

does not require the public to perform activities conducive to the use of VCS.

I. Submission to Congress and the Comptroller General

The Congressional Review Act, 5 U.S.C. 801 *et seq.*, as added by the Small Business Regulatory Enforcement Fairness Act of 1996, generally provides that before a rule may take effect, the agency promulgating the rule must submit a rule report, which includes a copy of the rule, to each House of the Congress and to the Comptroller General of the United States. EPA will submit a report containing this rule and other required information to the U.S. Senate, the U.S. House of Representatives, and the Comptroller General of the United States prior to publication of the rule in the **Federal Register**. A major rule cannot take effect until 60 days after it is published in the **Federal Register**. This rule is not a "major" rule as defined by 5 U.S.C. 804(2).

J. Petitions for Judicial Review

Under section 307(b)(1) of the Clean Air Act, petitions for judicial review of this action must be filed in the United States Court of Appeals for the appropriate circuit by December 30, 2002. Filing a petition for reconsideration by the Administrator of this final rule does not affect the finality of this rule for the purposes of judicial review nor does it extend the time within which a petition for judicial review may be filed, and shall not postpone the effectiveness of such rule or action. This action may not be challenged later in proceedings to enforce its requirements. *See* section 307(b)(2).

List of Subjects in 40 CFR Part 52

Environmental protection, Air pollution control, Incorporation by reference, Intergovernmental relations, Ozone, Reporting and recordkeeping requirements, Volatile organic compounds.

Dated: October 1, 2002.

Laura Yoshii,  
Deputy Regional Administrator, Region IX.

Part 52, chapter I, title 40 of the Code of Federal Regulations is amended as follows:

PART 52—[AMENDED]

1. The authority citation for part 52 continues to read as follows:

Authority: 42 U.S.C. 7401 *et seq.*

Subpart F—California

2. Section 52.220 is amended by adding paragraphs (c)(279)(i)(A)(9),

(284)(i)(C)(2), and (284)(i)(D)(2) to read as follows:

§ 52.220 Identification of plan.

- \* \* \* \* \*
- (c) \* \* \*
- (279) \* \* \*
- (i) \* \* \*
- (A) \* \* \*
- (9) Rule 415, adopted on September 14, 1999.
- \* \* \* \* \*
- (284) \* \* \*
- (i) \* \* \*
- (C) \* \* \*
- (2) Rule 346, adopted on January 18, 2001.
- (D) Ventura County Air Pollution Control District.
- (2) Rule 70, adopted on November 14, 2000.

\* \* \* \* \*

[FR Doc. 02-27343 Filed 10-28-02; 8:45 am]  
BILLING CODE 6560-50-P

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[FRL-7390-6]

RIN 2040-AD72

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

AGENCY: Environmental Protection Agency (EPA).  
ACTION: Final rule.

SUMMARY: This action approves EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry (Method 1631E) for determination of mercury in aqueous samples. Today's rule replaces the currently approved version of Method 1631 and includes revisions that address stakeholder concerns. EPA Method 1631E clarifies quality control and sample handling requirements and allows flexibility to incorporate additional available technologies. This rule also amends the requirements regarding preservation, storage, and holding time for low level mercury samples.

DATES: This final rule is effective on November 23, 2002. For judicial review purposes, this final rule is promulgated as of 1 p.m. Eastern Standard Time on November 12, 2002 in accordance with 40 CFR 23.7. The incorporation by reference of EPA Method 1631, Revision E, is approved by the Director of the

Federal Register as of November 23, 2002.

FOR FURTHER INFORMATION CONTACT: William Telliard; Engineering and Analysis Division (4303T); 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) 566-1061 or e-mail at [telliard.william@epa.gov](mailto:telliard.william@epa.gov).

SUPPLEMENTARY INFORMATION:

A. 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 an authorized State, Territory, or 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.
Industry .....	Private facilities required to monitor.

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 facility or organization is regulated by this action, you should carefully examine the applicability language at 40 CFR 136.1 (NPDES permits and CWA). 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.

## B. How Can I Get Copies of This Document and Other Related Information?

1. *Docket.* EPA has established an official public docket for this action under Docket ID No. W-01-05. The official public docket consists of the documents specifically referenced in this action, any public comments received, and other information related to this action. Although a part of the official docket, the public docket does not include Confidential Business Information (CBI) or other information whose disclosure is restricted by statute. The official public docket is the collection of materials that is available for public viewing at the Water Docket located at EPA West Building, Room B135, 1301 Constitution Avenue, Washington, DC. This Docket Facility is open from 8:30 a.m. and 4:30 p.m. Eastern Time, Monday through Friday, excluding Federal holidays. The Docket telephone number is 202-566-2426.

2. *Electronic Access.* You may access this **Federal Register** document electronically through the EPA Internet under the "Federal Register" listings at <http://www.epa.gov/fedrgstr/>.

An electronic version of the public docket is available through EPA's electronic public docket and comment system, EPA Dockets. You may use EPA Dockets at <http://www.epa.gov/edocket/> to view public comments, access the index listing of the contents of the official public docket, and to access those documents in the public docket that are available electronically. Although not all docket materials may be available electronically, you may still access any of the publicly available docket materials through the docket facility identified in Unit I.B. Once in the system, select "search," then key in the appropriate docket identification number.

## Outline of Document

- I. Statutory Authority
- II. Background
  - A. Regulatory History
  - B. Settlement Agreement
  - C. Proposed Rule
- III. Summary of Final Rule
  - A. EPA Method 1631, Revision E
  - B. Amendment to 40 CFR 136.3(e) Table II

- IV. Changes from the October 9, 2001 Proposed Rule
  - A. Additional Requirements for Clean Techniques and Quality Control Provisions
  - B. Election by a Permittee or Industrial User
  - C. Additional Revisions to EPA Method 1631
  - D. Extension of Holding Time for Unpreserved Samples
  - E. Clarifications and Corrections
- V. Response to Major Comments
  - A. Performance-Based Measurement System
  - B. Proposed Requirements for Clean Techniques
  - C. Election by a Permittee or Industrial User
  - D. Bottle Blanks
  - E. Range of Method Calibration
  - F. Acceptance Criteria Associated with Blank Samples
  - G. Flow-Injection Systems
  - H. Sample Containers
  - I. Carryover
  - J. Other Technical Details
- VI. Administrative Requirements
  - A. Executive Order 12866: Regulatory Planning and 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.*
  - C. Unfunded Mandates Reform Act
  - D. Paperwork Reduction Act
  - E. National Technology Transfer and Advancement Act
  - F. Executive Order 13045: Protection of Children from Environmental Health Risks and Safety Risks
  - G. Executive Order 13132: Federalism
  - H. Executive Order 13175: Consultation and Coordination with Indian Tribal Governments
  - I. Congressional Review Act
  - J. Executive Order 13211: Energy Effects
  - K. Plain Language Directive

## I. Statutory Authority

Today's rule is promulgated 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" or "CWA"). 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 History

On May 26, 1998, EPA proposed EPA 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). On March 5, 1999, EPA published a Notice of Data Availability that included additional data supporting the application of EPA Method 1631 to effluent matrices (64 FR 10596), and on June 8, 1999, 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. Following method promulgation, EPA published a technical correction replacing Revision B (Method 1631B) with EPA Method 1631, Revision C (66 FR 32774; June 18, 2001). Revision C clarified the method text regarding the reporting and use of field blanks.

