SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

APRIL 24, 2015

The Committee held a conference call on Friday, April 24, 2015, at 2:00 pm EST. Chair Richard Burrows led the meeting.

1 – Roll call

Richard Burrows, Test America (Lab)	Present
Francoise Chauvin, NYC DEP (Lab)	Absent
Brooke Connor (Other)	Present
Gale Warren, NYSDOH (Accreditation Body)	Present
Colin Wright, Florida DEP (Lab)	Absent
JD Gentry, ESC (Lab)	Absent
Nancy Grams, Advanced Earth Technologists, Inc. (Other)	Present
Anand Mudambi, USEPA (Other)	Present
John Phillips, Ford Motor Co. (Other)	Present
Scott Siders, IL DEP (Accreditation Body)	Absent
Valerie Slaven, Teklab (Laboratory)	Present
Gary Ward, OR DPH (Accreditation Body)	Absent
Ken Jackson, Program Administrator	Absent

Associate Committee Members present: Eric Davis; Arthur Denny; Diana Shannon

2 – Previous Minutes

It was moved by Anand and seconded by John to approve the minutes of April 10, 2015. All were in favor, except Nancy who abstained.

3 – Calibration Interim Standard

Discussion continued on when standards can be removed sequentially. Scott had suggested the following revised language for 1.7.1.1 d, that avoided the use of "sequentially": "*The laboratory may, only in consecutive order (e.g., standard concentration level 1 then standard concentration level 2), remove standard concentrations from the lowest level and/or highest level of the calibration curve for individual analytes on a case by case basis due to high or low sensitivity for the analyte. The laboratory shall not remove standard concentrations from the interior (e.g., remove standard concentration level 2 without removing standard concentration level 1) of the calibration curve for an individual analyte*." Richard, in turn, proposed a modification of Scott's language: "*The laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of interior levels is not permitted.*" Richard's language was adopted.

4 – Consideration of Comments on the Detection/Quantitation Working Draft Standard

The Discussion was continued from the previous call.

1.5.2.2.3 b "*Calculate the percent recovery for each result. What criteria is being set for % percent recovery, SD and mean?*." John had said no criteria were set, the requirement being only to document them. The laboratory will however need to meet their client requirements. He said perhaps we should add to 1.5.2.2.2 d), "to verify that client requirements are being met." The committee agreed with John's response, but with the last sentence deleted.

1.5.2.2.3.c.i There were two related comments: "*If any results were not included in the calculation, record the reason for exclusion*.", and "*Who decides which data points aren't used and what criteria are used to allow a lab to remove points? Shouldn't this be part of the procedure they have to establish to determine the LOD and LOQ".* Richard said Section 1.5.2.2.2 b says all results are to be tabulated for on-going sample verification testing. All data representative of the current operations must be used, if generated within the last two years. It was decided to remove the sentence "*If any results were not included in the calculation, record the reason for exclusion*."

1.5.2.2.3 d "*This isn't a sentence. And doesn't tell a lab what to do and what is required."* The Committee decided to strike the second sentence.

1.5.2.2.3 d *"Change the wording from "…or used to calculate project-specific precision and bias or measurement uncertainty statements." To "…or used to calculate method-specific precision and bias or analytical uncertainty statements." The generation of this data in this section uses quality system matrix and not a project specific matrix or sample collection precision and bias. Therefore all aspects of the measurement are not accounted for with this standard deviation and the standard should not state that these are project specific or a complete measurement uncertainty."* This sentence had been removed.

1.5.2.2 a *"The intent for the requirement of "qualitative identification" in 1.5.2.2.2.a is unclear in the context of LOQ discussions. It is suggested that this requirement is removed or clarified. It is also suggested that the requirement to calculate percent recovery (indicated in 1.5.2.2.2.3.b) is mentioned here. Or, if the intent of this language ONLY applies to analyses such as GC/MS then these references throughout the document to qualitative identification need some "notes" to better explain these requirements."* The committee discussed at length the question of qualitative identification. Richard said this could be difficult to define; e.g., for ICPAES it could just be getting a signal at the requisite wavelength. It was suggested it could be a result above the detection limit or above zero. The committee referred back to the fourth bullet of Section 1.5.2.1, and added "and shall be above zero"

1.5.2.2 b *"Section 1.5.2.2.2.b and c just don't make sense to me. Why tabulate once per year and what does the lab do with this data? The standard doesn't tell you want to do or how to treat it, just to tabulate it. And, why use all data generated within the last 2 years?"* The committee added "Update documentation as described in section 1.5.2.2.3."

1.5.2.2 c *"should either read "The concentration of the LOQ verification sample must be…" or "The concentration of the established LOQ must be…" You can see that some key words are missing and need to be added to ensure proper understanding and interpretation of this section. And, why is the lab running calibration standards to be used in the calibration routine that are below the limit of*

quantitation? Wouldn't this bias the calibration curve?" For clarification, it was agreed to say "The concentration of the established LOQ must be…"

1.5.2.2 c *"Requiring LOQ to be at or above the lowest calibration standard is not applicable to ICP methods."* Gale questioned if this means the established LOQ or the sample used to establish the LOQ? Richard said it is the LOQ value. This was added, with the exception of methods using a single-point calibration.

