ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[FRL–7074–6]

Guidelines Establishing Test Procedures for the Analysis of Pollutants; Measurement of Mercury in Water; Revisions to EPA Method 1631; Proposed Rule

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule.

SUMMARY: By this action, EPA is proposing modifications to EPA Method 1631, Revision C: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry (Method 1631C), which measures mercury in aqueous samples. The proposed modifications would require use of certain ''clean techniques'' and quality control requirements when using this test method. The Agency is proposing to modify EPA Method 1631C to fulfill obligations under a Settlement Agreement designed to resolve litigation challenging an earlier EPA rulemaking that standardized this test method. The proposed modifications are intended to improve performance of EPA Method 1631C by reducing opportunities for contamination during sample collection and analysis. In addition, EPA is proposing revisions to this test method based on comments received from method users following method approval.

DATES: Comments must be postmarked, delivered by hand, or electronically mailed on or before December 10, 2001. Comments provided electronically will be considered timely if they are submitted electronically by 11:59 p.m. Eastern Standard Time (EST) on December 10, 2001.

ADDRESSES: Mail written comments on the proposed rule to ''Method 1631—Proposed Rule'' Comment Clerk (W–01–05), Water Docket (4101); U.S. Environmental Protection Agency; Ariel Rios Building; 1200 Pennsylvania Avenue, NW., Washington, DC 20460. Hand deliveries should be delivered to: EPA's Water Docket at 401 M Street, SW., East Tower Basement (Room EB 57), Washington, DC 20460. If you wish to hand-deliver your comments, please call (202) 260–3027 between 9:00 a.m. and 4:00 p.m., Monday through Friday, excluding Federal holidays, to schedule an appointment. Comments also may be submitted electronically to: *OW-docket@epamail.epa.gov.*

FOR FURTHER INFORMATION CONTACT: Maria Gomez-Taylor, Ph.D.; Engineering and Analysis Division (4303); Office of Science and Technology; Office of Water; U.S. Environmental Protection Agency; Ariel Rios Building; 1200 Pennsylvania Avenue, NW; Washington, DC 20460, or call (202) 260–1639 or E-mail at *gomez-taylor.maria@epa.gov.*

SUPPLEMENTARY INFORMATION:

Potentially Regulated Entities

EPA Regions, as well as States, Territories and Tribes authorized to implement the National Pollutant Discharge Elimination System (NPDES) program, issue permits that comply with the technology-based and water quality-based requirements of the Clean Water Act. In doing so, NPDES permitting authorities, including authorized States, Territories, and Tribes, make a number of discretionary choices associated with permit writing, including the selection of pollutants to be measured and, in many cases, limited in permits. If EPA has ''approved'' (i.e., promulgated through rulemaking) standardized testing procedures for a given pollutant, the NPDES permitting authority must specify one of the approved testing procedures or an approved alternate test procedure for the measurements required under the permit. In addition, when a State, Territory, or authorized Tribe provides certification of Federal licenses under Clean Water Act section 401, States, Territories and Tribes are directed to use the approved testing procedures. Categories and entities that may be regulated include:

Category	Examples of potentially regulated entities
State, Territorial, and Indian Tribal Governments.	States, Territories, and Tribes authorized to administer the NPDES permitting program; States, Territories, and Tribes providing certification under Clean Water Act section 401.

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. If you have questions regarding the applicability of this action to a particular entity, consult the person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

Record and Commenting Procedures

The record for this rulemaking has been established under Docket Number W–01–05. A copy of the supporting documents cited in this proposal are available for review at EPA's Water Docket. The record is available for inspection from 9 a.m. to 4 p.m. EST, Monday through Friday, excluding Federal holidays at EPA's Water Docket, 401 M Street SW., East Tower Basement (Room EB 57), Washington, DC 20460. For access to docket materials, please call (202) 260–3027 to schedule an appointment.

Commenters are requested to submit any references cited in their comments. Commenters also are requested to submit an original and three copies of their written comments and enclosures, and to clearly identify the specific issue(s) and method section(s) to which the comment applies. Commenters who want a confirmed receipt of their comments should include a self-addressed, stamped envelope. All comments must be postmarked or delivered by hand. No facsimiles (faxes) will be accepted.

Electronic comments must be submitted as a Word Perfect for Windows 5/6/7/8 file or an ASCII file, avoiding the use of special characters and any form of encryption. Comments and data also will be accepted on disks in Word Perfect 5/6/7/8 or ASCII file format. Electronic comments on this notice may be filed online at many Federal Depository Libraries. All electronic comments must be identified by docket number. Electronic comments will be transferred into a paper version for the official record. EPA will attempt to clarify electronic comments if there is an apparent error in transmission.

Information on Internet Access

This Federal Register document has been placed on the Internet for public review and downloading at the following location: http//www.epa.gov/fedrgstr. Method 1631, Revision C; Draft Method 1631, Revision D; the Method 1631 Guidance; and a Fact Sheet are available at *www.epa.gov/ost/methods/1631.html* or from the EPA Sample Control Center (SCC), DynCorp I&ET, 6101 Stevenson Ave., Alexandria, VA 22304 (703–461–2100; *SCC@DynCorp.com*).

Outline of Notice

I. Statutory Authority

Today's proposal is pursuant to the authority of sections 301, 304(h), 307, and 501(a) of the Clean Water Act (CWA), 33 U.S.C. 1311, 1314(h), 1317, 1361(a) (the ''Act''). Section 301 of the Act prohibits the discharge of any pollutant into navigable waters unless the discharge complies with a National Pollutant Discharge Elimination System (NPDES) permit, issued under section 402 of the Act. Section 304(h) of the Act requires the Administrator of the EPA to ''promulgate guidelines establishing test procedures for the analysis of pollutants that shall include the factors which must be provided in any certification pursuant to section 401 of this Act or permit applications pursuant to section 402 of this Act.'' Section 501(a) of the Act authorizes the Administrator to ''prescribe such regulations as are necessary to carry out his function under this Act.'' EPA publishes CWA analytical method regulations at 40 CFR Part 136. The Administrator also has made these test procedures applicable to monitoring and reporting of NPDES permits (40 CFR Parts 122, §§ 122.21, 122.41, 122.44, and 123.25), and implementation of the pretreatment standards issued under section 307 of the Act (40 CFR Part 403, §§ 403.10 and 402.12).

II. Background

A. Regulatory Actions

On May 26, 1998, EPA proposed Method 1631 at 40 CFR Part 136 for use in determining mercury at ambient water quality criteria levels in EPA's Clean Water Act programs (63 FR 28867). Subsequently, on March 5, 1999, EPA published a Notice of Data Availability that included additional data supporting the application of Method 1631 to effluent matrices (64 FR 10596) in response to comments received at proposal. On June 8, 1999, EPA published a final rule promulgating EPA Method 1631, Revision B: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry (64 FR 30416) at 40 CFR Part 136. EPA published a technical correction revising EPA Method 1631B to EPA Method 1631C (66 FR 32774; June 18, 2001) to clarify the method text regarding the use of field blanks (see Settlement Agreement discussion below).

