

I. Identify the rulemaking by docket number and other identifying information (subject heading, **Federal Register** date and page number).

II. Follow directions—The agency may ask you to respond to specific questions or organize comments by referencing a Code of Federal Regulations (CFR) part or section number.

III. Explain why you agree or disagree; suggest alternatives and substitute language for your requested changes.

IV. Describe any assumptions and provide any technical information and/or data that you used.

V. If you estimate potential costs or burdens, explain how you arrived at your estimate in sufficient detail to allow for it to be reproduced.

VI. Provide specific examples to illustrate your concerns, and suggest alternatives.

VII. Explain your views as clearly as possible, avoiding the use of profanity or personal threats.

VIII. Make sure to submit your comments by the comment period deadline identified.

III. Where Can I Find More Information About This Proposal and the Corresponding Direct Final Rule?

For additional information see the direct final rule published in the rules section of this **Federal Register**.

List of Subjects in 40 CFR Part 52

Environmental protection, Air pollution control, Intergovernmental relations, Ozone, Volatile organic compounds.

Dated: September 16, 2004.

Norman Niedergang,

Acting Regional Administrator, Region 5.

[FR Doc. 04-24822 Filed 11-5-04; 8:45 am]

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ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[OW-2003-0003; FRL-7834-8]

Guidelines Establishing Test Procedures for the Analysis of Pollutants; Procedures for Detection and Quantitation

AGENCY: Environmental Protection Agency (EPA).

ACTION: Notice of document availability.

SUMMARY: EPA uses method detection limit (MDL) and minimum level of quantitation (ML) procedures to establish detection and quantitation capabilities of test procedures (*i.e.*,

analytical methods) under the Clean Water Act (CWA). The MDL is used to determine the lowest concentration at which a substance is detected or is “present” in a sample. The ML is used to describe the lowest concentration of a substance in a sample that can be measured with a known level of confidence. Today’s notice announces the availability of a document entitled *Revised Assessment of Detection and Quantitation Approaches*. This document presents EPA’s revised assessment of MDL, ML and other detection and quantitation procedures for use under the CWA, and EPA’s consideration of public comment received on an assessment document published by EPA in 2003.

ADDRESSES: The docket for today’s action is available under Docket ID No. OW-2003-0003. All documents in the docket are listed in the EDOCKET index at <http://www.epa.gov/edocket>.

Although listed in the index, some information is not publicly available, *i.e.*, CBI or other information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, is not placed on the Internet and will be publicly available only in hard copy form. Publicly available docket materials are available either electronically in EDOCKET or in hard copy at the Water Docket in the EPA Docket Center, (EPA/DC) EPA West, Room B102, 1301 Constitution Ave., NW., Washington, DC. This Docket Facility is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is (202) 566-1744, and the telephone number for the Water Docket is (202) 566-2426.

FOR FURTHER INFORMATION CONTACT: 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.

SUPPLEMENTARY INFORMATION:

I. General Information

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

1. *Docket.* EPA has established an official public docket for this notice under Docket ID No. OW-2003-0003. The official public docket consists of the documents specifically referenced in this notice, public comments received

on EPA’s assessment presented in the February 2003 Technical Support Document, and other supporting information related to this assessment. Information claimed as CBI and other information whose disclosure is restricted by statute, or which is not included in the official public docket, will not be available for public viewing in EPA’s public docket. The official public docket is available for public viewing at the Water Docket in the EPA Docket Center (EPA/DC), EPA West, Room B102, 1301 Constitution Ave., NW., Washington, DC 20460. This Docket Facility is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is (202) 566-1744, and the telephone number for the Water Docket is (202) 566-2426.

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 access the index listing of the contents of the official public docket, and to access those documents in the public docket that are available electronically. Once in the system, select “Search,” then key in the appropriate docket identification number. Information claimed as CBI and other information whose disclosure is restricted by statute, or which is not included in the official public docket, will not be available for public viewing in EPA’s electronic public docket. EPA’s policy is that copyrighted material will not be placed in EPA’s electronic public docket but will be available only in printed, paper form in the official public docket. Although not all docket materials may be available electronically, you may still access any of the publicly available docket materials through the docket facility identified in I.A.1.

