L	82.0	114.0	16.0	81.0	115.0	81.0	115.0	16.0		No data			
65.	Sulfate - Color/Auto	375.1	99.00	1.80	Single	MCAWW	3	10 %	100 mg/L	92.0	106.0	6.5	91.0	107.0	91.0	107.0	6.5		10 mg/L	Range		
	Sulfate - Grav	375.3	102.00	1.45	Single	MCAWW	3	10 %	100 mg/L	96.0	108.0	5.3	96.0	108.0	96.0	108.0	5.3		10 ug/L	Range		
	Sulfate - Turbid	375.4	96.99	7.15	Multi	MCAWW	3	10 %	100 mg/L	82.0	112.0	15.0	81.0	113.0	81.0	113.0	15.0		1 mg/L	DL		
66.	Sulfide - Turbid	376.1	100.00	10.00	No data	Default	3	10 %	10 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0		1 mg/L	DL		
	Sulfide - Color	376.2	100.00	10.00	No data	MCAWW	3	10 %	10 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0		No data			
67.	Sulfite - Turbid	377.1	100.00	10.00	No data	Default	3	10 %	10 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0		3 mg/L	DL		
68.	Surfactants	425.1	101.36	9.13	Multi	MCAWW	3	10 %	3 mg/L	83.0	120.0	19.0	81.0	122.0	81.0	122.0	19.0		25 ug/L	Range		
69.	Temperature	170.1	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---		N/A			
70.	Thallium - Flame	279.1	100.00	3.00	Single	MCAWW	3	10 %	600 ug/L	89.0	111.0	11.0	88.0	112.0	88.0	112.0	11.0		600 ug/L	Data		
	Thallium - Furnace	279.2	87.10	11.79	Multi	Apx D	5	25 %	100 ug/L	63.0	111.0	24.0	61.0	114.0	61.0	114.0	24.0		5 ug/L	Range		
	Thallium - ICP	200.7	82.90	28.34	Multi	Apx C	5	25 %	1 mg/L	26.0	140.0	57.0	20.0	146.0	20.0	146.0	57.0		20 ug/L	3.18 x MDL		
71.	Tin - Flame	282.1	96.00	6.25	Single	MCAWW	3	10 %	4 mg/L	73.0	119.0	23.0	71.0	121.0	71.0	121.0	23.0		10 mg/L	Range		
	Tin - Furnace	282.2	100.00	10.00	No data	Default	3	10 %	10 mg/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0		20 ug/L	Range		
72.	Tin - ICP	200.7	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0		7 ug/L	3.18 x MDL		
	Titanium - Flame	283.1	97.00	3.50	Single	MCAWW	3	10 %	2 mg/L	84.0	110.0	13.0	83.0	111.0	83.0	111.0	13.0		2 mg/L	Data		
73.	Titanium - Furnace	283.2	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0		50 ug/L	Range		
	Titanium - ICP	200.7	100.00	10.00	No data	Default	3	10 %	100 ug/L	64.0	136.0	36.0	60.0	140.0	60.0	140.0	36.0		1 ug/L	Range		
74.	Turbidity	180.1	100.00	2.31	Single	MCAWW	3	10 %	25 NTU	91.0	109.0	8.4	90.0	110.0	90.0	110.0	8.4		0.05 NTU	Est		
	Vanadium - Flame	286.1	100.00	5.00	Single	MCAWW	3	10 %	2 mg/L	82.0	118.0	18.0	80.0	120.0	80.0	120.0	18.0		2 mg/L	Range		
75.	Vanadium - Furnace	286.2	85.11	32.80	Multi	Apx D	5	25 %	100 ug/L	19.0	151.0	66.0	12.0	158.0	12.0	158.0	66.0		10 ug/L	Range		
	Vanadium - ICP	200.7	94.15	7.88	Multi	Apx C	3	10 %	100 ug/L	78.0	110.0	16.0	76.0	112.0	76.0	112.0	16.0		3 ug/L	3.18 x MDL		
76.	Vanadium - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---					
	Vanadium - Color	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---					
	Zinc - Flame	289.1	99.93	18.60	Multi	Apx D	3	10 %	100 mg/L	62.0	138.0	38.0	59.0	141.0	59.0	141.0	38.0		50 ug/L	Range		
77.	Zinc - Furnace	289.2	168.59	67.06	Multi	Apx D	7	25 %	100 ug/L	34.0	303.0	135.0	21.0	317.0	21.0	317.0	140.0		0.2 ug/L	Range		
	Zinc - ICP	200.7	93.26	12.89	Multi	Apx C	5	25 %	100 ug/L	67.0	120.0	26.0	64.0	122.0	64.0	122.0	26.0		2 ug/L	3.18 x MDL		
78.	Zinc - DCP	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---					
	Zinc - Color/Dilthiz	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---					
	Zinc - Color/Zincon	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---					

(c) Under certain circumstances, the Regional Administrator or the Director in the Region or State where the discharge will occur may determine that an additional parameter or pollutant of concern must be reported. Under such circumstances, an additional test procedure for the analysis of the pollutant may be specified by the Regional Administrator, or the Director, upon the recommendation of the Director of the Analytical Methods Staff.

(d) Sample preservation procedures, container materials, and maximum allowable holding times for parameters and pollutants cited in Tables IA, IB, IC, ID, and IE are prescribed in Table II. Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples collected from a specific discharge. An application for a variance may be made by letter to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to ensure such variance does not adversely affect the integrity of the sample. Such data will be forwarded by the Regional Administrator to the Director of the Analytical Methods Staff for technical review and recommendations for action on the variance application. Upon receipt of a recommendation from the Director of the Analytical Methods Staff, the Regional Administrator may grant a variance applicable to samples collected from the specific discharge for which the application for variance was made. A decision to approve or deny a variance will be made within 90 days of receipt of a complete application by the Regional Administrator.

\* \* \* \* \*

4. Section 136.4 is proposed to be revised to read as follows:

**§ 136.4 Modifications to reference methods.**

A reference method listed in tables IB, IC, or ID of this part 136 may be modified to improve separations, lower the costs of measurements, reduce or eliminate interferences, or for other purposes, provided that the modification is not explicitly prohibited in the reference method and provided that the laboratory modifying the reference method meets the requirements in this section, performs the standardized QC tests, and demonstrates that the QC acceptance criteria and the requirements specified at Appendixes E, F, and G of this part are met. A laboratory that wishes to use a new or modified wastewater method must demonstrate that the method detection limit (MDL) specified in the

reference method can be achieved. Alternatively, if the effluent limitation to be measured is above the MDL, laboratories must demonstrate that the minimum level (ML) determined with the new or modified wastewater method is at or below  $\frac{1}{3}$  the effluent limitation. Demonstration of a valid detection limit requires use of an MDL study in accordance with the procedure at 40 CFR part 136 Appendix B. If the MDL determined with the new or modified method is not acceptable, the method may not be used. Specified detection limits are usually analyte-specific. For any given analyte, the specified detection limit may vary between a wastewater and drinking water reference method.

(a) Tier 1: modification of a reference method for application in a single laboratory to one or more matrix types.

(1) Application to a single matrix type.

(i) A laboratory may modify a reference method listed in tables IB, IC, and ID for determination of an analyte of concern in a specific matrix type, provided that the laboratory:

(A) Performs the standardized QC tests, including a test of initial precision and recovery (IPR) on a reagent water matrix;

(B) Performs the matrix spike (MS) and matrix spike duplicate (MSD) tests on the matrix type to which the modification is to be applied;

(C) Meets the QC acceptance criteria in the reference method or that apply to the reference method in the table of QC acceptance criteria for wastewater methods at § 136.3 Table IF;

(D) Documents the results of the QC tests using the checklists in Appendix E of this part;

(E) Maintains the results of the QC tests and other tests on file for inspection by EPA and/or the approved State NPDES authority.

(ii) After the laboratory has demonstrated application of a method modification to a given matrix type by meeting the MS/MSD QC acceptance criteria, only that laboratory may subsequently apply that method modification to that given matrix type.

(iii) A laboratory may apply a given method modification to additional matrix types if the laboratory validates the modification on each matrix type by performing a matrix spike (MS) and matrix spike duplicate (MSD) test and meeting the MS/MSD QC acceptance criteria for precision and recovery for each matrix type.

(2) Application to multiple matrix types. After a laboratory has validated a given method modification on a minimum of nine (9) matrix types in

accordance with the procedures given in paragraph (a)(1) of this section, the laboratory may subsequently apply that method modification to other matrix types without validating the method modification on those subsequent matrix types, provided that:

(i) The following are included in the matrix types validated:

(A) Effluent from a publicly-owned treatment works (POTW);

(B) ASTM D 5905, Standard

Specification for Substitute Wastewater;

(C) Sewage sludge, if sludge will be in the permit; and

(D) ASTM D 1141, Standard Specification for Substitute Ocean Water, if ocean water will be in the permit.

(ii) At least one of the matrix types in paragraph (a)(2)(i) of this section has at least one of the following

characteristics: total suspended solids (TSS) greater than 40 mg/L, total dissolved solids (TDS) greater than 100 mg/L, oil and grease greater than 20 mg/L, sodium chloride (NaCl) greater than 120 mg/L, and calcium carbonate ( $\text{CaCO}_3$ ) greater than 140 mg/L.

(iii) The matrix spike (MS) and matrix spike duplicate (MSD) recovery and the relative percent difference are within the QC acceptance criteria given for the analyte in the reference method or as supplemented by the QC acceptance criteria specified for wastewater methods at § 136.3 Table IF. If the method modification is to be applied to multiple media, validation must include a minimum of one matrix type from each additional medium in addition to the matrix types listed in this paragraph. If all QC acceptance criteria are not met for a given matrix type, the modification may not be applied to that matrix type.

(b) Tier 2: modification of a reference method for application by all laboratories to one or more matrix types within a single industrial category or subcategory.

(1) A person may modify a reference method for application by all laboratories to determination of an analyte of concern in a single matrix type in a single industrial category or subcategory, provided that the modification is not explicitly prohibited in the reference method and provided that the modification is validated in a minimum of three (3) laboratories, each of which test the same three (3) matrix types and each matrix type is from a different facility in the industrial category or subcategory (a minimum of nine (9) tests). Each laboratory must meet the requirements in paragraph (a)(1)(i) of this section. After the tests in all three laboratories have met all QC acceptance criteria for the reference

method, the modified reference method may be applied by laboratories nationwide to that matrix type in that industrial category or subcategory only.

(2) A person who modifies and validates a method modification under Tier 2 may submit that modification to EPA for a letter of approval using the procedures specified in Appendix F and G of this part. The information that must be submitted includes the results of the performance tests required by paragraph (b)(1) of this section. This information and other detailed information that must be submitted and the format for submission are given in Appendixes E, F, and G of this part.

(3) A person who modifies and validates a method modification under Tier 2 may submit that modification to EPA and for approval and inclusion in a table in this part 136. The information that must be submitted includes the results of the performance tests required by paragraph (b)(1) of this section, and the detailed information specified in Appendixes E, F, and G of this part.

(4) A person may modify a reference method for application by all laboratories to determination of an analyte of concern in additional matrix types within a single industrial category or subcategory, provided that the modification is validated in each additional matrix type according to the requirements in paragraph (b)(1) of this section.

(c) Tier 3: modification of a reference method for application by all laboratories to all matrix types in all industrial categories and subcategories (nationwide modification).

(1) A person may modify a reference method for application by all laboratories to determination of an analyte of concern in all matrix types, provided that the modification is validated in an interlaboratory method validation study or in a study with a minimum of nine (9) different laboratories each of which test a minimum of one sample from a set representing a minimum of nine (9) different matrix types for a total of a minimum of nine unique samples. Each of the nine (9) matrix types must be from a different industrial category or subcategory. Each laboratory must meet the requirements in paragraph (a)(1)(i) of this section and, the nine matrix types must collectively meet all of the criteria in paragraphs (a)(2)(i), (ii), and (iii) of this section. After the modification has been validated, it may be applied by laboratories nationwide to all matrix types.

(2) A person who modifies and validates a method modification under Tier 3 may submit that modification to

EPA for a letter of approval. The information that must be submitted includes the results of the performance tests required by paragraph (b)(1) of this section. This information and other detailed information that must be submitted and the format for submission are given in Appendixes E, F, and G of this part.

(3) A person who modifies and validates a method modification under Tier 3 may submit that modification to EPA and for approval and inclusion in a table in this part 136. The information that must be submitted includes the results of the performance tests required by paragraph (c)(1) of this section. This information and other detailed information that must be submitted and the format for submission are given in Appendixes E, F, and G of this part.

(d) A decision to recommend proposal of a Tier 2 or Tier 3 method modification will be made by the Director of the Analytical Methods Staff within 90 days of receipt of a complete application.

5. Section 136.5 is proposed to be revised to read as follows:

#### **§ 136.5 New methods.**

A person may apply to EPA for use of a new method for determination of an analyte of concern, provided that the new method meets the requirements for validation and format as set forth in this section and in appendixes E, F, and G of this part. A new method must meet the MDL criteria specified at § 136.4. A new method must: be documented in accordance with requirements in appendixes E, F, and G of this part; contain standardized QC as defined at § 136.2; contain QC acceptance criteria that have been developed in accordance with the requirements detailed in appendixes E, F, and G of this part; employ a determinative technique for an analyte of concern with selectivity or sensitivity equal or superior to the selectivity or sensitivity of the determinative technique in any approved method, and that differs from the determinative techniques employed for that analyte in all approved methods; and be accompanied by the information specified at appendix G of this part. A decision to recommend proposal of a new method will be made by the Director of the Analytical Methods Staff within 90 days of receipt of a complete application.

(a) Tier 1: application of a new method in a single laboratory to one or more matrix types.

(1) A person may develop a new method for determination of an analyte of concern in one or more matrix types by validating the method and

developing QC acceptance criteria from an interlaboratory method validation study or from a single-laboratory validation study on each specific matrix type. Details of the single-laboratory method validation study and development of QC acceptance criteria from a single-laboratory or interlaboratory method validation study are specified at § 136.4(a) (1) and (2) and in appendixes E, F, and G of this part.

(2) A person who develops a new method under Tier 1 must submit the method to EPA for a letter of approval. The information that must be submitted and the format for submission is specified at in appendixes E, F, and G of this part.

(b) Tier 2: application of a new method in all laboratories to one or more matrix types within a single industrial category or subcategory.

(1) A person may develop a new method for determination of an analyte of concern in one or more matrix types within a single industrial category or subcategory by validating the method and developing QC acceptance criteria on each matrix type from an interlaboratory method validation study or from multiple, single-laboratory validation studies. Details of the multiple, single-laboratory method validation studies and development of QC acceptance criteria from these studies or from an interlaboratory method validation study are specified at § 136.4(b) (1) and (4) and in appendixes E, F, and G of this part.

(2) A person who develops a new method under Tier 2 must submit the method to EPA for approval and inclusion in a table in this part 136. The information that must be submitted includes the results of the performance tests required by paragraph (b)(1) of this section. This information and other detailed information that must be submitted and the format for submission are given in appendixes E, F, and G of this part.

(c) Tier 3: application of a new method by all laboratories to all matrix types in all industrial categories and subcategories (nationwide use).

(1) A person may develop for nationwide use a new method for determination of an analyte of concern in all matrix types by validating the method and developing QC acceptance criteria on the matrix type from an interlaboratory method validation study or from multiple, single-laboratory validation studies. Details of the multiple, single-laboratory method validation studies and development of QC acceptance criteria from these studies or from an interlaboratory method validation study are specified at

§ 136.4(c)(1) and in appendixes E, F, and G of this part.

(2) A person who develops a new method under Tier 3 must submit the method to EPA for approval and inclusion in a table in this part 136. The information that must be submitted includes the results of the performance

tests required by paragraph (c)(1) of this section. This information and other detailed information that must be submitted and the format for submission are given in appendixes E, F, and G of this part.

(d) The number and type of required tests, testing laboratories, matrices, and

replicate QC tests for the method validation specified at §§ 136.4, 136.5 (a), (b), and (c) and 141.27 depend on the tier at which the new or modified wastewater or drinking water method is validated. These requirements are summarized in the following table:

SUMMARY OF VALIDATION REQUIREMENTS FOR NEW METHODS AND METHOD MODIFICATIONS<sup>1</sup>

Method application	Number of			Number of analyses required			
	Labs	Matrix types	Facilities PWSs	IPR-reagent water <sup>2</sup>	IPR-sample matrix <sup>3</sup>	MS/MSD	MDL <sup>4</sup>
Tier 1-Single-lab:							
WW/DW—First matrix type or first PWS .....	1	1	1	4	4	<sup>5</sup> 2	7
WW—Each add'l matrix type (8 max.) from any industrial category	1	1	1	<sup>6</sup> 0	<sup>6</sup> 0	<sup>5</sup> 2	<sup>6</sup> 0
DW—Each add'l PWS (2 max.) .....	1	1	1	<sup>6</sup> 0	<sup>6</sup> 0	<sup>5</sup> 2	<sup>6</sup> 0
Tier 2-Multi-lab, single matrix type .....	3	1	3	12	0	<sup>7</sup> 6	21
WW/DW—Each matrix type in a single industrial category							
Tier 3-Multi-lab, multiple matrix types .....	<sup>8</sup> 9	9	9	36	0	<sup>7</sup> 18	63
WW only—All matrix types, all industrial categories							

<sup>1</sup> Numbers of analyses in this table do not include background analyses or additional QC tests such as calibration, blanks, etc. Validation requirements are based on the intended application of the method. Method application would be designated by tier for wastewater (WW) and drinking water (DW) programs. Three would be the maximum number of public water systems (PWSs) that would be required to validate a new or modified drinking water method at Tier 1 or 2. Nine would be the maximum number of matrix types (or facilities) that would be required to validate a new or modified wastewater method at Tier 1 or 3; at Tier 2 the number would be three matrix types.

<sup>2</sup> IPR reagent water analyses would be used to validate a method modification and to establish QC acceptance criteria for initial precision and recovery (IPR) and ongoing precision and recovery (OPR) for a new method. The required number of IPR analyses, except as noted under footnote 7, would be four times the number of laboratories required to validate a method modification or new method because each laboratory would perform a 4-replicate IPR test.

<sup>3</sup> IPR sample matrix analyses would be used to establish QC acceptance criteria for matrix spike/matrix spike duplicate (MS/MSD) recovery and precision for a Tier 1 new method only. Would not be required for validation of Tier 2 or 3 new methods because this variability data would be obtained from MS/MSD tests. Would not be required for validation of a method modification because MS/MSD data from the reference method would be used.

<sup>4</sup> A method detection limit (MDL) test would be performed in each laboratory using the new or modified method. 40 CFR part 136 Appendix B requires a minimum of seven analyses per laboratory to determine an MDL. Each lab involved in validation of a wastewater modification would demonstrate that the modified method would achieve the detection limits specified in the regulations at 40 CFR parts 136 and 141 and/or in chapter 6 of the Streamlining Guide (EPA 1996a).

<sup>5</sup> MS/MSD analyses would be required only for a method modification because, for new methods, the MS/MSD QC acceptance criteria would be established by the 4-replicate sample matrix IPR test. For modified methods, the MS/MSD test would demonstrate that the reference method MS/MSD QC acceptance criteria have been met.

<sup>6</sup> The MDL, reagent water IPR, and sample matrix IPR tests would not have to be repeated after the first matrix type, facility, or PWS was validated.

<sup>7</sup> For validation of a new method, the MS/MSD analyses would establish QC acceptance criteria for MS/MSD recovery and precision. For validation of a method modification, the MS/MSD analyses would demonstrate that reference method MS/MSD recovery and precision have been met. The required number of MS/MSD analyses would be two times the number of facilities, PWSs or matrix types tested.

<sup>8</sup> The number of laboratories and samples would vary if a conventional interlaboratory study is used.

6. Appendix A to 40 CFR part 136 is removed and reserved. Appendix E, Appendix F, and Appendix G are added to 40 CFR part 136 to read as follows:.

#### Appendix A to Part 136 [Removed and Reserved]

\* \* \* \* \*

#### Appendix E to Part 136—Equivalency Checklists

The Checklist for Initial Demonstration of Method Performance, Checklist for Continuing Demonstration of Method Performance, and Certification Statement (collectively called "Checklists") and instructions for their completion are

provided in this appendix. Because these checklists were developed by EPA's Environmental Monitoring Management Council (EMMC) for general application across all EPA programs, the lists contain categories that are not relevant to approval of drinking water or wastewater methods. Therefore, these categories are indicated in this appendix by "NA" (not applicable). The EMMC instructions have been annotated, where appropriate, to clarify each checklist item's applicability to the approval of drinking water and wastewater methods.

#### Checklist for Initial Demonstration of Method Performance

For the demonstration of equivalency, provide a checklist for each matrix in each medium.

Date:

Page \_\_\_\_\_ of \_\_\_\_\_

Laboratory Name & Address:

Facility Name:

Discharge Point ID:

EPA Program and Applicable Regulation:

Medium:

(e.g., wastewater, drinking water, soil, air, waste solid, leachate, sludge, other)

Analyte or Class of Analytes:

(e.g., barium, trace metals, benzene, volatile organics, etc.)