### B. Settlement Agreement

Following promulgation of EPA Method 1631B on June 8, 1999, several industry groups filed a petition for judicial review of the method. On October 19, 2000, EPA entered into a Settlement Agreement (*Alliance of Automobile Manufacturers, et al. v. EPA*, No. 99-1420, D.C. Dir.) with the Petitioners. The Settlement Agreement includes three clauses that address revisions to EPA Method 1631 (Clauses 2, 3, and 4). Clauses 2 and 3 committed EPA to sign a notice of final rulemaking by June 15, 2001, revising sections 12.4.2 and 9.4.3.3 of EPA Method 1631B to clarify the use of field blanks. EPA complied with that commitment. On June 18, 2001, EPA published a notice of final rulemaking announcing a revised version of EPA Method 1631 (Revision C; Method 1631C).

Clause 4 of the Settlement Agreement required that EPA sign a notice for publication in the **Federal Register** to propose additional requirements for certain clean techniques and quality control (QC) provisions on or before September 30, 2001, and to sign a notice

for final action on the proposal on or before September 30, 2002. On October 9, 2001, EPA published a notice proposing such revisions (66 FR 51518). At that time, EPA also made a draft of the method available to present the proposed revisions in context of EPA Method 1631 procedures (draft Method 1631, Revision D). Today's action satisfies EPA's obligation to take final action on the proposed rulemaking.

### C. Proposed Rule

On October 9, 2001, EPA proposed revisions to Method 1631 under the Settlement Agreement (66 FR 51518). The proposed revisions were listed in Appendix A of the Settlement Agreement and were presented in Section IV of the proposed rule (66 FR 51520). The proposed revisions would have converted certain of the recommendations and guidance in the method (specifically, certain clean techniques and quality control provisions) into requirements. The proposal would have allowed an NPDES permittee to forgo such requirements at their own discretion, but at their own risk. The proposal would not have allowed other method users (*e.g.*, State agencies) to forgo the proposed requirements.

EPA proposed several additional revisions that were not contested in the litigation. These latter proposals would clarify method procedures, increase method flexibility, and provide additional guidance for method implementation. To ensure consistency with analytical method requirements, EPA also proposed an amendment to Table II at 40 CFR 136.3(e) to address collection and handling of samples for analysis using EPA Method 1631. The additional proposed revisions and the proposed amendment to Table II at 40 CFR 136.3(e) were based on comments and recommendations submitted to EPA by various stakeholders since promulgation of EPA Method 1631B in June of 1999. EPA received 26 comment packages on the October 2001 proposed rule. Section V of this document summarizes the major comments. The administrative record supporting today's action responds to the public comments received on all the proposed changes.

### III. Summary of Final Rule

#### A. EPA Method 1631, Revision E

Today's action replaces all previously approved versions of EPA Method 1631 with EPA Method 1631, Revision E (Method 1631E) for measurement of mercury in aqueous samples. Today's action does not repeal any other

currently approved methods that measure mercury. EPA Method 1631E (the "Method") incorporates several revisions to increase method flexibility and improve data quality. These revisions:

- Allow the use of automated flow-injection systems (Sections 10.3 and 11.2.2);
- Incorporate system blanks for use with automated flow-injection systems (Sections 9.4.2 and 10.3.2);
- Incorporate definitions for blank samples (Sections 9.4 and 17);
- Incorporate a requirement for analysis of method blanks (Section 9.4);
- Include a requirement to analyze bottle blanks at a recommended minimum frequency of 5 percent (Sections 6.1.2.4 and 9.4.7);
- Allow extension of the calibration range (Sections 1.3 and 10.4);
- Remove requirements for immediate sample preservation, refrigeration of unpreserved samples, and collection of samples with zero headspace, provided sample bottles are tightly capped and samples are either preserved or analyzed within 48 hours of collection (Section 8.5);
- Allow extension of the time until preservation to 28 days if a sample is oxidized in its sample container (Section 8.5);
- Extend the maximum sample holding time (time from sample collection until sample analysis) from 28 days to 90 days (Section 8.5);
- Incorporate a carryover test for determining the amount of mercury that would be carried into a subsequent sample when a sample containing a high level of mercury is analyzed (Sections 4.2.8.1 and 11.2);
- Further clarify that samples must be completely oxidized prior to analysis (Section 8.1);
- Allow shipment of empty bottles for sample collection (Section 6.1.2.1);
- Incorporate a requirement for analysis of a minimum of two matrix spike/matrix spike duplicate (MS/MSD) sample pairs per analytical batch of twenty samples (Section 11.1.2);
- Reinforce the requirement that only glass or fluoropolymer bottles may be used for sample collection (Section 4.3.7.1 and 8.2);
- Allow both field and laboratory sample filtration (Sections 2.2 and 8.4);
- Correct part numbers (Sections 6.1.3.2 and 6.1.3.3); and
- Clarify that method users are permitted to omit steps or modify procedures provided that all performance requirements in the Method are met, but must not omit or modify any procedure defined by the term "shall" or "must," and must

perform all quality control tests (Method introductory note).

#### B. Amendment to 40 CFR 136.3(e), Table II

Today's rule also amends 40 CFR 136.3(e) by adding 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 includes the following requirements for mercury samples: samples must be collected in either fluoropolymer or glass containers; samples must be preserved with either HCl or BrCl within 48 hours of collection; time until preservation may be extended to 28 days if samples are oxidized in the sample bottles; samples have a maximum holding time of 90 days from the date of sample collection; and samples must be filtered in a clean area in the laboratory or in the field prior to sample preservation. This amendment provides consistency with requirements approved in previous versions of EPA Method 1631 and with the revisions promulgated today.

### IV. Changes from the October 9, 2001 Proposed Rule

#### A. Additional Requirements for Clean Techniques and Quality Control Provisions

Under the Settlement Agreement, EPA proposed certain clean techniques and quality control (QC) provisions as requirements. Under the then existing versions of Method 1631, these provisions were only recommendations. These provisions were presented in Section IV.A of the proposed rule and were indicated throughout draft Method 1631D by the word "must" in bracketed and italicized text. A summary of the comments received and EPA's response to the comments is presented in Section V.B of this document.

Commenters generally opposed the changes from "should" to "must," maintaining that Method 1631 and other EPA methods should be "performance based"; *i.e.*, that the method user should be accorded flexibility to improve method performance and lower the costs of measurements, provided all performance criteria are met. However, commenters supported specific requirements for analysis of bottle blanks (Sections 6.1.2.3 and 9.4.7), analysis of blanks to test for carryover (Sections 4.3.8.1 and 11.2), analysis of two MS/MSD pairs per each analytical batch of 20 samples (Section 11.1.2), and use of either fluoropolymer or glass containers for sample collection

(Section 4.3.7.1). In response to comments, EPA is incorporating these changes into EPA Method 1631E in today's rule.