II. Background

A. Test Procedures Used for Clean Water Act Programs

EPA proposes and promulgates test procedures at 40 CFR part 136 in accordance with Section 304(h) of the CWA, which requires that the EPA Administrator “promulgate guidelines establishing test procedures for the analysis of pollutants” to be monitored and regulated under the National Pollutant Discharge Elimination System (NPDES). Test procedures are also

known as analytical methods. EPA draws the analytical methods from a variety of sources, including methods developed by commercial vendors, EPA and other government agencies, as well as methods from voluntary consensus standards bodies such as the American Public Health Association, the Water Environment Federation, and the American Water Works Association, which jointly publish *Standard Methods for the Examination of Water and Wastewater*; the Association of Official Analytical Chemists; and the American Society for Testing and Materials (ASTM) International. An analytical method promulgated by EPA under CWA section 304(h) is considered approved by EPA for purposes of EPA's NPDES permitting regulations.

Among considerations for approval of an analytical method at 40 CFR part 136 are the demonstrated performance characteristics of precision, bias, and sensitivity (*i.e.*, detection and quantitation). EPA generally evaluates each of these characteristics to determine if the analytical method will yield results at concentrations of concern that are reliable enough to meet Agency needs for permitting and compliance monitoring under the Clean Water Act (CWA). Detection and quantitation limits have been the most controversial of these characteristics, particularly among members of the regulated community.

The method detection limit (MDL), which is specified at 40 CFR part 136, appendix B, is used to determine the lowest concentration at which a substance is detected or is "present" in a sample. The minimum level of quantitation (ML) is used to describe the lowest concentration in a sample of a substance that can be measured with a known level of confidence. The existing MDL procedure has been in place since 1984. Individual MDLs and MLs are included in many EPA-approved methods at 40 CFR part 136, and have provided laboratories and data users with limits for evaluating results of analytical measurements or analytical method selection.

B. EPA's Initial Assessment

In 2003, EPA completed an initial assessment of approaches for determining detection and quantitation capabilities of analytical methods and their application to CWA programs, and published the results in the *Technical Support Document for the Assessment of Detection and Quantitation Approaches* (EPA-821-R-03-005, February 2003). This assessment examined EPA's current MDL and ML procedures, as well as alternative

detection and quantitation concepts and procedures.

A draft of the initial assessment document was peer-reviewed in August 2002. EPA revised the document to incorporate comments from the peer review. On March 12, 2003 (68 FR 11791), EPA made *Technical Support Document for the Assessment of Detection and Quantitation Approaches* available to the public and provided for 150 days for public comment, including a 30-day extension of the comment period (68 FR 41988, July 16, 2003).

C. EPA's Revised Assessment

Today's notice announces the availability of the document entitled *Revised Assessment of Detection and Quantitation Approaches* (the Revised Assessment Document), EPA-821-B-04-005, October 2004. The revised assessment examines the procedures currently used by the Agency for determining detection and quantitation levels. It also evaluates alternative concepts and procedures, including two detailed procedures submitted by the U.S. Geological Survey (USGS) and the American Council of Independent Laboratories (ACIL). ACIL is an organization representing a large group of independent commercial laboratories.

This revised assessment evaluates several alternative concepts and procedures, some of which were submitted during the comment period on EPA's previous assessment. The Revised Assessment Document consists of the following parts:

- Chapter 1 provides background information regarding EPA's assessment of detection and quantitation procedures.
- Chapter 2 includes a discussion of additional concepts and procedures not included in *Technical Support Document for the Assessment of Detection and Quantitation Approaches*, February 2003.
- Chapter 3 contains a summary of and general response to public comments received on chemical, regulatory, and statistical issues.
- Chapter 4 addresses public comment on the six evaluation criteria that EPA used to evaluate each of the detection and quantitation procedures.
- Chapter 5 contains an evaluation of detection and quantitation procedures. It includes an evaluation of procedures suggested by ACIL, USGS, and the Inter-industry Analytical Group.
- Chapter 6 summarizes EPA's findings and outlines steps for a continuing dialogue about detection and quantitation issues.
- Appendix A contains a list of documents used in the assessment.