B. Settlement Agreement

In response to a petition for judicial review of EPA Method 1631B, EPA entered into negotiations with several industry groups. On October 19, 2000, EPA entered into a Settlement Agreement with the Alliance of Automobile Manufacturers, Inc., the Chemical Manufacturers Association, and the Utility Water Act Group (collectively, Petitioners), and the American Forest & Paper Association (Intervenor). The Settlement Agreement (*Alliance of Automobile Manufacturers, et al.* v. *EPA*, No. 99–1420, D.C. Dir.), is included in the rulemaking record in the Water Docket for today's proposal (see the **ADDRESSES** section of this proposal for details on the Water Docket). The Settlement Agreement includes four clauses that directly affect Method 1631 (Clauses 2, 3, 4, and 5).

Clauses 2 and 3 of the Settlement Agreement committed EPA to sign a notice of final rulemaking by June 15, 2001, revising sections 12.4.2 and 9.4.3.3 of Method 1631B to clarify the use of field blank subtraction (section 12.4.2) and the use of multiple field blanks (section 9.3.3.3) to determine whether test samples are acceptable for compliance monitoring purposes. EPA complied with that commitment, and on June 18, 2001, EPA published a final rule; technical correction notice (66 FR 32774) announcing a revised version of Method 1631 (Revision C; Method 1631C). That notice included the technical corrections about field blanks required by the Settlement Agreement. At that time, no other changes were made to the test method.

Clause 4 of the Settlement Agreement requires that EPA sign a notice for publication in the **Federal Register** on or before September 30, 2001 to propose additional requirements for certain clean techniques and quality control (QC) provisions in Method 1631. Today's proposed rule complies with EPA's obligation under Clause 4 of the Settlement Agreement. The additional requirements were listed in the Settlement Agreement as Appendix A and are discussed in Section IV of this preamble. Clause 4 also requires that EPA propose that ''a National Pollutant Discharge Elimination System (NPDES) permittee or an industrial user of a publicly-owned treatment works (POTW) may elect not to implement such provisions in its discretion and at its peril, unless specifically provided otherwise by the relevant permitting agency or pretreatment control authority, as the case may be.''

Clause 5 of the Settlement Agreement required that EPA publish a guidance document on or before March 1, 2001 specifying procedures for identifying, reducing, and demonstrating potential matrix interferences. On February 27, 2001, EPA published Guidance for Implementation and Use of EPA Method 1631 for the Determination of Low-Level Mercury (40 CFR Part 136) (EPA 821–R–01–023; March 2001) to address this clause and to assist regulatory agencies, dischargers, industrial users, and laboratories in the application of Method 1631 to ambient water and wastewater. In addition to providing information on potential matrix interferences, the guidance provides information on the use of clean techniques and method flexibility, and answers frequently asked questions regarding method implementation.

III. Summary of Today's Action

This rulemaking proposes to modify EPA Method 1631 to require the use of certain clean techniques and quality control (QC) provisions in accordance with clause 4 of the Settlement Agreement. These changes are in response to the petitioners' concerns

that results produced by laboratories using EPA Method 1631 would not be reliable unless the optional clean techniques and QC provisions are required. These revisions are discussed in Section IV of this proposal and are included in draft Method 1631, Revision D (Method 1631D).

Today's notice also proposes improvements and clarifications to EPA Method 1631 to make this test method more consistent with other approved methods and current practices, and easier to use. These proposed revisions are based on comments received from method users since promulgation of Method 1631. The proposed revisions are discussed in Section V and are included in draft Method 1631D.

IV. Proposed Revisions Based on the Settlement Agreement

A. Additional Requirements for Clean Techniques and Quality Control Provisions

Clause 4 of the Settlement Agreement requires EPA to sign a notice for publication in the **Federal Register** on or before September 30, 2001, proposing to require certain clean techniques and quality control (QC) provisions in EPA Method 1631. These requirements to propose clean techniques and QC provisions are listed in Appendix A to the Settlement Agreement and are reproduced below. The Petitioners believe that these additional requirements are necessary to prevent samples from becoming contaminated during the sampling and analysis process. EPA believes that these techniques may improve test performance. EPA refers readers to the appropriate section of draft EPA Method 1631, Revision D for the proposed revised language, which is indicated in the draft revised method in brackets and italics.

EPA solicits your comments and/or data on the proposed requirements, collectively or individually, and requests that you provide a reason to support your position.

1. Proposed Revision to Section 1.4

Item 1 of Appendix A to the Settlement Agreement states, ''Section 1.4 currently explains the importance of minimizing contamination of ambient water samples and explains that the Method includes suggestions for improvements to minimize contamination and maximize the ability of laboratories to make reliable measurements. The notice shall invite comment on revisions to this section to explain that certain sections contain suggestions and that other sections contain requirements to minimize contamination.'' Revision D, as proposed, includes a statement to that effect in Section 1.4.

2. Proposed Revision to Sections 2.3 and 8.5

Item 2 of Appendix A to the Settlement Agreement states, ''Sections 2.3 and 8.5 currently suggest that a sample used for the determination of methyl mercury should be preserved with 5 mL/L HCl solution only. The notice would invite comment on whether to change these sections to require the use of HCl for preservation if the sample is collected for the determination of methyl mercury.'' Revision D, as proposed, includes statements in Sections 2.3 and 8.5 corresponding to this requirement.

3. Proposed Revision to Section 4.3.3

Item 3 of Appendix A to the Settlement Agreement states, ''Section 4.3.3 currently suggests the use of a clean room or a clean bench. The notice would invite comment on whether to change this section to require a clean bench if a clean room is not available. The notice would not invite comment on whether to require a nonmetal hood because the Agency believes removal and replacement of existing metal hoods is unnecessary; use of a plastic awning in the hood prevents contamination during sample digestion.'' Revision D, as proposed, reflects this language in Section 4.3.3.

4. Proposed Revision to Section 4.3.4

Item 4 of Appendix A to the Settlement Agreement states, ''Section 4.3.4 currently suggests precautions to minimize exposure of the apparatus to contamination. The notice would invite comment on whether to change the 'shoulds' to 'musts.' '' Revision D, as proposed, changes the 'shoulds' to 'musts' in Section 4.3.4.

5. Proposed Revision to Section 4.3.5

Item 5 of Appendix A to the Settlement Agreement states, ''Section 4.3.5 currently recommends cleaning work surfaces before a batch of samples is processed. The notice would invite comment on whether to require the laboratory to clean work surfaces after processing a batch of samples with high levels of mercury.'' Revision D, as proposed, includes a requirement to this effect in Section 4.3.5.

6. Proposed Revision to Section 4.3.7.1

Item 6 of Appendix A to the Settlement Agreement states, ''Section 4.3.7.1 currently recommends that only fluoropolymer or borosilicate glass containers be used for samples. The notice would invite comment on whether to change 'should' to 'must' to respond to Petitioners' concern.'' Revision D, as proposed, changes the 'should' to 'must' in Section 4.3.7.1.

