SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

JANUARY 9, 2015

The Committee held a conference call on Friday, January 9, 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)	Present
Brooke Connor (Other)	Present
Gale Warren, NYSDOH (Accreditation Body)	Present
Colin Wright, Florida DEP (Lab)	Present
JD Gentry, ESC (Lab)	Present
Nancy Grams, Advanced Earth Technologists, Inc. (Other)	Present
Anand Mudambi, USEPA (Other)	Absent
John Phillips, Ford Motor Co. (Other)	Present
Scott Siders, IL DEP (AB)	Absent
Gary Ward, OR DPH (AB)	Absent
Ken Jackson, Program Administrator	Present

Associate Committee members present: Tim Fitzpatrick; Reed Jeffrey; Dixie Marlin.

2 – Previous Minutes

It was moved by John and seconded by Francoise to approve the minutes of December 12, 2014. All were in favor, except Nancy and Colin who abstained.

3 – Working Draft Standard on Detection and Quantitation

Several written comments on the Working Draft Standard were discussed.

Carl Kircher, 1.5.2.2.c. "*I would insert an additional word into the sentence as follows: "The LOQ must be at or above the corresponding lowest calibration standard concentration. The reason for this addition is that calibrations are oftentimes constructed on a digestate or extract basis (or even on a mass-loading basis), but LOQ is expressed on a whole-sample basis."* The committee agreed the existence of a preparation factor should be recognized, but Francoise did not like "corresponding". Gale said laboratories should understand it because that is the word generally used. It was agreed to leave it that way, with "corresponding", for now and perhaps change it later.

Carl Kircher, 1.5.2.2.d and 1.5.2.2.3. This was to correct a typographical mistake in the clause numbers.

Carl Kircher, 1.5.2.2.1. "*There are occasions where a laboratory might do the Initial LOQ Verification in one matrix but extrapolate the results to pertain to other matrices without performing an Initial LOQ Verification for each matrix. Was that the intent of this Expert Committee? I would recommend adding a new Section (e) to this section, to read as follows: "If the initial verification was not performed in the same quality system matrix as the matrix claimed for the LOQ, then verify any LOQ extrapolated to the claimed quality system matrix in that claimed quality system matrix of interest, as described in Section 1.5.2.2.3, below." A continuing LOQ verification in all laboratory sample matrices is better than initial and continuing LOQ verifications in one matrix only and no LOQ data for the other matrices."* John said it was not intended you could extrapolate to another matrix, though it might be a good idea. Richard thought the standard could say extrapolation is not allowed, but this could be discussed at the Crystal City meeting in February. Francoise pointed out that quality systems matrices are very different.

Carl Kircher, 1.5.2.2.2. "*See the comment mentioned above. If the laboratory does not receive a sample to analyze in a given quality system matrix for years, what is the minimum frequency with which an initial LOQ should be verified? Rather than quarterly, I recommend annually in this case. Please consider adding an additional Section (e) to this section, to read as follows: 'If samples are not being analyzed for each accredited quality system matrix, technology/method, and analyte quarterly, then perform the continuing LOQ verification at least annually for that matrix, technology/method, and analyte (on at least one instrument).'"* Richard pointed out the standard says verification is only required in quarters when samples are analyzed. Francoise was concerned that, if not required at least annually, at the end of the year there would be just one data point to the verification of the LOQ. Colin pointed out, with two PTs per year, they would have to do their verification for those. It was agreed the existing language, that does not require verification in any quarter in which samples for the given matrix are not analyzed, was sufficient.

Carl Kircher, 1.5.2.2.2a and c. "*These sections only have a subsection (i) but no subsequent (ii), (iii), etc. Is the subdivision to (i) really needed in these cases?"* The committee agreed. In subsection c the (i) would be removed and it would be made all one sentence.

Carl Kircher, 1.5.2.2.2.3.d. "*What is the "n value" that is supposed to be provided to clients upon request? If it is the number of data points, then add the following to Section "1.5.2.2.2.3(c)" above: "Total the number of data points (n) for each analyte…."".* It was agreed to clarify the language that it is the number of results used in the calculation.

Martin Hackman, 1.5.2.2.1.c.(i). This pointed out a typographical error in use of parentheses. The committee agreed.

Martin Hackman, 1.5.2.2.2.3. a. *"The documentation does not mention the method used."* John said both analytical and preparation methods are important, and this would be added.