Following review of comments, EPA believes that requiring the additional proposed requirements for clean techniques or quality control provisions would result in unnecessary economic burden and would limit future use of the Method. With the exceptions outlined above, EPA is not promulgating the clean techniques and quality control requirements proposed earlier. Instead, EPA has retained in the Method as recommendations that samples should be collected using clean hands/dirty hands collection procedures (Section 9.4.4.2); samples should be processed in a clean room or clean bench (Sections 4.3.3 and 8.5.3); exposure to sources of contamination should be minimized (Section 4.3); work surfaces should be cleaned prior to processing sample batches (Section 4.3.5); traps that tend to absorb large quantities of water vapor should be pre-dried or discarded (Section 4.3.3); outside air, if clean, should be brought into the clean bench air intake (Section 7.2); samples should be stored in clean, new polyethylene bags prior to use (Section 8.6); and samples collected for measurement of methylmercury should be collected and preserved according to procedures required in the analytical method that will be used (Sections 2.3 and 8.5).

#### *B. Election by a Permittee or Industrial User*

Under the Settlement Agreement, EPA also proposed that an NPDES permittee or an industrial user of a POTW be able to elect not to implement the clean techniques and QC 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." The election, if promulgated, would apply only to those clean techniques and QC provisions designated in the Settlement Agreement and designated by bracketed and italicized text throughout draft Method 1631D. Because EPA is not imposing such requirements, EPA has not included the proposed election revision in today's final rule. A summary of the comments regarding the proposed option and EPA's response to the comments is presented in Section V.C of this document.

#### *C. Additional Revisions to EPA Method 1631*

Since promulgation of EPA Method 1631B in June 1999, EPA received many suggestions from Method users for

improving method flexibility and clarifying certain method procedures. EPA proposed and discussed these improvements and clarifications in the October 9, 2001 proposal. In today's final rule, EPA is withdrawing or revising certain proposed Method revisions based on adverse comments. Specifically, EPA is (1) revising the term "calibration blank" to "system blank" for those blank samples required during calibration and batch analyses when using a flow-injection system, (2) revising the proposed QC acceptance criteria associated with system blanks and the use of these blanks, (3) withdrawing the frequency requirement associated with analysis of bottle blanks, and (4) withdrawing the requirement to commensurately raise the lowest calibration point when the upper end of the calibration range is raised. These four revisions and the corresponding comments on the proposed rule are described in more detail in Sections V.E through V.F of this document.

#### *D. Extension of Holding Times for Unpreserved Samples*

In the October 9, 2001 proposal, EPA stated that it was reviewing data that indicate samples collected for measurement of low level mercury may be stable for as long as 35 days prior to preservation, and included the data in the Record supporting the proposed rule. At that time, EPA also solicited additional data or comments regarding the stability of unpreserved samples.

EPA received comments on the proposed rule that support extension of the time prior to preservation and has completed review of the data discussed in the proposed rule. In response to these data and to submitted comments, EPA is requiring in Method 1631E that samples must be preserved within 48 hours of sample collection. However, EPA is allowing extension of the time until preservation to 28 days if samples are oxidized in the sample bottles. EPA has included this change in Section 8.5 of EPA Method 1631E and in Footnote 17 to Table II at 40 CFR 136.3(e).

#### *E. Clarifications and Corrections*

Minor clarifications and technical corrections are included in EPA Method 1631E to address errors and inconsistencies noted by commenters. These changes and corrections:

- Revise Section 9.4.2 to clarify that system blanks are specific to flow-injection systems;
- Revise Section 9.4.3.1 to clarify that in order to assess possible contamination from reagents, reagent blanks include hydroxylamine

hydrochloride solution in addition to BrCl solution;

- Revise Section 11.2.2.1 to clarify that the amount of  $\text{NH}_2\text{OH}$  required will be approximately 30 percent of the BrCl volume;

- Revise the QC acceptance criteria for reagent blanks in Section 9.4.3 from 0.25 ng/L to 0.2 ng/L for consistency with reporting requirements;

- Revise Section 12.2.1 to clarify that the mean peak response for bubbler blanks measured during calibration or with each analytical batch is used for calculating sample results;

- Correct the concentration units in the equations in Sections 12.2.2 and 12.3.2;

- Revise Section 9.3.2.2 to clarify that identical volumes of spiking solution must be used for MS/MSD samples;

- Revise Section 4.4.1 to clarify that, for those samples requiring pre-reduction with  $\text{SnCl}_2$  (*i.e.*, samples containing iodide concentrations greater than 3 mg/L), the  $\text{SnCl}_2$  should be added in a closed vessel or analysis should proceed immediately;

- Revise Section 11.1.1.2 to clarify that samples containing high organic content may also be diluted to reduce the amount of BrCl that may be required, provided that the resulting level of mercury is sufficient for reliable determination within the range of method calibration;

- Revise Section 7 to include a note clarifying that the quantities of reagents and the preparation procedures are for illustrative purposes. A laboratory may use quantities of reagents and procedures that differ, provided it is able to demonstrate equivalent performance;

- Revise Sections 7.9 and 7.10 to clarify that standard solutions should be replaced monthly, or longer if extended stability is demonstrated;

- Correct Section 2.7 to include the analytical trap in the description;

- Revise Section 9.1.7 and Section 10.1 to clarify that analysis of samples may proceed without recalibration, provided system performance is verified at the end of the analytical sequence;

- Revise Section 9.4 to address the performance criteria associated with blank samples in those circumstances when a method detection limit greater than 0.2 ng/L is sufficient to address compliance monitoring;

- Include a note in Section 9.1.2.1 to clarify that acceptance criteria associated with blank samples may be adjusted to support measurements at the compliance level; and

- Revise Section 12.5.1 to include specifications for reporting results of Method blanks.

## V. Response to Major Comments

EPA requested comments on the various EPA Method 1631 revisions detailed in the October 9, 2001 proposal, and requested data supporting comments, if available. Twenty five stakeholders provided comments on the proposal addressing over 50 separate issues. Stakeholders included 10 laboratories, 6 POTWs, 3 regulatory authorities, 3 industries/industry groups, one instrument manufacturer, a group of several POTWs, and the Petitioners (see Settlement Agreement discussion, Section II.B).

The following section summarizes major comments received on the proposed rule and EPA's response. The complete Response to Comments document can be found in the public record for this final rule (Record Section VI, DCN B.1).

### A. Performance-Based Measurement System

Several commenters on the October 9, 2001 proposed rule noted that Section 1.8 of EPA Method 1631 describes the method as performance-based, and that if certain recommendations for clean techniques included in the method were to become requirements as proposed, the method would no longer be performance-based. Commenters stated that requiring laboratories and sample collectors to adopt clean procedures that are unnecessary is contrary to a performance-based measurement system, and added that additional requirements would impose cost burdens that could result in reduced method implementation. Commenters stated further that performance-based measurements must not prescribe particular actions unless they are essential to the successful implementation of the method. Commenters added that many of the proposed requirements would lock users into current technology despite the many advances and improvements in techniques and equipment that are likely to occur in the coming decades. Commenters believe that if the performance-based nature of the method is not retained, further improvement of method performance would be hindered.