7. Proposed Revision to Sections 4.3.8.1 and 11.2.4

Item 7 of Appendix A to the Settlement Agreement states, ''Sections 4.3.8.1 and 11.2.4 currently suggest that a bubbler blank be used to check for carryover after encountering an unusually concentrated sample. The notice would invite comment on whether to change 'should' to 'must' in both sections to require analysis of the bubbler blank.'' Revision D, as proposed, changes the 'should' to 'must' in Section 4.3.8.1 and also changes Section 11.2 to correspond to this requirement.

8. Proposed Revision to Section 4.3.8.4

Item 8 of Appendix A to the Settlement Agreement states, ''Section 4.3.8.4 currently suggests that sample processing should occur as far as possible from sources of airborne contamination. The notice would invite comment on whether to change the 'should' to 'must.''' Revision D, as proposed, changes the 'should' to 'must' in Section 4.3.8.4.

9. Proposed Revision to Section 4.4.3

Item 9 of Appendix A to the Settlement Agreement states ''Section 4.4.3 currently explains a concern regarding condensation of water in the gold traps. The section explains that condensation can be avoided by predrying the gold trap, and by discarding those traps that tend to absorb large quantities of water vapor. The notice would invite comment on whether to change this Section to preclude the use of gold traps that tend to absorb large quantities of water vapor.'' Revision D, as proposed, changes Section 4.4.3 to include a requirement to this effect.

10. Proposed Revision to Sections 6.1.2.3 and 9.4.4.1

Item 10 of Appendix A to the Settlement Agreement states, ''Sections 6.1.2.3 and 9.4.4.1 currently recommend the analysis of bottle blanks. The notice would invite comment on whether to change 'should' to 'must' to require analysis of bottle blanks in these sections.'' Revision D, as proposed, changes the 'should' to 'must' in Section 6.1.2.3 and also changes Section 9.4.7 to correspond to this requirement because the requirements for bottle blanks are presented in this section.

11. Proposed Revision to Section 7.2

Item 11 of Appendix A to the Settlement Agreement states, ''Section 7.2 currently lists two ways to assure that laboratory air is low in both particulate and gaseous mercury: use of outside air that is very low in mercury and use of inside air recycled through a gold-coated filter. As presently written, outside air 'should' be brought into the class-100 clean bench air intake. The notice would invite comment on whether to change this 'should' in the outdoor air option to a 'must.''' Revision D, as proposed, changes the 'should' to 'must' in Section 7.2.

12. Proposed Revision to Section 8.5.3

Item 12 of Appendix A to the Settlement Agreement states, ''Section 8.5.3 currently requires handling of samples in a mercury-free clean bench. The notice would invite comment on whether to change 'should' to 'must.''' Revision D, as proposed, changes the 'should' to 'must' in Section 8.5.3.

13. Proposed Revision to Note at Section 8.5.3

Item 13 of Appendix A to the Settlement Agreement states ''Section 8.5.3 (note) currently states that samples 'should' be filtered and preserved in accordance with the procedures in Method 1669. The notice would invite comment on whether to change the 'should' to 'must' but only for the provisions of Method 1669 related to filtration and preservation of samples when circumstances prevent overnight sample shipment (i.e., sections 2.9 and 2.10 of EPA Method 1669).'' Revision D, as proposed, changes the 'should' to 'must' in the Section 8.5.3 note.

14. Proposed Revision to Section 8.6

Item 14 of Appendix A to the Settlement Agreement states, ''Section 8.6 currently suggests that sample bottles should be stored in clean (new) polyethylene bags until sample analysis. The notice would invite comment on whether to change this section to require storage in clean bags by changing 'should' to 'must.''' Revision D, as proposed, changes the 'should' to 'must' in Section 8.6.

15. Proposed Revision to Section 9.4.4.2

Item 15 of Appendix A to the Settlement Agreement states, ''Section 9.4.4.2 currently suggests the use of 'Clean Hands/Dirty Hands' when preparing sampler check blanks at the laboratory or cleaning facility. The notice would invite comment on whether to change 'should' to 'must' in this section for low-level mercury measurements.'' Revision D, as proposed, makes this change in Section 9.4.6.1 because the ''clean hands/dirty hands'' technique is referenced in that section.

16. Proposed Revision to Section 11.1.2

Item 16 of Appendix A to the Settlement Agreement states, ''Section 11.1.2 currently suggests that there should be 2 matrix spike/matrix spike duplicate pairs for each analytical batch of 20 samples. The notice would invite comment on whether to make the matrix spike/matrix spike duplicate pairs mandatory by changing 'should' to 'must be a minimum of' in this section.'' Revision D, as proposed, makes this change in Section 11.1.2.

B. Election by a Permittee/Industrial User

In EPA's ''Guidance for the Implementation and Use of EPA Method 1631 for the Determination of Low Level Mercury (40 CFR Part 136),'' dated March 2001, EPA recommends that State and Federal agencies measuring ambient water quality for compliance with water quality standards at very low concentrations should require, as a matter of internal agency protocol, that their personnel use clean techniques. EPA also suggests in this guidance, that NPDES permits specify the use of clean techniques, on a permit-by-permit basis, depending on the measurement level of concern, upon request by the permit applicant. The guidance also states that EPA will propose additional requirements for clean techniques by October 2001, and that EPA may revise the guidance in accordance with any requirements that are promulgated as a result.

Clause 4 of the Settlement Agreement requires EPA to propose that an NPDES permittee or an industrial user of a POTW may elect not to implement the clean techniques and QC provisions that are listed in Appendix A to the Settlement Agreement and that are proposed today ''in its discretion and at its peril, unless specifically provided otherwise by the relevant permitting agency or pretreatment control authority, as the case may be.'' Revision D, as proposed, includes this election in Section 1.13.

As required by the Settlement Agreement, the election is applicable to the clean techniques and QC provisions designated in Appendix A to the Settlement Agreement only. These provisions are discussed in Section IV.A of this notice and are designated throughout draft EPA Method 1631D by bracketed and italicized text. If Section 1.13 is promulgated as proposed, any text pertaining to clean techniques and quality control provisions that is also promulgated as proposed under the Settlement Agreement, would remain italicized and bracketed in the approved version of EPA Method 1631D to designate the techniques and provisions to which the election is applicable.

Users of EPA Method 1631 should be aware that the election in Clause 4 of the Settlement Agreement would apply to the permittee/industrial user only, and not to a regulatory/control authority or to other users of the Method. Regulatory/control authorities and other users of EPA Method 1631 would be required to use the clean techniques and QC provisions as designated by the italicized bracketed text in the affected sections of EPA Method 1631D. Permittees/industrial users should be aware of the potential disparity that could result if a sample is analyzed by a permittee/industrial user not using the clean techniques and QC provisions and also by a regulatory/control authority using the clean techniques and QC provisions. In addition, if a regulatory/control authority requires that a permittee/industrial user use the clean techniques and QC provisions, the burden would be on the regulatory/control authority to incorporate this requirement into regulations or permits.

EPA is soliciting comments on this election and on the specific techniques and provisions to which the election would be applicable. EPA is soliciting comments particularly from permittees/industrial users because the users would have the election, and from regulatory/control authorities and other users of EPA Method 1631 because they would not. EPA also seeks comment on whether the philosophical change embodied by the election (*i.e.,* to allow a permittee/industrial user to not use certain techniques and QC provisions of an analytical method, yet require regulatory/control authorities and other users to use these techniques) is desirable, in general. In addition, EPA solicits comments on alternatives to the Settlement Agreement approach, mainly on whether the additional clean techniques and QC requirements should be applicable to all users or whether the additional requirements should be optional for all users.