- Appendices B and C present analyses of the detection and quantitation limit procedures. These analyses have been updated to include data and comments submitted during the comment period on *Technical Support Document for the Assessment of Detection and Quantitation Approaches*, February 2003.

The Revised Assessment Document addresses comments and concerns from stakeholders and peer reviewers. Based on this new information, EPA plans to continue consideration of alternatives or improvements to current detection and quantitation procedures and uses. It is clear that there is a broad interest in improving current procedures and uses, but no consensus for a specific procedure or procedures has emerged among the laboratory, industry, regulatory or regulated communities. EPA currently is soliciting stakeholders to participate in further considerations of alternatives or improvements to current detection and quantitation procedures.

D. Settlement Agreement

EPA conducted this revised assessment, and took comment on the 2003 assessment, to partially fulfill the requirements of a settlement agreement with the Alliance of Automobile Manufacturers, *et al.* The settlement agreement required that EPA assess existing Agency and alternative procedures for determining detection and quantitation limits and sign a notice for publication in the **Federal Register** on or before February 28, 2003, and to invite comment on the assessment. On March 12, 2003, EPA published: (1) A **Federal Register** notice announcing the availability of, and requesting comment on, a document describing EPA's assessment (68 FR 11791); and (2) a **Federal Register** notice proposing and requesting comment on revisions to the MDL definition and procedure at 40 CFR part 136, appendix B (68 FR 11770), and a definition and procedure for calculation of an ML. EPA is discharging its settlement agreement obligation today by making available a Revised Assessment Document.

E. Proposed Rule; Withdrawal

In a separate notice published elsewhere in today's **Federal Register**, EPA is withdrawing the March 12, 2003, proposal to revise the MDL definition and procedure and to add a definition and procedure for calculation of an ML. While EPA believes that some revisions to the MDL definition and procedure are appropriate, the Agency also believes that further work, including a stakeholder consultation process, is

needed before EPA can determine how best to address the concerns that have been raised. EPA is exploring the feasibility and design of a process through which stakeholders could provide their suggestions, ideas and recommendations on procedures for the development of detection and quantitation limits and uses of these limits in CWA programs. The Agency believes that the body of public comment on the proposed rule provides a strong starting point for a continued consultation with stakeholders representing constituencies such as citizens, environmental organizations, permit writers, regulators and regulated industries. In a **Federal Register** notice published on September 15, 2004 (69 FR 55547), EPA announced that a neutral party is seeking a broad group of stakeholders willing to work together to define and address concerns about the way detection and quantitation values are calculated and used to support CWA programs. Such a process, if feasible, could begin as early as December 2004.

III. Summary of Major Comments

EPA received many comment letters raising issues, concerns or suggestions on *Technical Support Document for the Assessment of Detection and Quantitation Approaches*. Commenters included laboratories, wastewater treatment plants, Federal agencies, State and county agencies, industrial firms, instrument manufacturers, trade organizations, and others. A summary of public comments and EPA's responses are included in the Response-to-Comments Document, which is in the public docket supporting this notice (see Section I.A of this notice).

A. EPA's Assessment

Although several commenters stated that EPA clearly put a great deal of effort and thought into preparation of the initial assessment document and commended EPA for its efforts, many commenters disagreed with certain aspects of the assessment. Some specific concerns by commenters are briefly discussed in the following paragraphs. A more detailed discussion of commenters' concerns and suggestions is in the Response-to-Comments Document or in the Revised Assessment Document.

1. Peer Review

In August 2002, a draft assessment document was peer-reviewed in accordance with EPA's peer review policies, which are described in the Science Policy Council Handbook (EPA 100-B-00-001). The draft document was reviewed by a panel of four peer

reviewers, who were selected because of their expertise in the fields of statistics and/or analytical chemistry and absence of conflicts of interest. The peer review panel did not include any experts that directly or indirectly contributed to the development of EPA's MDL or ML. The peer review panel was generally supportive of EPA's approach and criteria, and made some suggestions that were incorporated into the February 2003 document.