EPA developed performance-based measurement systems as part of EPA's commitment to reducing unnecessary regulatory burden and encouraging the use of emerging and innovative technologies. Throughout development of these analytical methods, EPA recognized that allowance for this flexibility must be matched with controls to ensure that data quality is

maintained. For this reason, many approved methods include standardized QC tests and specific QC acceptance criteria that must be met when a method is modified to overcome interferences or lower the cost of measurement.

The QC acceptance criteria included in EPA Method 1631 were developed using method validation data from 12 laboratories. These criteria include precision and recovery performance requirements for the matrix spike and matrix spike duplicate (MS/MSD), initial and ongoing precision and recovery (IPR and OPR), calibration linearity, method detection limit (MDL), and quality control samples (QCS). EPA Method 1631 criteria also include requirements for several blanks (*i.e.*, equipment, bottle, field, method, reagent, system, and bubbler blanks) to monitor potential contamination during sample collection and analysis.

EPA acknowledges the concerns submitted by commenters and agrees that the QC requirements and acceptance criteria in EPA Method 1631 are sufficient to ensure data quality and preclude inadequate method implementation. For these and other reasons given in the Response to Comments document, EPA has decided not to require most of the clean techniques and quality control provisions proposed.

### B. Proposed Requirements for Clean Techniques

Only comments submitted on behalf of the Petitioners supported promulgation of all the proposed requirements for the additional clean techniques specified in the Settlement Agreement. This commenter stated that clean sampling and analytical techniques are critical to obtaining reliable results for use in the regulatory process. The commenter stated further that, although clean techniques result in additional expense, the consequences could be more serious if data users act upon test results that may be affected by contamination.

Nineteen commenters submitted comments opposing these additional requirements. These commenters believe that EPA Method 1631 contains sufficient QC to determine the source of any contamination and that if a laboratory can demonstrate it is capable of meeting the Method QC criteria without the additional proposed clean techniques, it should be allowed to do so. Several commenters stated that the reasoning behind the proposed requirements appears to be arbitrary and that it is unclear what scientific basis was used to determine which techniques should be requirements.

Commenters noted that the requirements would place a burden, operational and economic, on facilities with little or no gain in analytical performance, and could severely limit the ability of regulators to determine whether mercury discharges are being controlled effectively. At least one POTW commenter stated that, if these requirements were promulgated as proposed, it most likely would no longer use EPA Method 1631. Additionally, a regulatory authority noted that if costs escalate because of additional requirements, such costs would limit the ability of regulators to determine whether mercury discharges are being controlled effectively.

EPA agrees with the majority of commenters and believes that the additional requirements proposed to be included in EPA Method 1631 would be burdensome, and that the QC acceptance criteria included in the Method are sufficient to ensure data quality. EPA has not received data to support a decision that the proposed additional clean techniques and quality control provisions are necessary to ensure validity of data obtained through implementation of EPA Method 1631. The requirements and criteria associated with quality control and blank samples throughout the Method are the most appropriate and valuable means for identifying and controlling contamination.

Additionally, EPA believes that if all of the recommendations for clean techniques were required, compliance with the requirements would be extremely difficult to monitor. For example, EPA proposed to revise Method 1631 Section 4.3.8.4 as follows: “\* \* \* Whenever possible, sample processing and analysis [must] occur as far as possible from sources of airborne contamination.” Following review of comments, EPA believes that it does not have sufficient information to provide specific tests to determine compliance with such a requirement. EPA believes the most appropriate means for demonstrating that samples are processed and analyzed using procedures to minimize contamination are already included in the requirements and criteria for analysis of blanks, and that analysts are appropriately advised regarding how to avoid contamination by the recommendations for clean techniques in the Method.

EPA also recognizes that sample locations and laboratory environments can differ significantly and that the site-specific clean techniques necessary to meet the performance criteria included in EPA Method 1631 will be best

determined and improved upon by individual Method users. For these reasons, and to respond to the concerns of commenters, EPA is retaining the clean techniques provisions as recommendations but not requirements in EPA Method 1631E. EPA Method 1631E continues to require that all QC tests be performed and that all QC acceptance criteria are met, and continues to include the following as recommendations:

- Use a clean room or clean bench for sample preparation and analysis (Sections 4.3.3 and 8.5.3);
- Minimize exposure of the apparatus to contamination (Section 4.3.3);
- Clean work surfaces prior to processing sample batches (Section 4.3.5);
- Process samples as far as possible from sources of airborne contamination (Section 4.3.8.4);
- Ensure that laboratory air is low in mercury (Section 7.2);
- Store sample bottles in clean (new) polyethylene bags until sample analysis (Section 8.6); and
- Use "Clean Hands/Dirty Hands" techniques described in EPA's Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels for collection of equipment blanks (Section 9.4.4.2).

In addition to recommending these protocols for clean techniques throughout EPA Method 1631E, EPA published several guidance documents supporting the collection and analysis of samples for measurement of low-level mercury. These guidance documents include Guidance for Implementation and Use of Method 1631 for Determination of Low-Level Mercury (40 CFR Part 136) EPA 821-R-01-023, March 2001; Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels, EPA-821-R-96-001, July 1996; Guidance on Establishing Trace Metals Clean Rooms in Existing Facilities, EPA-821-B-96-001, January 1996; and Guidance on Documentation and Evaluation of Trace Metals Data Collected for Clean Water Act Compliance Monitoring, EPA-821-B-96-004, July 1996.

#### *C. Election by a Permittee or Industrial User*

Under the Settlement Agreement, EPA proposed to require specific clean techniques and QC provisions in draft Method 1631D and to provide only NPDES permittees and industrial users with the option not to implement those techniques and provisions.

Comments submitted on behalf of the Petitioners regarding this proposed option state that this approach would be

appropriate because (1) the liability associated with sampling lies with the permittee and, therefore, the permittee should have the discretion to determine what is or is not an acceptable contamination risk, (2) permittees are familiar with the characteristics of their effluent and the level to which clean techniques must be followed, and (3) EPA and State agencies lack this level of facility-specific understanding and therefore, should be required to follow clean procedures. The commenter added that, under the current system, permittees may be precluded from raising the contamination issue as a defense in an enforcement action or, at a minimum, would bear the very heavy burden of proving contamination for data generated by EPA or State agencies.

Eleven commenters stated that giving certain groups the option to eliminate certain requirements for clean techniques and QC provisions would result in a plethora of methods and would make it very difficult for contract testing laboratories who would bear the burden of the resulting confusion. Some permittees may elect to forgo required clean techniques while others would not; all laboratory customers, however, would benefit and bear costs of clean techniques, regardless of their election. These commenters believe that such an option would set a dangerous and undesirable precedent regarding what any particular person believes is "necessary" to achieve a scientifically valid result. These commenters stated further that implementation of this option would limit the quality and value of collective databases for environmental decision making.