V. Proposed Additional Revisions to EPA Method 1631

Since promulgation of EPA Method 1631 in June 1999, EPA has received many suggestions for method improvement and requests to clarify certain method procedures. In today's action, EPA is proposing revisions to clarify and improve the method in

response to these comments. This section explains the revisions included in draft EPA Method 1631D in response to these comments. EPA is soliciting comment on the proposed revisions described below.

A. Use of Automated Flow-Injection Systems

Automated flow-injection systems are currently available and have been used successfully for performing EPA Method 1631 procedures for several years. These systems use flow injection and a gas-liquid separator in place of the bubbler.

EPA has worked with several users of these systems to develop appropriate calibration and calculation procedures and blank sample requirements and has included these procedures and requirements in draft Method 1631D. The revisions incorporate calibration blanks (Section 9.4.2), calibration procedures (Section 10.2), and result calculation procedures (Section 2.2) that are specific for flow injection systems. The proposed method also refers to flow injection systems throughout the text, and includes a figure depicting the flow-injection system (Figure 3). The revisions will expand the use of the Method by providing appropriate procedures for the use of automated flow injection systems.

B. Blanks

EPA Method 1631C includes the use of bubbler blanks (Section 9.4.1), reagent blanks (Section 9.4.2), field blanks (Section 9.4.3), equipment blanks (Section 9.4.4), bottle blanks (Section 9.4.4.1), and sampler check blanks (Section 9.4.4.1). Several commenters noted that the blanks are not well defined and that blank requirements are inconsistent with common usage. In addition, commenters noted that the types and requirements for blanks are not appropriate for use with the flow-injection systems. To address these comments, EPA added definitions for all blanks in the Glossary of draft Method 1631D, and clarified these definitions throughout the Method text. EPA has also added requirements for calibration blanks (for use with flow injection systems only) and method blanks (for use with both bubbler and flow injection systems). The proposed revisions are discussed in the following sections.

1. Definitions

In response to several comments that the types of blank samples required by Method 1631 are defined inconsistently throughout the Method, EPA revised Section 17 of draft Method 1631D to include definitions for the calibration blank, method blank, reagent blank, field blank, and bottle blank samples. Section 17 of draft Method 1631D also includes a revised definition of the bubbler blank to clarify its specificity for use with bubbler systems. In addition, EPA revised Section 9.4 in draft Method 1631D to further clarify definitions and use of blank samples for both bubbler and flow-injection systems.

The proposed revisions address more accurately EPA's intent to allow the use of both bubbler and flow injection systems for determination of mercury using Method 1631. The revisions also clarify the application and use of blank samples to identify and handle potential contamination.

2. Calibration Blanks

In EPA Method 1631, bubbler blanks are used to establish a background for the bubbler system (*i.e.,* bubbler, traps, and cold-vapor atomic fluorescence detector) and can be used to identify potential carryover from one sample to the succeeding sample (see Section 9.4.1 of EPA Method 1631C). Results of bubbler blanks are subtracted from all raw calibration and sample results. Bubbler blanks, however, are not appropriate for flow injection systems. Hence, EPA added a requirement for calibration blanks, when using a flow injection system. The performance criteria and application requirements of the calibration blanks are identical to those for the bubbler blanks.

3. Method and Reagent Blanks

EPA Method 1631 requires reagent blanks to identify contamination from reagents, but these blanks are required only when a new batch of reagents is prepared, with verification in triplicate each month, and are not required with each analytical batch (see Section 9.4.2 of EPA Method 1631C). Method 1631 also requires field blanks to identify contamination from sample collection and transport (see Section 9.4.3 of EPA Method 1631C). These field blanks may be used to identify contamination introduced at some point during the entire measurement process from sample collection through mercury detection, but cannot isolate contamination caused by sample collection and transport from contamination that is introduced during sample processing and analysis.

Several method users commented that laboratories typically use method blanks to determine potential contamination in the analytical system during sample preparation and analysis. These method blanks are prepared and analyzed using procedures identical to those used to prepare and analyze the corresponding samples.

Because method blanks can be used to identify total analytical system contamination, and are subjected to all sample processing and analytical steps including digestion, reduction, and determination, EPA added a requirement for method blanks to draft Method 1631D. The proposed method includes a requirement that at least three method blanks be analyzed with each analytical batch. It also includes a requirement that any sample requiring increased oxidation (*e.g.,* an increased amount of reagent) be associated with at least one method blank that is processed and analyzed using the same amount of increased oxidation. The performance criteria for the method blanks is identical to the field blank criteria. This requirement provides method users with a more appropriate procedure for addressing contamination that may result during the entire analytical procedure.

EPA also proposes to revise the requirement for the frequency of reagent blanks. In draft Method 1631D, analysis of reagent blanks is required only when each new batch of reagents is prepared. EPA believes that the requirements for method blanks included in draft Method 1631D will be sufficient to identify contamination that may be introduced by reagent solutions during processing and analysis of an analytical batch.

4. Equipment and Bottle Blanks

EPA received several comments on Method 1631 expressing confusion over the use of the terms ''equipment blank,'' ''sampler check blank,'' and ''bottle blank.'' Commenters also noted that the terms ''equipment blank'' and ''sampler check'' blank are synonymous and that using two terms to identify blanks used to check sample collection equipment is confusing. Additionally, commenters were concerned that bottle blanks are listed in EPA Method 1631 under blanks specific for determination of contamination in sample collection equipment. These commenters noted that bottle blanks also are necessary to determine contamination in bottles used for sample preparation and analysis, and recommended that bottle blanks be analyzed at a frequency of at least 20 percent of each lot used.

In response to these comments, EPA proposes to change the term ''sampler check blank'' to ''equipment blank'' in Section 9.4.6 of draft Method 1631D, and to revise Section 9.4 of the method to expand the application of bottle blanks for determination of contamination in bottles used for both sample collection and analysis (Section

9.4.7). EPA also included a requirement in draft Method 1631D that a minimum of 20 percent of the bottles from a given lot shall be tested and demonstrated to be free of mercury at the Method MDL (Section 6.1.2.4).

C. Calibration Over a Different Range

Several users of Method 1631 stated that they prefer to use EPA Method 1631 for mercury determination because it is less prone to interferences than other available methods, and would like to apply EPA Method 1631 procedures across a higher calibration range. Other users of the method commented that when they are analyzing samples known to be within a narrower range of concentrations than that of the current method (*e.g.,* an analytical run consisting of ambient samples), they prefer to calibrate the analytical system across the narrower range. Additional users noted that they desire to calibrate to a lower point to measure mercury in blanks to a lower level.