Martin Hackman, 1.5.2.2.2.3. d. *"There is no referencing of the units to be used. Further, the units should be absolute units such as mg/L, µg/Kg, etc. not ppm, ppb, etc."*. Richard thought "spiking concentration" should imply units are required. Gale felt it should say the concentration of the spiked sample (not the concentration of the spiking solution). It was agreed to add units of concentration. This also needed changing in subsection a.

Lee Wolf, General. "*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 (?)."* Richard said the current TNI language has a requirement for LOD verification. It was his opinion that the whole idea of LOD verification was mistaken. He said the MDL, usually used as a TNI LOD, is equivalent to Currie's L_C, or critical value. There is no expectation that a true concentration at the L_C would give a detectable result. Only a result above L_C indicates there is 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 L_d, which is used in the DOD QSM, but not routinely in environmental analysis and they did not want to complicate the issue too much by adding it. He said L_d is the true concentration that will reliably (e.g., 95% confidence) give a result above L_C. Richard said the committee did ensure 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 L_d. Francoise pointed out the current WDS, in 1.5.2.1.d, states the LOD if required shall be verified annually, and this is confusing. Richard agreed and suggested striking that wording. He said language could be added to say if some procedure other than the EPA MDL is used for the LOD, the laboratory must ensure the LOD provides protection from false positives (greater than 1%). He said he would circulate language for the committee to consider. It was suggested if not reporting below the LOQ, the standard will not require an LOD verification. Nancy suggested if you do, it is necessary to evaluate false positives and document the procedure used.

Lee Wolf, 1.5.2.2.1. "*The initial LOQ verification (1.5.2.2.1) doesn't ever say what to do with the 7 replicates results in terms of the immediate LOQ verification being conducted, only that the qualitative criteria must be met (in each I assume) and the LOQ being 3x the LOD, which is already defined in 1.5.2.2. So I have to ask – why the 7 replicates? I guess 7 has always been considered that 'statistically significant' number, but if you're not doing anything with the results from them (perhaps later, but not for immediate LOQ verification sake), what's the point other than showing you can replicate qualitative identification 7 times? For the most part, this is just the same as the LOD study. I guess the whole point is that the LOD study data can be used to initially verify the LOQ as well, assuming the batches/instruments part was done."* In response to the question of why there are 7 replicates, Richard said it is to be consistent with the 7 replicate requirement of the MDL. It also provides an adequate number to provide a statement of precision and accuracy, and the committee will consider requiring generating a statement of precision and accuracy after the initial determination.

Lee Wolf *"The WDS states the LOQ verification samples need to be at or below the stated LOQ which is different from the current V1M4 of 1-2x LOQ. In cases where the lab has historically used 2x, it seems like a large burden to the labs to have to repeat seven LOQ replicates only to show they can meet*

qualitative criteria, which has already been established in the LOD study." Richard said this may be so, but laboratories will have at least two years to get ready, and if they spike their MDL replicates at or a bit below the LOQ, they will be all set. He noted that the same replicates can be used to calculate the LOD and verify the LOQ.

Lee Wolf, 1.5.2.1. "*In section 1.5.2.1 the WDS does not state that analyses used in the determination of LOD must meet qualitative identification criteria. Since section 1.5.2.2.1.a indicates the same replicates used to generate an LOD may be used for LOQ verification, and the LOQ requires qualitative identification criteria, then the LOD must too. But this is not stated in the LOD section."* It is the committee's intent that replicates used to calculate the LOD must meet qualitative identification criteria, and it was commented this is required in the MDL procedure. On John's suggestion the committee added a subsection to 1.5.2.1 stating that results above the LOD shall meet the qualitative identification criteria of the method. However, this would be given further thought.

Lee Wolf, 1.5.2.2.3.d. "*Recommend section 1.5.2.2.2.3.d be revised as follows: "The spiking concentration, mean percent recovery, standard deviation of percent recovery and n value shall be provided to clients upon request, or may be used to calculate project-specific precision and bias or measurement uncertainty statements." As currently written with the word 'shall' in the first part of the sentence it could imply that 'shall' also applies to calculating project-specific precision and bias or uncertainty statements, which is not the case. Project-specific precision and bias or uncertainty are typically established by other means."* The committee added "These data may be also be used to calculate project-specific precision and bias or measurement uncertainty statements."

4 – Other Business

John said he had analyzed more data sets for the LOQ criteria, and hoped there would be interest from industry groups to analyze other sets. Nancy suggested John raise this issue at the Crystal City meeting. It was agreed that Richard, Brooke and John would meet on January 16 to discuss.

5 – Adjournment

The meeting was adjourned at 3:35 pm EST, with the next meeting scheduled for January 23.