INITIAL DEMONSTRATION OF METHOD PERFORMANCE <sup>1</sup>

Category	Performance criteria <sup>2</sup> based on		Results obtained	Perf. spec. achieved (✓)
	Measurement quality objective	Reference method		
1. Written method (addressing all elements in the EMMC format) attached 2. Title, number and date/rev. of "reference method", if applicable <sup>3</sup> 3. Copy of the reference method, if applicable, maintained at facility 4. Differences between PBM and reference method (if applicable) attached 5. Concentrations of calibration standards 6. %RSD or correlation coefficient of calibration regression 7. Performance range tested (with units) 8. Sample(s) used in initial demonstration have recommended preservative, where applicable. 9. Sample(s) used in initial demonstration met recommended holding times, where applicable 10. Interferences 11. Qualitative identification criteria used 12. Performance Evaluation studies performed for analytes of interest, where available: Latest study sponsor and title: Latest study number: 13. Analysis of external reference material 14. Source of reference material 15. Surrogates used, if applicable 16. Concentrations of surrogates, if applicable 17. Recoveries of surrogates appropriate to the proposed use, if applicable 18. Sample preparation 19. Clean-up procedures 20. Method Blank Result 21. Matrix (reagent water, drinking water, sand, waste solid, ambient air, etc.) 22. Spiking system, appropriate to method and application 23. Spike concentrations (w/ units corresponding to final sample concentration) 24. Source of spiking material 25. Number of replicate spikes 26. Precision (analyte by analyte) 27. Bias (analyte by analyte) 28. Detection Limit (w/ units; analyte by analyte) 29. Confirmation of Detection Limit, if applicable 30. Quantitation Limit (w/ units: analyte by analyte) 31. Qualitative Confirmation 32. Frequency of performance of the Initial Demonstration 33. Other criterion (specify) 34. Other criterion (specify)				

<sup>1</sup> Provide a detailed narrative description of the initial demonstration.

<sup>2</sup> For multi-analyte methods, enter "see attachment" and attach a list or table containing the analyte-specific performance criteria from the reference method or those needed to satisfy measurement quality objectives.

<sup>3</sup> If a reference method is the source of the performance criteria, the reference method should be appropriate to the required application, and the listed criteria should be fully consistent with that reference method.

Name and signature of each analyst involved in the initial demonstration of method performance (includes all steps in the proposed method/modification):

Name

Signature

Date

Name

Signature

Date

Name

Signature

Date

The certification above must accompany this form each time it is submitted.

### Checklist for Continuing Demonstration of Method Performance

For the demonstration of equivalency, provide a checklist for each matrix in each medium.

Page \_\_\_\_ of \_\_\_\_

Date:

Laboratory Name & Address:

Facility Name:

Discharge Point ID:

EPA Program and Applicable Regulation:

Medium:

(e.g., wastewater, drinking water, soil, air, waste solid, leachate, sludge, other)

Analyte or Class of Analytes:

(e.g., barium, trace metals, benzene, volatile organics, etc.)

## CONTINUING DEMONSTRATION OF METHOD PERFORMANCE

Category	Required frequency	Specific performance criteria	Results obtained	Performance specific achieved (✓)
1. Method blank result (taken through all steps in the procedure) 2. Concentrations of calibration standards used to verify working range (with units), where applicable 3. Calibration verification 4. Laboratory control sample 5. External QC sample (where available) 6. Performance evaluation (PE) studies, if applicable Latest study sponsor and title: Latest study number: 7. List analytes for which results were "not acceptable" in PE study 8. Surrogates used, if applicable 9. Concentration of surrogates, if applicable 10. Recovery of surrogates (acceptance range for multi-analyte methods), if applicable 11. Matrix 12. Matrix spike compounds 13. Concentration of matrix spike compounds 14. Recoveries of matrix spike compounds 14a. Recoveries of matrix spike duplicate compounds 15. Qualitative identification criteria used 16. Precision (analyte by analyte) 17. Other category (specify) 18. Other category (specify)	.....	.....	.....	.....

Name and signature of each analyst involved in continuing demonstration of method performance (includes all steps in the proposed method/modification):

Name

Signature

Date

Name

Signature

Date

Name

Signature

Date

The certification above must accompany this form each time it is submitted.

#### Certification Statement

Page \_\_\_\_ of \_\_\_\_

Date:

Laboratory Name & Address:

Facility Name:

Discharge Point ID:

EPA Program and Applicable Regulation:  
Medium:

(e.g., water, soil, air)

Analyte or Class of Analytes:

(e.g., barium, trace metals, benzene, volatile organics, etc.; Attach separate list, as needed.)

We, the undersigned, CERTIFY that:

1. The method(s) in use at this facility for the analysis/analyses of samples for the programs of the U.S. Environmental Protection Agency have met the Initial and any required Continuing Demonstration of Method Performance Criteria specified by EPA.

2. A copy of the method used to perform these analyses, written in EMMC format, and copies of the reference method and laboratory-specific SOPs are available for all personnel on-site.

3. The data and checklists associated with the initial and continuing demonstration of method performance are true, accurate, complete and self-explanatory.<sup>1</sup>

4. All raw data (including a copy of this certification form) necessary to reconstruct and validate these performance related analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Facility Manager's Name and Title

Signature

Date

Quality Assurance Officer's Name

Signature

Date

This certification form must be completed when the method is originally certified, each time a continuing demonstration of method performance is documented, and whenever a

<sup>1</sup> True: Consistent with supporting data.

change of personnel involves the Facility Manager or the Quality Assurance Officer.

*Accurate:* Based on good laboratory practices consistent with sound scientific principles/practices.

*Complete:* Includes the results of all supporting performance testing.

*Self-Explanatory:* Data properly labeled and stored so that the results are clear and require no additional explanation.

#### EMMC Checklists Instructions

##### Checklists Overview

The Checklists were arrived at through consensus among EPA's programs by developing performance "categories" that allow use of the same Checklists across the Agency's various programs/projects. The Checklists may be applied to screening and field techniques as well as laboratory procedures.

Implementation of the Checklists is program-specific and a category that does not apply within a given EPA program will be indicated by NA (not applicable). Criteria for a specific EPA program are to be filled in under the "Performance Criteria" column; e.g., an Office of Water Reference Method may specify 20% RSD or a correlation coefficient of 0.995 for the category that specifies calibration linearity, whereas an Office of Solid Waste Project may specify a Measurement Quality Objective of 12% RSD or a correlation coefficient of 0.998 for this category.

For each EPA program, the Checklists are to be completed for each matrix within each medium for all matrices and media to which an alternate method or method modification applies. The EMMC definition of media is equivalent to the definition at 136.2 of matrix type. Each completed Checklist must be retained on file at the laboratory that uses the

performance-based method (PBM) or method modification and at the regulated facility from which samples are collected, and must be submitted to the appropriate Regulatory Authority upon request to support analysis of those samples to which the PBM or modified method was applied. (For wastewater and drinking water methods, the term "PBM method" in the preceding sentence is replaced with the term "new method".)

#### Header:

Each page of the checklist contains seven lines of header information, consisting of:

(1) Date (enter the date that the checklist was completed—Program/Project implementation plans should indicate whether the checklist must be submitted to the Regulatory Authority, as well as, retained on file at the laboratory and regulated facility).

(2) Laboratory Name & Address (If a commercial contract laboratory uses the method on behalf of one or more applicable clients, enter the name and address of the laboratory.)

(3) Facility Name (enter the name of the water treatment facility, system, or regulated facility or other program or project specified entity where the facility maintains an on-site analytical laboratory. If the method is being employed by a commercial contract laboratory on behalf of one or more applicable clients, enter the name of the laboratory followed by a listing of the appropriate clients).

(4) Discharge Point Identification Number (enter the discharge point identification number, if applicable).

(5) EPA Program & Applicable Regulation (enter the name of the Agency Program or Project to whom the results will be reported, or under the auspices of which the data are collected, e.g., "CAA" for Clean Air Act monitoring and "SDWA" for analyses associated with the Safe Drinking Water Act).

(6) Medium (enter the type of environmental sample, e.g., drinking water—**Note:** A separate checklist shall be prepared for each medium, e.g., for checklists associated with performance-based methods for SDWA, enter "Drinking Water" as the matrix type. As the evaluations of a performance-based method involve matrix-specific performance measures, a separate checklist shall be prepared for each matrix. The "medium" is the environmental sample type to which the performance-based method applies, whereas the performance category "matrix", appearing in the body of the checklists refers to the specific sample type within the "Medium" that was spiked, e.g., for "medium" hazardous waste, the checklist category "matrix" may be solvent waste. For wastewater and drinking water methods, the term "medium" is replaced with the term "matrix".

(7) Analyte or Class of Analytes, where available. (As many methods apply to a large number of analytes, it is not practical to list every analyte in this field, as indicated on the form, the class of analytes may be specified here, i.e., volatile organics. However, if such a classification is used, a separate list of analytes and their respective Chemical Abstract Service Registry Numbers (CAS #) must be attached to the checklist).

#### Initial Demonstration of Method Performance Checklist

The Initial Demonstration of Method Performance involves multiple spikes into a defined sample matrix (e.g., wastewater medium, paper plant effluent matrix), to demonstrate that the Performance-based Method meets the Program or Project Performance Criteria based on the performance of established "Reference Method" or based on "Measurement Quality Objectives" (formerly called Data Quality Objectives). This exercise is patterned after the "Initial Demonstration of Capability" delineated in a number of the Agency's published methods (Reference Methods).

Footnote #1 indicates that a detailed narrative description of the initial demonstration procedure is to be provided.

Footnote #2 indicates that for multi-analyte methods, the range of performance criteria for the analytes may be entered, but an analyte-specific performance criteria is to be attached. In general, when using the checklists, if the criteria or performance are lengthy, attach as a separate sheet, and enter "see attached" for this item.

Footnote #3 indicates that if a reference method is the source of the performance criteria, the reference method should be appropriate to the required application and the listed criteria should be fully consistent with that reference method. The reference method name and EPA number (where applicable) should be delineated in the program/project implementation plan, e.g., by the Program Office or the Project Officer/Manager.

There are 34 numbered entries in the body of the checklist—NOTE: Under normal circumstances, it would never be acceptable to answer "No" to any of these performance categories, or fail to attach the requested materials (categories not applicable to drinking or wastewater methods are marked with "NA"):

#1. Written Method (addressing all elements in the EMMC format)

The details of the method used for analysis must be described in a version of the method written in EMMC format, which is specified for drinking water and wastewater methods at 40 CFR part 136 Appendix F. The EMMC method format includes the following: 1.0 Scope & Application; 2.0 Summary of Method; 3.0 Definitions; 4.0 Interferences; 5.0 Safety; 6.0 Equipment & Supplies; 7.0 Reagents & Standards; 8.0 Sample Collection, Preservation & Storage; 9.0 Quality Control; 10.0 Calibration & Standardization; 11.0 Procedures; 12.0 Data Analysis & Calculations; 13.0 Method Performance; 14.0 Pollution Prevention; 15.0 Waste Management; 16.0 References; 17.0 Tables, Diagrams, Flowcharts & Validation Data. While this format may differ from that used in standard operation procedures (SOPs) in a given laboratory, the use of a consistent format is essential for the efficient and effective evaluation by inspectors, program and project managers/officers.

#2. Title, Number and date/revision of "Reference Method" if applicable.

For Example Polychlorinated Dioxins and Furans, EPA Method 1613, Revision B, October, 1994.

#3. Copy of the reference method, if applicable, maintained at the facility.

A copy of the reference method must be kept available for all laboratory personnel, however, it need not be attached to the checklist itself.

#4. Differences between PBM and reference method attached.

The laboratory must summarize the differences between the reference method and the performance-based method and attach this summary to the checklist. This summary should focus on significant difference in techniques (e.g., changes beyond the flexibility allowed in the reference method), not minor deviations such as the glassware used.

#5. Concentrations of calibration standards.

The range of the concentrations of materials used to establish the relationship between the response of the measurement system and analyte concentration. This range must bracket any action, decision or regulatory limit. In addition, this range must include the concentration range for which sample results are measured and reported (when samples are measured after sample dilution/concentration).

#6. % RSD or Slope/Correlation Coefficient of Calibration Regression.

This performance category refers to quantitative measures describing the relationship between the amount of material introduced into the measurement system and the response of the system, e.g., analytical instrument. A linear response is generally expected and is typically measured as either a linear regression or inorganic analytes, or as the relative standard deviation (or coefficient of variation) of the response factors or calibration factors for organic analytes. Traditional performance specifications considered any regression line with a correlation coefficient (r) of 0.995 or greater as linear. Also, for organic analytes, a relative standard deviation (RSD) of 25% or less is considered linear. The calibration relationship is not necessarily limited to a linear relationship. However, it should be remembered if the Program/Project Office or Officer/Managers specifies other calibration relationships, e.g., quadratic fit, more calibration standards are generally necessary to accurately establish the calibration. If applicable, a calibration curve, graphical representation of the instrument response versus the concentration of the calibration standards, should be attached.

#7. Performance Range Tested (with units).

This range must reflect the actual range of sample concentrations that were tested and must include the concentration units. Since the procedures may include routine sample dilution or concentration, the performance range may be broader than the range of the concentrations of the calibration standards.

#8. Sample(s) used in initial demonstration have recommended preservative, where applicable.

Unless preservation have been specifically evaluated, this entry should be taken directly from the reference method/standard. If preservation has been evaluated, include the study description and conclusions of that evaluation, with a reference to the specific study description. The data must be attached.

#9. Sample(s) used in the initial demonstration must be within the recommended holding times, where applicable.

Unless holding time (time from when a sample is collected until analysis) has been specifically evaluated, this entry should be taken directly from the reference method/standard. If holding time has been evaluated, include the study description and conclusions of that evaluation here, with a reference to the specific study description. The data must be attached.

#10. Interferences.

Enter information on any known or suspected interferences with the performance-based method. Such interferences are difficult to predict in many cases, but may be indicated by unacceptable spike recoveries in environmental matrices, especially when such recovery problems were not noted in testing a clean matrix such as reagent water. The inferences associated with the reference method are to be indicated, as well as, the affect of these interferences on the performance-based method.

#11. Qualitative identification criteria used.

Enter all relevant criteria used for identification, including such items as retention time, spectral wavelengths, ion abundance ratios. If the instrumental techniques for the performance-based method are similar to the reference method, use the reference method as a guide when specifying identification criteria. If the list of criteria is lengthy, attach it on a separate sheet, and enter "see attached" for this item.

#12. Performance Evaluation Studies performed for analytes of interest, where available (last study sponsor and title; last study number:).

Several EPA Programs conduct periodic performance evaluation (PE) studies. Organizations outside of the Agency also may conduct such studies. Enter the sponsor, title, and date of the most recent study in which the performance-based method was applied to the matrix of interest. For the performance-based method to be acceptable, the performance on such studies must be "fully successful", i.e., within the study QC acceptance criteria.

#13. Analysis of external reference material.

Enter the results of analyses on reference material from a source different from that used to prepare calibration standards (where applicable). This performance category is especially important if Performance Evaluation Studies are not available for the analytes of interest. Analysis of a reference sample is one of standardized QC elements specified for wastewater and drinking water methods at 40 CFR 136.4, 136.5 and 141.27. A common (and recommended) reference sample is a Reference Material from the National Institute of Standards and Technology.

#14. Source of reference material.

Enter criteria, if applicable, for traceability of materials used to verify the accuracy of the results, e.g., obtained from the National Institute of Science and Technology (NIST).

#15. Surrogates used if applicable.

Surrogates may be added to samples prior to preparation, as a test of the entire analytical procedure. These compounds are typically brominated, fluorinated or isotopically labeled compounds, with structural similarities to the analytes of interest. Also, they are not expected to be present in environmental samples. Surrogates are often used in the analysis for organic analytes. Enter the names of the surrogate compounds in this category.

#16. Concentrations of surrogates (if applicable).

Enter the concentration of surrogates once spiked into the sample (i.e., final concentration).

#17. Recoveries of Surrogates appropriate to the proposed use (if applicable).

Enter the summary of the surrogate recovery limits and attach a detailed listing if more space is needed.

#18. Sample Preparation.

Enter necessary preliminary treatments necessary, e.g., digestion, distillation and/or extraction. A detailed listing may be attached if more space is needed.

#19. Clean-up Procedures.

Enter necessary intermediary steps necessary prior to the determinative step (instrumental analysis), e.g., GPC, copper sulfate, alumina/Florasil treatment, etc.

#20. Method Blank Result.

A clean matrix (i.e., does not contain the analytes of interest) that is carried through the entire analytical procedure, including all sample handling, preparation, extraction, digestion, cleanup and instrumental procedures. The volume or weight of the blank should be the same as that used for sample analyses. The method blank is used to evaluate the levels of analytes that may be introduced into the samples as a result of background contamination in the laboratory. Enter the analyte(s) and concentration measured in the blank.

#21. Matrix (reagent water, drinking water, soil, waste solid, air, etc.).

Refers to the specific sample type within the broader "Medium" that was spiked, e.g., for the Medium "Hazardous Waste," an example matrix spiked as part of the initial demonstration of method performance might be "solvent waste". For wastewater and drinking water methods, the term "medium" is replaced with "matrix".

#22. Spiking System, appropriate to the method and application.

Enter the procedure by which a known amount of analyte(s) ("spike") was added to the sample matrix. This may include the solvent that is employed and the technique to be employed (e.g., permeation tube, or volumetric pipet delivery techniques spiked onto a soil sample and allowed to equilibrate one day, etc.). Solid matrices are often difficult to spike and considerable detailed narrative may be necessary to delineate the procedure. For spikes onto aqueous samples, generally a water miscible solvent is specified.

#23. Spike levels (w/units corresponding to final sample concentration).

Enter the amount of the analyte(s) ("spike") that was added to the sample matrix in terms of the final concentration in the sample matrix. For wastewater and

drinking water methods, initial spikes, also known as initial precision and recovery (IPR) standards, will be performed in reagent water. Using reagent water will allow the comparison of IPR spike recoveries determined with the modified method against IPR criteria specified in the reference method because reference method IPR specifications are developed from reagent water spikes.

#24. Source of spiking material.

Enter the organization or vendor from which the "spiking" material was obtained. This should include specific identification information, e.g., lot#, catalogue number, etc.

#25. Number of Replicate Spikes.

The initial demonstration of method performance involves the analyses of replicate spikes into a defined sample matrix category #21. Enter the number of such replicates. In general, at least four replicates should be prepared and analyzed independently.

#26. Precision (analyte by analyte).

Precision is a measure of agreement among individual determinations. Statistical measures of precision include standard deviation, relative standard deviation or percent difference.

#27. Bias (analyte by analyte).

Bias refers to the systematic or persistent distortion of a measurement process which causes errors in one direction. Bias is often measured at the ratio of the measured value to the "true" value or nominal value. Bias is often (erroneously) used interchangeably with "accuracy", despite the fact that the two terms are complementary, that is, high "accuracy" implies low "bias", and vice versa. Enter the name of the Bias measure (% recovery, difference from true, etc.), the numeric value with associated units for each analyte obtained for each analyte spiked in the initial demonstration procedure.

#28. Detection Limit (w/units; analyte by analyte).

A general term for the lowest concentration at which an analyte can be detected and identified. There are various measures of detection which include Limit of Detection and Method Detection Limit. Enter the detection measure (e.g., "MDL") and the analytical result with units for each analyte in the matrix (#21). For wastewater and drinking water methods MDL requirements are specified at 40 CFR 136.4 and 141.27.

#29. Confirmation of Detection Limit.

In addition to spikes into the matrix of interest (#21) it may be beneficial to perform the detection measurements in a clean matrix, e.g., laboratory pure water. Results of the spikes in the clean matrix are frequently available in the Agency's published methods. Determining MDLs in a clean matrix using the performance-based method will allow a comparison to the MDLs published in the Agency methods.

Also, the detection limit technique may specify specific procedures to verify that the obtained limit is correct, e.g., the "iterative process" detailed in the 40 CFR part 136, Appendix B, MDL procedures.

#30. Quantitation Limit (w/units; analyte by analyte).

The lowest concentration that the analyte can be reported with sufficient certainty that

an unqualified numeric value is reportable. Measures of Quantitation limits include the Minimum Level (ML), Interim Minimum Level (IML), Practical Quantitation Level (PQL), and Limit of Quantitation (LOQ). Enter the measure of quantitation limit, and the units for each analyte.

**#31. Qualitative confirmation.**

Enter all relevant criteria used for identification, including such items as: retention time; use of a second chromatographic column; use of second (different) analytical technique; spectral wavelengths; and ion abundance ratios. If the instrumental techniques for the modified method are similar to those of the reference method, use the reference method as a guide when specifying confirmation criteria. If the list of criteria is lengthy, attach it on a separate sheet, and enter "see attached" for this item.

**#32. Frequency (initial Demonstration to be performed).**

Enter the frequency that the initial demonstration has to be repeated, e.g., with each new instrument or once a year, which ever is more frequent.

**#33–#34. Other Criteria.**

Enter other necessary program/project specific method performance categories. For wastewater and drinking water methods, Categories 33 and 34 are used as follows:

**#33. Matrix Spike/Matrix Spike Duplicate.**

Enter the percent recoveries of analytes spiked into the sample matrix. For method modifications, only one set of matrix spike/matrix spike duplicate (MS/MSD) samples are required. For new methods, two sets of MS/MSD samples must be analyzed to provide sufficient data for QC acceptance criteria development.

**#34. Matrix Spike/Matrix Spike Duplicate Relative Percent Deviation.**

Enter the calculated relative percent deviation between the MS and MSD analyte recoveries.

**Signatures:**

The name, signature and date of each analyst involved in the initial demonstration of method performance is to be provided at the bottom of the check sheet.

**Continuing Demonstration of Capability Checklist**

The process by which a laboratory documents that their previously established performance of an analytical procedure continues to meet performance specifications as delineated in this checklist.