In response to these comments and to allow expanded use of EPA Method 1631, EPA included a provision in draft Method 1631D to allow calibration over ranges other than the range currently specified (Section 10.4). EPA included certain criteria with this provision to ensure that this allowance does not compromise data quality. These criteria are: (1) there must be a minimum of five, non-zero calibration points; (2) the difference between successive calibration points must be no greater than a factor of 10 and no less than a factor of 2 and should be approximately evenly spaced on a logarithmic scale over the calibration range; (3) the relative standard deviation (RSD) of the calibration factors for all calibration points must be less than 15%; (4) the calibration factor for any calibration point at a concentration greater than 100 ng/L must be within plus or minus 15% of the average calibration factor for the points at or below 100 ng/L; (5) the calibration factor for any point less than 5 ng/L must be within plus or minus 25% of the average calibration factor for all points; (6) if the highest calibration point is increased above 100 ng/L, the lowest calibration point (ML) must be increased commensurately above 0.5 ng/L; and, (7) if the calibration is to a higher range and this Method is used for regulatory compliance, the ML must be less than one-third the regulatory compliance limit.

D. Sample Preservation, Refrigeration, Headspace, Collection Containers, and Storage

Section 8.5 of the currently approved EPA Method 1631 requires that samples are preserved upon collection, or alternatively, are collected only in fluoropolymer bottles, with zero headspace, capped tightly, and stored at 0–4°C until they can be preserved within 48 hours of collection.

Since promulgation of EPA Method 1631, EPA has received numerous comments on the sample preservation, refrigeration, headspace, and holding time requirements in the method and in Table II at 40 CFR 136.3(e). EPA has considered these comments and has included revisions in today's draft Method 1631D to address the recommendations from method users. Specific proposed revisions regarding preservation, refrigeration, container type, headspace, and holding time requirements are discussed below. The proposed changes are based on the comments received from method users and EPA requests data to support whether the changes would affect the quality of results. EPA is not proposing to revise the requirements that samples collected for determination of total mercury must be capped tightly and must be preserved or analyzed within 48 hours of collection. EPA is requesting comment on whether this requirement should be kept.

1. Sample Preservation

Sections 2.3 and 8.5 of EPA Method 1631 currently require that samples are either preserved with hydrochloric acid (HCl) or bromine monochloride (BrCl) solution immediately upon collection, or alternatively, are collected and stored under specific conditions (i.e., zero headspace, fluoropolymer bottles, capped tightly, and stored at 0–4°C) until they can be preserved in the laboratory within 48 hours of collection. As discussed in Section IV.A.2, EPA is also proposing to revise the sample preservation requirement in Sections 2.3 and 8.5 of the method for the determination of methyl mercury.

Commenters claim there is no need to preserve samples for total Hg if BrCl is added to the sample in the laboratory and the sample is allowed to stand for a minimum of 24 hours to oxidize all forms and species of mercury to $Hg^{(II)}$. Commenters also noted that the immediate preservation of samples collected for total or dissolved mercury determination is unnecessary, provided the samples are preserved or analyzed within 48 hours of collection, and have requested elimination of the requirement for preservation so that solutions of HCl or BrCl do not need to be shipped to the sampling site.

EPA currently is reviewing data that indicate that unpreserved samples collected for measurement of low level mercury may be stable for as long as 35 days. Additionally, EPA does not have data demonstrating that results of samples for total or dissolved mercury that are not preserved immediately are compromised, and solicits such data to determine whether immediate preservation should be required. Therefore, EPA has included revisions in draft Method 1631D (Section 8.5) to eliminate the requirement for immediate preservation of samples collected for determination of mercury using EPA Method 1631. Today's proposed method does not include a revision to the requirement for immediate preservation of samples collected for methyl- and dimethyl mercury determination.

EPA is also proposing to amend Table II of 40 CFR 136.3(e) to include requirements for preservation of samples collected for mercury measurement using Method 1631 within 48 hours of sample collection, using BrCl or HCl (see Section VI of this notice).

2. Sample Refrigeration

Users of EPA Method 1631 claim that there is no need to refrigerate unpreserved samples for total or dissolved mercury because the bromine monochloride (BrCl) digestion converts all forms of Hg to $Hg^{(II)}$. Therefore, if a given form or species of Hg were converted to another form or species in the absence of refrigeration (e.g., through biological activity), the BrCl digestion would convert the new form, as well as any remaining portion of the old form, to $Hg^{(II)}$. Commenters have requested that EPA eliminate the requirement for refrigeration of unpreserved samples because of costs and logistics problems (i.e., refrigeration requires purchase of ice, shipment of the sample in a cooler, and testing of the sample at the laboratory to make certain that the temperature remains in the range specified, 0–4 °C).

EPA currently does not have data demonstrating that refrigeration of unpreserved samples for measurement of total or dissolved mercury using Method 1631 is necessary, and is seeking such data to determine if refrigeration should be required. In the absence of data, EPA has revised draft Method 1631D (Section 8.5) to eliminate the requirement for refrigeration of unpreserved samples, provided that the sample is tightly capped and is either preserved or analyzed within 48 hours of collection.

EPA also has received comments that samples collected for measurement of mercury using Method 1631 are stable for up to 30 days prior to either preservation or analysis. EPA is

requesting data to support this comment. EPA will consider submitted data, and if appropriate, will re-evaluate the requirement for preservation or analysis of samples within 48 hours.

3. Sample Headspace

Section 8.5 of EPA Method 1631 requires that mercury samples are collected with zero headspace if they are not preserved immediately. A laboratory involved in the development of EPA Method 1631 commented that, although it is necessary to collect samples for methyl- and dimethyl-mercury with zero headspace, it is not necessary to collect samples for total or dissolved mercury with zero headspace. For total or dissolved mercury, the partitioning of volatile forms of mercury into a relatively small headspace volume is negligible.

EPA has removed the requirement for collecting unpreserved samples with zero headspace in draft Method 1631D (Section 8.5.1), provided the sample is tightly capped and is preserved or analyzed within 48 hours of sample collection. The proposed method does not include a revision of the requirement to collect samples for methyl mercury with no headspace.

4. Sample Collection Containers

Section 8.5.1 of EPA Method 1631 requires that unpreserved samples must be collected in fluoropolymer sample containers. Several users of EPA Method 1631 have commented that, in addition to fluoropolymer bottles, glass bottles can be used successfully for collection of unpreserved samples, provided the containers are demonstrated to be clean, are tightly capped, and are preserved or analyzed within 48 hours of sample collection.

EPA is soliciting data demonstrating that the use of glass containers for collection of mercury samples that are not preserved immediately does not compromise the quality of results obtained using EPA Method 1631. EPA has revised Sections 2.1, 4.3.7.1, and 8.5.1 in draft Method 1631D, to allow collection of unpreserved samples in either clean fluoropolymer or clean glass sample containers.

As discussed previously in Section V.D.1 of this document, EPA is requesting data to support the comment that samples collected for measurement of mercury using Method 1631 are stable for up to 30 days prior to preservation or analysis. EPA will consider submitted data, and if appropriate, will re-evaluate the requirement for preservation or analysis of samples within 48 hours.

5. Holding Time

Section 8.5 of EPA Method 1631 states that acid- and BrCl-preserved samples are stable for a period of 28 days. Several laboratories that assisted in the development of EPA Method 1631 believe that samples are stable for at least three months and have provided data to EPA demonstrating this stability in mercury samples that have been preserved with either BrCl or HCl. These data are included in the Record supporting today's rule.