2. Evaluation of Data

Some commenters questioned EPA's analysis of the data used to evaluate levels of detection and quantitation. They stated that they were unable to replicate EPA's data evaluations and expressed confusion regarding certain aspects of EPA's data handling (e.g., analytical sequence, data censoring, and calculations using their suggested quantitation procedure). In response to the comments that EPA's data evaluations could not be replicated or were confusing, EPA has revised or clarified the steps in its data evaluation in appendix B of the Revised Assessment Document and/or in the Response-to-Comments Document.

Some commenters submitted their evaluation of the data used by EPA, while others submitted data and evaluations from other studies to support the premise that their evaluations demonstrate that EPA's MDL does not do what it purports to do, i.e., provide a one percent (1%) false positive rate. In conducting the revised assessment, EPA considered this new information and agrees that the one percent false positive rate appears not to be achieved in some circumstances. EPA will continue to study this issue and notes that other commenters submitted a blank correction approach that could potentially improve false positive rate performance under certain conditions, and which we will explore in future consultations with stakeholders.

3. Evaluation and Selection of Alternative Concepts and Procedures

Several commenters stated that EPA inappropriately evaluated or rejected the International Union of Pure and Applied Chemistry and the International Standards Organization (IUPAC/ISO) critical value, and ASTM's Committee D19 on Water Interlaboratory Estimate of Detection (IDE) and Interlaboratory Estimate of Quantitation (IQE). Commenters also criticized EPA's choice of evaluation criteria. EPA agrees that some revisions to the evaluation criteria may be appropriate and will

explore this in future discussions with stakeholders.

EPA used the same evaluation criteria to evaluate all detection and quantitation approaches, including the IUPAC/ISO and ASTM IDE/IQE approaches. EPA did not reject either ASTM's approach or the concepts adopted by IUPAC and ISO. As described in the *Technical Support Document for the Assessment of Detection and Quantitation Approaches*, EPA identified many approaches that have been used to describe the sensitivity (i.e., the detection and quantitation capabilities) of analytical methods. EPA had focused the 2003 assessment on four sets of approaches that were either widely referenced or provided detailed instructions for use in the laboratory. The four approaches were: (1) ASTM's D19 committee IDE and IQE; (2) the LOD (limit of detection) and LOQ (limit of quantitation) adopted by the American Chemical Society; (3) the critical value, minimum detectable value, and limit of quantitation adopted by IUPAC and ISO; and (4) EPA's MDL and ML procedures. For the revised assessment, the Agency has expanded this evaluation to include three additional approaches. These three approaches were respectively submitted by the Inter-industry Analytical Group, U.S. Geological Survey, and the American Council of Independent Laboratories. The Revised Assessment Document also describes a quantitation procedure that is being developed by EPA's Office of Ground Water and Drinking Water. This quantitation procedure is known as the minimum reporting level or MRL. The Agency anticipates proposing the details of the MRL procedure in a rulemaking for public comment by mid-2005.

Several commenters requested that EPA continue its assessment by working with stakeholders to improve procedures for determining the detection and quantitation capabilities of analytical methods. EPA believes that there is benefit in continuing discussions and, in section II.E. of this notice, describes the beginning of a process for a series of discussions about these issues with stakeholders, such as permittees, permit writers, state regulators, nongovernmental organizations, and environmental groups.

B. Consensus Principles

Stakeholders commenting on EPA's 2003 assessment of detection and quantitation approaches expressed their support of a set of "consensus principles" submitted by 36 signatories.

The “consensus principles” described in this letter include the following:

- The definition of “quantitation” should account for both precision and bias.
- EPA should consider different uses of the MDL and ML in the Clean Water Act program (as a start-up test for a single laboratory, as a figure of merit to characterize an analytical method, as a permit compliance level, *etc.*), and evaluate the applicability of the MDL and ML to these uses.
- Definitions of and procedures for determining quantitation levels should take into account their use as regulatory compliance levels in NPDES permits, and the effects of routine variability within a laboratory on the results generated by the laboratory.

EPA notes that some of these “consensus principles” highlight existing aspects of approaches to detection and quantitation and provide a framework for future discussions with stakeholders. A more detailed description and additional discussion of these “consensus principles” is in Chapter 4 of the Revised Assessment Document.

