Working Draft Standard

EL-V1M4 Section 1.5.2

November 2014

Description

This proposed standard is designed to replace Section 1.5.2 of EL-V1M4-2009-Rev1.1.

1.5.2 Limit of Detection and Limit of Quantitation (However Named)

1.5.2.1 Limit of Detection (LOD)

If a mandated test method or applicable regulation includes protocols for determining detection limits, these shall be followed. If the protocol for determining the LOD is not specified the laboratory shall document how LODs are to be determined. One option is to use the spiked blanks required for verification of the LOQ (Sec. 1.5.2.2) to calculate an LOD following EPA's MDL procedure.

a) An LOD study is not required for any analytical parameter for which spiking solutions or quality control samples are not available such as temperature, or where a detection limit is not applicable such as pH or flashpoint.

b) The LOD shall be initially determined for the analytes of interest in each test method in a quality system matrix in which there are neither target analytes nor interferences at a concentration that would impact the results or the LOD shall be performed in the sample matrix of interest.

c) When method modifications and/or instrumentation modifications or changes occur that may affect detection sensitivity or selectivity, the LOD determination shall be repeated prior to processing samples.

d) The LOD, if required, shall be verified annually for each quality system matrix, technology, method, and analyte.

1.5.2.2 Limit of Quantitation (LOQ)

The laboratory shall select a LOQ for each analyte, consistent with the needs of their clients, and at least three times the LOD. An LOQ is required for each quality system matrix, technology, method, and analyte, except: An LOQ is not required for any component or property for which spiking solutions or quality control samples are not available such as temperature, or where a quantitation limit is not applicable such as pH or flashpoint.

a) Each selected LOQ shall be verified through analysis of initial verification samples. An initial verification sample consists of a blank or matrix spiked at or below the selected LOQ.

b) All sample processing and analysis steps shall be included in the LOQ verification testing.

c) The LOQ must be at or above the lowest calibration standard concentration.

1.5.2.2.1 Initial Verification of the LOQ

When first establishing an LOQ or when an LOQ concentration has been selected that is lower than the concentration of the LOQ verification spikes previously performed, an initial verification shall be performed as follows:

a) Process a minimum of 7 initial verification samples spiked at or below the LOQ concentration through all steps of the method, including any required sample preservation. Both preparation and analysis of these samples must include at least 3 batches on 3 separate days.
   Note: Spiking slightly below the LOQ may help ensure that the results are also suitable for LOD determination.
   Note: If spiked blanks have been analyzed in order to generate a LOD, the same spiked blanks may be used to verify the LOQ.

   i) If there are multiple instruments that will be assigned the same LOQ, then the initial verification samples must be distributed across all of the instruments

   ii) A minimum of two initial verification samples prepared and analyzed on different days is required for each instrument

b) Existing data may be used if compliant with the requirements for at least 3 batches, generated within the last 2 years and representative of current operations

c) The LOQ is verified if the following criteria are met:

   i) All results meet the qualitative identification criteria of the method (e.g., recognizable spectra, signal to noise requirements, and presence of qualifier ions

   ii) The LOQ must be at least 3X the current LOD.

   iii) If the results from the LOQ verification samples do not meet the qualitative identification criteria in the method, the problem must be corrected and the verification repeated, or the LOQ and LOQ spikes must be repeated at a higher concentration.

   iv) If the LOQ is less than 3 times the LOD, the LOQ shall be raised to at least 3 times the LOD.

   Note: It is not necessary to repeat the LOQ verification at a higher concentration when it is necessary to raise the LOQ to 3 times the LOD.

d) Document the results of the initial LOQ verification as described in section 1.5.2.2.2.3

1.5.2.2.2 Continuing verification of the LOQ

Prepare and analyze a minimum of one LOQ verification sample on each instrument during each quarter in which samples are being analyzed for the quality system matrix, technology, and analyte.

a) Results of each LOQ verification sample analysis must be evaluated at the time of the testing and must meet the qualitative identification criteria in the method.

   i) If a continuing LOQ verification test does not meet this requirement, the laboratory must take corrective action. Corrective action shall be either (i) raising the spiking level (and the quantitation limit if the spiking level is above it) and repeating the initial verification study, or (ii) correcting method or instrument performance and repeating the verification test one time. In the event of second failure of a quarterly verification sample, the quantitation limit must be raised and the initial study repeated.

b) At least once per year tabulate all results of the ongoing verification sample testing. Use all data representative of the current operations, if generated within the last two years.

c) If an LOD has been newly established or changed within the year, verify that the LOQ is still at least 3X the LOD

d) Document the results of the continuing LOQ verification as described in section 1.5.2.2.2.3

1.5.2.2.2.3 Documentation

a) Include the dates of preparation and testing, the batch identifiers, the testing instrument, quality system matrix, technology, analyte, spiking concentration and the test result (if any) for each LOQ verification test made.

b) Calculate the percent recovery for each result.

c) Total the number of data points for each analyte. Determine the mean and the standard deviation of the percent recovery for each analyte.

   i) If any results were not included in the calculation, record the reason for exclusion.

d) The spiking concentration, mean percent recovery, standard deviation of percent recovery and n value shall be provided to clients upon request, or used to calculate project-specific precision and bias or measurement uncertainty statements.