EPA revised Section 8.5 of draft Method 1631D to recognize that acid- or BrCl-preserved samples that are collected for measuring mercury using Method 1631 are stable for a period of 90 days. EPA is also proposing to amend Table II of 40 CFR 136.3(e) to include a maximum holding time of 90 days for samples collected for determination of mercury using Method 1631 (see Section VI of this preamble).

E. Shipment of Empty Sample Containers

Section 6.1.2.1 of EPA Method 1631 requires that sample bottles be filled with 0.4% HCl solution and stored until use. EPA Method 1631 also references Section 6.3.1 of EPA Method 1669, which suggests that clean sample bottles should be filled with reagent water for shipment to the sampling site prior to sample collection.

Commenters have stated that EPA Methods 1631 and 1669 should allow shipment of empty sample bottles to avoid shipping acid and to save shipping weight. As with sample refrigeration and preservation, EPA is soliciting data demonstrating whether shipping sample bottles full of dilute acid or reagent water is necessary and should be required. We have revised Section 6.1.2.1 in draft Method 1631D to allow shipment of empty bottles for sample collection based on comments from method users.

F. Scope of ''Should'' and ''May''

The introduction to EPA Method 1631 contains a note that addresses the performance-based aspects of the Method. The note states that the terms ''shall'' and ''must'' define procedures required for producing reliable data at water quality criteria levels and that 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 the unmodified method. As discussed in Section IV of today's notice, EPA is proposing additional requirements for clean techniques and quality control that would, if implemented, change certain ''should'' and ''may'' to ''shall'' and ''must.''

Some commenters have interpreted the terms ''should'' and ''may'' as limiting the applicability of the performance-based allowances in the Method. EPA does not intend this restriction. As stated in Sections 1.8 and 9 of EPA Method 1631, any procedure may be modified, except for procedures required as defined by the terms ''shall'' and ''must'' and all QC tests.

To preclude ambiguity, EPA has revised the note in draft Method 1631D to clarify that the laboratory is permitted to omit steps or modify procedures provided that all performance requirements in this Method are met, but that the laboratory must not omit or modify any procedure defined by the term ''shall'' or ''must'' and must perform all quality control tests.

G. Field Filtration for Dissolved Metals

Both EPA Method 1669 (Section 8.3) and the current version of EPA Method 1631 (Method 1631C, Section 8.5.3) recommend that filtration of samples collected for dissolved Hg should be performed in the clean room of the laboratory. In contrast, 40 CFR 136.3(e), Table II, footnote 7, says that samples for dissolved metals should be filtered immediately on-site before adding preservative.

Since promulgation of EPA Method 1631, some commenters have noted that it is preferable to filter samples for dissolved Hg in the laboratory under controlled clean conditions. Other commenters, however, have noted that it is preferable to filter samples for dissolved Hg immediately upon collection, thereby allowing for in-line filtration and immediate preservation if desired.

EPA believes that filtration of mercury samples in either the field or laboratory is appropriate, provided the filtration is performed in a clean area, and provided that samples are accompanied by a blank that has been filtered under the same conditions. EPA has revised Sections 2.2 and 8.4 of draft Method 1631D to allow for both in-field and laboratory sample filtration under these provisions.

EPA is also proposing to amend 40 CFR 136.3(e) Table II to include requirements for filtration of samples for measurement of dissolved mercury using Method 1631, in a clean area in the laboratory or in the field (see Section VI of this preamble for proposed revisions to 40 CFR 136.3(e), Table II).

H. Carryover Test

The bubbler blank carryover test in EPA Method 1631 is recommended ''when an unusually concentrated sample is encountered'' (Method 1631C, Section 4.3.8.1) or ''after very high samples'' (Method 1631C, Section 11.2.4).

Several commenters stated that ''unusually concentrated'' and ''high sample'' are not defined. Commenters also have noted that it may not be practical to stop a run and analyze a bubbler blank immediately after these samples. Often a sample is determined to have a high Hg concentration that could result in carryover only after subsequent samples have been analyzed or at the completion of an analytical batch.

To quantify the concentration of mercury in a sample that would carry a concentration into a subsequent sample, EPA has included a carryover test in draft Method 1631D (Section 4.3.8.1) that is similar to that in Section 8.5.1 of EPA Method 1624B for analysis of volatile organic compounds using a gas chromatograph/mass spectrometer (40 CFR Part 136, appendix A). In this test, successively greater concentrations of mercury in reagent water are analyzed to determine the concentration at which more than 0.2 ng/L (the MDL in Method 1631) would be measured in a subsequent bubbler blank.

EPA also has included revisions in draft Method 1631D (Section 4.3.8.1 and Section 11.2.1.3) to require that when an unusually concentrated sample is encountered, a bubbler blank must be analyzed to check for carryover and that samples run immediately following a sample that has been determined to result in carryover must be reanalyzed using a bubbler that is demonstrated to be free of Hg at the 0.2 ng/L level.

I. Correction of Part Numbers

Users of Method 1631 have informed EPA that the supplier does not recognize part numbers for the peristaltic pump or tubing suggested in the method. The supplier has informed us that the leading letter in the part number signifies the version of the catalog and should be omitted from the part number. EPA has corrected these part numbers in proposed Method 1631D (Sections 6.1.3.2 and 6.1.3.3).

J. Use of Polyethylene or Polypropylene Vessels for Sample Digestion

Most methods for determination of metals allow polyethylene bottles as sample containers (see 40 CFR Part 136, Table II, footnote 1). EPA Method 1631 requires use of glass or fluoropolymer because mercury can diffuse in and out of polyethylene bottles (see Section 16.4 of EPA Method 1631).

Commenters have stated that, although fluoropolymer or glass bottles are necessary for sample collection, this type of labware is not necessary for sample digestion and other laboratory uses, because mercury will not diffuse through these materials in the relatively short time during which the sample is analyzed. Because polyethylene and polypropylene is less expensive than fluoropolymer and is less susceptible to breakage than glass, EPA has included a revision to this requirement in draft Method 1631D (Section 4.3.7.1) to allow use of polyethylene or polypropylene labware for sample digestion and preparation, but not for sample collection.

K. Indication of Complete Oxidation

Section 8.1 of EPA Method 1631 currently states that the pH of all aqueous samples must be tested immediately before analysis to ensure that the sample has been properly preserved.

Users of EPA Method 1631 have noted that the pH of a sample provides an insufficient indication of whether or not the sample is completely oxidized or ready for analysis, and cite as an example, samples containing high concentrations of sulfides or other reducing compounds that can consume BrCl, but still have a pH less than 2. These commenters have stated that color is a better indication of complete oxidation (see Sections 11.1.1.1 and 11.1.1.2 of EPA Method 1631).

EPA proposes to revise Section 8.1 in EPA Method 1631D to recognize that samples must be completely oxidized prior to direct analysis and that pH alone is not sufficient for determination of complete oxidation.