**#1. Method Blank.**

A clean matrix (i.e., one that does not contain the analytes of interest) that is carried through the entire analytical procedure, including all sample handling, preparation, extraction, digestion, cleanup and instrumental procedures. The volume or weight of the blank should be the same as that used for sample analyses. The method blank is used to evaluate the levels of analytes that may be introduced into the samples as a result of background contamination in the laboratory. Enter the analyte(s) and concentration measured in the blank.

**#2. Concentrations of calibration standards used to verify working range, where applicable (include units).**

The range of the concentrations of materials used to confirm the established relationship between the response of the measurement system and analyte concentration. This range must bracket any action, decision or regulatory limit. In addition, this range must include the concentration range for which sample results are measured and reported (when samples are measured after sample dilution/concentration). Enter the concentrations of the calibration standards.

**#3. Calibration Verification.**

A means of confirming that the previously determined calibration relationship still holds. This process typically involves the analyses of two standards with concentrations which bracket the concentrations measured in the sample(s). Enter the procedure to be used to verify the calibration and the results obtained for each analyte.

**#4. Calibration check standard.**

A single analytical standard introduced into the instrument as a means of establishing that the previously determined calibration relationship still holds. Enter the concentrations and result for each analyte.

**#5. External QC sample (where applicable).**

Enter the results of analyses for reference material (e.g., Quality Control samples/ ampules) from a source different from that used to prepare calibration standards (where applicable). Enter the concentration, as well as, the source of this material. This performance category is of particular importance if Performance Evaluation studies are not available for the analytes of interest.

**#6. Performance Evaluation studies performed for analytes of interest, where available (Last study sponsor and title: Last study number:).**

Several EPA Programs conduct periodic performance evaluation (PE) studies. Other organizations, outside of the Agency, also conduct such studies. Enter the sponsor, title, and date of the most recent study in which the performance-based method was applied to the matrix of interest. For the Performance-based method to be acceptable the performance on such studies must be "fully successful", i.e., within the study QC acceptance criteria.

**#7. List of analytes for which results were "not acceptable" in PE study.**

**#8. Surrogate Compounds used (if applicable).**

Surrogates may be added to samples prior to preparation, as a test of the entire analytical procedure. These compounds are typically brominated, fluorinated or isotopically labeled compounds, with structural similarities to the analytes of interest. They are compounds not expected to be present in environmental samples. Surrogates are often used in analyses for organic analytes. Enter the names of the surrogate compounds in this performance category.

**#9. Concentration of surrogates (if applicable).**

Enter the concentration of surrogates once spiked into the sample (i.e., final concentration), with units.

**#10. Recoveries of Surrogates appropriate to the proposed use (if applicable).**

Enter the summary of the surrogate recovery limits and attached a detailed listing (each surrogate compound), if more space is needed.

**#11. Matrix (reagent water, drinking water, soil, waste solid, air, etc.).**

Refers to the specific sample type within the broader "Medium" that was spiked, e.g., for the Medium "Hazardous Waste," an example matrix spiked as part of the initial demonstration of method performance might be "solvent waste".

**#12. Matrix Spike Compounds.**

In preparing a matrix spike a known amount of analyte is added to an aliquot of a real-world sample matrix. This aliquot is analyzed to help evaluate the effects of the sample matrix on the analytical procedure. Matrix spike results are typically used to calculate recovery of analytes as a measure of bias for that matrix. Enter the analytes spiked.

**#13. Matrix Spike Concentrations (w/units corresponding to final sample concentration).**

Enter the amount of the analyte(s) ("spike") that was added to the sample matrix in terms of the final concentration in the sample matrix.

**#14. Recovery of Matrix Spike (w/units).**

The ratio of the standard deviation of a series of at least three measurements to the mean of the measurements. This value is often expressed as a percentage of the mean.

**Note:** Some programs/projects have utilized matrix spike duplicates (a separate duplicate of the matrix spike) to help verify the matrix spike result and to provide precision data for analytes which are not frequently found in real-world samples, i.e., duplication of non-detects provides little information concerning the precision of the method.

**#15. Qualitative identification criteria used.**

Enter all relevant criteria used for identification, including such items as retention times, spectral wavelengths, ion abundance ratios. If the instrumental techniques for the Performance-based method are similar to the reference method, use the reference method as a guide when specifying identification criteria. If the list of criteria is lengthy, attach it on a separate sheet, and enter "see attached" for this item.

**#16. Sample Preparation.**

Enter necessary preliminary treatments necessary, e.g., digestion, distillation and/or extraction. A detailed listing may be attached if more space is needed.

**#17. Clean-up Procedures.**

Enter intermediary steps necessary to prior to the determinative step (instrumental analysis), e.g., GPC, copper sulfate, alumina/florisil treatment, etc.

**#18. Confirmation.**

Qualitative identification criteria used. Enter all relevant criteria used for identification, including such items as: retention time; use of second chromatographic column; use of second (different) analytical technique; spectral wavelengths, ion abundance ratios. If the instrumental techniques for the Performance-based method are similar to the reference method, use the reference method as a guide when specifying confirmation criteria. If the

list of criteria is lengthy, attach it on a separate sheet, and enter "see attached" for this item.

#19–20. Other.

Enter other necessary program/project specific method performance categories. Signatures:

The name, signature and date of each analyst involved in the continuing demonstration of method performance is to be provided at the bottom of the checklist.

#### **Appendix F to Part 136—Guidelines and Format for Methods to be Proposed at 40 CFR Part 136 or Part 141**

This appendix has been prepared to promote consistency among analytical methods and to streamline the method promulgation process. The elements in this appendix are mandatory for all methods proposed for approval at 40 CFR part 136 or 141. The appendix has four sections. The first section specifies standard elements that must be included in the method, the second section specifies the required method format, the third section specifies conventions to be used when preparing the method, and the fourth section specifies the required method content.

#### **1.0 Elements**

##### **1.1 Cover Page**

For methods submitted to EPA from other organizations or individuals, no cover page is required. Prior to method publication, EPA will prepare the cover page in the standard EPA format. The cover page will use black ink on white or colored paper stock and may include a cover graphic that illustrates the method.

EPA will assign a three- or four-digit method number that correlates with the EPA method series to which the method belongs. The method number is included as the first part of the method title on the cover page.

##### **1.2 Title Page**

There are two types of title page: a title page prepared by an organization or individual that is submitting a method to EPA, and the final title page that appears in the EPA-published method.

1.2.1 Individuals or organizations submitting methods to EPA should include the following information on the title page of the method: Method title, Date, and Sponsoring organization with address and telephone number.

1.2.1.1 When titling the method, use a concise title that cites (in sequence) the particular analyte(s) or property being determined, the type of sample or sample matrix(es) to which the method is applicable, as appropriate, and the determinative technique or instrumentation. Apply the following guidelines in titling methods:

1.2.1.1.1 If the method applies to numerous matrices (such as water, soil, sediment, sludge, tissue, and others), it may not be practical to include matrices in the title. However, if the method applies to a single matrix or a limited number of matrices, the matrix(es) should be specified in the title.

1.2.1.1.2 If the method is used to determine a number of analytes or properties,

analytes or properties can be named as a group (e.g., trace elements), and the names of specific analytes or properties omitted.

1.2.1.1.3 Avoid the use of the terms "analysis of..." or "determination of..." in method titles, since these terms are understood within the context of the term "method."

1.2.1.1.4 Method titles should use abbreviations or acronyms for familiar parts of the method title, e.g., HRGC/HRMS. The acronym or abbreviation should be defined at first use in the method. Examples of suitable method titles are: "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry" and "Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS".

1.2.1.2 For a methods manual, use a title that identifies the category of methods included in the manual. Examples of suitable methods manual titles are: "Analytical Methods for Pulp and Paper Industry Wastewater" and "Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewater".

1.2.2 Before publishing the method, EPA will generate a title page that mimics the cover page (excluding any cover graphics).

#### **11.3 Acknowledgments**

Acknowledgments should identify the author and editor, and provide credit to researchers, peer reviewers, and organizations or individuals that contributed directly and substantively in the development and writing of the method. These acknowledgments are independent of references listed at the end of the method.

#### **1.4 Disclaimer**

The disclaimer may appear on the same page with the acknowledgments or may be on the page following the acknowledgments. It may contain one or more disclaimer statements. All disclaimers should include the following statement: "The mention of trade names or commercial products does not constitute endorsement or recommendation for use."

The disclaimer may not state explicitly or imply that EPA has granted any approval of the method. Once the method has been validated and submitted to EPA for proposal, however, the following statement may be included: "This method has been submitted to the U.S. Environmental Protection Agency for use in EPA's water programs but has not been approved for use by EPA."

For draft methods, include the following statement: "This method is in draft form. It has not been released by the U.S. Environmental Protection Agency and should not be construed as an Agency-endorsed method. It is being circulated for comments on its technical merit."

When preparing the method for proposal at 40 CFR part 136 or 141, EPA will edit the disclaimer to cite the Agency review process that the method has undergone.

#### **1.5 Table of Contents**

A table of contents is required for methods manuals and is recommended for single methods that exceed 25 pages in length. The table of contents should cite the titles and

page numbers of all first- and second-order headings (see section 2.9 of this appendix) and all tables and figures.

#### **1.6 Introduction**

In the introduction, provide background on the method, describe the purpose of the method, and include a summary-level description of the method. Identify the name, organization, address, and telephone number to contact for questions regarding the method.

When preparing the validated method for submission to EPA for proposal at 40 CFR part 136 or 141, include the following instructions at the end of the introduction:

Questions concerning this method or its application should be addressed to: W. A. Telliard, USEPA Office of Water, Analytical Methods Staff, Mail Code 4303, 401 M Street, S.W., Washington, DC 20460, 202/260-7120.

Requests for additional copies of this publication should be directed to: Water Resource Center, Mail Code RC-4100, 401 M Street, S.W., Washington, DC 20460, 202/260-7786.

#### **1.7 Notice of Performance-based Method**

All methods prepared should be performance-based and should contain the following notice on a separate page directly preceding the body of the method: "Note: This method is performance-based. The laboratory is permitted to modify or omit any step or procedure, provided that all performance requirements set forth in this method and in the applicable regulations at 40 CFR parts 136 and 141 are met. The laboratory may not omit any quality control analyses. The terms "shall," "must," and "may not" indicate steps and procedures required for producing reliable results. The terms "should" and "may" indicate optional steps that may be modified or omitted if the laboratory can demonstrate that the modified method produces results equivalent or superior to results produced by this method."

#### **1.8 Body of Method**

The body of the method must be presented in the EMMC format. See Section 4.0 of this appendix for a detailed description of this format.

#### **2.0 Format**

##### **2.1 Page Numbering**

Page numbers should appear in the bottom center of the page. For methods prepared double-sided, page numbers may appear on the outside bottom corner of the page (i.e., on the bottom right for right-hand pages and on the bottom left for left-hand pages). 2.1.1 Numbering front matter—Number the front matter (i.e., everything preceding the body of the method) consecutively using lower-case Roman numerals. The numerals should appear on the bottom of each page of the front matter, except for the cover and title pages. The cover page is unnumbered. The title page holds the place of page i, but the numeral is not displayed.

2.1.2 Numbering body of method—Number the body of the method consecutively with Arabic numerals on the bottom of each page, starting with the number 1.

## 2.2 Method Identification

2.2.1 The method introduction page(s) should contain a header that identifies the method number and revision number or letter. The first page of the body of the method (preceding 1.0 Scope and Application) should start with the method number and title in the top center of the page with no header. Each subsequent page of the method should contain a header that identifies the method number and revision number or letter. The header also must be separated from the main body of the method by a horizontal line running the width of the page.

2.2.2 If the method was assigned a non-EPA method number during its development and validation, when preparing the method for submission to EPA for proposal at 40 CFR part 136 or 141, edit the header to reflect the method number assigned by EPA (i.e., Method 1664).

## 2.3 Method Date

The date of the method (month and year) should appear on the bottom of each page of the method.

## 2.4 Font

For text, use an 11-point Times Roman font (typeface). For first-order headings, use a bold, 14-point Univers font. For section numbering, use a bold, 12-point Univers font. For headers and footers, use an italics, 9-point Univers font. Univers or Times Roman fonts may be used in tables as appropriate. If Univers is unavailable, Helvetica may be substituted.

## 2.5 Margins

Left and right margins should be one inch. The header should be 0.5 inch from the top of the page, with the text starting one inch from the top of the page. The page number should appear 0.5 inch from the bottom of the page, with the text starting one inch from the bottom of the page.

## 2.6 Justification

Use left justification for text. This results in a ragged-right margin.

## 2.7 Line Spacing

The method should be single-spaced. (If preferred, 1.1 line spacing can be used to enhance readability.) One blank line should appear between each paragraph and section.

## 2.8 Method Sections

Each method must contain the sections given in the EMMC method format. See Section 4.0 for a detailed description of this format. If a section does not apply to a particular method, include the section with a statement that it is not applicable to that method.

## 2.9 Section Headings and Numbering

Use the Modified Decimal Numbering (MDN) system to organize material presented in methods and methods manuals. In this system, each method section and subsection is assigned a unique number that shows the relationship of a specific section/subsection to all previous sections/subsections and allows for easy reference. This numbering system is used in this document.

The first-order headings are the 17 sections identified in Section 4, starting with "1.0

Scope and Application". First-order headings must appear on a separate line, with a blank line appearing between the heading and the section text. Subsections are numbered and may or may not have a heading preceding the text. Second-order headings or sections are numbered 1.1, 1.2, 1.3, 1.4, etc. Third-order headings or sections are numbered 1.1.1, 1.1.2, 1.1.3, etc. Fourth-order headings or sections are numbered 1.1.1.1, 1.1.1.2, 1.1.1.3, etc.

Do not number beyond the fourth-order heading or section. If additional subdivisions are necessary, use (a), (b), (c), etc. to identify further divisions. Use of subdivisions below the fourth-order heading or section should be avoided where possible by organizing the material differently.

## 2.10 Indentation

First-order headings should appear flush left. Each subsequent order heading should be block-indented to align with the text of the previous order heading. This indentation method is illustrated in this document.

## 2.11 Electronic Submission

Methods and methods manuals must be prepared and submitted to EPA in both hardcopy and electronic formats.

2.11.1 Hardcopy methods should be produced in black type on white or off-white recycled paper and printed or copied double-sided.

2.11.2 Electronic methods must be submitted in machine-readable format, either ASCII or Agency standard (Novell WordPerfect® 6.1 or later).

2.11.3 To enable anyone accessing a method electronically to be certain they have retrieved the entire section or method accessed, include a "section end" notice at the end of each first-order section. This is illustrated as follows:

## 2.12 References

Use the following format for order, content, and punctuation when listing references.

2.12.1 Books—author's name or names (initials last), title of book (underline, period, no quotation marks), name of publisher, address of publisher (city and state), year of publication, and page number, if applicable

2.12.2 Magazines and Journals—author's name or names (initials last), "title of paper" (quotation marks, comma), volume number, issue number (this may be omitted if the journal page numbers are continuous throughout the volume), date of publications, and page numbers. Example: Jones, J.J., and Smith, R.R., "Correlation of Brinell Hardness and Tensile Strength, Materials in Design Engineering. Vol. 10, No. 2, February 1958, pp. 52–67.

2.12.3 Proceedings, Transactions, Reports, Bulletins, etc.—author's name or names (initials last), "title of paper" (in quotation marks), name of publication (underline, no quotation marks, comma), name of publisher, volume number, if any date of publication, and page numbers.

2.12.4 Symposium Volumes or Other Books Comprising Collections of Papers—Follow style for books, above and add title of paper, in quotes, after author's name.

2.12.5 Patents—patent number and data.

2.12.6 EPA methods—Method number and name, EPA report number, U.S.

Environmental Protection Agency, laboratory and/or office, location, date.

## 3.0 Conventions

### 3.1 Capitalization, Italics, Underlining, and Boldface

#### 3.1.1 Capitalization

3.1.1.1 For first-order headings (numbered 1.0, 2.0, 3.0, etc.), use initial capitalization of major words.

3.1.1.2 For second-, third-, or fourth-order headings, capitalize the first word of the heading only.

3.1.2 Italics—Italicize words or blocks of text for emphasis. Equations and notes interspersed in the text also should be italicized.

3.1.3 Underlining—Underline words that are defined in the Definitions section (or glossary). Use underlining in tables as appropriate for clear presentation of material. Do not use underlining for emphasis; use italics instead but avoid overuse of emphasis.

3.1.4 Boldface—Boldface the following items:

3.1.4.1 The method number and title on the cover page, title page, and page 1 of the method.

3.1.4.2 Acknowledgments, Disclaimer, and Introduction headings.

3.1.4.3 First-order headings.

3.1.4.4 Section numbering.

3.1.4.5 Equation numbers.

3.1.4.6 The word "Note:" preceding text notes.

## 3.2 Punctuation

3.2.1 Always use a comma after the second to last entry in a series.

3.2.2 A dash may be used between a subheading and text that directly follows the subheading. There should be no blank space before or after the dash, e.g., "Matrix Spikes—The laboratory must spike..."

3.2.3 As a general rule, use a hyphen in compound modifiers to avoid ambiguity, e.g., 1–L flask. (In some cases, the hyphen can be left out without ambiguity, e.g., toxic chemical waste.) Do not use a hyphen after an adverb ending in "ly," e.g., commonly accepted practice.

3.2.4 Bullets are not to be used in the body of the method. If used in introductory material, the text following the bullet should start with a capital letter. Short bullets do not require periods at the end; long (multiple-line) bullets do. Semicolons or commas should not be used after bulleted text.

## 3.3 Footnotes

Use footnotes only in tables. Footnotes should be designated with numbers or lower case letters in superscript, and should appear below the body of the table.

## 3.4 Text Notes

Notes may be used within the text to highlight important information regarding use of the method. Use a margin-to-margin line across the page both preceding and following the note to set it off from the text.

## 3.5 Equations

Equations should be numbered Equation 1, Equation 2, etc., consecutively as they appear in the text. Use a margin-to-margin line across the page both preceding and following

the equation to set it off from the text. Equations should be presented in italics. The equation is followed by "where:" and a list of terms used in the equation (e.g., where:  $n$  = number of samples,  $x$  = concentration in each sample).

### 3.6 Tables and Figures

Tables and figures appear in Section 17.0.

3.6.1 Number tables and figures consecutively with Arabic numerals, and give each a title that is complete and descriptive.

3.6.2 In table column headings, specify the quantity being tabulated, followed by the units of measurement shown in parentheses. For example, "Amount spiked ( $\mu\text{g/L}$ )".

3.6.3 Place table and figure titles above the information presented.

3.6.4 Figures may be enclosed in a box if desired.

### 3.7 Trademarks

3.7.1 Avoid the use of trademarks or brand names whenever possible. For examples, use the term "borosilicate glass" rather than the trademarks Pyrex or Kimax; use "fluoropolymer" rather than Teflon. (See Section 4.6.4.)

3.7.2 When a trademark or brand name is used, capitalize it.

### 3.8 Text References

Text references are references to other locations within the method, not references to any outside source. References to other sources appear in Section 16.0. Do not incorporate essential information into the method by referring to another method.

In the method text, refer to other sections of the method capitalizing the word "Section." Section references should appear in parentheses at the end of the phrase or sentence to which the reference applies, for example, (Section 9.6).

### 3.9 Units, Symbols, Abbreviations, and Acronyms

3.9.1 Units and symbols from the international metric system (SI, from the French name, *Le Systeme International des Unites*) are to be used. SI is based on seven basic units that are dimensionally independent. The SI unit of time is the second (symbol =  $s$ ) which should be used if practical. The SI unit of volume is the cubic meter (symbol =  $\text{m}^3$ ) but the spectral name liter (symbol =  $L$ ) can be used for liquids and gases. Although the SI unit for mass is kilogram (symbol =  $\text{kg}$ ), the use of gram ( $g$ ) with or without prefixes is appropriate.

3.9.2 Symbols, not abbreviations, should be used for units. Symbols are not followed by a period except when used at the end of a sentence. Unit symbols are written in lower case except for the symbol for liter ( $L$ ) or where the unit name was derived from a proper name, such as  $\text{Pa}$ , from Pascal. When a quantity is expressed as a numerical value and a unit symbol, a space should be left between them, except between the number and symbol for degree Celsius (e.g.,  $20^\circ\text{C}$ ) and for degree, minute, and second of plane angle.

3.9.3 Use commonly accepted acronyms and abbreviations in text and tables. An acronym is a word formed from the first or

first few letters of other words; everything else is an abbreviation. In many cases, an acronym or abbreviation is more readily identifiable than its narrative counterpart. Always spell out the term the first time it is used and follow it with the acronym or abbreviation shown in parentheses, e.g., material safety data sheet (MSDS), relative percent difference (RPD), or United States Environmental Protection Agency (EPA). Acronyms and nearly all abbreviations have no periods or spaces between letters. As depicted in these examples, although the acronym or abbreviation is capitalized, the narrative version of it is not capitalized unless it is a proper name such as a government agency, society, or association. Once an acronym or abbreviation is introduced in this manner, use only the acronym or abbreviation subsequently.

3.9.4 When a long word or phrase for which there is no standard acronym or abbreviation is used frequently, it may be replaced by an acronym or abbreviation that is explained when it first occurs. For example, relative centrifugal force (RCF).

