

to the pre-test procedure are intended to address this issue. These revisions now appear in section 4.1 of the proposed procedure. Specifically, the pre-test has been modified to suggest analysis of three aliquots. Results from these analyses are evaluated by calculating a preliminary MDL based on the standard deviation of the analyses, and then determining if this preliminary MDL is within 0.2 to 1.0 times the concentration spiked in the sample. This revision is consistent with the current procedure's recommendation that samples used to determine an MDL contain the analyte at a concentration that is "between 1 and 5 times the estimated method detection limit."

C. Editorial Changes to the MDL Procedure

This notice proposes editorial changes to the MDL procedure at 40 CFR part 136, Appendix B that are designed to clarify the existing procedure and improve readability. These editorial changes include changes to the numbering scheme, the addition of clearer titles to some of the procedural steps, and minor clarifications. Specifically, the proposed changes would:

1. Add a summary section to provide an overview of the various steps included in the MDL procedure. The summary section is consistent with the current format for analytical methods and should be particularly helpful to non-laboratory readers.

2. Clarify in the Scope and Application discussion that the MDL procedure is intended for use in EPA's Clean Water Act programs, and that alternative procedures may be used to establish test method sensitivity provided the resulting detection value meets the sensitivity needs for a specific application.

3. Clarify throughout the procedure that not all of the steps are required for every application. This clarification provides consistency with the proposed revisions in the Scope and Application section of the procedure.

4. Expand the discussion of matrix selection to address use of an MDL in either a reference matrix or an alternate matrix. (See Step 2 of the current procedure and section 4.2 of the proposed revisions.) Use of matrices other than reagent water are not discussed until Step 3b of the current procedure. The expanded discussion is intended to provide additional clarity and consistency with the description of the four applications added to the MDL Scope and Application section (see section VII.B.2 above).

5. Expand the discussion of establishing the test concentration range to more clearly describe the steps required and prepare the test samples. (See Step 3 of the current procedure and section 4.3 of the proposed revisions.) These proposed changes are editorial and describe the process that may be used for determining a matrix-specific MDL as well as determining an MDL in a reference matrix such as reagent water. Additional clarifications include recognition that some analytes may require that seven aliquots be prepared individually, as opposed to preparing a bulk sample of sufficient volume to be split into seven aliquots. EPA is proposing this clarification in response to questions from laboratories regarding the appropriate means for preparing the MDL aliquots.

6. Expand the discussion of performing the analyses to include a brief introduction clarifying that the samples used for MDL analyses must be processed using the sample handling, processing, and result calculations specified in the analytical method. (See Step 4 of the current procedure and section 4.4 of the proposed revisions.) This proposed change includes moving this statement from the Reporting section of the current procedure to the more appropriate location in section 4 of the revised procedure. This proposed change also would clarify that blank-correction or recovery-correction procedures are applied to the MDL analyses only when those procedures are employed for routine sample analyses, and precludes their use if they are not specified in the test method. EPA is proposing these changes in response to questions raised by laboratories, the Petitioners, and as a result of the recent assessment.

7. Reorganize the procedural steps contained in Step 4 of the current procedure, such that the optional pre-test of the MDL is discussed before the procedure for performing the full seven-replicate test. (See section 4.4 of the proposed revisions.) EPA is proposing this change strictly to improve ease of use.

8. Expand and reorganize the description of the seven-replicate version of the MDL described in Step 4(a) of the current procedure. The revised version would appear in section 4.5 and reflects comments from stakeholders that the discussion in the current procedure is not sufficiently clear. The revised procedure also would state explicitly that all analytical results must be positive numbers, and that the results from all aliquots analyzed must be used in the calculations, except those identified as outliers using the

procedures described in section 4.9 of the revised procedure (see the discussion regarding outliers in VII.B above). These proposed changes would clarify stakeholder concerns regarding those analytical methods (e.g., for metals) that may produce negative numbers at very low concentrations and would emphasize the revision made in response to concerns regarding inappropriate screening of results used for MDL determinations.

9. Simplify the calculations of standard deviation of replicate measurements in Step 5 of the current procedure. (See section 4.6 of the proposed revisions.) For example, the current procedure details the calculation of the sample variance (s^2), and then details the calculation of the sample standard deviation (s) in a separate equation. Given that the variance term does not factor into the MDL calculation directly, the proposed revision would require only calculation of the standard deviation. The proposed revision also would include a caution warning the reader to calculate the sample standard deviation (s), not the population standard deviation (σ), when using automated programs such as spreadsheets. This error was not as likely to occur in 1984, prior to the ready availability of personal computers and laboratory data systems, but is commonly seen today.

10. Move the table of Student's t -values from its current location following the text in Step 7 to section 4.7, where the t -value is employed. EPA is proposing this change to improve ease of use and increase readability.

11. Add a table of F-statistic values to the iterative procedure described in section 4.8. EPA is proposing this change to improve ease of use and address those instances in which an iterative MDL might be determined from other than seven replicates per data set.

12. Delete the "Reporting" section of the MDL procedure. The existing procedure includes a section listing the information that must be provided with the MDL for each analyte. EPA is proposing to delete this section because it is not relevant to the procedure and it is generally duplicative of reporting and recordkeeping requirements that States, other regulatory entities, or laboratory certification officials already require.

D. Definition and Procedure for Determining the Minimum Level of Quantitation

Although ML values for analytes were published in 1984 in EPA Methods 1624 and 1625 (49 FR 43234, October 26, 1984), the definition of the ML was