L. Adjustment for Amount of Bromine Monochloride to Blanks

Section 9.4.2.2 of EPA Method 1631 currently requires that the amount of reagent that is added to a reagent blank must be the same as the amount of reagent that is added to the samples. One of the laboratories responsible for the development of EPA Method 1631 commented that this requirement is inconsistent with Section 12.3 which allows adjustment during calculation of reagent blanks for greater amounts of reagent that may be added to samples requiring increased oxidation. Users also have commented that, although the correction allowed in Section 12.3 is appropriate for volume adjustment, it is not necessarily appropriate for adjustment of increased reagent concentration.

In draft Method 1631D, EPA has clarified that a sample requiring increased oxidation via an increased amount of reagents must be associated with at least one blank sample that has been analyzed using procedures identical to those used to prepare and analyze the sample. This requirement is included with the requirements for method blanks (draft Method 1631D, Section 9.4.4.3).

M. Addition of Method 1631 Guidance as a Reference

On March 2001, EPA published guidance to assist users with the implementation and use of EPA Method 1631. This guidance, Guidance for Implementation and Use of EPA Method 1631 for the Determination of Low-Level Mercury (40 CFR Part 136) (EPA 821–R–01–023, March 2001), was developed and published largely in response to the October 19, 2000 Settlement Agreement. EPA has added a reference for this guidance to draft Method 1631D (Section 16.22).

VI. Proposed Amendment to 40 CFR 136.3(e) Table II

EPA is today proposing to amend Table II at 40 CFR 136.3(e), which lists required containers, preservation techniques, and maximum holding times for biological and chemical parameters. This amendment provides consistency with previously approved requirements in EPA Method 1631 and with requirements proposed today (see Section V of this preamble). This proposal would add a footnote (17) to Table II to include requirements for collection, filtration, preservation, and maximum holding times that are specific to samples collected for determination of mercury using EPA Method 1631. This footnote would include the following requirements for mercury samples: samples must be collected using either fluoropolymer or glass containers, samples must be preserved with either HCl or BrCl within 48 hours of collection, preserved samples have a maximum holding time of 90 days, and samples must be filtered in a clean area in the laboratory or in the field prior to sample preservation. EPA invites comment on the proposed text to be added to Table II at 40 CFR 136.3(e).

VII. Administrative Requirements

A. Executive Order 12866—Regulatory Planning and Review

Under Executive Order 12866 [58 FR 51735 (October 4, 1993)], the Agency must determine whether the regulatory

action is ''significant'' and therefore subject to Office of Management and Budget (OMB) review and the requirements of the Executive Order. The Executive Order defines ''significant regulatory action'' as one that is likely to result in a rule that may:

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

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

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

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

It has been determined that this rule is not a ''significant regulatory action'' under the terms of Executive Order 12866 and is therefore not subject to OMB review.

B. Regulatory Flexibility Act (RFA), as Amended by the Small Business Regulatory Enforcement Fairness Act of 1996 (SBREFA), 5 U.S.C. 601 et seq.

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

For purposes of assessing the impacts of today's rule on small entities, small entity is defined as: (1) A small business as defined by the U.S. Small Business Administration definitions at 13 CFR 121.201; (2) a small governmental jurisdiction that is a government of a city, county, town, school district or special district with a population of less that 50,000; and (3) a small organization that is any not-for-profit enterprise which is independently owned and operated and is not dominant in its field.

After considering the economic impacts of today's proposed rule on small entities, I certify that this action will not have a significant economic impact on a substantial number of small entities. Today's rule proposes a revised version of a currently approved EPA Method to include additional requirements for clean techniques and quality control and to improve and clarify method procedures. Today's rule also proposes an amendment to Table II at 40 CFR 136.3(e) to provide consistency with previously approved requirements in Method 1631 and with revisions proposed today for collection, preservation, and storage of samples collected for determination of mercury using Method 1631 procedures.

Overall, the cost of these revisions are minimal. While some of the revisions may increase cost (e.g., clean technique and quality control requirements), others will provide flexibility and actually lower the overall analytical costs (e.g., use of new, less expensive equipment). Only NPDES permitting authorities must use the clean techniques. Permittees, including small entities, are not required to use them unless required to do so by their permitting authority. Many of the laboratories that analyze for mercury are already using the clean techniques, further minimizing any potential cost increases. EPA estimates that any costs associated with clean techniques would be alleviated or eliminated by the additional flexibility resulting from some of the proposed revisions to the Method that are discussed in Section V. Therefore, EPA believes that this proposed rule will not have a significant economic impact on a substantial number of small entities. We continue to be interested in the potential impacts of the proposed rule on small entities and welcome comments on issues related to such impacts.

C. Unfunded Mandates Reform Act

Title II of the Unfunded Mandates Reform Act of 1995 (UMRA), Public Law 104–4, establishes requirements for Federal agencies to assess the effects of their regulatory actions on State, Tribal, and local governments and the private sector. Under section 202 of the UMRA, EPA generally must prepare a written statement, including a cost-benefit analysis, for proposed and final rules with ''Federal mandates'' that may result in expenditures to State, Tribal, and local governments, in the aggregate, or to the private sector, of $100 million or more in any one year.

Before promulgating an EPA rule for which a written statement is needed, section 205 of the UMRA generally requires EPA to identify and consider a reasonable number of regulatory alternatives and adopt the least costly, most cost-effective or least burdensome alternative that achieves the objectives of the rule. The provisions of section 205 do not apply when they are inconsistent with applicable law. Moreover, section 205 allows EPA to adopt an alternative other than the least costly, most cost-effective or least burdensome alternative if the Administrator publishes with the final rule an explanation of why that alternative was not adopted. Before EPA establishes any regulatory requirements that may significantly or uniquely affect small governments, including Tribal governments, it must have developed under section 203 of the UMRA a small government agency plan. The plan must provide for the notification of potentially affected small governments, enabling officials of affected small governments to have meaningful and timely input in the development of EPA regulatory proposals with significant Federal intergovernmental mandates, and informing, educating, and advising small governments on compliance with the regulatory requirements.

EPA has determined that today's proposed rule does not contain a Federal mandate that may result in expenditures of $100 million or more for State, Tribal, and local governments, in the aggregate, or the private sector in any one year. This rule proposes revisions to a previously approved method for measuring mercury in wastewater. This rule also proposes to revise Table II at 40 CFR 136.3(e) to clarify requirements for sample collection, preservation, and storage, and to make these requirements consistent with previously approved requirements in EPA Method 1631 and with today's proposed method revisions. As discussed in Section VII.B regarding RFA analysis, EPA expects the cost of these revisions to Method 1631 to be minimal. Thus, today's rule is not subject to sections 202 and 205 of the UMRA. For the same reasons, EPA has also determined that this rule contains no regulatory requirements that might significantly or uniquely affect small governments. Thus, today's rule is not subject to the requirements of section 203 of the UMRA.

D. Paperwork Reduction Act

This action does not impose an information collection burden under the provisions of the Paperwork Reduction Act, 44 U.S.C. 3501 *et seq.* This rule proposes to revise a currently approved test method for use in water monitoring programs but does not require the use of the test method.

