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January 12, 2015

Richard Burrows

Chemistry Expert Committee Chair

The NELAC Institute

Sent via Electronic Mail to: Richard.Burrows@testamericainc.com

Subject: NACWA Comments on The NELAC Institute - Working Draft Standard; EL-V1M4 Section 1.5.2 (Limit of Detection and Limit of Quantitation)

Dear Mr. Burrows,

The National Association of Clean Water Agencies (NACWA) appreciates the opportunity to comment on The NELAC Institute's (TNI) Working Draft Standard: EL-V1M4 Section 1.5.2 (Limit of Detection and Limit of Quantitation). As you know, NACWA has carefully tracked efforts to better define how the limit of detection (LOD) and limit of quantitation (LOQ) should be established, including participating together with you as a member of EPA's Federal Advisory Committee on Detection and Quantitation.

NACWA, with input from Dr. James Pletl, Director of Water Quality for the Hampton Roads Sanitation District and Chair of NACWA's Water Quality Committee, would like to offer the following comments for your consideration.

General Comments

- The purpose of the document is not clear and it could be used in at least two different ways. One would be to determine the sensitivity of instrumentation and the ability to quantify a specific component at a given level. The LOQ verification samples would not need to go through the entire analytical process if this were the purpose and should be handled and processed the same way as calibration standards. Another way the document could be used would be to determine the ability of an analytical system to quantify at a specific concentration. The LOQ verification samples for this purpose would need to go through the entire analytical process, including sample preparation. The document needs to be clearer in its purpose given these and other possible interpretations.

- It is not clear whether the LOD section of the text applies to analyses that are not censored to LOD. It is recommended that language clarifying this point be added.
- Acceptance ranges for accuracy and precision should be specified in the standard given that the LOQ verification procedure is quantitative.
- The requirement that the LOQ be at least three times the LOD is arbitrary. It should only be stated that the LOQ must be greater than the LOD and verified whenever operations change and the LOD or LOQ are no longer representative of current performance.
- The text does not include criteria for selecting LOD or LOQ calculation statistics. Such criteria will be extremely important in ensuring that LODs and/or LOQs are reliable for their use.

Specific Comments by Section

Section 1.5.2.1

- There are a number of requirements that apply to calculation of the LOQ that should apply to the calculation of the LOD. Text should be added to address these points:
 - Need to represent current operations;
 - Need to include the entire analytical process;
 - Minimum number of samples and batch;
 - Requirement to analyze samples over multiple days; and
 - Requirement to analyze samples for multiple instruments and combine results to calculate one limit.
- Section 1.5.2.2 is structured to address initial and continuing verification for LOQ. The text for this section should be similarly structured.
- It is not clear as to whether a laboratory must recalculate the LOD when the quality system matrix changes. Language addressing this question should be added to the document.
- The text makes clear that LOD and LOQ are matrix dependent but procedures to determine whether matrices are different and require different LODs and LOQs has not been provided. The text needs to address how matrices are compared and differences established.
- It is recommended that the text use the terms “analyze”, “analyzing”, and “analysis” globally to replace “processing”, “test”, and “testing” to be consistent and avoid confusion in interpretation.
- A requirement to annually determine operational variability is arbitrary. The text needs to provide criteria that a lab can use to determine if LOD or LOQ are no longer representative of

operations. For example, the text could provide procedures for how a laboratory determines if detection sensitivity or selectivity has changed.

Section 1.5.2.2

- The text does not address the implications of mandated methods and regulations similar to the approach used in section 1.5.2.1. Since mandated methods and regulations can impact the use and development of the LOQ the inclusion of such language will be important and is recommended.
- The text does not address whether a lab needs to document how a LOQ is calculated; however, the text regarding the LOD addresses this point. Documentation of the LOQ development procedure is important and should be a requirement of this standard.

Section 1.5.2.2.1

- The requirements for number of samples, number of batches and number of days have seemingly been set independent of the calculation procedure used. This is not defensible unless a demonstration has been made that meeting these criteria will satisfy all data quality objectives associated with setting LODs and LOQs regardless of calculation procedure. This demonstration needs to be provided for the current requirements to be set or the text needs to be changed to simply state that the calculation procedure must address minimum number of samples, batches and days of analysis.
- Section 1.5.2.2.1(b) states that data up to 2 years old can be used in the calculation. This is arbitrary. The data used in the calculation needs to be representative of current operations. The text needs to provide a procedure for determining whether data is representative of current conditions and the text regarding data up to 2 years of age needs to be deleted.

Section 1.5.2.2.2

- The text does not address the situation where only one of three instruments does not meet the qualitative identification criteria required. This may occur and requirements should be set if these circumstances (where one of several instruments does not perform similarly to the other instruments) occur.
- As stated above, delete the language regarding the use of data up to 2 years of age and provide text that will guide a lab on how to determine if data are representative of current conditions.

Section 1.5.2.2.2.3

- The text for this section is unclear as to whether “shall” only applies to client requests or whether it also applies to calculating project-specific precision and bias as well as measurement uncertainty statements. The text needs to be adjusted to clarify intent.

NACWA Comments on TNI LOD/LOQ Standard

January 12, 2015

Page 4 of 4

Again, NACWA appreciates the opportunity to comment on the Working Draft Standard, "Limit of Detection and Limit of Quantitation". Please contact me at 202-833-9106 or chornback@nacwa.org should you have any questions about our comments or would like to discuss further.

Sincerely,

A handwritten signature in black ink, appearing to read "Chris Hornback", with a stylized, cursive script.

Chris Hornback

Senior Director, Regulatory Affairs