In today's final rule, EPA is not requiring most of the proposed clean techniques and QC provisions for any users of the Method. EPA agrees with the majority of commenters that applying such requirements to only some users would create unwanted inconsistency, would severely impair laboratories serving multiple clients, and would ultimately cause misinterpretation of data and confusion among regulators and laboratories. Most importantly, EPA disagrees with the comment that all of the proposed clean techniques are necessary to obtain reliable results. EPA also disagrees with the comment that Federal and State laboratories lack the necessary information (or even need facility-specific information) to minimize contamination. In today's final rule, EPA has not included this option in EPA Method 1631E.

Rather than requiring the clean techniques and QC provisions for some users but not for others, EPA instead is

providing equal flexibility for all users of the Method. Most of the clean techniques and QC provisions are included only as recommendations in the final rule. Because EPA is not requiring most of the proposed clean techniques and QC provisions for any users of the Method, there is no reason to include the option for permittees and industrial users to elect not to use them, and in today's final rule, EPA has not included this option.

#### *D. Bottle Blanks*

EPA proposed to revise EPA Method 1631 to include requirements to assess cleanliness of bottle blanks and to require analysis of bottle blanks at a minimum frequency of 20 percent from a given lot. Most commenters agreed that requiring analysis of bottle blanks is appropriate and good practice. However, only one comment submitted regarding these blank samples supported a requirement that bottle blanks be analyzed at a frequency of 20 percent. EPA received comments from 7 laboratories, 1 instrument manufacturer, and 1 regulatory authority that this frequency requirement is excessive, and would result in unnecessary additional equipment costs. Commenters also provided cost information suggesting that, if one assumes a low cost of between \$40 and \$50 per Method 1631 analysis performed, this requirement would add a cost of \$800 to \$1000 per lot of clean bottles, or approximately \$8 to the cost of each bottle purchased. Commenters also recommended alternate frequency requirements for bottle blank testing ranging from a minimum of 1 percent to 10 percent.

EPA's proposal to include a requirement that a minimum of 20 percent of the bottles from a given lot be tested for cleanliness was based on current practices implemented in a single laboratory. Because method and field blanks also are used to monitor contamination and are required in EPA Method 1631E, EPA agrees that requiring testing of 20 percent of the bottles from each lot is unnecessary and probably excessive. Although laboratories and cleaning facilities may choose to test at this frequency as a means of ensuring contamination control, EPA is not requiring that frequency in today's rule. EPA believes that testing a lot of bottles at a minimum frequency of 5% is sufficient and has included this frequency as a recommendation in EPA Method 1631E. While EPA is recommending that bottle blanks be analyzed at a frequency of 5%, laboratories have demonstrated the ability to meet EPA Method 1631 performance criteria and data quality

objectives analyzing bottle blanks at a minimum frequency of 1%. Therefore, if a laboratory is able to meet performance criteria using a minimum frequency of 1%, it should be allowed.

#### *E. Range of Method Calibration*

In response to several requests from stakeholders to apply EPA Method 1631 across a broader range, EPA proposed to allow calibration to a lower point (below the ML) to more accurately measure mercury in blank samples, and to a higher point (above 100 ng/L) to measure concentrations presently measured with other approved mercury methods. EPA also proposed certain criteria to ensure that this allowance would not compromise data quality. These criteria included: (1) 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.

Several commenters expressed concern regarding the proposed option to extend the lower end of calibration in EPA Method 1631. Commenters noted that in the proposed rule preceding the June 8, 1999, promulgation of EPA Method 1631B, EPA proposed to allow users to calculate lower MDLs and MLs. Based on comments, however, EPA "removed the option for laboratories to calculate their own lower MDLs and MLs. \* \* \*" 64 FR 30420 (June 8, 1999). In support, EPA stated its belief that "this will avoid confusion and preclude lower MDLs and MLs from being used for NPDES permitting or regulatory compliance determinations." Commenters stated that authorization for Method users to calibrate instruments to below the Method ML would result in regulatory uncertainty. For these reasons and in response to

comments, EPA has clarified this provision in EPA Method 1631E by stating that, for the purpose of measuring the level of mercury in blank samples only, calibration may be extended to a lower level.

One commenter on the proposed rule expressed concern that allowing extension of the high end of the calibration range could jeopardize low-level compliance determinations by increasing the potential for bias resulting from cross contamination or carryover. The commenter pointed out that EPA acknowledged this increased risk by proposing a new method section that specified a carryover test (see Proposed Rule, Section IV.A.7). This commenter believes that bubbler blank and method blank analyses are insufficient to identify and control contamination if calibration is extended to levels greater than 100 ng/L. EPA proposed to add a carryover test to Method 1631 in response to comments from stakeholders who requested an allowance for extension of the method calibration range, but were concerned about potential contamination that could result from extension of the upper end of calibration. EPA believes that the carryover test, the requirements associated with blank analyses, and the calibration criteria included in Method 1631, Revision E are sufficient to prevent effects that could result from cross contamination or carryover. For this reason, and in response to additional comments, EPA has included an allowance for extension of the upper end of the calibration range in EPA Method 1631, Revision E.

Three additional commenters supported extension of the upper end of the calibration range. These commenters believe the carryover test and ongoing blank determinations will ensure the analytical system remains sufficiently clean or that carryover will be detected should it occur. The commenters stated, however, that the corresponding criterion that the lowest calibration point must be raised commensurately when the upper end of calibration is raised is inappropriate. Commenters stated further that this criterion is unnecessary, particularly if an analytical system is demonstrated to be sufficiently linear and clean as specified by QC requirements included in the Method. Commenters added that commensurate raising of the lower end of calibration is unnecessary, particularly if two of the additional proposed corresponding criteria for extended calibration are met (*i.e.*, the calibration factor (CF) for any calibration point at a concentration greater than 100 ng/L must be within

plus or minus 15 percent of the average CF for the points at or below 100 ng/L and the CF for any point less than 5 ng/L must be within plus or minus 25 percent of the average CF for all points). EPA agrees that commensurate raising of the lower end of calibration is unnecessary, provided the remaining calibration criteria are met. EPA also points out that (1) there is no similar restriction in other methods; (2) the carryover test included in Section 4.3.8.1 of Method 1631, Revision E will allow a laboratory to determine the level at which Hg will be carried into a succeeding sample or blank; (3) the extensive requirements for blanks will detect contamination; and (4) a laboratory can run a blank before each sample, if desired, to demonstrate that a preceding sample did not carry Hg into the next sample. For these reasons, EPA is not including the proposed requirement for increasing the low end of the calibration range when the upper end is increased in today's rule.

Following review of these comments, EPA has determined that allowing Method users to raise the Method calibration range, provided the performance criteria specified in Section 10.4 of the Method are met, will increase method flexibility without compromising data quality. Such an allowance is consistent with EPA protocol for approval of new methods and with the International Union of Pure and Applied Chemists (IUPAC) guidelines for calibration. EPA agrees with most commenters and believes that Method 1631 contains QC requirements that are sufficient to detect and preclude carryover from samples or standards containing high levels of mercury (*i.e.*, greater than 100 ng/L). Additionally, EPA agrees that the criteria included in Section 10.4 of the Method is sufficient to ensure data quality without the requirement to raise the lower end of calibration if an extended upper end of calibration is used. For these reasons, EPA has removed the requirement to commensurately raise the low end of the calibration range when the upper end is raised from Section 10.4 of Method 1631E and is approving the provision to allow extension of the upper end of the calibration range. EPA also agrees with most commenters that the proposed procedure for identifying and controlling carryover will assist Method users, and is promulgating the procedures in Section 4.3.8.1 (see Section V.I. below).