1.5.2.2 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. Does this allow for a liquid LOQ sample even with soil analyses? In other words, does this have to be matrix specific or can a single liquid spike for say metals verify all matrices? The prior section seems to negate that, but if a lab is using blank water for a method blank for soil sample prep batches, then the wording of this section would allow the same blank matrix for use in the LOQ verification."* John said you must use either the specific matrix or a quality control matrix. Richard added if you cannot get a solid matrix that is uncontaminated (e.g., metals), you have to use a water blank, so then it is acceptable to use the same matrix that is being used for the method blank. After discussion the Committee decided the language did not need to be changed.

1.5.2.2 a "*Sounds as though a blank can be used to verify the LOQ. Does it actually mean spiked matrix or spiked blank?"* John said perhaps the word "spiked" should be added after blank for clarification. It was agreed to say "spiked matrix blank".

1.5.2.2 c "*The LOQ must be at or above the lowest calibration standard concentration. Why would this be limited to at or above? Why can't it be below the lowest level of quantitation? If proving a lower level and it meets the criteria for acceptance, then wouldn't it also be implied that a higher level would also be acceptable for LOQ? This is actually contradicted in the second note of 1.5.2.2.1 a)."* John disagreed it is contradictory. The reason the LOQ must be at or above the lowest calibration standard is so that it falls within the calibration range. It was decided the language did not need to be clarified.

Several general comments (not referencing a specific section) were discussed. An Accreditation Body member had made a number of comments, and Richard thought some of the revisions would address those concerns. Some comments were about the LOD section and it had been revised.

A commenter said "*There are numerous comments regarding raising the LOQ whenever the verification doesn't meet the requirement. THE LABORATORY DOES NOT HAVE THE OPTION TO RAISE THE LOQ WHENEVER THEY CHOOSE. These are set by regulation or project. There simply is no difference in the usability of a data point that was "Non-detect" with the LOQ at 3.5X the LOD and one with the LOQ at 2X the LOD yet the later data would be deemed invalid."* Richard agreed the laboratory does not have the option to raise the LOQ , but if the LOQ does not meet criteria then they need to improve their methodology so it does.

It was suggested the committee offer a webinar to explain the standard, and Richard agreed.

A commenter was concerned that the standard depended on the EPA acceptance of the updated MDL procedure.

It was commented: "*Conspicuously absent is how to do the LOD verification, yet it does say 'how to' on the LOQ verification. That is, the draft goes from describing the LOD and LOQ (1.5.2.1 and 1.5.2.2) straight to how to verify the LOQ. I assume that is on purpose to allow LOD verification however the lab defines (?). In my opinion, the whole idea of LOD verification is mistaken. The MDL, usually used as a TNI LOD, is equivalent to Currie's Lc, or critical value. There is no expectation that a true concentration at the Lc would give a detectable result. Only that if you get a result above Lc that indicates that there is actually something in the sample. Therefore, it makes no sense to use a spike to "verify" the LOD. The way to verify the LOD (MDL) is to ensure that your method blanks do not give a result above the MDL. There is a concentration above the MDL that could in principle be verified with spikes, and that is Currie's Ld. Ld is used in the DOD QSM, but not routinely in environmental analysis and we did not want to complicate the issue too much by adding it. Ld is the true concentration that will reliably (say 95% confidence) give a result above Lc*." The Committee ensured that a true concentration at or above the LOQ would give results above the MDL; i.e., the LOQ would have at least the properties of Currie's Ld. Section 1.5.2.1d had been modified.

A commenter said: "*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*." Richard pointed out it does say you have to go through the whole process.

A commenter thought it was not clear whether the LOD section of the text applies to analyses that are not censored to LOD. It was recommended that language clarifying this point be added. John said this was addressed with the requirement for both blanks and low level spikes. It says you do have to do initial in that case, but not continuing.

It was commented that 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. John responded while any multiplier of the LOD (MDL) will be arbitrary, 3x does have some basis based on a fixed set of assumptions, used by the USEPA. While not perfect a 3x multiplier provides greater protection against false negatives at the LOQ than would a requirement that the LOQ > LOD.

It was criticized that 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. John said this is true, and for this reason the committee has considered making the proposed revisions to 40 CFR Part 136, Appendix B procedure a requirement for determining the LOD. Richard added that most laboratories do the MDL anyway.

Another comment was that the MDL_b was not described in the draft procedure, and is it required to analyze seven method blanks along with seven LOD/LOQ spikes? The committee responded this would be the primary way the LOD would be determined.

This completed the discussion. Richard said he would make all the agreed changes and then send it out for everyone to read and check if anything else needs to done. In particular, it should be checked that the general comments were addressed. This would then be considered during the next call, and then it was hoped a Voting Draft Standard would be ready. Also on the next call, the comments to EPA on 40 CFR Part 136 needed to be finalized.

5 – Adjournment

The meeting was adjourned at 3:20 pm EST.