Method Detection Limit Verification

1.0 BACKGROUND

The 2016 TNI Standard, Volume 1, Module 4, section 1.5.2 requires that all laboratories perform a minimum of two verification spikes and two blanks every quarter to evaluate their Method Detection Limit (MDL) 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 MDL verification is required rather than the LOQ verification.

In the 2016 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 discriminate from a blank with a predetermined confidence level". Chapter 40, Part 136 Appendix B, Revision 2 (2017) 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 those of a blank with a 1% false positive rate (Type 1 error).

2.0 IMPLEMENTATION

The following interpretation of the requirements in the 2016 TNI Standard, Volume 1, Module 4, section 1.5.2 and additional guidance shall be implemented when verifying the MDL:

2.1 The MDL shall be verified every quarter (4 times a year) for each analyte/matrix/test combination and on each analytical instrument used to report results. A minimum of two blanks and two low-level spikes should be prepared in separate batches and analyzed in separate batches every quarter. 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 MDL.

2.2 The analyte in the applicable laboratory control sample (LCS) shall be fortified at a concentration near the MDL; that concentration shall be no more than 3 times the MDL (from the previous year) for single component tests and no more than 4 times the MDL for multi-component tests. Because the laboratory defines the MDL 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 MDL (where 50% of the measurements will yield a non-detect). A spike that is at least twice the value of the MDL is recommended.

2.3 For MDL verifications carried out for tests on non-potable water, the LCS shall consist of fortified laboratory grade water. For tests on solids or biological

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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 MDL verification shall be performed in the absence of a matrix (e.g., using laboratory grade water instead of another matrix or surrogate matrix).

2.4 MDL verifications shall consist of positive detection equal to or greater than the previously established method detection limits (i.e., a signal distinctly greater than the method blank) 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 MDL verification at acceptable ratios defined by method or SOP relative to primary quantification ions.

2.5 All routine and quarterly MDL verification data must be uploaded and to the LIMS Statistics Tracking module.

2.6 The spiking level should be re-evaluated at least once a year. Recalculate the MDL at least once every thirteen months using the procedure described in SOP LB-007. Include data generated within the last twenty-four months, but only data with the same spiking level and acceptable calibrations and batch QC.

2.7 If the verified (recalculated) MDL is within 0.5 to 2.0 times the existing MDL, and fewer than 3% of method blank results have values above the existing MDL, then the existing MDL may be left unchanged. Otherwise, adjust the MDL to the new verification MDL.

2.8 An initial MDL 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. The procedure for determining initial MDLs is documented in SOP LB-007.

3.0 REFERENCES

3.1 DEP SOP LB-007, *Procedure and Policy for Demonstration of Capability for Methods, Instruments and Laboratory Staff.*

3.2 2016 TNI Standard, Volume 1, Module 4, section 1.5.2.

3.3 EPA 821-R-16-006, *Definition and Procedure for the Determination of the Method Detection Limit, Revision 2, December 2016.*

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