### 3.10 Numerals

3.10.1 Spell out single-digit numbers (one through nine), with the following exceptions:

3.10.1.1 Use numerals when the quantity is partly fractional, e.g., 1.15,  $1\frac{1}{2}$ .

3.10.1.2 Use numerals when the number is followed by a unit symbol, e.g., 1 m, 9%, 3 ppm. In the method text, units should be spelled out, so the numbers one through nine associated with the units would be spelled out also (e.g., one meter, nine percent, three parts per million).

3.10.1.3 Use numerals to identify equations and tables (e.g., Equation 2, Table 5).

3.10.1.4 In sentences containing multiple numbers, if some numbers must be numerals, use numerals for all (e.g., 2 tests and 16 weighings).

3.10.2 Use numerals for multiple-digit numbers (10 and above), with the following exceptions:

3.10.2.1 Do not begin a sentence with a numeral. When the numeral is spelled out, also spell out the unit following (e.g., One gram is usually sufficient.)

3.10.2.2 Spell out round numbers that are used in an indefinite sense (e.g., a hundred feet or so).

3.10.3 When a number is used as an adjective, insert a hyphen between the number and the unit symbol (e.g., 100-mL volumetric flask, 1-L sample).

3.10.4 When writing decimal numbers of value less than one, place a zero before the decimal point (e.g., 0.45 g).

3.10.5 Do not point-off numbers of four figures (1234) except in tables when they occur in a column containing numbers of more than four figures. Point-off numbers of more than four figures, using commas with no spaces (e.g., 1,325,000).

3.10.6 In expressing ranges and ratios in text, use 1 to 10 or 1:10, not 1–10. A hyphen may be used for ranges in tables.

### 3.11 Significant Digits

Handle numbers with careful regard for correspondence between the data accuracy and the given number of digits. The number

of significant digits should neither sacrifice nor exaggerate accuracy.

3.11.1 Any digit that is necessary to define the specific value or quantity is significant and should be used. For example, when measured to the nearest 1 m, a distance may be 157 m, which has three significant figures; when measured to the nearest 0.1 m, the distance may be 157.4 m, which has four significant figures.

3.11.2 When adding or subtracting numbers with different degrees of precision, the answer should contain no digits farther to the right than the least precise number. Numbers should first be rounded to one digit farther to the right than that of the least precise number. The answer is then rounded to the same number of significant figures as the least precise number.

3.11.3 For multiplication and division, the product or quotient should contain no more significant figures than are contained in the number with the fewest significant figures.

3.11.4 Examples to distinguish the addition/subtraction and multiplication/division rules are:

Addition:  $113.2 + 1.43 = 114.63$ , rounded to 114.6

Subtraction:  $113.1 - 1.43 = 111.77$ , rounded to 111.8

Multiplication:  $113.2 \times 1.43 = 161.876$ , rounded to 162

Division:  $113.1 \div 1.43 = 79.16$ , rounded to 79.2

**Note:** The product and quotient above should contain only three significant figures because the number 1.43 contains only three significant figures. The above sum and difference, however, contain four significant figures, because digits that occur to the right of the last significant in the least precise number are rounded.

### 3.12 Order of Magnitude

Zeros may be used to indicate a specific value or to indicate the order of magnitude of a number. For example, in the number 203,185,000, representing population rounded to thousands, the first six digits are significant. The last three digits are zeros that indicate the order of magnitude.

### 3.13 Rounding

3.13.1 When the first digit discarded is less than five, the last digit retained is not changed.

3.13.2 When the first digit discarded is five or greater, or when five is followed by a digit other than zero, the last digit retained is increased by one.

3.13.3 When the first digit discarded is exactly five followed only by zeros, the last digit retained is rounded upward if it is an odd number and is not adjusted if it is an even number.

### 4.0 Content

In accordance with EMMC format, each analytical method must contain 17 specific topical sections in a designated order. The required order and content of these sections are listed and described below. All of these sections are mandatory for all methods.

1.0 Scope and Application

2.0 Summary of Method

3.0 Definitions

4.0 Interferences



- 5.0 Safety
- 6.0 Equipment and Supplies
- 7.0 Reagents and Standards
- 8.0 Sample Collection Preservation and Storage
- 9.0 Quality Control
- 10.0 Calibration and Standardization
- 11.0 Procedure
- 12.0 Data Analysis and Calculations
- 13.0 Method Performance
- 14.0 Pollution Prevention
- 15.0 Waste Management
- 16.0 References
- 17.0 Tables, Diagrams, Flowcharts, and Validation Data

Starting with section 11.0 Procedure, additional numbered sections may be inserted as required by the particular method; however, the sections listed above must appear in each method in the order listed.

**Note:** Subsections within each of the 17 required sections do not need to correlate directly to the subsections included here. In other words, the information mentioned in 4.1.1 below might be covered in two or more subsections in a method.

#### 4.1 Scope and Application

This section outlines the purpose, range, limitations, and intended use of the method, and identifies target analytes.

4.1.1 Define the purpose and intended use of the method. State what the method is based upon, noting any relationship of the method to other existing analytical methods. Indicate whether the method is associated with a sampling method. Include the following statement: "This method is for use in the Environmental Protection Agency's (EPA's) data gathering and monitoring programs under the Clean Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation, and Liability Act, and the Safe Drinking Water Act."

4.1.2 List analytes that can be measured by the method, including each analyte's Chemical Abstracts Service Registry Number (CASRN). If regulations cite other than the most commonly used analyte name, refer to the regulation. For pesticides, use "acceptable common names." The use of registered trade names is permitted.

4.1.3 Identify the matrix(ces) for which the method has been found satisfactory.

4.1.4 Indicate the statistically determined method detection limit (MDL) and the analyte concentration range over which the method is applicable. State the matrix(ces) in which MDL was determined. If the MDL is not available, report an instrumental detection limit and define how it was derived. Indicate the minimum level (ML) and water quality criteria if appropriate to the analyte and method.

4.1.5 Describe method limitations, such as "This method is not applicable to saline water," or "This method is not intended for determination of metals at concentrations normally found in treated and untreated discharges from industrial facilities." Indicate any means of recognizing cases where the method may not be applicable to the sample under test.

4.1.6 List any restrictions that may apply, such as "This method is restricted to use by

or under the supervision of analysts experienced in \* \* \*

4.1.7 Include the following statement regarding performance-based methods: "This method is performance-based. The laboratory is permitted to omit any step or modify any procedure (e.g., to overcome interferences, to lower the cost of measurements), provided that all performance requirements in this method are met. Requirements for establishing method equivalency are given in Section 9.1.2."

4.1.8 Include the following statement: "Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in section 9.1.2."

#### 4.2 Summary of Method

This section provides an overview of the method procedure and quality assurance.

4.2.1 Outline, specifying amounts of sample and reagent, the procedure that is followed to determine the presence or absence of the listed analytes. Include any sample pretreatment, such as filtration or digestion. In this description, identify the basic steps involved in performing the method, but omit the details that are a necessary part of the complete statement of procedure.

4.2.1.1 For chemical methods, state the type of procedure (colorimetric, electrometric, volumetric, etc.) and describe the source of color, major chemical reaction, including pertinent chemical equations, etc. For instrumental methods, state the technique.

4.2.1.2 In the "Summary of Method" section, use the passive voice, e.g., "Instrumental drift is corrected by using internal standardization," rather than "Correct instrumental drift by using internal standardization."

4.2.2 Identify the determinative step in the method.

4.2.3 State in a summary fashion how quality is assured in the method.

4.2.4 List options to the method, if applicable.

#### 4.3 Definitions

This section includes definitions of terms, acronyms, and abbreviations used in the method. If preferred, definitions may be provided in a glossary at the end of the method or manual. In this case, the definitions section must still appear in the method, with a notation that definitions are provided in a glossary at the end of the method. Refer to the specific section number of the glossary.

4.3.1 Include an introductory statement as follows: "The definitions and purposes below are specific to this method, but have been conformed to common usage as much as possible."

4.3.2 List units of weight and measure and their abbreviations or acronyms used in the method.

4.3.3 Alphabetically list and define terms, acronyms, and abbreviations used in the method. Where appropriate, include the purpose (e.g., the purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample).

4.3.4 Include definitions of the terms may, may not, must, and should, as follows:

4.3.4.1 May: This action, activity, or procedural step is neither required nor prohibited.

4.3.4.2 May not: This action, activity, or procedural step is prohibited.

4.3.4.3 Must: This action, activity, or procedural step is required.

4.3.4.4 Shall: This action, activity, or procedural step is required.

4.3.4.5 Should: This action, activity, or procedural step is suggested but not required.

#### 4.4 Interferences

This section identifies known or potential interferences that may occur during use of the method, and describes ways to reduce or eliminate interferences.

4.4.1 Describe any known or potential problem(s) (e.g., sample or equipment contamination, instrument noise) that may be encountered during the performance of the method and the source of the problem(s). Recommend techniques to avoid or minimize the problem(s) (e.g., ways to reduce sample or equipment contamination, or instrument noise).

4.4.2 Identify any substances, ions, or properties that are known to or likely to cause interference and the amounts that are known to or likely to interfere. Sometimes this information can be obtained only by observation during the analysis. In such cases, include appropriate notes under "Procedure" or "Data Analysis and Calculations."

#### 4.5 Safety

This section describes special precautions needed to ensure personnel safety during the performance of the method. Procedures described here should be limited to those which are above and beyond good laboratory practices. The section must contain information regarding specific toxicity of analytes or reagents.

4.5.1 Identify and warn analysts of potential hazards associated with using the method (e.g., toxicity or carcinogenicity of analytes or reagents, explosions, fire, radiation). Recommend techniques to minimize hazards where possible (e.g., performing operations in a hood or glove box).

4.5.2 Where the toxicity or carcinogenicity of each compound or reagent has not been precisely determined, include the following statement: "The toxicity or carcinogenicity of each analyte or reagent has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that the laboratory perform personal hygiene monitoring of each analyst using this method and that the results of this monitoring be made available to the analyst."

4.5.3 Indicate the steps in the procedure at which hazards that could damage equipment may occur by use of the word CAUTION in boldface type, followed by the details of the precautionary measures that must be taken. If any step in the procedure could result in personal injury or death, include the word WARNING in boldface type, followed by the details of the protective measures that must be taken.

4.5.4 Include the following statements: "This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References \_\_\_\_."

#### 4.6 Equipment and Supplies

This section lists and describes all nonconsumable supplies and equipment needed to perform the method.

4.6.1 Include the following statement as a note preceding the list of equipment and supplies:

**Note:** Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory."

4.6.2 Categorize and list required equipment and supplies by the logical order of use; e.g., sampling equipment, equipment for glassware cleaning, equipment for calibration, equipment for sample extraction, etc. Do not list common laboratory equipment, but do include special or modified forms of unusual sizes or numbers of common equipment that are required or that may require special preparation.

4.6.3 Describe the essential features of each required item. Include schematic drawings as needed to clarify or supplement apparatus descriptions.

4.6.4 Avoid the use of trademarks, brand names, trade names, or suppliers unless a specific manufacturer's product is required for a well-defined reason or the availability of the product is limited (i.e., the apparatus is unique or unusual). For example, when special types of glassware are required, such as heat-resistant, chemical-resistant, etc., state the significant characteristic desired rather than a trademark ("borosilicate glass" rather than Pyrex or Kimax). If only a single source is known, that supplier may be identified.

4.6.5 Whenever a brand name is used, include "or equivalent" following the brand name or part number to demonstrate that use of another product is acceptable.

4.6.6 Include any special glassware cleaning instructions.

4.6.7 List special facilities required, such as a special room for handling hazardous materials.

#### 4.7 Reagents and Standards

This section lists and describes all reagents and standards required to perform the method, and provides preparation instructions and/or suggested suppliers as appropriate.

4.7.1 List the name of the reagent and the necessary purity, followed by any descriptive terms. List reagents in a logical order (e.g., by order of occurrence or use, by group). The

method should require that reagents be ACS Reagent Grade unless otherwise specified.

4.7.2 Spell out the full name of inorganic reagents when first used, and include within parentheses the exact chemical formula, showing its water of crystallization, etc. Subsequently, refer to inorganic compounds by formula if they can be specified clearly in this way. As exceptions, always spell out the word "water" and the names of substances in their elemental state (e.g., "lead" not "Pb," "oxygen" not "O<sub>2</sub>").

4.7.3 Spell out organic, organometallic, or complex inorganic compounds; chemical formulae are not necessary. Cite the CASRN to avoid ambiguity.

4.7.4 Avoid the use of trademarks and names of patented products. Use chemical names and common names, unless a specific product is required for a well-defined reason. The use of registered trade names is permitted.

4.7.5 Unique and unusual reagents can be named by brand. Whenever a brand name is used, include "or equivalent" following the brand name to demonstrate that another product can be used.

4.7.6 Specify the concentration of inorganic reagents in applicable terms, as follows:

Concentrated acids and bases: density  
Dilute acids and bases: volume ratio, x+y (x volume of reagent added to y volume by water)

Nonstandardized solutions: normality, expressed decimally; or the equivalent of 1 mL of solution in terms of grams of a given element expressed as 1 mL = x.xx g of  
\* \* \*

4.7.7 Specify filter paper by describing the significant characteristic such as porosity, rate of filtering ash content, etc., or by reference to ASTM Specification D1100 for Filter Paper for Use in Chemical Analysis.

#### 4.8 Sample Collection, Preservation, and Storage

This section provides requirements and instructions for collecting, preserving, and storing samples.

4.8.1 Give detailed directions for collecting, filtering (if applicable), preserving, shipping, and storing samples.

4.8.2 Use preservation procedures and holding times consistent with those specified in current EPA publications or regulations and with other methods for the same analytes.

#### 4.9 Quality Control

This section cites the procedures and analyses required to fully document the quality of data generated by the method. The required components of the laboratory's quality assurance (QA) program and specific quality control (QC) analyses are described in this section. For each QC analysis, the complete analytical procedure, the frequency of required analyses, and interpretation of results are specified.

**Note:** To ensure data quality, water methods must specify a comprehensive laboratory QA program. The minimum QC requirements that must be included in methods proposed at 40 CFR part 136 or part 141 are specified at 40 CFR 136.3 table IF,

136.4, 136.5 and 141.27. The method should specify QC acceptance criteria.

4.9.1 Include the following statements in the first subsection (Section 9.1): "Each laboratory that uses this method is required to operate a formal quality assurance program (Reference \_\_\_\_). The minimum requirements of this program consist of an initial demonstration of laboratory capability, ongoing analyses of standards and blanks as a test of continued performance, and [complete as appropriate to the method]. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method."

"The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2."

4.9.2 In Section 9.1, cite any options that the analyst is permitted, e.g., alternate extraction, concentration, or cleanup procedures; changes in columns or detectors. Specify that the analyst is required to repeat the required initial demonstration of laboratory capability each time a modification is made to the method. Include the following statements: "Each time a modification is made to the method, the analyst is required to repeat the procedure in section 9.2. If the change will affect the detection limit of the method, the laboratory is required to demonstrate that the MDL (40 CFR part 136, Appendix B) is lower than the MDL for that analyte in this method, or one-third the regulatory compliance level, whichever is higher. If the change will affect calibration, the analyst must recalibrate the instrument according to section 10.1."

"Changes that degrade method performance are not allowed. If an analytical technique other than the techniques specified in this method is used, that technique must have a specificity equal to or better than the specificity of the techniques in this method for the analytes of interest."; and

"The laboratory is required to maintain records of modifications made to this method. These records include the following, at a minimum:

- The names, titles, addresses, and telephone numbers of the analyst(s) who performed the analyses and modification, and of the quality control officer who witnessed and will verify the analyses and modification.
- A listing of analytes measured, by name and CASRN.
- A narrative stating reason(s) for the modification(s).
- Results from all QC tests comparing the modified method to this method, including:
  - (a) Calibration (section 10)
  - (b) Calibration verification (section 9.5)
  - (c) Initial precision and recovery (section 9.2.2)
  - (d) Analysis of blanks (section 9.4)
  - (e) Accuracy assessment (section 9.3)
  - (f) Ongoing precision and recovery (section 9.6)
- Data that will allow an independent reviewer to validate each determination by tracing the instrument output (weight or

other signal) to the final result. These data are to include:

- (a) Sample numbers and other identifiers
- (b) Extraction dates
- (c) Analysis dates and times
- (d) Analysis sequence/run chronology
- (e) Sample weight or volume
- (f) Extract volume
- (g) Make and model of analytical balance and weights traceable to NIST
- (h) Copies of logbooks, printer tapes, and other recordings of raw data
- (i) Data system outputs, and other data to link the raw data to the results reported

4.9.3 In the remainder of section 9.1, outline the QC requirements that will be described in the section, and the purpose for each type of QC (e.g., blanks, matrix spikes/matrix spike duplicates, calibration verification).

4.9.4 In section 9.2, describe in detail the initial demonstration of laboratory capability.

4.9.5 Describe the procedure for matrix spikes, calculating percent recoveries, and calculating relative percent difference for duplicates.

4.9.6 Provide instructions for analysis of blanks, e.g., laboratory reagent blanks, method blanks.

4.9.7 Specify requirements for calibration verification.

4.9.8 Provide instructions for analysis of ongoing precision and recovery standards.

4.9.9 Include requirements for analysis of quality control samples (QCS).

4.9.10 Include the following statement at the end of section 9.0: "Depending upon specific program requirements, field replicates and field spikes of the analytes of interest into samples may be required to assess the precision and accuracy of the sampling and sample transporting techniques."

#### 4.10 Calibration and Standardization

This section describes the method/instrument calibration and standardization process, and required calibration verification. Corrective actions are described for cases when performance specifications are not met.

4.10.1 Specify operating conditions or refer to manufacturer's recommended operating conditions. If appropriate, specify a precalibration routine as needed to document instrument stability.

4.10.2 Give detailed instructions for the use of standards to prepare calibration lines or tables. Include the number of calibration standards, the need for blanks, the frequency of calibration checks, the critical range, etc.

4.10.3 Give detailed instructions for internal standardization, including number and concentration of internal standards.

4.10.4 Include instructions for calibration data storage.

#### 4.11 Procedure

This section describes the sample processing and instrumental analysis steps of the method, and provides detailed instructions to analysts.

4.11.1 For methods used for determination of a method-defined analyte, include the following statement in the introductory portion of Section 11.0 Procedure: "This method is entirely empirical. Acceptable results can be obtained

only by strict adherence to all details." Do not include this statement in methods for which the analyte is a chemical or physical parameter, the characteristics of which are known (e.g., oil and grease, COD, BOD).

4.11.2 Include in proper sequence detailed directions for performing the analysis.

4.11.2.1 Include steps that are essential to the process and avoid unnecessarily restrictive instructions.

4.11.2.2 Organize the procedure by logical order of activity, e.g., sample preparation, extraction, analysis.

4.11.2.3 Describe the procedure in the imperative mood, present tense, e.g., "Heat the sample aliquot," rather than "The sample aliquot should be heated." Comments and descriptive information that are not in the imperative mood may be included, as appropriate.

4.11.2.4 Write the text so that it is concise and easily understandable.

4.11.2.5 When alternative procedures are given, state which is preferred.

4.11.3 In chemical methods, specify the size of sample aliquot and indicate the required measurement accuracy. (There is no need to weigh a sample to five significant figures in a spectrophotometric method where the final absorbance measurement yields data with only three significant figures).

4.11.4 Include "Notes" throughout the procedure to highlight critical points. Include notes of "WARNING" or "CAUTION" as appropriate to identify known or potential hazards to the analyst or the equipment, respectively.

4.11.5 Indicate steps in which timing is critical, e.g., if a determination may not be interrupted overnight. For a color reaction, indicate how long the color is stable.

#### 4.12 Data Analysis and Calculations

This section provides instructions for analyzing data, and equations and definitions of constants used to calculate final sample analysis results.

4.12.1 Calculations—Provide directions for calculating the results of the analysis, including any equations.

4.12.1.1 Use the imperative mood, e.g., "Report results to three significant figures," rather than "Results should be reported to three significant figures."

4.12.1.2 Where there may be ambiguity of meaning, spell out names in the text (e.g., total Kjeldahl nitrogen) but use the abbreviations (e.g., TKN) in text where the meaning is clear, and in equations.

4.12.1.3 Define the symbols used in the equation immediately under the equation.

4.12.1.4 Use numerical values for any constants. Identify dilution factors, titration factors, etc.

#### 4.12.2 Reporting Results

4.12.2.1 Indicate the units in which the results are to be reported (e.g., >g/L, mg/kg).

4.12.2.2 If the sample is a solid material such as a sediment or sludge, indicate whether results are to be reported as wet weight or dry weight.

4.12.2.3 Specify the number of significant figures to be reported.

4.12.2.4 Require that all values obtained by various QC procedures are reported along with the calculated results of the analysis.

4.12.3 Interpretation of results—Use this heading in place of Calculations when the results of the analysis must be expressed in descriptive form, relative terms, or abstract values. List and define the descriptive terms or classifications used.

#### 4.13 Method Performance

This section provides method performance criteria for the method, including precision/bias statements regarding detection limits and source/limitations of data produced using the method.

**Note:** Requirements for validating new methods are specified in [cite the volume and page number of the **Federal Register** in which the streamlining initiative is promulgated].

4.13.1 Explain how the method was validated. Provide a detailed description of method performance, including data on precision, bias, detection limits (including the method by which they were determined and matrices to which they apply), and statistical procedures used to develop performance specifications.

**Note:** This information can be provided through reference to the method validation study.

4.13.2 At a minimum, state single-operator precision and accuracy on reagent water. If other sample types have been investigated, also provide this information for them.

