SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

JULY 14, 2015

The Committee met at the Environmental Measurement Symposium, Chicago IL, on Tuesday July 14, 2015. Chair Richard Burrows led the meeting.

1 – Roll call

Richard Burrows, Test America (Lab)	Present
Francoise Chauvin, NYC DEP (Lab)	Present
Brooke Connor (Other)	Absent
Gale Warren, NYSDOH (Accreditation Body)	Absent
Colin Wright, Florida DEP (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 (Accreditation Body)	Present
Valerie Slaven, Teklab (Laboratory)	Present
Gary Ward, OR DPH (Accreditation Body)	Absent
Ken Jackson, Program Administrator	Present

2 – Introductions

The meeting was convened at 9:00 am CDT, when Richard welcomed the audience and the Committee members introduced themselves. Richard announced that the Voting Draft Standard on Detection and Quantitation had passed the voting, and the day's agenda was to present the voters' comments for discussion.

3 – Presentation on the Volume 1 Module 4 Detection/Quantitation Sections

Richard presented the major changes to the sections, and compared the old and new standards. The committee answered clarification questions.

4 – Discussion of Voters' comments

Comment 1 It was suggested the use of active voice throughout the standard, and the committee agreed to consider appropriate editorial changes.

Comment 2 *"General comment on Verification of MDL and LOQ. I don't think that the spiked sample from the initial level in the MDL verification is necessary to verify the ongoing MDL. However, if the committee wants to combine the verification of the MDL and the verification of the LOQ into one event it can do so by specifying a combined verification with criteria. Example: Quarterly ongoing verification of the MDL and LOQ. Prepare and analyze one method blank for MDL verification and one LOQ verification sample spiked at 3x MDL. The MDL and LOQ are verified when; a) The result of the*

method blank analysis shall be a value less than (<) the MDL. b) The result of the LOQ Verification sample shall a value greater than (>) the MDL. Then continue with the current language for when the verifications don't work, tabulations, etc." The committee commented if there are no spikes for the MDL then there would be no data for recalculation at the end of the year. They recognized that the LOQ spikes could be used for the MDL, but attempting to combine the language would probably be confusing. Therefore, it would be left as written.

Comment 7 "*We disagree with LOQ = 3 x MDL. The way we currently determine LOQ/RL is by using the lowest standard on the initial calibration curve (ICAL). We are unsure why this is not considered a reliable number. The low level of the ICAL has meaning. It is verified each time an ICAL is run, which is at least monthly. Instead of ruling out the use of the low level of the ICAL being used as an LOQ/RL, instead propose than an LOQ/RL standard be run daily. The criteria for this can be the same as for passing an ICAL or daily check standard. This is done in many wet chemistry tests and in NJDEP LLTO-15. Changing the definition of LOQ/RL has an effect on all RLs, with many of them increasing. Increases in LOQ/RL will cause many, if not all labs, to exceed the necessary limits for regulatory cleanup criteria. Also, it will change the way labs run their calibration curves because the current low-level standard would be meaningless. Sure, LOQ/RL could be artificially increased so that the low-level standard could be fairly consistent across the board, but that would surely lead to even more regulatory criteria being exceeded. It is in our opinion that if this change is made, it will promote cheating on the calculation of MDLs, since the lower your MDL is, the lower the LOQ/RL is. Another proposal for use of LOQ is to use the LOQ calculation as verification for the RL (= lowest point on the ICAL). The RL must not exceed +/-50% of the LOQ. The attached file shows the impact of this on one instrument for one test. Three regulatory criteria cannot be met with the standard change in place. This will be magnified across the lab."* Richard presented several sets of data showing gross errors at the lowest point of the calibration curve, even when the correlation coefficient was good. He showed examples where the old criterion would not be satisfactory, but the new criterion would be.

Comment 19 "*The standard requires that the LOQ must be at least 3X MDL. This works in most cases but may not apply with GCMS where the secondary ion has sufficiently lower intensity than the primary quant ion. In these cases, when the concentration is high enough to qualitatively identify a compound by the presence of the seconday quant ion, the primary ion has sufficient intensity for accurate quantitation. As such the LOQ can legitimately be close to the MDL. I would recommend that you lower the requirement from 3x to 2x the MDL. "* The committee agreed the comment is accurate. The FACA on detection and quantitation showed where 2x could be done, but it would be very complicated. The committee was not planning to change the 3x multiplier, and contended it would not affect the MDL in most cases.

Comment 5 *"I support the plans for changes and the intent behind the changes presented.*
• I support clearer language regarding MDL/LOD.
• I appreciate the committee's diligence to present a VDS and recognize the labor and dedication you have demonstrated.
• My "No" vote is given because this language has flaws or lack of clarity that will prevent it from being enforceable to laboratories. These flaws need to be addressed before this section is adopted. Since clause 1.56.2.1.1 has apparent conflicting language, Richard agreed a language change was needed.

