

provided in a footnote to the tables within those methods. The original definition was intended to define a minimum level of quantitation for these isotope dilution GC/MS methods. However, as described in the Assessment Document, EPA has changed the definition of the ML over the years and has expanded its applicability to other 40 CFR part 136 methods. This proposal requests comment on whether to add the following definition of the ML to Appendix B of 40 CFR part 136: “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.” In addition to the definition, EPA requests comment on whether Appendix B should contain an explicit explanation of the calculation of the ML from an MDL value determined using the revised MDL procedure, including a table of multiplier values that may be used when the MDL value is calculated from other than seven replicate analyses.

An alternative is to not incorporate a definition in § 136.2 but to continue to specify the ML on a method-by-method basis. In this case, the ML may continue to be determined and supported with data gathered during method validation studies. This approach would allow maximum flexibility to design studies that are appropriate for the intended use of the method.

A second alternative is to incorporate into § 136.2 the definition of limit of quantitation as “The lowest concentration of an analyte that can be measured with a defined level of confidence” and to incorporate the definition of ML (minimum level) as “The procedure set forth in Appendix B of this part of the same name, which can be used to estimate the limit of quantitation.”

In this proposal, EPA is also requesting comment on whether it should encourage or require that laboratories periodically demonstrate target analyte recovery at the ML by preparing and analyzing a reference matrix sample spiked at the ML using all sample handling and processing steps described in the method. EPA recognizes that existing methods do not provide acceptance criteria for such “ML standards.” Therefore, EPA suggests that, if the method does not provide acceptance criteria for an ML standard, acceptance criteria for other spiked reference matrix samples (e.g.,

laboratory control samples, laboratory fortified blanks, ongoing precision and recovery samples, etc.) may be used to evaluate analyte recovery at the ML. EPA is soliciting comment on whether this recommendation should be made into a mandatory requirement, retained as a recommendation, or replaced by an alternative recommendation for demonstrating recovery at the ML.

#### *E. Acceptance of Test Methods Employing Alternative Detection and Quantitation Procedures*

This proposed rule would allow use of alternative detection and quantitation procedures to establish detection and quantitation limits in an analytical method, provided that the resulting detection and quantitation limits meet the sensitivity needs for the specific application. The use of detection and quantitation approaches from voluntary consensus standards bodies (VCSBs) and other organizations is encouraged under the National Technology Transfer and Advancement Act (NTTAA), because it facilitates the approval of analytical methods from these organizations at 40 CFR part 136 without requiring that these organizations specifically employ EPA’s MDL and ML procedures to establish method sensitivity. This allowance would result in greater flexibility to establish or improve the sensitivity of methods for use under the Clean Water Act. It also would facilitate approval of analytical methods from VCSBs and other organizations. In selecting an appropriate test method for a specific purpose, the laboratory must always consider the sensitivity of the approved test methods. Only those test methods with the desired sensitivity should be used to meet the objective of the CWA “to restore and maintain the chemical, physical, and biological integrity of the Nation’s waters.”

EPA recognizes that there are alternative detection and quantitation approaches that may be used for determining test method sensitivity. EPA has included test methods at 40 CFR part 136 that employ alternative approaches, although some of these approaches have not been rigorously defined. In its recent assessment of detection and quantitation approaches, EPA evaluated the interlaboratory detection estimate (IDE) and the interlaboratory quantitation estimate (IQE) procedures published by ASTM International. However, EPA is not aware at this time of any published test methods from any source that include specific values for the IDE and the IQE, including test methods published by ASTM International. EPA will consider

test methods that include these procedures for use in CWA programs when such methods are available. If ASTM International is successful in developing single-laboratory adaptations of the IDE and IQE that may be used to verify the ability of a given laboratory to achieve the IDE and IQE, then EPA also may consider those single-laboratory approaches in evaluating both method and laboratory performance.

#### **VIII. Industry Proposal**

On December 27, 2002, the Inter-Industry Analytical Group (IIAG) submitted a proposal that recommends (1) a sensitivity test intended to “replace the MDL as a test of whether an individual laboratory is performing adequately,” and (2) an interlaboratory validation study design intended to characterize precision and accuracy of methods used for regulatory compliance. EPA did not have the opportunity to evaluate IIAG’s proposal against the criteria discussed in Section IV of this preamble, but intends to do so prior to publication of a final rule. EPA is providing a summary of the recommendations contained in the “Inter-Industry Analytical Group Proposal for Sensitivity Test and Full-Range Interlaboratory Validation Study” here. The complete text of the recommendations has been placed in the docket supporting this proposed rule. EPA is soliciting comment on the industry recommendations.

IIAG is proposing a sensitivity test in place of the MDL for determining laboratory performance capability. The proposed sensitivity test includes the provision that EPA first determine the lowest calibration point of a method, prescribe a dilution of that calibration point as the spike level (e.g., at one-half or two-thirds the lowest calibration point), specify a required number of replicates, and set a quality control acceptance criterion. IIAG asserts that an advantage of such a test is that it would provide all laboratories with a single spike level and an “unambiguous pass or no-pass test.” EPA is soliciting comment on approaches that might be considered appropriate for such determinations (i.e., the lowest calibration point of a method, an appropriate dilution, a number of replicates, and an acceptance criterion for standard deviation between measurements of the replicates). EPA also is soliciting comment on how IIAG’s recommended sensitivity test would be either more appropriate or less appropriate than either the current MDL and ML procedures or the MDL and ML