Burden means the total time, effort, or financial resources expended by persons to generate, maintain, retain, or disclose or provide information to or for a Federal agency. This includes the time needed to review instructions; develop,

acquire, install, and utilize technology and systems for the purposes of collecting, validating, and verifying information, processing and maintaining information, and disclosing and providing information; adjust the existing ways to comply with any previously applicable instructions and requirements; train personnel to be able to respond to a collection of information; search data sources; complete and review the collection of information; and transmit or otherwise disclose the information.

An Agency may not conduct or sponsor, and a person is not required to respond to a collection of information unless it displays a currently valid OMB control number. The OMB control numbers for EPA's regulations are listed in 40 CFR Part 9 and 48 CFR Chapter 15.

E. National Technology Transfer and Advancement Act

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

This proposed rulemaking involves technical standards. Therefore, the Agency conducted a search to identify potentially applicable voluntary consensus standards. However, we identified no standard for the measurement of mercury at low water quality criteria levels or for the use of ''clean techniques.'' Therefore, EPA proposes to use EPA Method 1631, Revision D: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. EPA welcomes comments on this aspect of the proposed rulemaking and, specifically, invites the public to identify potentially applicable voluntary consensus standards for measuring low levels of mercury and for ''clean techniques'' and to explain why such standards should be used in this regulation.

F. Executive Order 13045—Protection of Children From Environmental Health Risks and Safety Risks

Executive Order 13045 (62 FR 19885, April 23, 1997) applies to any rule that: (1) is determined to be ''economically significant'' as defined under Executive Order 12866, and (2) concerns an environmental health or safety risk that EPA has reason to believe may have a disproportionate effect on children. If the regulatory action meets both criteria, the Agency must evaluate the environmental health or safety effects of the planned rule on children, and explain why the planned regulation is preferable to other potentially effective and reasonably feasible alternatives considered by the Agency. This proposed rule is not subject to the Executive Order because it is not economically significant as defined in Executive Order 12866, nor does it concern an environmental health or safety risk that EPA has reason to believe may have a disproportionate effect on children.

G. Executive Order 13132—Federalism

Executive Order 13132, entitled ''Federalism'' (64 FR 43255, August 10, 1999), requires EPA to develop an accountable process to ensure ''meaningful and timely input by State and local officials in the development of regulatory policies that have federalism implications.'' ''Policies that have federalism implications'' is defined in the Executive Order to include regulations that have ''substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government.''

This proposed rule does not have federalism implications. It will not have substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government, as specified in Executive Order 13132. Today's rule proposes revisions to EPA's Method 1631, Revision C, for measuring mercury at low levels for compliance monitoring under the Clean Water Act. As discussed in Section VII.B regarding RFA analysis, EPA expects the cost of these revisions to Method 1631 to be minimal. Thus, Executive Order 13132 does not apply to this rule.

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

H. Executive Order 13175—Consultation and Coordination With Indian Tribal Governments

Executive Order 13175, entitled ''Consultation and Coordination With Indian Tribal Governments'' (65 FR 67249, November 6, 2000), requires EPA to develop an accountable process to ensure ''meaningful and timely input by tribal officials in the development of regulatory policies that have tribal implications.'' ''Policies that have tribal implications'' is defined in the Executive Order to include regulations that have ''substantial direct effects on one or more Indian tribes, on the relationship between the Federal government and the Indian tribes, or on the distribution of power and responsibilities between the Federal government and Indian tribes.''

This proposed rule does not have tribal implications. It will not have substantial direct effects on tribal governments, on the relationship between the Federal government and Indian tribes, or on the distribution of power and responsibilities between the Federal government and Indian tribes, as specified in Executive Order 13175. As discussed in Section VII.B regarding RFA analysis, EPA expects the cost of these revisions to Method 1631 to be minimal. Thus, Executive Order 13175 does not apply to this rule.

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

I. Executive Order 13211: Actions Concerning Regulations That Significantly Affect Energy Supply, Distribution, or Use

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

J. Plain Language Directive

Executive Order 12866 requires each agency to write all rules in plain language. We invite your comments on how to make this proposed rule easier to understand. For example, have we organized the material to suit your needs? Are the requirements in the rule clearly stated? Does the rule contain technical language or jargon that isn't clear? Would a different format (grouping and order of sections, use of

headings, paragraphing) make the rule easier to understand? Would more (but shorter) sections be better? Could we improve clarity by adding tables, lists, or diagrams? What else could we do to make the rule easier to understand?

VIII. Request for Comments

EPA encourages public participation in this rulemaking and is requesting comments on the various EPA Method 1631 revisions detailed in the proposal. EPA is also requesting data supporting comments, if available. Specifically, EPA is soliciting comments on: the proposed requirements for certain quality control and clean techniques that are currently recommended in the method and that are detailed in Section IVA of this preamble; the proposal to allow a discharger to elect not to implement the requirements at Section IVA; the proposed revisions to Method 1631 that address stakeholder comments and are detailed in Section V of this preamble; the proposed amendment to 40 CFR 136.3(e) Table II to include requirements for preservation and storage that are specific to aqueous samples collected for measurement of mercury using Method 1631; and the testing costs that may be associated with any of the proposed method modifications.

To ensure that EPA can properly respond to comments, commenters should cite, where possible, the paragraph(s) or section(s) in this proposal or in Method EPA 1631 to which each comment refers.

List of Subjects at 40 CFR Part 136

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

Dated: September 28, 2001

Christine Todd Whitman,

Administrator.

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

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

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

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

2. Section 136.3 is amended by revising paragraph (b) (41) and by revising the ''Metals'' entry in Table II of paragraph (e) to read as follows:

§ 136.3 Identification of test procedures.

* * * * *

(b) * * *

(41) USEPA. 2001. Method 1631, Revision D, ''Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry.'' September 2002. Office of Water, U.S. Environmental Protection Agency (EPA–821–R–xx–xxx). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161. Publication No. PB2001–xxxxxx. Cost: $25.50 (subject to change). Table IB, Note 43.

* * * * *

Parameter No./name	Container [1]	Preservation [2, 3]	Maximum holding time [4]
* * *	*	* *	*
Metals			
18. Chromium VI [7]	P, G	Cool, 4°C	24 hours.
35. Mercury [17]	P, G	HNO_3 to pH<2	28 days.
3, 5–8, 12,13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals except boron, chromium VI and mercury [7].	P, G	.....do	6 months.
* * *	*	* *	*

[1] Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic), except for samples collected for trace-level mercury (see footnote 17).

[2] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed, except for samples collected for trace-level mercury (see footnote 17).

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

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. (See footnote 17 for samples collected for trace level mercury). Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory , is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details. The term ''analyze immediately'' usually means within 15 minutes or less of sample collection.

* * * * *

[7] Samples should be filtered immediately on site before adding preservative for dissolved metals, except for samples collected for trace-level mercury (see footnote 17).

* * * * *

[17] Samples collected for the determination of trace level mercury using EPA Method 1631, must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. Samples for dissolved trace level mercury must be filtered in a clean area in the field or the laboratory prior to sample preservation. Samples that have been preserved for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

[FR Doc. 01–24886 Filed 10–5–01; 8:45 am]

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