#### *F. Acceptance Criteria Associated With Blank Samples*

EPA received several comments regarding Method 1631 QC acceptance



criteria associated with the analysis of blank samples. Numerous commenters stated that an acceptance criterion stipulated as less than the Method MDL (<0.2 ng/L) is inappropriate. Commenters note that measurements below the minimum level of quantitation (*i.e.*, the lowest calibration point) of an analytical method are inherently problematic and will result in failures attributable to random variation alone. Commenters also state that such a criterion is inappropriate because an MDL of 0.2 ng/L is not required to meet performance specifications in the Method. These commenters point out that Section 9.2.1 of EPA Method 1631 states that an MDL less than or equal to 0.2 ng/L or one-third the regulatory compliance limit, whichever is greater, is acceptable. Commenters note further that if a compliance level were 4 ng/L, an MDL determination of 1.3 ng/L would be sufficient. In such a case, monitoring levels of mercury in blanks against a criterion of 0.2 ng/L would be inconsistent.

Except for those criteria associated with calibration and method blank analyses, the QC acceptance criteria for blank samples included in EPA Method 1631 are identical to those originally proposed in May 1998. EPA received, reviewed, and responded to numerous comments prior to promulgation of EPA Method 1631B in June of 1998. Since promulgation, use of EPA Method 1631 has increased significantly, as has the ability to meet Method QC acceptance criteria. For this reason, EPA did not include revisions to these acceptance criteria in the October 2001 proposed rule, and is not promulgating such revisions in today's final rule. In response to comments on the October 2001 proposed rule, however, EPA is clarifying that the QC acceptance criteria for blank samples may be adjusted (*i.e.*, raised) to support measurements at the compliance level (see EPA Method 1631E, Note to Section 9.1.2.1). For example, if the compliance level is 4 ng/L, an appropriate MDL would be 1.3 ng/L, the corresponding lowest calibration standard would be at the ML of 4 ng/L, and appropriate QC acceptance criteria for blank samples would be 1.3 ng/L (bubbler and reagent blanks) or 4 ng/L (method, field, and bottle blanks).

#### G. Flow-Injection Systems

Commenters were supportive of EPA's proposed revision to incorporate automated flow-injection systems into EPA Method 1631. Commenters stated that, without EPA's acknowledgment that these systems are in use,

implementation of such systems could be curtailed.

#### 1. Calibration or System Blanks

EPA Method 1631, Revision C included bubbler blanks to establish a background for the bubbler system (*i.e.*, bubbler, traps, and cold-vapor atomic fluorescence detector). Bubbler blanks, however, are not appropriate for flow-injection systems. Hence, EPA proposed a requirement for analysis of calibration blanks when using flow-injection systems. The proposed QC acceptance criteria and application requirements for the calibration blanks were identical to the existing QC acceptance criteria and application requirements for the bubbler blanks (*i.e.*, the mean result of bubbler or calibration blanks is subtracted from results of calibration standards and samples and must be < 0.25 ng/L).

Since proposal, EPA has determined that the term "system" blank is more appropriate for the blank samples associated with flow-injection systems because these blank samples are used to assess contamination during calibration and during analysis of analytical batches. Therefore, EPA has replaced the proposed term "calibration blank" with "system blank" throughout Method 1631E.

Numerous commenters strongly objected to the requirements associated with the system blanks that EPA proposed to accompany flow-injection systems. Commenters stated that bubbler blanks and calibration (*i.e.*, system) blanks are not identical in either composition or purpose, and emphasized that it would be inappropriate and impractical to treat these samples as identical. Commenters noted that, unlike bubbler blanks which analyze previously-purged water to measure the level of mercury remaining in the bubbler system, system blanks measure residual mercury in reagent water as well as mercury in the reagents used in the calibration standards and samples. Commenters added that for this reason, the proposed system blank criteria for flow-injection systems are at least twice as restrictive as those placed on the use of bubbler systems.

In response to comments and upon further review of automated flow-injection systems, EPA has revised the proposed requirements associated with system blanks. EPA recognizes that flow injection systems require that reagents are added to all samples including the calibration standards, and has included a criterion in Section 9.4.2 of EPA Method 1631E that system blanks containing levels of mercury equal to or greater than 0.5 ng/L demonstrate that the system is out of control.

#### 2. Terminology

Two commenters on the proposed rule stated that EPA should revise the term used for these systems from "Automated Flow-Injection" to "Continuous Flow" throughout EPA Method 1631. Although EPA agrees with these commenters that "Continuous Flow" is descriptive of the flow-injection systems used for determination of mercury in EPA Method 1631, it is a generic term that includes other systems such as sequential injection, sequential flow, and bead injection systems. EPA believes that the system described in EPA Method 1631 is consistent with the definition of a flow injection system, and has retained the proposed "flow-injection" terminology throughout EPA Method 1631E.

#### H. Sample Containers

Twelve commenters submitted comments regarding the proposed requirement to use either glass or fluoropolymer containers for collection of samples for measurement of mercury using EPA Method 1631. Commenters generally emphasized support for the performance-based nature of sample container selection to preclude unnecessary expense and allow for development of future materials. However, most commenters also expressed preference for fluoropolymer, glass, or fluoropolymer-lined glass containers for sample collection and preparation, particularly because of the possibility for some forms of mercury to move in or out of containers composed of other materials.

Four commenters on the proposed rule requested that EPA Method 1631 be revised to allow collection of samples in high density polyethylene (HDPE) containers if the samples are shipped to the laboratory for preservation and transferred to fluoropolymer containers within 48 hours. These commenters submitted identical data comparing summary results of samples collected using fluoropolymer sample bottles to samples collected using HDPE sample bottles to support this request. These data are included in Section V.E.1.10 of the Rulemaking Record. EPA has reviewed these data, and notes that although the results of the composite samples collected using either container type do not demonstrate a significant trend in mercury increase or loss, the results of the grab samples indicate a consistent increase in mercury concentration, ranging from 15 to 240 percent, in samples collected using HDPE containers.



EPA recognizes the concern that some forms of mercury can move in or out of containers composed of materials other than fluoropolymer or glass, and believes this concern is emphasized by commenters requesting that samples collected using HDPE containers be allowed only if the samples are transferred to fluoropolymer containers within 48 hours. In response to comments, and in recognition of current Method implementation practices, EPA is including a requirement in EPA Method 1631E that only fluoropolymer or glass containers be used for collection of samples to avoid artificial increases in mercury levels prior to measurement.

