STATE OF CONNECTICUT
DEPARTMENT OF PUBLIC HEALTH

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EHS Circular Letter #2013-65

Date: December 27, 2013

To: All Connecticut In-state Certified Laboratories

From: Dermot Jones, MS, MPH, CPH
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Environmental Laboratory Certification Program (ELCP)

Re: New Policy regarding MDL, LOD, and LOQ Calculations

The Environmental Laboratory Certification program (ELCP) office has been working with the laboratory advisory committee, in considering a policy change with respect to a laboratory's ability to characterize and document instrument response to a low level analyte concentration for specific test methods. The ELCP office will now allow laboratories the option to use two statistical formulas – method detection limit (MDL), and limit of detection (LOD) – to demonstrate the minimum concentration of an analyte that can be identified, statistically measured, and qualitatively reported by a method with 99% confidence that the analyte concentration is greater than zero. Laboratories will also perform the limit of quantitation (LOQ) study to demonstrate instrument response to the lowest level calibration standard.

Currently laboratories certified by the ELCP office are asked to demonstrate instrument response to low level concentration standards by annually calculating the MDL for a given analyte for which the laboratory is certified. National Environmental Laboratory Accreditation Program (NELAP) accredited out-of-state laboratories (certified in Connecticut through reciprocity with the accrediting state) calculate the LOD and LOQ annually and have been submitting these results to the ELCP office in lieu of the MDL calculation. At best every 2 years during the registration renewal process, the ELCP office is able to get MDL results from these out-of-state laboratories and seemingly this is the only time when MDL studies are calculated by these laboratories.

Since the LOD and LOQ calculations are more efficient and more meaningful to perform compared to the MDL study and as well provide good information on instrument performance, this letter will be notification for when and at what frequency the MDL, LOD and LOQ calculations will be performed by Connecticut in-state certified environmental laboratories.

MDLs shall be determined as required by EPA methodology, when a new laboratory analyst is hired to perform analyses by a specific method where the calculation of an MDL is indicated, when a new analysis using specific instrumentation is performed, or whenever, in the judgment of the Director, a change in analytical performance caused by either a change in instrument hardware, operating conditions or the analytical procedure would dictate they be recalculated.

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It is expected that for the methods involving the use of essential instrumentation an MDL must be calculated. For example, it would not be necessary for a laboratory to have an MDL established for a manual titration method but for an automated titration method it would be necessary to calculate an MDL. The procedures outlined in Appendix B to 40 CFR Part 136 of the Federal Register have been routinely used to calculate MDL's.

As noted above, the LOD is defined by EPA as the lowest concentration that can be determined to be statistically different from a blank. This concentration is recommended to be three standard deviations above the measured average difference between the sample and blank signals which corresponds to the 99% confidence level. In practice, detection of an analyte by an instrument is often based on the extent to which the analyte signal exceeds peak-to-peak noise (Keith et al., 1983). This definition is similar to how an MDL is defined.

Since the definition of the LOD and MDL are very similar the analytical verification of the LOD for the various methods laboratories perform can be associated to existing MDL data on file for these methods. The LOD study may also be calculated based on the standard deviation of the response (SD) and the slope of the calibration curve (S) at concentrations approximating the LOD according to the formula: LOD = 3.3(SD/S).

Annually a validation check of the LOD needs to be performed and typically analyzing a standard at $^1/_3$ to $^1/_2$ the LOQ would be an acceptable way to perform this check. The recovery for the LOD validation check must be at a minimum of 50% of the true value and/or have a response of at least three times the instrument's "noise". However, if the LOD validation check does not pass, the MDL study or an acceptable alternate procedure must be performed to redetermine and verify the LOD level.

A laboratory is required to annually validate its LOD only if a test result is reported below the LOQ. **Although for drinking water methods it is recommended that this check be done regardless of the established LOQs for these methods.**

The LOQ is defined by EPA as the level above which quantitative results may be obtained. The corresponding sample/blank difference is recommended to be 10 standard deviations above the blank which corresponds to the 99% confidence level (Keith et al., 1983) and to an uncertainty of ±30% in the measured value at the LOQ. LOQ is typically used to define the lower limit of the useful range of the measurement technology in use or the low level calibration standard. The LOQ is considered the reporting limit of a method.

The LOQ must be validated at a minimum annually and required method criteria would apply to acceptance of the LOQ check. The formula based on the standard deviation of the response (SD) and the slope of the calibration curve (S) according to the formula: LOQ = 10(SD/S) can be used to calculate the LOQ.

The implementation of both the LOD and LOQ verifications and annual validation checks would replace the annual MDL study requirement beginning in 2014.

If you have any concerns with this notification please contact the program office at 860-509-7389 or e-mail dermot.jones@ct.gov or philip.schlossberg@ct.gov .

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