C. Technical Issues

EPA considered, and is continuing to consider, several technical issues related to the development of detection and quantitation approaches. These issues are discussed in chapter 3 of the Revised Assessment Document. Commenters expressed concern regarding EPA’s consideration of several of these technical issues, specifically how these issues are, or are not, addressed by EPA’s MDL and ML. Specific concerns or suggestions expressed by commenters dealt with technical aspects of EPA’s assessment, such as treatment of sample blanks, instrument data censoring, false positive and false negative rates, and calculation of MLs. EPA addressed these comments in the Revised Assessment Document and/or the Response-to-Comments Document, and the Agency expects to further address these issues in a continued consultation with stakeholders.

IV. Next Steps

It is clear that there is a strong interest in improving current procedures and uses, but no consensus for a specific procedure or procedures has emerged among the laboratory, industry, regulatory or regulated communities. The Agency looks forward to working with stakeholders. Based on an analysis of comments received on the 2003 assessment and proposed revisions to the MDL procedure, issues for

consideration in future stakeholder consultations may include, but are not limited to, development of detection and quantitation procedures that:

- Vary in the nature and extent of statistical rigor and laboratory performance checks depending on the end use of a calculated limit in CWA programs;
- Account for more sources of variability, such as the variability between and within laboratories;
- Require more than seven samples and collect samples over a long period of time; and
- Use routine blank samples collected over long periods of time to account for background signals and temporal variability.

EPA has engaged a neutral third party to ask stakeholders for suggestions for additional issues, and about their interest in working with EPA to revise existing procedures and/or adopt one or more alternative procedures.

Dated: November 1, 2004.

Benjamin H. Grumbles,

Acting Assistant Administrator for Water.

[FR Doc. 04-24824 Filed 11-5-04; 8:45 am]

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ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[OW-2003-0002; FRL-7834-9]

RIN 2040-AD53

Guidelines Establishing Test Procedures for the Analysis of Pollutants; Procedures for Detection and Quantitation

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule; withdrawal.

SUMMARY: On March 12, 2003, EPA published a document in the **Federal Register** that proposed revisions to the regulations for the definition and procedure for EPA’s method detection limit (MDL). The document also proposed to add to these regulations a definition of minimum level of quantitation (ML) and a procedure for developing it. The proposed rule requested comment on the revisions and additions. The MDL and ML are used to characterize the capabilities of analytical test procedures applied under the Clean Water Act (CWA). The proposed revisions were based on EPA’s 2003 assessment of approaches to determining detection and quantitation capabilities of analytical methods.

Today’s document withdraws the proposed revisions. The proposed revisions were disfavored by the vast majority of commenters on the March 2003 proposed rule, and the Agency has determined that these proposed revisions do not represent the most effective way to address the public’s and EPA’s concerns regarding approaches to, and use of, detection and quantitation values. The Agency believes, preliminarily, that new approaches submitted in comments to the proposed rule might better address the issues EPA sought to address in its proposed revisions and that these new approaches warrant further consideration and refinement. Hence, EPA plans to work with stakeholders to evaluate one or more approaches to detection and quantitation that will satisfy the needs of programs, regulations, and initiatives at the Federal level for use of detection and quantitation procedures, and to revise its existing procedures, as appropriate.

DATES: For judicial review purposes, this action is considered issued as of November 8, 2004. Under section 509(b)(1) of the Clean Water Act, judicial review of the Administrator’s action regarding guidelines establishing test procedures for analysis of pollutants can only be had by filing a petition for review in the United States Court of Appeals within 120 days after the decision is considered issued for purposes of judicial review. Under 40 CFR 23.12, if within ten days of the issuance date of this action for purposes of judicial review EPA’s General Counsel receives two or more petitions filed in two or more United States Courts of Appeals, the General Counsel will notify the United States Judicial Panel of Multidistrict Litigation of all petitions received within the ten day period.

ADDRESSES: The docket for today’s action is available for public inspection under Docket ID No. OW-2003-0002 at the Water Docket in the EPA Docket Center, (EPA/DC) EPA West, Room B102, 1301 Constitution Ave., NW., Washington, DC.

FOR FURTHER INFORMATION CONTACT: William A. 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.

SUPPLEMENTARY INFORMATION: