Limit of Detection Verification

Background

The 2009 TNI Standard, Volume 1, Module 4, section 1.5.2 requires that all laboratories perform annual evaluations of their Limit of Detection (LOD) or Limit of Quantitation (LOQ) for each quality system matrix, technology, and analyte (where appropriate). In Florida, laboratories are required to report their detection limits, thus, pursuant to section 1.5.2, the LOD verification is required rather than the LOQ verification.

In the 2009 TNI Standard, Volume 1, Module 2, Section 3.1 the LOD is defined as "A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility". The intent of this definition is ambiguous, primarily because the 'detection limits' have been defined and interpreted in different manners by various organizations and rules. Chapter 40, Part 136 Appendix B of the Code of Federal Regulations, which has been adopted for use in Florida, sets 'method detection limits' as the critical value separating methodological signals from noise with a 1% false positive rate (Type 1 error). The International Union of Pure and Applied Chemistry (IUPAC) and the International Standards Organization define the minimum detectable value or detection limit as the level defined by a false negative rate (Type II error) relative to a critical value with a defined false positive rate. Until this ambiguity is resolved the laboratory will interpret the TNI definition of 'LOD' to be the corresponding concentration at the critical value which separates signals from noise with a 1% false positive rate.

Implementation

The following interpretation of the requirements in the 2009 TNI Standard, Volume 1, Module 4, section 1.5.2 and additional guidance shall be implemented when verifying the LOD:

1. The LOD shall be verified annually for each analyte/matrix/test combination and on each analytical instrument used to report results. This includes identical analytical instruments that have the same detection limits and are used to report the same suite of analytes such as multiple GC-ECD instruments. All routine sample processing steps shall be performed when verifying the LOD.

2. The analyte in the applicable laboratory control sample (LCS) shall be fortified at a concentration near the LOD; that concentration shall be no more than 3 times the LOD (from the previous year) for single component tests and no more than 4 times the LOD for multi-component tests. Because the laboratory defined the LOD as the critical value where the false negative rate is 50%, it is not recommended to fortify the laboratory control sample (LCS) used for this verification at a level equal to the LOD (where 50% of the measurements will yield a non-detect).

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3. For LOD verifications carried out for tests on non-potable water, the LCS shall consist of fortified laboratory grade water. For tests on solids or biological materials, where no matrix sample is readily available, the LCS shall consist of fortified silica sand or Teflon or glass beads with sufficient solvent or laboratory grade water to support the fortification. For cases where a surrogate matrix which does not contribute analyte or contaminate the fortified sample with the analyte of interest (at levels greater than the detection limit) cannot be found, the LOD verification shall be performed in the absence of a matrix (e.g., using laboratory grade water instead of another matrix or surrogate matrix).

4. LOD verification shall consist of positive detection equal to or greater than the previously established method detection limit (i.e., a signal distinctly greater than the methodological noise distribution) or qualitative identification of the analyte signal based on mass identification, retention time or other unique characteristic deemed appropriate by the unit supervisor. In addition, where required by methods employing mass spectrometric detection (such as EPA 8260, 8270, etc.), the secondary identification ions must be present in the LOD verification at acceptable ratios defined by method or SOP relative to primary quantification ions.

5. Unless verified with each analytical run, annual LOD verifications shall be documented in the maintenance logbook of each analytical instrument used to report results, where LOD verification is applicable. The logbook documentation shall contain information sufficient to retrieve the raw analytical data files.

6. An initial LOD shall be determined for each analyte/matrix/test combination whenever a new instrument is set-up, any time an existing instrument undergoes major repairs or modifications and whenever major method modifications are implemented.

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