#### *I. Carryover*

EPA proposed to include a carryover test in Method 1631 to determine the concentration at which greater than 0.2 ng/L mercury would be carried into the subsequent sample (see draft EPA Method 1631D, Section 4.3.8.1). EPA also proposed to require that each time a laboratory analyzes a sample containing the concentration of mercury determined to result in 0.2 ng/L carryover, the laboratory must run a bubbler or calibration blank to ensure carryover does not affect the results of the analysis of the subsequent samples.

Commenters generally supported incorporation of a carryover test to identify and control contamination from carryover that can result from analysis of samples containing high levels of mercury. Commenters also supported the proposed requirement for analysis of a blank sample following a sample containing a high level of mercury to demonstrate that the analytical system is clean. Several commenters, however, noted that the proposed standard for determining carryover (*i.e.*, the level at which the analytical system will carry greater than 0.2 ng/L of Hg into a succeeding bubbler or calibration blank) is inappropriate because measurements below the ML of 0.5 ng/L are unreliable.

EPA has reviewed these comments and agrees with commenters' concerns regarding this performance standard. EPA recognizes that the carryover test is designed to target samples containing levels of mercury that could cause carryover, and believes that a performance standard of 0.5 ng/L is appropriate for the purposes of this test. EPA also believes, however, that in order to ensure data quality at the low levels of detection achievable by EPA Method 1631, levels of mercury no greater than 0.2 ng/L should be permitted to be carried over into succeeding samples. For this reason, EPA is requiring that a bubbler blank or system blank be analyzed following a

sample containing a level of mercury that is one-half or greater than the level identified in the carryover test. Specifically, EPA is requiring that when a sample is analyzed that contains one-half or greater of the level of mercury that has been determined to result in 0.5 ng/L carryover, a blank must be analyzed to demonstrate no carryover at the 0.2 ng/L level (see Section 4.3.8.1 of EPA Method 1631E). For example, if the carryover test determines that samples containing 150 ng/L result in carryover greater than 0.5 ng/L, then the laboratory must analyze a blank sample following analysis of any sample containing more than 75 ng/L mercury.

EPA received additional comments presenting other concerns related to the carryover test. Commenters noted that it is unnecessary to require analysts to order samples from the lowest to the highest concentration. Commenters stated that analysts will define the order of samples according to information made available to them, and that there are other factors besides mercury concentration that are important in determining the order of samples. Commenters also noted that EPA should clarify that blanks should be run on the same bubbler used to run the high-concentration sample and that the requirements for the carryover test were not included in procedures for implementation of flow injection systems. EPA acknowledges that analysts often are not aware of the concentration levels of mercury contained in samples and that analysts may wish to order samples from least complex matrix to most complex matrix (*e.g.*, ambient water to influent). For this reason, EPA has removed the requirement, and is instead recommending that samples known or suspected to contain the lowest concentration of mercury should be analyzed first followed by samples containing higher levels. EPA also has clarified that a bubbler blank should be analyzed using the same bubbler as that used to analyze the high-concentration sample and has added Section 11.2.2.3 to EPA Method 1631E to clarify that the carryover test and associated blank analysis are required.

#### *J. Other Technical Details*

Several commenters requested revisions and clarifications to the Method that were already addressed in the Guidance or were beyond the scope of the proposed rule. Specifically, EPA received comments requesting the inclusion of all QC acceptance criteria into a single table in Method 1631; clarification of site-specific frequency requirements associated with MS/MSD

samples; additional recommendations for sample filtration, clean techniques, and sample handling procedures; and approval of EPA Method 245.7: Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry.

Following promulgation of today's rule, the Agency plans to revise the Guidance to reflect today's final rule and to incorporate further clarification of Method procedures. EPA also has completed a study to validate Method 245.7 in multiple laboratories and is planning to continue efforts towards approval of this additional test procedure.

EPA reviewed all the additional recommendations and comments and has provided responses to each in the Comment Response Document.

### **VI. 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 than 50,000; and (3) a small organization that is any not-for-profit enterprise which is independently owned and operated and is not dominant in its field.

After considering the economic impacts of today's 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 promulgates a revised version of an already approved EPA Method to improve and clarify method procedures. Today's rule also promulgates an amendment to Table II at 40 CFR 136.3(e) to provide consistency with previously approved requirements in EPA Method 1631 and with method revisions promulgated today for collection, preservation, and storage of samples for determination of mercury using Method 1631.

Overall, the costs of these revisions are minimal. While some of the revisions may increase cost (e.g., quality control requirements), other revisions will offset any increases and provide flexibility to lower the overall analytical costs (e.g., use of new, less expensive equipment). Many of the laboratories that analyze for mercury are already implementing the additional requirements, further minimizing any potential cost increases. EPA estimates that any costs associated with the additional requirements will be alleviated or eliminated by the additional flexibility. Therefore, EPA believes that this rule will not have a significant economic impact on a substantial number of small entities.

#### C. Unfunded Mandates Reform Act

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, 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.

Today's rule contains no Federal mandate (under the regulatory provisions of Title II of the UMRA) for State, Tribal, and local governments or the private sector in any one year. This rule imposes no enforceable duty on any State, local or Tribal governments or the private sector. This rule promulgates revisions to a previously approved method for measuring mercury in wastewater. This rule also revises 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 promulgated method revisions. 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 also 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.* It does not impose any information collection, reporting or record keeping requirements. This action revises a currently approved test method for use in water monitoring programs but does not add additional burden nor specifically 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 numbers for EPA's regulations are listed in 40 CFR Part 9 and 48 CFR Chapter 15.

#### E. National Technology Transfer and Advancement Act

As noted in the proposed rule, Section 12(d) of the National Technology Transfer and Advancement Act of 1995 ("NTTAA"), Pub. L. 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 rulemaking involves technical standards. Therefore, the Agency conducted a search to identify potentially applicable voluntary consensus standards. However, EPA

identified no such standards for the measurement of mercury at water quality criteria levels, and none were brought to our attention in comments. Therefore, EPA has decided to promulgate EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry.

*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 rule is not subject to Executive Order 13045 because it is neither “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 final 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 promulgates EPA Method 1631, Revision E to replace an already approved version of the method for measuring mercury at low levels for compliance monitoring under the Clean Water Act and provide additional

flexibility for use of currently available technologies. The costs of this rule for State and local governments are minimal. Thus, Executive Order 13132 does not apply to this rule.

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

Executive Order 13175, titled “Consultation and Coordination With Indian Tribal Governments” (65 FR 67249, November 9, 2000), requires EPA to develop an accountable process to ensure “meaningful and timely input by Tribal officials in the development of regulatory policies that have Tribal implications.” “Policies that have Tribal implications” is defined in the Executive Order to include regulations that have “substantial direct effects on one or more Indian Tribes, on the relationship between the Federal government and the Indian Tribes or on the distribution of power and responsibilities between the Federal government and Indian Tribes.”

This final 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. Today’s rule promulgates EPA Method 1631, Revision E to replace an already approved version of the method for measuring mercury at low levels for compliance monitoring under the Clean Water Act, and provide additional flexibility for use of currently available technologies. The costs of this rule for Tribal governments are minimal. Thus, Executive Order 13175 does not apply to this rule.

*I. Congressional Review Act*

The Congressional Review Act, 5 U.S.C. 801 *et seq.*, as added by the Small Business Regulatory Enforcement Fairness Act of 1996, generally provides that before a rule may take effect, the agency promulgating the rule must submit a rule report, which includes a copy of the rule, to each House of the Congress and to the Comptroller General of the United States. EPA will submit a report containing this rule and other required information to the U.S. Senate, the U.S. House of Representatives, and the Comptroller General of the United States prior to publication of the rule in the **Federal Register**. A major rule cannot take effect until 60 days after it is published in the **Federal Register**. This action is not a “major rule” as

defined by 5 U.S.C. 804(2). This rule will be effective on November 29, 2002.

*J. Executive Order 13211: Energy Effects*

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.

*K. Plain Language Directive*

Executive Order 12866 directs each agency to write its rules in plain language. Readable regulations help the public find requirements quickly and understand them easily. Plain language increases compliance, strengthens enforcement, and decreases mistakes, frustration, phone calls, appeals, and distrust of government. EPA made every effort to write this preamble to the final rule in as clear, concise, and unambiguous manner as possible. Specifically, EPA used active voice and avoided the use of technical terms except when necessary. EPA solicited but received no comments on the Plain Language aspects of this rule.

**List of Subjects in 40 CFR Part 136**

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

Dated: September 30, 2002.

**Christine Todd Whitman,**  
*Administrator.*

For the reasons set out in the preamble, title 40, chapter I of the Code of Federal Regulations, is 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:

- a. By revising Item 35 in Table IB of paragraph (a).
- b. By revising Footnote 43 to Table IB of paragraph (a).
- c. By revising paragraph (b)(41).
- d. By revising entries 18 and 35 under the heading “Metals” in Table II of paragraph (e).

**§ 136.3 Identification of test procedures.**

\* \* \* \* \*

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, units and method	Reference (method number or page)				
	EPA <sup>1, 35</sup>	Standard methods [edition(s)]	ASTM	USGS <sup>2</sup>	Other
	*	*	*	*	*
35. Mercury—Total, <sup>4</sup> mg/L:					
Cold vapor, manual or ..	245.1 .....	3112 B [18th, 19th]	D3223-91 .....	I-3462-85 .....	977.22 <sup>3</sup>
Automated .....	245.2.				
Oxidation, purge and trap, and cold vapor atomic fluorescence spectrometry (ng/L).	1631E <sup>43</sup> .				
	*	*	*	*	*

## Table 1B 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 samples 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 TO TABLE 1B NOTE 4: 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:

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

<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>43</sup> USEPA. 2002. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry," September 2002. Office of Water, U.S. Environmental Protection Agency (EPA-821-R-02-019). The application of clean techniques described in EPA's draft Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels (EPA-821-R-96-011) are recommended to preclude contamination at low-level, trace metal determinations.

* * * * *	Spectrometry." September 2002. Office of Water, U.S. Environmental Protection Agency (EPA-821-R-02-019). Available from: National Technical Information Service, 5285 Port Royal Road,	Springfield, Virginia 22161. Publication No. PB2002-108220. Cost: \$25.50 (subject to change).
(b) * * *		* * * * *
(41) USEPA. 2002. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence		(e) * * *

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

Parameter No./name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
	*	*	*
Metals			
18. Chromium VI <sup>7</sup> .....	P, G .....	Cool, 4°C .....	24 hours.
35. Mercury <sup>17</sup> .....	P, G .....	HNO <sub>3</sub> 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 <sup>7</sup> .	P, G .....	do .....	6 months.

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

Parameter No./name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
*	*	*	*

<sup>1</sup> 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).

<sup>2</sup> 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).

<sup>3</sup> 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<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) 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).

<sup>4</sup> 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.

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

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

[FR Doc. 02–27136 Filed 10–28–02; 8:45 am]

BILLING CODE 6560–50–P

## ENVIRONMENTAL PROTECTION AGENCY

### 40 CFR Part 141

[FRL–7398–4]

RIN 2040–AD81

### Unregulated Contaminant Monitoring Regulation: Approval of Analytical Method for *Aeromonas*; National Primary and Secondary Drinking Water Regulations: Approval of Analytical Methods for Chemical and Microbiological Contaminants

**AGENCY:** Environmental Protection Agency.

**ACTION:** Final rule.

**SUMMARY:** Today's rule approves the analytical method and an associated Minimum Reporting Level (MRL) to support the Unregulated Contaminant Monitoring Regulation's (UCMR) List 2 *Aeromonas* monitoring. This List 2 monitoring will be conducted at 120 large and 180 small Public Water Systems (PWS) from January 1, 2003 through December 31, 2003.

Today's rule also approves EPA Method 515.4 to support previously required National Primary Drinking Water Regulation (NPDWR) compliance

monitoring for 2,4-D (as acid, salts and esters), 2,4,5-TP (Silvex), dinoseb, pentachlorophenol, picloram and dalapon. In addition, EPA Method 531.2 is approved to support previously required NPDWR monitoring for carbofuran and oxamyl.

Minor changes have been made in the format of the table of methods required to be used for organic chemical NPDWR compliance monitoring to improve clarity and to conform to the format of other methods tables. In addition, the Presence-Absence (P–A) Coliform Test listed in the total coliform methods table was inadvertently identified as Method 9221. This has been corrected to 9221 D. Also, detection limits for "Cyanide" were added in the "Detection Limits for Inorganic Contaminants" table for the two cyanide methods, and minor editorial corrections were made.

EPA is approving seven of the eight additional industry-developed analytical methods that were proposed to support previously required NPDWR compliance monitoring. These seven methods include: A method for the determination of atrazine, two methods for the determination of cyanide, two methods for the determination of total coliforms and *E. coli*, a method for the determination of heterotrophic bacteria, and a method for the determination of turbidity. With respect to the eighth industry-developed method proposed on March 7, 2002, EPA is deferring a

decision on its approval until additional clarifying information from the vendor is evaluated.

Finally, EPA is updating the information concerning the inspection of materials in the Water Docket to reflect its new address.

**DATES:** This regulation is effective November 29, 2002. The incorporation by reference of the methods listed in the rule is approved by the Director of the Federal Register as of November 29, 2002. For purposes of judicial review, this final rule is promulgated as of 1 p.m. Eastern Time on November 12, 2002, as provided in 40 CFR 23.7.

**ADDRESSES:** The official public docket for this rule is located at EPA West Building, Room B102, 1301 Constitution Avenue, NW., Washington DC.

**FOR FURTHER INFORMATION CONTACT:** For information regarding the actions included in this final rule contact David J. Munch, EPA, 26 West Martin Luther King Dr. (MLK 140), Cincinnati, Ohio 45268, (513) 569–7843 or e-mail at [munch.dave@EPA.gov](mailto:munch.dave@EPA.gov). General information may also be obtained from the EPA Safe Drinking Water Hotline. Callers within the United States may reach the Hotline at (800) 426–4791. The Hotline is open Monday through Friday, excluding Federal holidays, from 9 a.m. to 5:30 p.m. Eastern Time.

**SUPPLEMENTARY INFORMATION:**