

IDE—interlaboratory detection estimate
 IIAG—Inter-Industry Analytical Group
 IQE—interlaboratory quantitation estimate
 ISO—International Organization for Standardization
 IUPAC—International Union of Pure and Applied Chemistry
 LOD—limit of detection
 LOQ—limit of quantitation
 MDL—method detection limit
 MDV—minimum detectable value
 ML—minimum level of quantitation
 NBS—National Bureau of Standards (now NIST)
 NIST—National Institute of Standards and Technology (formerly NBS)
 NPDES—National pollutant discharge elimination system
 NTTAA—National Technology Transfer and Advancement Act
 OMB—Office of Management and Budget
 POTW—Publicly-owned treatment works
 RFA—Regulatory Flexibility Act
 SBREFA—Small Business Regulatory Enforcement Fairness Act
 SCC—Sample Control Center
 TSD—technical support document
 UMRA—Unfunded Mandates Reform Act
 USATHAMA—U.S. Army Toxic and Hazardous Materials Agency (now the U.S. Army Environmental Center [USAEC])
 U.S.C.—United States Code
 WQBEL—water-quality-based effluent limit
 WEF—Water Environment Federation

List of Subjects at 40 CFR Part 136

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

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Administrator.

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

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

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

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

2. Section 136.2 is amended by revising paragraph (f) and adding paragraph (g) to read as follows:

§ 136.2 Definitions.

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(f) *Detection limit* means the method detection limit (MDL), as determined by the procedure set forth at Appendix B of this part. The MDL is an estimate of the measured concentration at which there is 99% confidence that a given analyte is present in a given sample matrix.

(g) *Minimum level of quantitation* (ML) means the lowest level at which the entire analytical system gives a recognizable signal and acceptable calibration point for the analyte, as determined by the procedure set forth at Appendix B of this part. The ML represents the lowest concentration at which an analyte can be measured with a known level of confidence.

3. Appendix B of part 136 is revised to read as follows:

Appendix B to Part 136

A. Definition and Procedure for the Determination of the Method Detection Limit—Revision 2

1.0 Definition

The method detection limit (MDL) is an estimate of the measured concentration at which there is 99% confidence that a given analyte is present in a given sample matrix. The MDL is the concentration at which a decision is made regarding whether an analyte is detected by a given analytical method. The MDL is calculated from replicate analyses of a matrix containing the analyte and is functionally analogous to the “critical value” described by Currie (1968, 1995) and the Limit of Detection (LOD) described by the American Chemical Society (Keith et al., 1980, McDougal et al., 1983).

2.0 Scope and Application

2.1 This procedure is for the determination of an MDL for a given analyte (parameter) in a given matrix (the component or substrate that contains the analyte) using a given test procedure (analytical method). It is applicable to a wide variety of analytes, matrices, and instruments, and to a broad variety of physical and chemical analytical methods, with some exceptions (e.g., pH, temperature). This procedure is intended for use in EPA’s Clean Water Act (CWA) programs. An alternative procedure may be used (e.g., from a voluntary consensus standards body) to establish the sensitivity of an analytical method, provided the resulting detection limit meets the sensitivity needs for the specific application.

2.2 This procedure requires a complete, specific, and well-defined analytical method. It is essential that all sample processing steps of the analytical method that are applied to routine analyses be included in determination of an MDL.

2.3 This procedure may be used for a variety of applications, including, but not limited to:

2.3.1 Demonstrating laboratory capability with a particular method. A laboratory using this procedure to demonstrate capability with a particular method is not required to perform the iterative verification of the MDL

(section 4.8) if the laboratory-determined MDL is less than or equal to either the MDL in the method, the MDL required to support a regulation, or the objectives of a study (see section 4.8.5).

2.3.2 Monitoring trends in laboratory performance. When used in this manner, the MDL for a given analyte measured using a given analytical method may vary as a function of laboratory experience and the matrix tested.

2.3.3 Characterizing method sensitivity in a particular matrix. An MDL is typically determined in a reference matrix. However, it also may be determined in a real-world matrix to verify that the target MDL can be achieved in that matrix.

2.3.3.1 If the MDL required for a specific application can be achieved in a real-world matrix, that MDL may be used in lieu of a reference-matrix MDL, and iteration (section 4.8) is not necessary.

2.3.3.2 If the MDL needed for a specific application cannot be achieved in the real-world matrix (i.e., if the purpose of the MDL study is to demonstrate the effects of matrix interferences in a real world sample), the laboratory must (1) perform an MDL study in a reference matrix to demonstrate the laboratory’s ability to apply the method in the absence of interferences, and (2) verify the matrix-specific MDL through the iterative procedure given in section 4.8.

2.3.3.3 Refer to section 4.2 for additional information concerning the selection of test matrices.

2.3.4 Establishing an MDL for a new or revised method for nationwide use. When the procedure is used to establish an MDL for a new or revised method, the MDL should be derived from data obtained from multiple laboratories. Organizations developing or revising methods must document and make available the data and procedures used to establish an MDL to obtain approval for use under Clean Water Act programs.

3.0 Summary of the Procedure

3.1 The procedural steps required for determining an MDL vary with the intended application of the MDL. However, regardless of the intended application, all MDL determinations must include the following steps:

- (a) Estimating the detection limit of the method as practiced,
- (b) Selecting the appropriate matrix to be used in the determination,
- (c) Selecting the appropriate test concentration,
- (d) Preparing and analyzing a minimum of seven replicate aliquots of a blank or spiked matrix,
- (e) Calculating the mean concentration of the analyte, the standard deviation of that mean, and the MDL, using the formula provided in this procedure,
- (f) Comparing the calculated MDL to a method-specified MDL, relevant regulatory requirements, or project-specific objectives, as appropriate.

3.2 When developing MDLs for new or revised methods, or developing matrix-specific MDLs for nationwide use, the procedure also may include:

- (a) Conducting an optional pre-test using fewer replicates to verify that an appropriate