4.13.3 If a collaborative study has been completed, describe the study and report the number of participating operators and laboratories, spike concentrations, level of replication, types of background waters, and any other significant aspects. If the study has been documented, cite the study report and include it in the References section. When citing reference documentation, the details of the study do not have to be included in this section.

#### 4.14 Pollution Prevention

This section describes aspects of the method that minimize or prevent pollution known to be or potentially attributable to the method.

4.14.1 Cite potential sources of pollution attributable to the method.

4.14.2 Recommend ways to minimize pollution.

#### 4.15 Waste Management

This section describes minimization and proper disposal of waste and samples.

4.15.1 Include the following statement as the first subsection: "It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required."

4.15.2 Provide instructions for sample and waste handling and disposal.

4.15.3 Include the following statement as the last subsection: "For further information on waste management, consult The Waste Management Manual for Laboratory Personnel and Less is Better: Laboratory Chemical Management for Waste Reduction, both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW., Washington DC, 20036."

#### 4.16 References

This section lists references for source documents and publications that contain ancillary information.

**Note:** Each method should be a free-standing document, providing all information necessary for the method user to perform the method may be found. References within a method should be restricted to associated or source material. Procedural steps or instructions should not be referenced as being found elsewhere, but should be included in total within the method.

4.16.1 Include references for other, related EPA methods; and published studies/articles relating to method performance, techniques, or analytes, and health and safety.

4.16.2 List references in the order cited in the method, and assign each reference an identification number using Arabic numerals.

4.16.3 As a rule, do not list documents that are not readily accessible to the reader (e.g., unpublished theses, personal communications, private correspondence). If it is important to list these types of documents, identify where the reader may obtain a copy of the document.

#### 4.17 Tables, Diagrams, Flowcharts, and Validation Data

This section contains all method tables and figures (diagrams and flowcharts), and may contain validation data referenced in the body of the method.

4.17.1 In addition to tables and figures, include additional useful information. Examples of such information include:

4.17.1.1 Notes on significance and interpretation of the method, used to amplify the statement in the text.

4.17.1.2 Development of equations used in the calculations.

4.17.1.3 Charts or supplementary information for computations.

#### 4.18 Glossary

This optional section contains a glossary of terms, acronyms, abbreviations, and symbols used in the method.

**Note:** This information may appear in the Definitions section of the method (Section 3.0) or may be included in a glossary at the end of the method.

4.18.1 In the first subsection of the glossary, identify units of weight and measure used in the method and their abbreviations.

4.18.2 In the second subsection, define key terms and all acronyms used in the method.

4.18.2.1 List terms, acronyms, and abbreviations alphabetically.

4.18.2.2 Definitions should appear only once. Where an acronym or abbreviation

represents a term that is defined under its full name, reference the full name as the definition for acronym or abbreviation.

### Appendix G to Part 136—Method Flexibility, Equivalency and Approval

Section 1 of this appendix defines the analyst's flexibility to modify certain steps in a reference method. Section 2 specifies requirements for assessing the equivalency of a method modification. Section 3 specifies requirements for submitting method modifications or new methods for approval.

#### 1.0 Method Flexibility

This section specifies requirements for exercising method flexibility (i.e., "allowable" method modifications). Under requirements specified at 40 CFR 136.4 and 141.27(b), an analyst is allowed to modify a reference method without seeking formal approval through the regulatory process provided the modification is not explicitly forbidden in the reference method and provided the analyst demonstrates and documents that the modified method produces results that are equal or superior to results produced by the reference method. An EPA-designated reference method that contains (or is supplemented with) QC acceptance criteria against which to measure performance of a method modification is the primary control used to ensure data quality. Other controls include specific multi-laboratory and multi-matrix requirements for validating modified methods (as specified at 40 CFR 136.4, 136.5(d), 141.27 (b) and (e)) checklists for documenting equivalency (as specified at Appendix E of this part).

The QC elements and associated QC acceptance criteria (e.g., calibration, sensitivity, accuracy, precision) necessary to demonstrate the equivalency of a modified method to a reference method are defined at 40 CFR 136.2 and 141.2 and specified at 40 CFR 136.3 Table IF, 136.4, 141.27 (b) and (d).

#### 1.1 Types of Method Modifications

There are two types of method modifications:

1.1.1 Explicit modifications to approved methods may be made as explicitly specified within those methods. Explicit flexibility exists for all approved methods including EPA, Standard Methods, ASTM, AOAC-International, and other methods approved at 40 CFR parts 136 and 141.

1.1.2 Allowable modifications beyond those explicitly allowed in an approved method that has been designated as a reference method are allowed provided that the modification meets the requirements specified in this appendix, at 40 CFR 136.4, 136.5(d) or 141.27(b) and (e), and at Appendixes E, F, and G of this part. Allowable modifications do not apply to Standard Methods, ASTM, and AOAC-International methods, none of which have been designated as reference methods.

#### 1.2 Controls on Allowable Modifications (Method Flexibility)

The controls on method flexibility are:

1.2.1 A requirement to demonstrate and document equivalency when method modifications are used.

1.2.2 Designation of a reference method that contains (or is supplemented with) QC

acceptance criteria for use in demonstrating equivalency.

1.2.3 Standard procedures for validating new methods and demonstrating equivalency of method modifications, based on the intended use of the method.

1.2.4 Detailed requirements for preparing the method validation package and supporting data when new or modified methods are validated.

1.2.5 Requirements for assessing equivalency of method modifications.

#### 1.3 Reference Method

All methods approved for use at 40 CFR parts 136 and 141 have been categorized as either a "reference method" or an "other approved method"; both types of methods carry equal regulatory status. The difference between the methods is that the reference method contains (or is supplemented with) detailed QC acceptance criteria that are required to assess the equivalency of an allowable method modification.

A reference method is specified at 40 CFR 136.3, 141.23(k), 141.24(e) and 141.40(n). For some determinative techniques, no currently approved method contains either all of the QC acceptance criteria proposed in today's rule (e.g., Table ID in 40 CFR part 136) or sufficient data from which to develop such criteria. In these cases, no reference method has been designated; therefore, all of these methods are classified as other approved methods. Without a reference method, analysts are not allowed to modify approved methods that use that determinative technique.

Only one reference method is designated for each unique combination of analyte and determinative technique to avoid the possible confusion if two or more reference methods contained different QC acceptance criteria. The QC acceptance criteria associated with the reference method are the sole criteria against which a method modification is tested.

#### 1.4 Categories of Allowable Method Modifications

The four categories of allowable method modifications are (1) sample collection and holding procedures, (2) front-end techniques, (3) determinative techniques, and (4) analyte addition. These categories are defined below and described in terms of allowed flexibility to modify the procedures or techniques included in each category.

The first category, sample collection and holding procedures, includes procedures and reagents used in the field, in transit, and at the laboratory. This category includes sample containers, sample holding times, preservation reagents and procedures, and shipping and storage procedures and conditions. Requirements for modifying sample collection and preservation conditions are specified at 40 CFR 136.3(c) and 141.27(b).

Front-end techniques, the second category of method modifications, include any step in the analytical process used at the laboratory that precedes the determinative technique and includes all procedures, equipment, solvents, etc., that are used to prepare a sample for analysis. The third category is the determinative technique, which is defined as

the physical and/or chemical process by which an analyte is identified and its concentration measured. For most methods, the determinative technique consists of an instrumental measurement (e.g., a detector). The fourth category covers increasing the analytical scope of a reference method to include additional analytes.

A person may modify any and all front-end techniques in the reference method, provided the modification is not explicitly prohibited in the reference method and provided the analyst demonstrates and documents that the modification produces results equal or superior to results produced by the reference method. The person must keep on file the documents that demonstrate equivalency. Method developers are cautioned that modifications to the front-end chemistry of the method (e.g., extraction solvents, solvent-to-sample volumes, extraction media, and pH) require a thorough understanding of the measurement science that was used to develop and validate the reference method. The developer of a modified method may ask EPA or another regulatory authority for a technical opinion on the acceptability of the validation data that supports the method modification(s).

A reference method may be modified to allow use of an alternate determinative technique that is not explicitly prohibited in the reference method, provided that equivalency with the reference method performance is demonstrated and documented, and provided that four conditions are met: (1) the alternate determinative technique must measure a property similar to the prescribed technique, (2) the alternate technique must be demonstrated to be more specific (i.e., provides better separation of the analyte from interferences) and/or more sensitive (i.e., produces a lower detection limit) for the analyte of concern than the determinative technique in the reference method, (3) there is not another approved method that uses the alternate determinative technique for the determination of that analyte, and (4) use of the alternate determinative technique would not result in a nonsensical combination of analyte, front-end technique, and determinative technique.

Examples of allowed changes to a determinative technique are substitution of a photoionization detector for a flame ionization detector for determination of polynuclear aromatic hydrocarbons, substitution of a nitrogen-phosphorous detector for an electron capture detector (ECD) for determination of analytes containing nitrogen or phosphorous, and substitution of a fluorescence detector for an ultraviolet or visible wavelength detector. Substitution of a mass spectrometer (MS) for an ECD would not be allowed if there is an approved MS method that measures the analyte of concern.

Modifications to the determinative technique are limited by the four conditions described above to preclude nonsensical combinations of analyte and determinative technique, to encourage a net benefit (increased sensitivity and/or specificity), and to preclude multiple reference methods with the same determinative technique but with

different QC acceptance criteria for the same analyte(s) of concern. For example, if a mass spectrometer were substituted for the conventional detectors in EPA Methods 601–612, all of these methods would become GC/MS methods, but all would contain different QC acceptance criteria. Further, they would all conflict with approved EPA GC/MS Methods 625 and 1625. Another reason for limiting changes to the determinative technique is that there are techniques, such as immunoassay, for which EPA has no reference method and therefore no history to ensure that the standardized QC proposed in today's rule would be germane to, or adequate for, assurance of the quality of data produced by the novel determinative technique. A new method must be written and submitted to EPA for approval when a novel determinative technique is developed.

An analyst may add a new target analyte to a reference method provided (1) it is demonstrated that the analyte does not interfere with determination of the analytes of concern in that method, (2) QC acceptance criteria are developed and employed for determination of the target analyte, (3) there is not another approved method that uses the same determinative technique for that analyte, and (4) that the reason for adding the analyte is not to avoid the sample preservation or sample (or extract) holding time conditions that are already required for that analyte in another approved method. The third and fourth conditions preclude "method shopping", whereby an analyst might add analytes to a reference method with less rigid QC acceptance, sample collection, or holding time criteria. Thus, if a reference method for an analyte of concern required acidification of the sample, an analyst does not have the flexibility to modify a method that does not require sample acidification to include analysis of the analyte of concern. Modifications of this type require EPA approval as a new method.

If QC acceptance criteria do not exist to allow addition of a new analyte, the requirements specified at 40 CFR part 136 Appendix E, and at 40 CFR 136.4, 136.5, and 141.27 must be used to develop and obtain approval for these criteria. Alternatively, QC acceptance criteria for the new analyte could be transferred from the criteria for an analyte with similar chemical characteristics in the same method or from the criteria for the analyte in another approved method.

#### 1.5 Method-Defined Analytes

Some techniques may not produce results equivalent to results produced by techniques employed for "method-defined analytes". A method-defined analyte is an analyte that does not have a specific, known composition so that the analytical result depends totally on how the measurement is made. Therefore, a change to either the front-end steps or the determinative technique for a method-defined analyte has the potential of changing the numerical value of the result for a given sample. Examples of method-defined analytes include adsorbable organic halides (AOX), biochemical oxygen demand (BOD), total radioactivity, and whole effluent toxicity (WET).

Until the nature and extent of allowable flexibility for method-defined analytes is

defined by EPA, these methods may not be modified using the requirements specified in this section unless the modified method is reviewed and approved by EPA. A person may attempt to demonstrate that the new technique produces results that are equivalent to the reference method on a matrix-by-matrix basis. When these data are submitted to EPA, EPA will work with the method developer to determine whether the submitted combination of analyte and determinative technique is new and whether a new method for a method-defined analyte is desirable.

**1.6 New Methods, Screening Methods, and Modified Methods** This section clarifies the differences between new and modified methods and the requirements that pertain to each. This section also describes how screening methods might be approved in the future for compliance monitoring under the CWA and the SDWA.

A new method is a set of procedures that:

- (1) Is documented in accordance with the requirements detailed as specified at Appendix F of this part,
- (2) Contains the standardized QC elements defined at 40 CFR 136.2 and 141.2,
- (3) Contains QC acceptance criteria that have been developed in accordance with the requirements at 40 CFR 136.5 and 141.27(c),
- (4) Employs a determinative technique for an analyte of concern that differs from determinative techniques employed for that analyte in methods previously approved at 40 CFR part 136 or 141, and
- (5) Employs a determinative technique that is more sensitive and/or selective (specific) than the determinative techniques in all methods previously approved for the analyte.

Methods that meet all five of these characteristics are considered to be definitive methods, if the method also is sufficiently selective and quantitative that most positive results do not have to be verified by analysis with another method. The term "definitive" is used to distinguish these methods from screening methods. All methods currently approved at 40 CFR parts 136 and 141 are definitive methods.

In this appendix, a screening method is defined as a method that meets the first four of the five conditions described above for new methods and that has been demonstrated to produce a false negative probability of no more than one percent (1%) at the limit(s) of regulatory concern. Methods can fail the fifth condition for a new method, if they are non-selective or not quantitative for the target analyte. A non-selective method is a method in which the determinative (or other step) technique in the method may produce a result for any one of several analytes that share common physical or chemical characteristics with the target analyte. For example, an atrazine immunoassay might respond to any triazine (atrazine, simazine, cyanazine) pesticide in the sample.

In the future, screening methods may be considered for approval as compliance monitoring methods provided: (1) the method meets all the requirements described in the regulations at 40 CFR 136.5 and 141.27(c), (2) all positive sample results obtained with the method are confirmed and

reported using an approved definitive method, and (3) the probability of the method producing a false negative result at concentrations of regulatory interest is no more than one percent (1%). For part 141 approval, these criteria may be amended when the Agency implements the requirements for screening methods that are in the August 2, 1996 amendments to the SDWA.

## 2.0 Assessing Method Equivalency

This section provides requirements for assessing the equivalency of a method that has been modified according to the requirements specified at 40 CFR 136.4, 136.5(d), 141.27 (b) and (e). Analysts and regulatory authorities may use these equivalency requirements to verify and document that equivalent or better method performance relative to the reference method has been achieved and documented by the laboratory using the method modification. This section also specifies requirements for documenting the performance of new methods and method modifications.

Good communication among analytical laboratories, regulated entities, and regulatory authorities is essential for the method modification assessment process. Although many compliance monitoring analyses are performed by contract laboratories on behalf of the regulated entity, the responsibility for maintaining validation documentation for new and modified methods rests with the regulated entity. Regulated entities, therefore, must inform their contract laboratories about the requirements for detailed documentation of method modifications.

### 2.1 Requirements for Documenting Validation of New and Modified Methods

Although validation requirements vary depending on the intended use of the new or modified method, the documentation requirements are the same. A validation study report must be prepared for every study conducted to validate new or modified methods. The primary basis for documenting method validation studies are the Checklist for Initial Demonstration of Method Performance, the Checklist for Continuing Demonstration of Method Performance, and the Certification Statement (collectively called the "Checklists"). The Checklists must be used by auditors, drinking water laboratory certification officers, and other reviewers to evaluate new methods and method modifications against reference methods promulgated at 40 CFR parts 136 and 141.

The Checklists and instructions for their completion are provided at Appendix E in this part. Regulated entities must make the Checklists available to the contract laboratories to document method modifications. In turn, contract laboratories are responsible for returning validation study reports including completed Checklists to the regulated entities.

The data reviewer should verify that all validation and documentation requirements appropriate to the intended tier of the new or modified have been met as specified at 40 CFR 136.4, 136.5(d), 141.27 (b) and (e). For Tier 1 method modifications, the completed

Checklists are adequate to document method equivalency. For all other validation tiers, the data reviewer must ensure that the validation study report is complete and includes all supporting raw data. The following sections must be included in the report:

2.1.1 A background section that describes the method and the responsible organization.

2.1.2 A section that describes the study design and objectives.

2.1.3 A section that describes the study methodology and implementation

2.1.4 A section that describes the procedures that were used to report and validate data.

2.1.5 A results section. (Note: Since different instruments provide different data, the specific form of the supporting analytical data will differ according to the method. For example, gas chromatography/mass spectrometry procedures produce chromatograms, while colorimetric determinations do not.)

2.1.6 A section for a discussion of the study results.

2.1.7 A section that describes conclusions from the study.

2.1.8 An appendix that contains the Checklists.

### 2.2 Data Review Guidance for EPA Water Methods

This section provides guidance for reviewing data submitted to EPA and state authorities under CWA and SDWA. The guidance provides a tool for authorities who want to perform detailed inspection of data analyzed by methods under 40 CFR parts 136 and 141. The material presented in this section is technically detailed and is intended for data reviewers familiar with analytical methods.

#### 2.2.1 Standardized Quality Assurance/Quality Control

Standardized QA/QC is specified for each reference method and contains the following elements:

2.2.1.1 Calibration linearity.

2.2.1.2 Calibration verification.

2.2.1.3 Absolute and relative retention time precision (for chromatographic analyses).

2.2.1.4 Initial precision and recovery or "start-up" tests.

2.2.1.5 Ongoing precision and recovery.

2.2.1.6 Analysis of blanks.

2.2.1.7 Surrogate or labeled compound recovery.

2.2.1.8 Matrix spike and matrix spike duplicate precision and recovery (for non-isotope dilution analyses).

2.2.1.9 Demonstration of method detection limits.

2.2.1.10 Analysis of reference sample. When reviewing method validation data, the permit writer, PWS, or other individual or organization has the authority and responsibility to ensure that the test data submitted contain the elements listed above; otherwise, the data can be considered noncompliant.

#### 2.2.2 Details of Data Review

The details of the data review process depend to a great extent upon the specific analytical method. Even for data from the same method, there may be many approaches

to data review. However, given the standardized QC requirements of the streamlined methods approval program, a number of basic concepts apply. The following sections provide the details for reviewing data submitted and a rationale for the QC tests. Results from all QC tests must be within the QC acceptance criteria specified in, or associated with, the reference method to validate that results produced by a method modification are equivalent or superior to results produced by the reference method.

##### 2.2.2.1 Calibration linearity

The relationship between the response of an analytical instrument to the concentration or amount of an analyte introduced into the instrument typically is represented by an averaged response or calibration factor, a calibration line, or a calibration curve. An analytical instrument can be said to be calibrated in any instance in which an instrumental response can be related to a single concentration of an analyte. The response factor or calibration factor is the ratio of the response of the instrument to the concentration (or amount) of analyte introduced into the instrument.

Nearly all analytical methods focus on the range over which the response is a linear function of the concentration of the analyte. This range usually extends from the minimum level of quantitation (ML) on the low end to the point at which the calibration becomes non-linear on the high end. For regulatory compliance, it is important that the concentration of regulatory interest (e.g., permit limit; MCL) fall within this range. Calibration can also be modeled by quadratic or higher order mathematical functions. The advantage of a calibration line that passes through the origin is that an averaged response factor or calibration factor can be used to represent the slope of this line. Use of a single factor simplifies calculations and the interpretation of the data. Also, it is easier to discern when an inaccurate calibration standard has been prepared if the calibration function is a straight line.

Many analytical methods, particularly recent methods, specify some criterion for determining the linearity of the calibration. When this criterion is met, the calibration function is sufficiently close to a straight line that passes through the origin to permit the laboratory to use an averaged response factor or calibration factor. Linearity is determined by calculating the relative standard deviation (RSD) of the response factor or calibration factor for each analyte and comparing this RSD to the limit specified in the method. If the RSD does not exceed the specification, linearity through the origin is assumed. If the specification is not met, a calibration curve must be used.

For whatever calibration range is used, a reference method should contain a specification for the RSD of the response or calibration factor to establish the breakpoint between linear calibration through the origin and a line not through the origin or a calibration curve. For new methods, the method developer must provide the RSD results by which one can judge linearity, even in instances where the laboratory is using a calibration curve. In instances where

the laboratory employs a curve rather than an average response or calibration factor, the data reviewer should review each calibration point to ensure that the response increases as the concentration increases. If it does not, the instrument is not operating properly, or the calibration curve is out of the range of that instrument, and data are not considered valid.

#### 2.2.2.2 Calibration Verification

Calibration verification involves the analysis of a single standard at the beginning of each analytical shift or after the analysis of a fixed number of samples (e.g., 10). The concentration of each analyte in this standard is normally at the same level as in one of the calibration standards, typically at 1–5 times the ML. The concentration of each analyte in this standard is calculated using the calibration data. The calculated concentration is compared to the concentration of the standard. Calibration is verified when the concentration is within the calibration verification limits specified in the method. If the results are within the specifications, the laboratory is allowed to proceed with analysis without recalibrating and allowed to use the calibration data to quantify the sample concentration or amount of each analyte in samples, blanks, and QC tests.

If calibration cannot be verified, the laboratory may either recalibrate the instrument or prepare a fresh calibration standard and make a second attempt to verify calibration. If calibration cannot be verified with a fresh calibration standard, the instrument must be recalibrated. If calibration is not verified, subsequent data are considered to be invalid until the instrument is recalibrated.

#### 2.2.2.3 Absolute and Relative Retention Time Precision

Retention time specification aid in the identification of analytes in chromatographic analyses. In some methods, a minimum retention time is specified to ensure adequate separation of analytes in complex mixtures. If retention time QC criteria cannot be verified, chromatographic identification of analytes is suspect and reanalysis is necessary.

#### 2.2.2.4 Initial Precision and Recovery

The laboratory must demonstrate that it can meet the IPR QC acceptance criteria in the method. This test is required prior to the use of the method by a laboratory. It is sometimes termed the "start-up test." Difficulty in passing the start-up test frequently leads to marginal performance by the laboratory in the routine operation of the method. Performing the start-up test "after the fact" or after samples have been analyzed is not acceptable.

The start-up test consists of spiking the analytes of interest into a set of four or more aliquots of a reference matrix and analyzing these four aliquots. The reference matrix simulates the medium being tested. A separate IPR test must be performed for each medium. The mean concentration and the standard deviation of the concentration are calculated for each analyte and compared to QC acceptance criteria in the method. If the

mean and standard deviation are within the limits specified, the analysis system is in control and the laboratory can use the system for analysis of blanks, field samples, and other QC tests samples. For some methods (e.g., EPA Methods 625 and 1625), a repeat test is allowed because of the large number of analytes being tested simultaneously.

If there are no start-up test data, or if these data fail to meet the QC acceptance criteria in the method, all data produced by that laboratory using that method are not considered valid. It is important to remember that if a change is made to a method, the start-up test must be repeated with the change as an integral part of the method. Such changes may involve alternative extraction, concentration, or cleanup processes; alternative GC columns, GC conditions, or detectors; or other procedures designed to address a particular matrix problem. If the start-up test is not repeated when a procedure is changed, added, or deleted, data produced by the modified method are considered invalid.

#### 2.2.2.5 Ongoing Precision and Recovery

An ongoing precision and recovery (OPR) standard (also termed a "laboratory control sample" (LCS) or a "laboratory fortified blank" (LFB)) must be analyzed with each sample batch prior to the analysis of a blank, sample, or matrix spike or duplicate. The number of samples in the batch is usually 10 or 20, depending on the method, or the OPR is required at the beginning of an analysis shift, regardless of the number of samples analyzed during that shift. The data reviewer must determine if the OPR standard has been run with each sample batch or at the beginning of the shift and if all criteria have been met. If the standard was not run with a given set of samples, or if the criteria are not met, the results for that set of samples are considered invalid.

#### 2.2.2.6 Analysis of blanks

Blanks must be analyzed either on a periodic basis or with each sample batch, depending on the method. Blanks may contain contamination at levels no higher than specified in the method. Samples associated with a contaminated blank must be reanalyzed.

#### 2.2.2.7 Surrogate or Labeled Compound Recovery

Surrogate or labeled compounds are used to assess the performance of the method on each sample. Recoveries of these compounds from each sample must be within QC acceptance criteria to demonstrate acceptable method performance on the sample. If the recovery is not within the criteria, the sample is normally diluted and the dilute sample analyzed to demonstrate that a matrix effect precluded reliable analysis of the undiluted sample.

#### 2.2.2.8 Matrix Spike and Matrix Spike Duplicate

Non-isotope dilution methods require a spike of the analytes of interest into a separate aliquot of the sample for analysis with the sample. The purpose of the matrix spike (sometimes termed a "laboratory fortified sample matrix" (LFM)) is to determine if the method is applicable to the

sample in question. While many of the approved methods were tested using effluents from a wide variety of industries, samples from some sources may not yield acceptable results. It is, therefore, important to evaluate method performance in the sample matrix of interest. If the recovery for the MS/MSD is not within the QC acceptance criteria, a matrix interference may be the cause. The sample is usually diluted and the diluted sample spiked and analyzed. If the QC acceptance criteria are met with the diluted MS/MSD, a matrix problem exists. Cleanup and other processing of the sample are then required to overcome the matrix interference if analysis of the undiluted sample is required to establish compliance.

#### 2.2.2.9 Demonstration of Method Detection Limits

A laboratory that wishes to use a new or modified wastewater method must demonstrate that the method detection limit (MDL) specified in the reference method can be achieved. Alternatively, if the regulatory wastewater compliance limit is above the MDL, laboratories must demonstrate that the minimum level (ML) determined with the new or modified method is at or below  $\frac{1}{3}$  the compliance limit. A laboratory that wishes to use a new or modified drinking water method must demonstrate that the MDL determined with that method meets the detection limits specified at 40 CFR parts 141.23, 141.24 and 141.89 and/or as specified at 40 CFR 141.27(d). For both drinking water and wastewater determinations, demonstration of a valid detection limit requires use of an MDL study in accordance with the procedure at 40 CFR part 136 Appendix B. If the MDL determined with the new or modified method is not acceptable, the method may not be used because the laboratory has not demonstrated an ability to detect the analyte at the level required.

**Note:** Required detection limits specified in regulations and/or in the reference method(s) are usually analyte-specific; for the same analyte, the requirement may differ between the wastewater and the drinking water reference method.

#### 2.2.2.10 Reference Sample Analysis

Provided such acceptance limits are specified by EPA or other regulatory authorities, a laboratory must be able to demonstrate the ability to quantitate the analyte in a reference material to within the acceptance range specified for the reference material. Currently, EPA specifies at 40 CFR 141.23, 141.24 and 141.89 acceptance limits for analysis of performance evaluation (PE) samples that are provided by EPA under the drinking water studies (WS) PE-sample program.

### 3.0 Method Approval Process

Use of the procedures specified in this section will expedite the approval of drinking and wastewater methods by ensuring that methods submitted to EPA for approval contain the appropriate elements, have been validated, and contain all supporting documentation. This section details procedures for preparing and submitting method documentation, and describes the rulemaking process required to



approve a new method or method modification. All new wastewater and drinking water methods are subject to EPA review. New methods recommended for approval will be subject to one of two actions: an approval letter or an Agency rulemaking. Tier 1 new methods will receive a letter of approval from EPA/EAD. Tier 2 and 3 new methods will be approved in a formal rulemaking. Rulemaking involves publishing in the **Federal Register** a proposed rule containing the method(s) for public comment, responding to public comment, and approving the method(s) in a final rule. The approved method(s) will be cited in the applicable parts of the CFR. The text of the approved method(s) will be incorporated by reference rather than published in the CFR. The method submitter will be responsible for developing, writing, and validating the method; providing information in a format suitable for a proposed rule; providing the necessary supporting documentation; and assisting EPA in responding to public comments to support approval. EPA will review the method and supporting documents, draft the regulatory language, and submit the proposed rule to the Office of the Federal Register (OFR) for publication in the **Federal Register**. New methods must undergo the processes detailed above; no other types of action will be substituted.

Method modifications can be used directly after the method validation study confirms method equivalency. EPA, only upon request, will review Tier 2 and Tier 3 method modifications. The option to request EPA review of a modified method is provided to allow interested parties to substantiate EPA approval of a method modification. Any party associated with method modification and/or development can request review, including: permittees, publicly owned treatment works (POTWs), public water systems (PWSs), commercial laboratories, vendors, or States. Upon determination that a method modification is appropriate, EPA either will issue a letter of approval or conduct a rulemaking, whichever action is requested by the method submitter. The text of the approved method(s) will be incorporated by reference rather than published in the CFR. The method submitter will be responsible for developing, writing, and validating the method; providing information in a format suitable for a proposed rule; providing the necessary supporting documentation; and assisting EPA in responding to public comments to support approval. EPA's role will be to review the method and documentation; to write the rule language; and to submit the rule to the OFR, if appropriate.

### 3.1 Pre-Submission Procedures

EPA must review all new methods, and will review Tier 2 and Tier 3 method modifications upon request. Prior to EPA review, a party developing a new or modified method will proceed through up to four steps: (1) method development, (2) method validation, (3) information in a format suitable for proposal in the **Federal Register** (if appropriate), and (4) submission to EPA.

#### 3.1.1 Method Development

Any person can develop a new method or method modification if they identify a new or improved procedure or technique for analyzing an analyte of interest. A new method must be a unique combination of analyte and determinative technique, as discussed in Section 1. Otherwise, it would qualify as a modification of an existing method. The method development process will typically include drafting, checking, and modifying testing procedures. Once the person has confidence in the new or modified testing procedures, the procedures should be finalized into a standardized format. The method description should identify the anticipated application of the new procedures: single laboratory; multi-laboratory, single matrix type; or multi-laboratory, multiple matrix types.

The requirement to provide the method in standard format is needed to preclude confusion. Specific details on the standard format for the new or modified method can be found at Appendix F of this part. Appendix F specifies the analytical methods format developed by EPA's Environmental Monitoring Management Council (EMMC). The EMMC format is directed at standardizing all Agency analytical methods.

For new methods submitted for approval at 40 CFR part 136 or part 141, a format from another organization may be used provided that it is standardized and contains the same elements specified in the Method Guidelines and Format. For example, the method format documents from Standard Methods, ASTM, AOAC-International, or USGS are acceptable because these formats are documented and routinely followed by these organizations. However, method submitters other than these organizations must use the EPA format specified at Appendix F of this part. Reserving method formats for those specific organizations avoids misleading the analytical community concerning the authorship of the method. EPA will review and approve standardized formats from governmental authorities and industrial associations upon request, but will not approve miscellaneous formats written by instrument manufacturers, individual laboratories, and others because of the potential proliferation of different method formats. The format provided in Appendix F of this part meets the needs of a format for new methods.

#### 3.1.2 Method Validation

Each new method or method modification must be tested to assess its performance. The process of establishing or substantiating method performance is called validation. To approve a new method or method modification, EPA must be provided with a report describing and including results of the validation study. When undertaking method validation, the method submitter is responsible for performing the validation study at the appropriate tier as specified at §§ 136.4, 136.5 and 141.27. The study will be detailed in a method validation report submitted to EPA that includes the required Checklist and Certification Statement as specified at Appendix E of this part.

For new methods, QC acceptance criteria must be included in the method and the

details of development of these criteria must be included in the validation report. QC acceptance criteria are used to ensure that a method produces results that are reliable, defensible, and suitable for regulatory decisions. QC acceptance criteria must be developed from data gathered in a method validation study. When an analyte is being added to an approved method, however, QC acceptance criteria may be (1) developed from validation data, (2) transferred from another analyte already included in the approved method, (3) transferred from another analyte in another approved method, or (4) transferred from another approved method for the same analyte. For a transfer from another analyte to be appropriate, the chemical characteristics of the analyte from which the criteria are transferred should simulate, as closely as possible, the chemical characteristics of the newly regulated analyte. For example, if 2,4-dimethyl-3-chlorophenol is added to Method 625, and data from a method validation study are not available from which QC acceptance criteria can be derived, QC acceptance criteria can be transferred from 2,4-dimethylphenol or 4-chloro-3-methylphenol in Method 625. For newly regulated analytes added to an existing method, it is highly likely that EPA would require that the QC acceptance criteria be developed from validation data rather than transferring criteria from another analyte to ensure proper validation.

#### 3.1.3 Draft **Federal Register** Preamble

When Tier 2 and Tier 3 methods are to undergo the rulemaking process (e.g., for all new methods and modified methods requests), the submitter must provide information in a format suitable for proposal of the method at 40 CFR parts 136 or 141. This information should describe the basis and purpose for the proposed rule and should be written to communicate the import of the rule to the general public. The OFR requires a specific format for the preamble. Examples of appropriate and pertinent preambles include 49 FR 43234, October 26, 1984; 56 FR 5090, February 7, 1991; 60 FR 53988, October 18, 1995; and 61 FR 1730, January 23, 1996.

#### 3.1.4 Submission to EPA

When all the pre-submission steps are completed, the method submitter should generate a single packet for submission to EPA. This packet will include the method in a standard format, the method validation report, the draft preamble (if rulemaking will occur), and any necessary supporting documents. If this streamlining proposal is promulgated, the submission packet will be submitted to the Director of the Analytical Methods Staff in EPA's Office of Water.

### 3.2 EPA Review

EPA must review all new methods, and will review Tier 2 and Tier 3 method modifications if requested. When a method package is submitted for review, EPA will first check the documentation for completeness. The documentation must include the final method in standard format, the validation report, and information that would facilitate EPA's drafting of a proposed rule (if rulemaking will occur). If all of the



documentation is in order, EPA will begin an internal review of the method for scientific merit, consistency, and appropriateness. The internal review may involve multiple programs and workgroups. Should any problems or questions arise, EPA will

communicate with the submitter to resolve the outstanding issues. Depending on the circumstances, EPA may return the submission to the submitter for revision.

If internal review recommends acceptance, EPA will issue a letter of acceptance for a

Tier 1 new method. For Tier 2 and Tier 3 new methods, EPA will begin the rulemaking process. For Tier 2 and Tier 3 method modifications, the method submitter has the option of receiving a letter of approval or proceeding with the rulemaking process.

TABLE 1—1.—EPA REVIEW AND APPROVAL OF METHODS

	New method	Modified method
Tier 1: Single-lab .....	<ul style="list-style-type: none"> <li>• EPA review required .....</li> <li>• EPA issues a letter of approval .....</li> </ul>	No EPA review.
Tier 2: Multi-lab, single matrix type .....	<ul style="list-style-type: none"> <li>• EPA review required .....</li> <li>• Approved through rulemaking .....</li> </ul>	<ul style="list-style-type: none"> <li>• If requested, EPA reviews and issues letter of approval, or conducts rulemaking.</li> </ul>
Tier 3: Multi-lab, multiple matrix type .....	<ul style="list-style-type: none"> <li>• EPA review required .....</li> <li>• Approved through rulemaking .....</li> </ul>	<ul style="list-style-type: none"> <li>• If requested, EPA reviews and issues letter of approval, or conducts rulemaking.</li> </ul>

### 3.3 Limited Use Methods

Currently, EPA reviews single-laboratory, limited-use methods only for special applications. Examples of special circumstances could include procedures to remove sulfate interferences in drinking water matrices and, as described below, technologies that can eliminate total cyanide false positives in some wastewater measurements.

Use of limited-use methods as Tier 1 methods for both wastewater and drinking water methods is allowed. The purpose of this allowance is to provide the means by which (1) a new technology can be introduced and (2) specific matrix interference problems can be overcome. Furthermore, additional single laboratories can use the technology until a sufficient number of devices are available for interlaboratory validation.

Tier 1 new methods must be submitted to EPA for review. Upon recommendation for approval, a letter of approval will be issued. Tier 1 modified methods can be used directly upon validation. EPA will not review Tier 1 method modifications.

### 3.4 Rulemaking Process

The customary rulemaking process consists of four phases: proposal of the rule, public comment, response to comments, and publication of the final rule. The proposed rule requests public comment and allows a specified comment period, for example, 30 to 90 days depending on the magnitude of the proposed change. At the end of the comment period, EPA will forward any significant comments to the method submitter. The submitter would then provide technical assistance to EPA in drafting responses to comments. All comments that have scientific or legal merit, or raise substantive issues with the proposed rule, must be answered to complete the rule-making process.

EPA will review the comment responses and complete a response-to-comments document that must be included in the final rule. EPA will prepare and submit the final rule to the OFR for publication. The final rule will state the date that the rule becomes effective; as of this date, the method is approved.

### 3.5 Proprietary Components

Proprietary components can be classified into three categories: proprietary reagents,

proprietary instruments, and proprietary methods. Proprietary reagents and instruments are allowed in the approval of analytical methods for compliance purposes to the extent that such inclusion still provides an adequate opportunity for public review and comment under the Administrative Procedure Act. Use of proprietary methods for determining compliance with regulatory requirements where the entire method is claimed as "confidential business information" (CBI) is not allowed. However, if the proprietary method is patented it could be considered for approval because the public would have the opportunity to comment on the patented method.

Proprietary reagents and instruments are allowed in approved methods. The details of the proprietary elements must be disclosed to EPA, but will be withheld from the public if the person requesting protection for the CBI demonstrates that the information is entitled to confidential treatment under the applicable regulations. Examples of these proprietary components are immunoassay reagents and antibodies, and liquid phases in GC columns, e.g., DB-1®, SPB-octyl, Dextsil®, etc. A new or modified method submitted for EPA approval must include language stating that the proprietary reagent or instrument can be replaced by an equivalent. Changes made to the method after EPA approval would require the manufacturer to demonstrate through supporting documentation that the new proprietary equipment, substance, or reagent would produce results equal or superior to results produced with the material originally tested and on which the method approval is based. For proprietary reagents, a method must contain accurate, specific instructions for the safe handling of each proprietary reagent listed in the method, and for safe disposal of each spent proprietary reagent and/or reagent product. When a material safety data sheet (MSDS) accompanies the proprietary material, the MSDS will serve as these instructions, and the submission of an MSDS with the method shall be evidence that the requirements for instructions for safe handling and disposal of the reagent have been met.

## 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, and 300j-9.

2. Section 141.2 is proposed to be amended by adding the following definitions in alphabetical order to read as follows:

### § 141.2 Definitions.

As used in this part, the term:

**Accuracy** means the degree of agreement between an observed value and an accepted reference value. Accuracy includes random error (precision) and systematic error (bias) that are caused by sampling and analysis.

\* \* \* \* \*

**Administrator** means the Administrator of the U.S. Environmental Protection Agency (EPA).

**Analyte or Analyte of concern** means a substance or property that is to be measured by an analysis.

**Approved method** means a testing procedure (analytical method) promulgated at this part or at 40 CFR part 142 or 143.

**Assistant Administrator (AA)** means the EPA Assistant Administrator for Water.

\* \* \* \* \*

**Calibration (CAL)** means the process of establishing the relationship between the concentration or amount of material introduced into an instrument or measurement process and the output signal.

**Calibration linearity** means the degree to which calibration points lie along a straight line.

**Calibration verification** means the means of establishing that instrument

performance remains within pre-established limits.

\* \* \* \* \*

*Determinative technique* means the process (physical or chemical or both) to measure the identity and concentration of an analyte. In test methods, the determinative technique follows the front-end techniques.

\* \* \* \* \*

*Front-end technique* means any technique in the analytical process that precedes the determinative technique, including all procedures, equipment, solvents, etc. that are used in the laboratory in the preparation and cleanup of a sample but this excludes conditions and/or procedures for the collection, preservation, shipment and storage of the sample.

\* \* \* \* \*

*Initial precision and recovery test (IPR)* means analysis of a minimum of four spiked reagent water samples under the same conditions as will be used for analysis of environmental samples. The IPR is used to demonstrate that a laboratory is able to produce reliable results with the method prior to analysis of environmental samples.

*Interference* means a positive or negative effect on a measurement caused by a substance other than the analyte being investigated.

\* \* \* \* \*

*Matrix* means the component or substrate that contains the target analyte.

*Matrix spike (MS)* means a sample prepared by adding a known mass of target analyte to a specified amount of a sample matrix for which an independent estimate of target analyte concentration is available.

*Matrix spike duplicate (MSD)* means a duplicate of the matrix spike. The MS/MSD are used in combination to test the precision of an analysis.

*Matrix type* is any potable water sample provided by a PWS.

\* \* \* \* \*

*Medium* means the physical phase of a sample matrix. Air, water, soil, sediment, rock, and sludge are sample media.

\* \* \* \* \*

*Method* means an orderly and systematic arrangement of procedures and techniques for performing an analysis.

*Method blank (or blank)* means a sample absent the analytes of interest and interferences that is processed through all steps of a method simultaneously with and under the same conditions as samples that may contain an analyte of interest.

*Method detection limit (MDL)* means the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero as determined by the procedure set forth in appendix B of this part.

*Method Guidelines and Format* means the procedures set forth in appendix F of part 136.

*Method modification* means a change to a reference method. The change may be to a front-end technique or to the determinative technique.

*Method validation* means a process by which a laboratory or vendor establishes the performance of a new method or substantiates the performance of a reference method modification.

*Minimum level (ML)* means the lowest level at which an entire analytical system gives a recognizable signal and acceptable calibration point for an analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and clean-up procedures have been employed.

\* \* \* \* \*

*New method* means a combination of analyte of concern and determinative technique that is different from those in the approved methods.

\* \* \* \* \*

*Ongoing precision and recovery sample (OPR)* means a spiked reference matrix sample that is processed through all steps of a method simultaneously with and under the same conditions as samples that may contain an analyte of interest. Also called a laboratory control sample (LCS), the OPR/LCS is used to demonstrate that a laboratory is able to produce reliable results continuously.

\* \* \* \* \*

*Organic Methods* means the document titled: Methods for the Determination of Organic Compounds in Drinking Water—Supplement III (available from the National Technical Information Service (NTIS), U.S. Department of Commerce, Springfield, Virginia, 22161, 703/487-4600, at NTIS publication PB97-125298).

*Other approved method* means a promulgated method that is not designated as a reference method.

*Percent recovery* means the recovery multiplied by one hundred.

\* \* \* \* \*

*Precision* means the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation,

variance, or range, in either absolute or relative terms.

*Preparation* means processing performed on a sample prior to analysis, including extraction, concentration, and cleanup.

*Procedure* means a set of systematic instructions for performing an activity.

*Promulgated method* means a method that has been published or incorporated by reference into 40 CFR parts 141, 142, or 143.

\* \* \* \* \*

*Quality assurance (QA)* means an integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

*Quality control (QC)* means the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of a user. The aim is to provide quality that is satisfactory, adequate, dependable, and economical.

*QC acceptance criteria* means performance specifications developed from validation data and used to control the limits within which an analytical method is operated.

*Range* means the amounts or concentrations over which an instrument or analytical system is calibrated.

*Recovery* means the total amount of analyte found divided by the amount of analyte added as a spike.

*Reference method* means an approved method that is designated as a standard to which a modified method can be compared. A reference method will include standardized QC and QC acceptance criteria as well as sample preparation, cleanup, and other procedures.

*Regional Administrator* means an EPA Regional Administrator.

\* \* \* \* \*

*Sample* means a portion of a larger whole or a single item of a group; a finite part or subset of a statistical population; the medium subjected to analysis. A sample serves to provide data or information concerning the properties of the whole or population.

*Sample matrix effect validation* means to verify that the performance of a modified or new analytical method on samples obtained from different PWSs does not differ from the results validated in reagent water samples.

\* \* \* \* \*

*Screening method* means a method that employs a qualitative determinative technique for an analyte of interest that

is different from the determinative techniques used in all approved methods for that analyte. The screening method must produce a false negative probability less than 1%.

\* \* \* \* \*

*Selectivity* means the capability of a method or instrument to respond to an analyte in the presence of interferences.

*Sensitivity* means the capability of a method or instrument to differentiate between different amounts or concentrations of an analyte.

\* \* \* \* \*

*Spike* means the process of adding a known amount of an analyte to a sample to determine the recovery.

*Spike amount* means a known mass of analyte added to a sample and used to determine the recovery of a method.

*Standard deviation* means the measure of the dispersion of observed values expressed as the positive square root of the sum of the squares of the

difference between the individual values of a set and the arithmetic mean of the set, divided by one less than the number of values in the set.

\* \* \* \* \*

*Standardized quality control (standardized QC)* means a uniform set of performance testing procedures that ensure reliable results. Depending on the method, standardized QC procedures include, but are not limited to, the following: calibration, calibration linearity, calibration verification, absolute retention time, absolute and relative retention time precision, initial precision and recovery, ongoing precision and recovery (laboratory control sample), surrogate or labeled compound recovery, analysis of blanks, matrix spike and matrix spike duplicate recovery and precision, demonstration of method detection limit(s), and analysis of a reference sample.

\* \* \* \* \*

*Surrogate* means a substance with properties that mimic the behavior of an analyte, that is unlikely to be found in an environmental sample, and that is added to the sample for quality control purposes.

\* \* \* \* \*

*Tier 1* means the application of a new or modified method in a single laboratory to one or more PWSs.

*Tier 2* means the application of a new or modified method by all laboratories to all PWSs (nationwide use).

\* \* \* \* \*

3. Section 141.23, paragraph (k)(1), is proposed to be amended by revising the table to read as follows:

**§ 141.23 Inorganic chemical sampling and analytical requirements.**

\* \* \* \* \*

(k) \* \* \*

(l) \* \* \*

TABLE 141.23(k)(1)—LIST OF APPROVED INORGANIC TEST PROCEDURES

Contaminant	Methodology	Ref- erence method	Other approved methods			
			EPA	ASTM <sup>3,13</sup>	SM <sup>4,13</sup>	Other
Antimony	ICP-Mass Spectrometry	2 200.8				
	Hydride-Atomic Absorption			D-3697-92		
Arsenic	Atomic Absorption; Platform	2 200.9				
	Atomic Absorption; Furnace				3113B	
	Inductively Coupled Plasma	2 200.7			3120B	
	ICP-Mass Spectrometry	2 200.8				
	Atomic Absorption; Platform	2 200.9				
Asbestos	Atomic Absorption; Furnace			D-2972-93C	3113B	
	Hydride-Atomic Absorption			D-2972-93B	3114B	
	Transmission Electron Microscopy	10 100.2	9 100.1			
Barium	Inductively Coupled Plasma	2 200.7			3120B	
	ICP-Mass Spectrometry	2 200.8				
Beryllium	Atomic Absorption; Direct				3111D	
	Atomic Absorption; Furnace				3113B	
	Inductively Coupled Plasma	2 200.7			3120B	
	ICP-Mass Spectrometry	2 200.8				
Cadmium	Atomic Absorption; Platform	2 200.9				
	Atomic Absorption; Furnace			D-3645-93B	3113B	
	Inductively Coupled Plasma	2 200.7				
	ICP-Mass Spectrometry	2 200.8				
Chromium	Atomic Absorption; Platform	2 200.9				
	Atomic Absorption; Furnace				3113B	
	Inductively Coupled Plasma	2 200.7			3120B	
	ICP-Mass Spectrometry	2 200.8				
Cyanide	Atomic Absorption; Platform	2 200.9				
	Atomic Absorption; Furnace				3113B	
	Manual Distillation followed by				4500-CN-C	
	Spectrophotometric, Amenable			D-2036-91B	4500-CN-G	
Fluoride	Spectrophotometric, Manual			D2036-91A	4500-CN-E	5 I-3300-85
	Semi-automated	6 335.4				
	Selective Electrode				4500-CN-F	
	Ion Chromatography	6 300.0		D4327-91	4110B	
Mercury	Manual Distill.; Color. SPADNS				4500-F-B,D	
	Manual Electrode			D1179-93B	4500-F-C	
	Automated Electrode				4500-F-E	11 380-75WE
	Automated Alizarin					11 129-71W
Nickel	Manual, Cold Vapor	2 245.1		D3223-91	3112B	
	Automated, Cold Vapor	1 245.2				
Nickel	ICP-Mass Spectrometry	2 200.8				
	Inductively Coupled Plasma	2 200.7			3120B	
	ICP-Mass Spectrometry	2 200.8				
	Atomic Absorption; Platform	2 200.9				
	Atomic Absorption; Direct				3111B	

TABLE 141.23(k)(1)—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Contaminant	Methodology	Ref- erence method	Other approved methods			
			EPA	ASTM <sup>3,13</sup>	SM <sup>4,13</sup>	Other
Nitrate	Atomic Absorption; Furnace				3113B	
	Ion Chromatography	<sup>6</sup> 300.0		D4327-91	4110B	<sup>8</sup> B-1011
	Automated Cadmium Reduction	<sup>6</sup> 353.2		D3867-90A	4500-NO <sub>3</sub> -F	
	Ion Selective Electrode				4500-NO <sub>3</sub> -D	<sup>7</sup> 601
Nitrite	Manual Cadmium Reduction			D3867-90B	4500-NO <sub>3</sub> -E	
	Ion Chromatography	<sup>6</sup> 300.0		D4327-91	4110B	<sup>8</sup> B-1011
	Automated Cadmium Reduction	<sup>6</sup> 353.2		D3867-90A	4500-NO <sub>3</sub> -F	
	Manual Cadmium Reduction			D3867-90B	4500-NO <sub>3</sub> -E	
Selenium	Spectrophotometric				4500-NO <sub>2</sub> -B	
	Hydride-Atomic Absorption			D3859-93A	3114B	
	ICP-Mass Spectrometry	<sup>2</sup> 200.8				
	Atomic Absorption; Platform	<sup>2</sup> 200.9				
Thallium	Atomic Absorption; Furnace			3859-93B	3113B	
	ICP-Mass Spectrometry	<sup>2</sup> 200.8				
Lead	Atomic Absorption; Platform	<sup>2</sup> 200.9				
	Atomic Absorption; Furnace			D3559-90D	3113B	
Copper	ICP-Mass Spectrometry	<sup>2</sup> 200.8				
	Atomic Absorption; Platform	<sup>2</sup> 200.9				
	Atomic Absorption; Furnace			D1688-90C	3113B	
	Atomic Absorption; Direct Aspiration			D1688-90A	3111B	
pH	ICP	<sup>2</sup> 200.7			3120B	
	ICP-Mass Spectrometry	<sup>2</sup> 200.8				
	Atomic Absorption; Platform	<sup>2</sup> 200.9				
	Electrometric	<sup>1</sup> 150.2	<sup>1</sup> 150.1	D1293-84	4500-H <sup>+</sup> -B	
Conductivity	Conductance			D1125-91A	2510B	
Calcium	EDTA Titrimetric			D511-93A	3500-Ca-D	
	Atomic Absorption; Direct Aspiration			D511-93B	3111B	
Alkalinity	Inductively Coupled Plasma	<sup>2</sup> 200.7			3120B	
	Titrimetric			D1067-92B	2320B	
Orthophosphate <sup>12</sup>	Electrometric Titration					<sup>5</sup> I-1030-85
	Colorimetric, Automated, Ascorbic Acid	<sup>6</sup> 365.1			4500-P-F	
	Colorimetric, Ascorbic Acid, Single Reagent			D515-88A	4500-P-E	
	Colorimetric, Phosphomolybdate; Automated-segmented flow;					<sup>5</sup> I-1601-85
Silica	Automated discrete					<sup>5</sup> I-2601-90
	Ion Chromatography	<sup>6</sup> 300.0		D4327-91	4110	<sup>5</sup> I-2598-85
	Colorimetric, Molybdate Blue; Automated-segmented flow					<sup>5</sup> I-1700-85
	Colorimetric			D859-88		<sup>5</sup> I-2700-85
Temperature	Molybdosilicate				4500-Si-D	
	Heteropoly Blue				4500-Si-E	
	Automated Method for Molybdate-Reactive Silica				4500-Si-F	
	Inductively Coupled Plasma	<sup>2</sup> 200.7			3120B	
Sodium	Thermometric				2550	
	Inductively Coupled Plasma	<sup>2</sup> 200.7				
	Atomic Absorption; Direct Aspiration				3111B	

<sup>1</sup> Methods 150.1, 150.2 and 245.2 are available from U.S. EPA, NERL, Cincinnati, OH 45268. The identical methods were formerly in "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, March 1983, which is available at NTIS, PB84-128677.

<sup>2</sup> "Methods for the Determination of Metals in Environmental Samples—Supplement 1", EPA-600/R-94-111, May 1994. Available at NTIS, PB 94-184942.

<sup>3</sup> The procedures shall be done in accordance with the *Annual Book of ASTM Standards*, 1994, Vols. 11.01 and 11.02, American Society for Testing and Materials. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, S.W., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, N.W., Suite 700, Washington, DC.

<sup>4</sup> The procedures shall be done in accordance with the 18th edition of *Standard Methods for the Examination of Water and Wastewater*, 1992, American Public Health Association. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street, N.W., Washington, DC 20005. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, S.W., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, N.W., Suite 700, Washington, DC.

<sup>5</sup> Available from Books and Open-File Reports Section, U.S. Geological Survey, Federal Center, Box 25425, Denver, CO 80225-0425.

<sup>6</sup> "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA-600/R-93-100, August 1993. Available at NTIS, PB94-121811.

<sup>7</sup> 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. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from ATI Orion, 529 Main Street, Boston, MA 02129. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, S.W., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, N.W., Suite 700, Washington, DC.

<sup>8</sup> Method B-1011, "Waters Test Method for Determination of Nitrite/Nitrate in Water Using Single Column Ion Chromatography", Millipore Corporation, Water Chromatography Division, 34 Maple Street, Milford, MA 01757.

<sup>9</sup>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.

<sup>10</sup>Method 100.2, "Determination of Asbestos Structure Over 10  $\mu$ m in Length in Drinking Water", EPA-600/R-94-134, June 1994. Available at NTIS, PB94-201902.

<sup>11</sup>The procedures shall be done in accordance with the 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. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from the Technicon Industrial Systems, Tarrytown, NY 10591. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, S.W., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, N.W., Suite 700, Washington, DC.

<sup>12</sup>Unfiltered, no digestion or hydrolysis.

<sup>13</sup>Methods published by this organization and approved for use under this part may not be modified beyond the modifications expressly allowed and defined.

\* \* \* \* \*

4. Section 141.24, paragraph (e), is proposed to be amended by revising the table to read as follows:

**§ 141.24 Organic chemicals other than total trihalomethanes, sampling and analytical requirements.**

(e) \* \* \*

\* \* \* \* \*

TABLE 141.24(e).—LIST OF APPROVED ORGANIC TEST PROCEDURES

Parameter/methodology	Reference method	Other approved methods		
		EPA	Standard method 18th Ed. <sup>1</sup>	Other
1. Benzene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
2. Carbon Tetrachloride				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
GC/ECD .....	551			
3. Chlorobenzene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
4. 1,2-Dichlorobenzene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
5. 1,4-Dichlorobenzene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
6. 1,2-Dichloroethane				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
7. cis-Dichloroethylene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
8. Trans-Dichloroethylene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
9. Dichloromethane				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
10. 1,2-Dichloropropane				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
11. Ethylbenzene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
12. Styrene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
13. Tetrachloroethylene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
GC/ECD .....	551			
14. 1,1,1-Trichloroethane				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
GC/ECD .....	551			
15. Trichloroethylene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
GC/ECD .....	551			
16. Toluene				
GC/ELCD .....	502.2			

TABLE 141.24(e).—LIST OF APPROVED ORGANIC TEST PROCEDURES—Continued

Parameter/methodology	Reference method	Other approved methods		
		EPA	Standard method 18th Ed. <sup>1</sup>	Other
GC/MS .....	524.2			
17. 1,2,4-Trichlorobenzene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
18. 1,1-Dichloroethylene				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
19. Vinyl chloride				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
20. Xylenes (total)	.....	515.1		
GC/ELCD .....	502.2			
GC/MS .....	524.2			
21. 2,3,7,8-TCDD (dioxin)	.....	515.1		
GC/MS .....	1613			
22. 2,4-D				
GC/ECD .....	515.2			
HPLC/UV .....	555			
23. 2,4,5-TP (Silvex)				
GC/ECD .....	515.2			
HPLC/UV .....	555			
24. Alachlor				
GC/NPD .....	507			
GC/ECD .....	508.1	505		
GC/MS .....	525.2			
25. Atrazine				
GC/NPD .....	507			
GC/ECD .....	508.1	505		
GC/MS .....	525.2			
26. Benzo(a)pyrene				
GC/MS .....	525.2			
HPLC/FI-UV .....	550.1	550	6610	
27. Carbofuran				
HPLC/FI .....	531.1			
28. Chlordane				
GC/NPD .....	507			
GC/ECD .....	508.1	505		
GC/MS .....	525.2			
29. Dalapon				
GC/ECD .....	515.1	552.1		
30. Di-(2-ethylhexyl) adipate				
GC/PID .....	506			
GC/MS .....	525.2			
31. Di-(2-ethylhexyl) phthalate				
GC/PID .....	506			
GC/MS .....	525.2			
32. Dibromochloropropane (DBCP)				
GC/ECD .....	504.1	551		
33. Dinoseb				
GC/ECD .....	515.2	515.1		
HPLC/UV .....	555			
34. Diquat				
HPLC/UV .....	549.1			
35. Endothall				
GC/MS .....	548.1			
36. Endrin				
GC/NPD .....	507			
GC/ECD .....	508.1	505		
GC/MS .....	525.2			
37. Ethylene Dibromide (EDB)				
GC/ECD .....	504.1	551		
38. Glyphosate				
HPLC/FI .....	547		6651	
39. Heptachlor				
GC/ECD .....	508.1	505, 508		
GC/MS .....	525.2			
40. Heptachlor Epoxide				
GC/ECD .....	508.1	505, 508		

TABLE 141.24(e).—LIST OF APPROVED ORGANIC TEST PROCEDURES—Continued

Parameter/methodology	Reference method	Other approved methods		
		EPA	Standard method 18th Ed. <sup>1</sup>	Other
GC/MS .....	525.2			
41. Hexachlorobenzene				
GC/ECD .....	508.1	505, 508		
GC/MS .....	525.2			
42. Hexachlorocyclopentadiene				
GC/ECD .....	508.1	505, 508		
GC/MS .....	525.2			
43. Lindane				
GC/ECD .....	508.1	505, 508		
GC/MS .....	525.2			
44. Methoxychlor				
GC/ECD .....	508.1	505, 508		
GC/MS .....	525.2			
45. Oxamyl				
HPLC/FI .....	531.1		6610	
46. PCBs				
GC/ECD, As decachlorobiphenyl .....	508A			
GC/ECD, As Aroclors .....	508	505		
47. Pentachlorophenol		515.1		
GC/ECD .....	515.2			
HPLC/UV .....	555			
GC/MS .....	525.2			
48. Picloram		515.1		
GC/ECD .....	515.2			
HPLC/UV .....	555			
49. Simazine		505		
GC/NPD .....	507			
GC/ECD .....	508.1			
GC/MS .....	525.2			
50. Toxaphene		505		
GC/ECD .....	508			
GC/MS .....	525.2			
51. Total Trihalomethanes				
GC/ELCD .....	502.2			
GC/MS .....	524.2			
GC/ECD .....	551			

<sup>1</sup> Methods published by this organization and approved for use under this part may not be modified beyond the modifications expressly allowed and defined in each method.

**Note:** The following acronyms are used in this table:

ECD—Electron Capture Detector.  
 ELCD—Electrolytic Conductivity Detector.  
 FI—Fluorescence.  
 GC—Gas Chromatography.  
 GC/MS—Gas Chromatography/Mass Spectrometry.  
 HPLC—High Performance Liquid Chromatography.  
 NPD—Nitrogen Phosphorous Detector.  
 PID—Photoionization Detector.  
 UV—Ultraviolet Detector.

\* \* \* \* \*

5. Section 141.27 is proposed to be revised to read as follows:

**§ 141.27 New and alternate analytical methods.**

(a) Sample preservation procedures, container materials, and maximum allowable holding times for contaminants cited in tables in §§ 141.23(k)(1), 141.24(e) and 141.40(n)(11) are prescribed in these methods except as specified in the table in § 141.23(k)(2). Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times

applicable to samples collected from a public water system (PWS) supply or tap water. An application for a variance may be made by letter to the Regional Administrator in the Region in which the water supply system is located. Sufficient data should be provided to ensure such variance does not adversely affect the integrity of the sample. Such data will be forwarded by the Regional Administrator to the Director of the Analytical Methods Staff for technical review and recommendations for action on the variance application. Upon receipt of a recommendation from the Director of the Analytical Methods Staff,

the Administrator may grant a variance applicable to samples collected from the specific PWS for which the application for variance was made. A decision to recommend approval or denial of a variance will be made within 90 days of receipt of a complete application.

(b) A reference method listed in the tables in §§ 141.23(k)(1), 141.24(e), and 141.40(n)(11) of this section may be modified to improve separations, lower the costs of measurements, reduce or eliminate interferences, or for other purposes, provided that the modification is not explicitly prohibited in the reference method and provided

that the laboratory modifying the reference method meets the requirements in this section, performs the standardized QC tests, and demonstrates that the QC acceptance criteria and the requirements specified at Appendixes E, F, and G of 40 CFR part 136 are met. A laboratory that wishes to use a new or modified drinking water method must demonstrate that the MDL determined with that method meets the detection limits specified at §§ 141.23, 141.24 and 141.89 and/or at § 141.27(d). Demonstration of a valid detection limit requires use of an MDL study in accordance with the procedure at 40 CFR part 136, Appendix B. If the MDL determined with the new or modified method is not acceptable, the method may not be used. Specified detection limits are usually analyte-specific. For any given analyte, the specified detection limit may vary between a wastewater and a drinking water reference method.

(1) Tier 1: modification of a reference method for application in a single laboratory to one or more PWSs.

(i) Application to a single PWS.

(A) A laboratory may modify a reference method listed in the tables in §§ 141.23(k)(1), 141.24(e) and 141.40(n)(11) of this section for determination of an analyte of concern in a specific PWS, provided that the laboratory:

(1) Performs the standardized QC tests, including a test of initial precision and recovery (IPR) on a reagent water matrix;

(2) Performs the matrix spike (MS) and matrix spike duplicate (MSD) tests on a sample from the PWS to which the modification is to be applied;

(3) Meets the QC acceptance criteria in the reference method as supplemented in the table of QC acceptance criteria for drinking water methods at § 141.27(d);

(4) Documents the results of the QC tests using the Checklist for Initial Demonstration of Method Performance and the Checklist for Continuing Demonstration of Method Performance which are specified in 40 CFR part 136, Appendix E; and

(5) Maintains the results of the QC tests and other tests on file for inspection by EPA and/or the State.

(B) After the laboratory has demonstrated application of a method modification to a given PWS by meeting the MS/MSD QC acceptance criteria, only that laboratory may subsequently apply that method modification to samples from that PWS.

(C) A laboratory may apply a given method modification to additional

PWSs if the laboratory validates the modification on a sample from each PWS by performing a matrix spike (MS) and matrix spike duplicate (MSD) test and meeting the MS/MSD QC acceptance criteria for precision and recovery for each PWS.

(ii) Application to multiple public water systems (PWSs). After a laboratory has validated a given modification on samples from a minimum of three (3) PWSs in accordance with the procedures given in paragraph (b)(1)(i)(A) of this section, the laboratory may subsequently apply that method modification to other PWSs, provided that the matrix spike (MS) and matrix spike duplicate (MSD) recovery and the relative percent difference are within the QC acceptance criteria given for the analyte in the reference method as supplemented by the applicable QC acceptance criteria for drinking water methods at § 141.27(d). If all QC acceptance criteria are not met for a sample from a given PWS, the modification may not be applied to samples from that PWS.

(iii) To test the modified method for potential matrix effects, the three (3) PWS samples must be collected from PWSs with water quality characteristics that are sufficiently different that sample matrix effects, if any, can be observed. In all cases, the laboratory must try to determine if the measurement result for the target analyte using a new or modified method differs from the result obtained in a reagent water matrix or in a previously validated matrix type or PWS sample. Selection of suitable PWSs requires a knowledge of the chemistry of the method. Analysts may review an applicable approved or published method for indications of matrix effects that are unique to the analyte separation and measurement technologies used in the new or modified method. Water quality characteristics that can affect analysis of drinking water samples include, but are not limited to pH, total organic carbon content, turbidity, total organic halogen content, ionic strength, sulfate contamination, metal contamination, and trihalomethane contamination of the drinking water sample.

(2) Tier 2: modification of a reference method for application by all laboratories to all PWSs in the water supply and distribution industry (nationwide modification).

(i) A person may modify a reference method for application by all laboratories to determination of an analyte of concern in sample matrices from any PWS provided that the modification is validated in a minimum

of three (3) laboratories each of which test a sample from each of three (3) different PWS for a minimum of nine (9) tests. To test the modified method for potential matrix effects, the three (3) PWS samples must be collected from PWSs with sufficiently different water quality characteristics according to criteria specified at paragraph (b)(1)(iii) of this section. Each laboratory must meet the requirements in paragraph (b)(1)(i)(A) of this section. After the tests in all three laboratories have met all QC acceptance criteria for the reference method, the modified method may be applied by laboratories nationwide to PWSs in the water supply and distribution industry.

(ii) A person who modifies a reference method and validates the method modification under Tier 2 may submit that modification to EPA for a letter of approval. The information that must be submitted includes the results of the performance tests required by paragraph (b)(1)(i)(A) of this section. This information and other information that must be submitted and the format for submission are specified at 40 CFR part 136, Appendixes E, F, and G.

(iii) A person who modifies a reference method and validates the method modification under Tier 2 may submit that modification to EPA for approval and inclusion in a table in this part 141. The information that must be submitted includes the results of the performance tests required by paragraph (b)(1)(i)(A) of this section. This information and other detailed information that must be submitted and the format for submission are specified at 40 CFR part 136, Appendixes E, F, and G.

(iv) A decision to recommend proposal of a Tier 2 method modification will be made by the Director of the Analytical Methods Staff within 90 days of receipt of a complete application.

(c) A person may apply to EPA for use of a new method for determination of an analyte of concern, provided that the new method meets the requirements for validation and format as specified in this section and in 40 CFR part 136, Appendixes E, F, and G.

(1) The new method must demonstrate an acceptable MDL for each analyte as specified in § 141.27(b).

(i) A new method must:

(A) Be documented in accordance with requirements in 40 CFR part 136, Appendixes E, F, and G.

(B) Contain standardized QC as defined at § 141.2.

(C) Contain QC acceptance criteria that have been developed in accordance



with the requirements detailed in 40 CFR part 136, Appendixes E, F, and G.

(D) Employ a determinative technique for an analyte of concern with selectivity or sensitivity equal or superior to the selectivity or sensitivity of the determinative technique in any approved method, and that differs from the determinative techniques employed for that analyte in all approved methods.

(E) Be accompanied by the information specified at 40 CFR part 136, Appendix G.

(ii) A decision to recommend proposal of a new method will be made by the Director of the Analytical Methods Staff within 90 days of receipt of a complete application.

(2) Tier 1: application of a new method by a single laboratory to one or more PWSs.

(i) A person may develop a new method for determination of an analyte of concern by a single laboratory by validating the method and developing QC acceptance criteria from an interlaboratory method validation study or from a single-laboratory validation study on a drinking water sample. Details of the single-laboratory method

validation study and development of QC acceptance criteria from a single-laboratory or interlaboratory method validation study are specified at paragraph (b)(1) of this section and at 40 CFR part 136, Appendix E.

(ii) A person who develops a new method under Tier 1 must submit the method to EPA for a letter of approval. The information that must be submitted and the format for submission are specified at 40 CFR part 136, Appendixes E, F, and G.

(3) Tier 2: application of a new method by all laboratories to all PWSs in the water supply and distribution industry (nationwide use).

(i) A person may develop a new method for determination of an analyte of concern in all PWSs in the water supply and distribution industry by developing QC acceptance criteria from an interlaboratory method validation study or from multiple, single-laboratory validation studies as specified in the Streamlining Guide, and by validating the new method in a minimum three (3) laboratories each of which test samples from a minimum of three (3) different PWS for a minimum of nine (9) tests. In the method

validation study, each laboratory will test all of the samples from the same set of PWS samples and this set will contain samples from a minimum of three (3) different PWSs. To test the modified method for potential matrix effects, the three (3) PWS samples must be collected from PWSs with sufficiently different water quality characteristics according to criteria specified at paragraph (b)(2) and (b)(1)(iii) of this section.

(ii) A person who develops a new method under Tier 2 must submit the method to EPA for approval and inclusion in a table in this part 141. The information that must be submitted includes the results of the performance tests required by paragraph (b)(2)(i) of this section. This information and other detailed information that must be submitted and the format for submission are specified at 40 CFR part 136, Appendixes E, F, and G.

(d) Standardized QC and QC acceptance criteria for modifications of inorganic contaminant reference methods at § 141.23(k)(1) of this section are as follows:

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Standardized QC and QC Acceptance Criteria for Methods in 40 CFR 141.23(k)(1)

No.	Analyte	Reference Method	Data		Prec-ision	Labs	Source	Specs				IPR				OPR				MSMSD				ML Value	Calc	
			Recovery	ision				CAL	Points	Lin	MCL (ug/L)	Spike conc	Recovery		Prec-ision	Recovery		High	RPD	ML MDL	ML Value					
													Low	High		Low	High					Low	High			
1.	Alkalinity - Titr/Man	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---			
2.	Alkalinity - Titr/Auto	---	---	---	---	---	---	---	---	---	6.0	---	---	---	---	---	---	---	---	---	---	---	---			
	Antimony - Furnace	---	---	---	---	---	---	---	---	---	6.0	---	---	---	---	---	---	---	---	---	---	---	---			
3.	Antimony - Hydride	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---			
	Antimony - ICP/MS	200.8	98.8	8.067	Multi	Tbl 12	3	10 %	3	10 %	6.0	6 ug/L	82.0	115.0	17.0	81.0	117.0	81.0	117.0	81.0	117.0	17.0	0.4 ug/L	1 ug/L	3.18 x MDL	
	Antimony - STGFAA	200.9	95.4	2.8	Single	Tbl IE	3	10 %	3	10 %	6.0	20 ug/L	85.0	106.0	11.0	84.0	107.0	84.0	107.0	84.0	107.0	11.0	0.8 ug/L	2 ug/L	3.18 x MDL	
	Arsenic - Furnace	---	---	---	---	---	---	---	---	---	50	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
4.	Arsenic - Hydride	---	---	---	---	---	---	---	---	---	50	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Arsenic - ICP	200.7	98.27	13.59	Multi	Apx C	3	10 %	3	10 %	50	200 ug/L	71.0	126.0	28.0	68.0	129.0	68.0	129.0	68.0	129.0	28.0	53 ug/L	200 ug/L	3.18 x MDL	
	Arsenic - ICP/MS	200.8	100.44	6.9	Multi	Tbl 12	3	10 %	3	10 %	50	50 ug/L	86.0	115.0	14.0	85.0	116.0	85.0	116.0	85.0	116.0	14.0	1.4 ug/L	5 ug/L	3.18 x MDL	
	Arsenic - STGFAA	200.9	88.4	10	Single	Tbl IE	3	10 %	3	10 %	50	10 ug/L	52.0	125.0	36.0	48.0	129.0	48.0	129.0	48.0	129.0	36.0	0.5 ug/L	2 ug/L	3.18 x MDL	
5.	Asbestos - TEM	100.1	---	---	---	---	---	---	---	---	7 MFL	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Asbestos - TEM	100.2	---	---	---	---	---	---	---	---	7 MFL	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
6.	Barium - Flame	---	---	---	---	---	---	---	---	---	2000	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Barium - Furnace	---	---	---	---	---	---	---	---	---	2000	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Barium - ICP	200.7	76.88	18.47	Multi	Apx C	3	10 %	3	10 %	2000	1 mg/L	39.0	114.0	37.0	36.0	118.0	36.0	118.0	36.0	118.0	37.0	2 ug/L	5 ug/L	3.18 x MDL	
7.	Barium - ICP/MS	200.8	96.31	4.55	Multi	Tbl 12	3	10 %	3	10 %	2000	1 mg/L	87.0	106.0	9.1	86.0	107.0	86.0	107.0	86.0	107.0	9.1	0.8 ug/L	2.0 ug/L	3.18 x MDL	
	Beryllium - Flame	---	---	---	---	---	---	---	---	---	4.0	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Beryllium - ICP	200.7	97.54	25.11	Multi	Apx C	3	10 %	3	10 %	4.0	4 ug/L	47.0	148.0	51.0	42.0	153.0	42.0	153.0	42.0	153.0	51.0	0.3 ug/L	1 ug/L	3.18 x MDL	
	Beryllium - ICP/MS	200.8	110.50	12.70	Multi	Tbl 12	3	10 %	3	10 %	4.0	4 ug/L	85.0	136.0	26.0	82.0	139.0	82.0	139.0	82.0	139.0	26.0	0.3 ug/L	1 ug/L	3.18 x MDL	
8.	Beryllium - STGFAA	200.9	106	9.4	Single	Tbl IE	3	10 %	3	10 %	4.0	2.5 ug/L	72.0	140.0	34.0	68.0	144.0	68.0	144.0	68.0	144.0	34.0	0.02 ug/L	0.05 ug/L	3.18 x MDL	
	Cadmium - Furnace	---	---	---	---	---	---	---	---	---	5.0	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Cadmium - ICP	200.7	95.14	45.97	Multi	Apx C	3	10 %	3	10 %	5.0	5 ug/L	3.0	188.0	92.0	d	197.0	d	197.0	d	197.0	92.0	4 ug/L	10 ug/L	3.18 x MDL	
	Cadmium - ICP/MS	200.8	100.5	16.1	Multi	Tbl 12	3	10 %	3	10 %	5.0	5 ug/L	68.0	133.0	33.0	65.0	136.0	65.0	136.0	65.0	136.0	33.0	0.5 ug/L	2 ug/L	3.18 x MDL	
9.	Cadmium - STGFAA	200.9	105.2	6.3	Single	Tbl IE	3	10 %	3	10 %	5.0	0.5 ug/L	82.0	128.0	23.0	80.0	131.0	80.0	131.0	80.0	131.0	23.0	0.05 ug/L	0.2 ug/L	3.18 x MDL	
	Calcium - Flame	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
10.	Calcium - ICP	200.7	89.22	22.38	Multi	Apx C	3	10 %	3	10 %	---	100 ug/L	44.0	134.0	45.0	39.0	139.0	39.0	139.0	39.0	139.0	45.0	10 ug/L	20 ug/L	3.18 x MDL	
	Calcium - Titr	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Chromium - Furnace	---	---	---	---	---	---	---	---	---	100	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Chromium - ICP	200.7	98.54	9.39	Multi	Apx C	3	10 %	3	10 %	100	100 ug/L	79.0	118.0	19.0	77.0	120.0	77.0	120.0	77.0	120.0	19.0	7 ug/L	20 ug/L	3.18 x MDL	
11.	Chromium - ICP/MS	200.8	100.45	3.69	Multi	Tbl 12	3	10 %	3	10 %	100	100 ug/L	93.0	108.0	7.4	92.0	109.0	92.0	109.0	92.0	109.0	7.4	0.9 ug/L	2 ug/L	3.18 x MDL	
	Chromium - STGFAA	200.9	105.7	3.1	Single	Tbl IE	3	10 %	3	10 %	100	2.5 ug/L	94.0	117.0	12.0	93.0	119.0	93.0	119.0	93.0	119.0	12.0	0.1 ug/L	0.2 ug/L	3.18 x MDL	

Standardized QC and QC Acceptance Criteria for Methods in 40 CFR 141.23(k)(1)

No.	Anal/te	Data		Specs										ML		ML	
				Reference Method	Prec- Recovery	Labs	Source	CAL Points	MCL (ug/L)	IPR		OPR		MS/MSD Recovery	High	RPD	Calc
										Low	High	Prec- ision	Low				
10.	Conductivity	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
11.	Copper - Flame	---	---	---	---	---	---	---	1000	---	---	---	---	---	---	---	---
	Copper - Furnace	---	---	---	---	---	---	---	1000	---	---	---	---	---	---	---	---
	Copper - ICP	200.7	92.94	4.71	Multi	Apex C	3	10 %	1000	1 mg/L	83.0	103.0	9.5	82.0	104.0	9.5	20 ug/L 3.18 x MDL
	Copper - ICP/MS	200.8	97.56	6.39	Multi	Tbl 12	3	10 %	1000	1 mg/L	84.0	111.0	13.0	83.0	112.0	13.0	0.2 ug/L 3.18 x MDL
	Copper - STGFAA	200.9	111.5	10	Single	Tbl IE	3	10 %	1000	10 ug/L	75.0	148.0	36.0	71.0	152.0	36.0	2 ug/L 3.18 x MDL
12.	Cyanide - CATC	---	---	---	---	---	---	---	200	---	---	---	---	---	---	---	---
	Cyanide - Spectro/Man	---	---	---	---	---	---	---	200	---	---	---	---	---	---	---	---
	Cyanide - Spectro/Auto	335.4	100	10	No data	Default	3	10 %	200	200 ug/L	64.0	136.0	36.0	60.0	140.0	36.0	5 ug/L Range
	Cyanide - ISE	---	---	---	---	---	---	---	200	---	---	---	---	---	---	---	---
13.	Fluoride - Elec/man	---	---	---	---	---	---	---	2000	---	---	---	---	---	---	---	---
	Fluoride - Elec/auto	---	---	---	---	---	---	---	2000	---	---	---	---	---	---	---	---
	Fluoride - SPADNS	---	---	---	---	---	---	---	2000	---	---	---	---	---	---	---	---
	Fluoride - Auto/Aliz	---	---	---	---	---	---	---	2000	---	---	---	---	---	---	---	---
	Fluoride - IC	300.0	87.7	5	Single	MCAWW	3	10 %	2000	2 mg/L	69.0	106.0	18.0	67.0	108.0	18.0	20 ug/L 3.18 x MDL
14.	pH - Electrode	150.1	---	---	---	---	---	---	6.5-8.5	---	---	---	---	---	---	---	---
	pH - Auto	150.2	---	---	---	---	---	---	6.5-8.5	---	---	---	---	---	---	---	---
15.	Lead - Furnace	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
	Lead - ICP/MS	200.8	100.20	12.10	Multi	Tbl 12	3	10 %	---	10 ug/L	76.0	125.0	25.0	73.0	127.0	25.0	2 ug/L 3.18 x MDL
	Lead - STGFAA	200.9	101.80	4.00	Single	Tbl IE	3	10 %	---	10 ug/L	87.0	117.0	15.0	85.0	118.0	15.0	2 ug/L 3.18 x MDL
	Mercury - CV/Man	245.1	100.34	43.82	Multi	MCAWW	3	10 %	2.0	2 ug/L	12.0	188.0	88.0	3.0	197.0	88.0	0.2 ug/L Range
	Mercury - CV/Auto	245.2	102	4.5	Single	MCAWW	3	10 %	2.0	2 ug/L	85.0	119.0	17.0	84.0	120.0	17.0	0.2 ug/L Range
	Mercury - ICP/MS	200.8	100	10	No data	Default	3	10 %	2.0	2 ug/L	64.0	136.0	36.0	60.0	140.0	36.0	No data
17.	Nickel - Flame	---	---	---	---	---	---	---	100	---	---	---	---	---	---	---	---
	Nickel - Furnace	---	---	---	---	---	---	---	100	---	---	---	---	---	---	---	---
	Nickel - ICP	200.7	95.48	10.44	Multi	Apex C	3	10 %	100	100 ug/L	74.0	117.0	21.0	72.0	119.0	21.0	50 ug/L 3.18 x MDL
	Nickel - ICP/MS	200.8	95.11	5.16	Multi	Tbl 12	3	10 %	100	100 ug/L	84.0	106.0	11.0	83.0	107.0	11.0	2 ug/L 3.18 x MDL
	Nickel - STGFAA	200.9	103.8	4.3	Single	Tbl IE	3	10 %	100	20 ug/L	88.0	120.0	16.0	86.0	121.0	16.0	2 ug/L 3.18 x MDL
	Nitrate - IC	300.0	100.7	5	Single	MCAWW	3	10 %	10000	10 mg/L	82.0	119.0	18.0	80.0	121.0	18.0	50 ug/L 3.18 x MDL
	Nitrate - Cd/Auto	353.2	97.31	7.10	Multi	MCAWW	3	10 %	10000	2.5 mg/L	83.0	112.0	15.0	81.0	113.0	15.0	50 ug/L Range
	Nitrate - ISE	---	---	---	---	---	---	---	10000	---	---	---	---	---	---	---	---
19.	Nitrite - IC	300.0	97.7	5	Single	MCAWW	3	10 %	1000	0.1 ug/L	79.0	116.0	18.0	77.0	118.0	18.0	10 ug/L 3.18 x MDL

Standardized QC and QC Acceptance Criteria for Methods in 40 CFR 141.23(k)(1)

No.	Analyte	Reference Method	Data		Specs													
			Recovery	Precision	Labs	Source	CAL Points	MCL (ug/L)	Spike conc	IPR Recovery		Precision	OPR Recovery		ML MDL	ML Value	Calc Range	
										Low	High		Low	High				
20.	Nitrite - Cd/Auto	353.2	97.31	7.10	Multi	MCAWW	3	10 %	1000	2.5 mg/L	83.0	112.0	15.0	81.0	113.0	15.0	50 ug/L	200 ug/L 3.18 x MDL 10 ug/L Range
	Nitrite - Spec/Auto	---	---	---	---	---	---	---	1000	---	---	---	---	---	---	---	---	
	Nitrite - Spec/Auto	---	---	---	---	---	---	---	1000	---	---	---	---	---	---	---	---	
	O-phosphate - IC	300.0	100.4	3.8	Single	MCAWW	3	10 %	---	500 ug/L	86.0	115.0	14.0	85.0	116.0	14.0	61 ug/L	
	O-phosphate - Asc/Auto	365.1	87.2	22	Multi	MCAWW	3	10 %	---	300 ug/L	43.0	132.0	45.0	38.0	136.0	44.0	---	
	O-phosphate - Asc/Sing	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
21.	O-phosphate - Phos/Mo	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
	O-phosphate - Auto/seg	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
	O-phosphate - Auto/Dis	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
	Selenium - Furnace	---	---	---	Multi	---	---	50	---	---	---	---	---	---	---	---	---	
	Selenium - Hydride	---	---	---	Multi	---	---	50	---	---	---	---	---	---	---	---	---	
	Selenium - ICP/MS	200.8	102.48	9.8	Multi	Tbl 12	3	10 %	50	50 ug/L	82.0	123.0	20.0	80.0	125.0	20.0	7.9 ug/L	
22.	Selenium - STGFAA	200.9	88.9	10	Single	Tbl IE	3	10 %	50	25 ug/L	52.0	125.0	36.0	48.0	129.0	36.0	2 ug/L	
	Silica - ICP	200.7	53.86	45.38	Multi	Apx C	5	25 %	---	1 mg/L	d	145.0	91.0	d	154.0	91.0	200 ug/L	
	Silica - Color	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
	Silica - Color/Mo Blue	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
	Silica - Molybdosil	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
	Silica - Heteropoly	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
23.	Silica - Auto/Mo react	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
	Sodium - Flame	---	---	---	Multi	Apx C	5	25 %	---	1 mg/L	51.0	149.0	49.0	46.0	154.0	49.0	100 ug/L	
24.	Sodium - ICP	200.7	99.77	24.27	Multi	---	---	25 %	---	---	---	---	---	---	---	---	3.18 x MDL	
25.	Temperature	---	---	---	Multi	Tbl 12	3	10 %	2.0	2 ug/L	72.0	131.0	29.0	69.0	134.0	29.0	1 ug/L	
	Thallium - ICP/MS	200.8	101.5	14.5	Multi	Tbl IE	3	10 %	2.0	20 ug/L	85.0	106.0	11.0	84.0	107.0	11.0	2 ug/L	
	Thallium - STGFAA	200.9	95.4	2.8	Single	Tbl IE	3	10 %	2.0	---	---	---	---	---	---	---	3.18 x MDL	

(e) The number and type of required tests, testing laboratories, matrices, and replicate QC tests for method validation depend on the tier at which the new or modified wastewater or drinking water method is validated. These

requirements are specified at paragraphs (a), (b), (c) of this section and in the table at § 136.5(d).

6. Section 141.40, paragraph (n)(11), is proposed to be amended by revising the table to read as follows:

**§ 141.40 Special monitoring for inorganic and organic contaminants.**

\* \* \* \* \*

(n) \* \* \*

(11) \* \* \*

TABLE 141.40(n)(11)

Parameter/methodology	Reference method	Other approved methods		
		EPA	Standard methods 18th ed. <sup>1</sup>	Other
1. aldicarb HPLC/FI	531.1		6610	
2. aldicarb sulfone HPLC/FI	531.1		6610	
3. aldicarb sulfoxide HPLC/FI	531.1		6610	
4. aldrin GC/ECD GC/MS	508.1 525.2	505, 508		
5. butachlor GC/MS GC/NPD	525.2 507			
6. carbaryl HPLC/FI	531.1		6610	
7. dicamba GC/ECD HPLC	515.2	515.1		
12. metribuzin GC/ECD GC/MS GC/NPD	508.1 525.2 507			
13. propachlor GC/ECD GC/MS	508.1 525.2	508		

**Note:** The following acronyms are used in this table:

ECD—Electron Capture Detector.

FI—Fluorescence.

GC—Gas Chromatography.

GC/MS—Gas Chromatography/Mass Spectrometry.

HPLC—High Performance Liquid Chromatography.

NPD—Nitrogen Phosphorous Detector.

UV—Ultraviolet Detector.

\* \* \* \* \*

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