Comment 9 Referring to cause 1.5.2.1.1 (e), it was commented *""The MDL determination must include evaluation of routine method blanks," is a vague statement. It does not have sufficient information to be auditable. The term "evaluation" needs further definition or explanation of the minimum requirement for laboratories."* Richard responded that the committee had chosen not to write the MDL language into the standard, so it became a question of what could be done to clarify the language. John thought more detail could be added. Richard suggested adding language after "blanks" to read: "..to ensure that the blank results are normally lower than the MDL". He agreed that would not be quite the right language, but could be along those lines. There were a couple of language suggestions from the floor. The committee would finalize the language later.

Comment 23 *1.5.2.2.2.b lacks clarity.*
• There is an instruction to "tabulate" all results but there is no instruction about what to do with this tabulation.
• There is a "minimum of 7 samples required" but no provision for what happens if there are less. For example, if a laboratory failed to collect some of the quarterly verification data during that period and does not have 7 samples, what happens then? [This will likely happen! I'd expect this quarterly process to be part of a learning curve for laboratories, therefore laboratories and evaluators need to know what do if 7 data points representing evenly distributed periodic sampling over a 2 year period are not available.]
• The second paragraph of this section refers to a LOQ value (which is an apparent result of the "tabulated" data?) but there is no instruction about where this value came from. If it is calculated from the tabulated data, this instruction (and what calculation is done) needs to be clearly communicated. In response to the first bullet, the committee agreed to reference the documentation section. Scott said he would provide suggested language for the second bullet. Richard said if there are less than 7 samples, an assessor should require that laboratory to run more. Valerie did not see how this would be different from any finding requiring corrective action. Richard was doubtful if anything could really be added, but he said the committee would look at the language. For the third bullet, Richard said the LOQ is the value chosen by the laboratory, and perhaps wording could be added to clarify this. Richard suggested adding a statement that if the MDL is changed, the laboratory must ensure the LOQ is still at least 3x the MDL.

Comment 11 Referring to 1.5.2.1.1, the commenter remarked *"Gravimetric methods should be included in the list of these that do not require a MDL. The MDL will vary with the uncertainty of the equipment used and not the method."* Richard responded, since it cannot be spiked, it could say you only have to do the blank part for gravimetric methods. Possible language was discussed. Richard proposed separating the methods where spiking solutions are not available from those where a detection limit is inappropriate, and drafting language.

The meeting was now adjourned from 12:00 until 1:30 pm

Comment 12 This addressed clause 1.5.2.1.1. "*1.) The third sentence, "One option is to follow EPA's MDL procedure specified at 40 CFR Part 136 Appendix B." should be converted to a note. The statement is not assessable and reads like guidance. 2.) The last sentence before a)-g), "The determination, at a minimum, shall incorporate language addressing the following requirements:" I find it a little confusing how a determination incorporates language. Do you mean the SOP for MDL shall incorporate language? I suggest revising to, "The determination, at a minimum, shall incorporate*

*language addressing the following requirements:"*O*r "The Laboratory SOP for determining the MDL, at a minimum, shall incorporate language addressing the following requirements:" 3.) Items a) through e) use the term "must" and item f) uses the term "shall". I think 'shall' is the better word and should be used in a) through f). 4.) I think item g) should be moved into the ongoing verification of the MDL section 1.5.2.1.2 as the second paragraph because it addresses method alterations after an MDL has been established and whether or not the alteration merits another verification of the MDL.*
5) In Item g) I think the phrase "reasonably expected" will be difficult to explain and apply consistently among assessors. The term "method alteration" is already a strong phrase reserved for significant change in the analytical system. When a method is altered, the MDL should be verified as a rule. Consider changing the first sentence in item g) to, "Verify the MDL after method alteration by preparing and analyzing a method blank and a spike at the LOQ concentration." This way we don't have to judge what's reasonable and what isn't over whether or not to run two extra samples." The committee agreed with points #1, #2, #3, and #4. On point #5, there was some discussion how to define what can be reasonably expected to change the detection limit. The commenter proposed "method alteration", but Richard questioned what that really means. He asked for suggestions from the audience on how to say the MDL should be changed if there is a "significant" change. Marlene Moore said you cannot say what change would be significant to affect the sensitivity. The best suggestion was to add "..in a way other than routine maintenance that can be expected to elevate the detection limit." Richard said the committee might have another look at the language.

Comment 14 This addressed clause 1.5.2.1.2. "*Quarterly verification of the MDL creates a large burden on labs and provides little or no value. As a DOD assessor, I often discuss this issue with labs, and without question, the resources required to comply with this requirement far exceeds it value.*" The committee ruled this non-persuasive. Without the quarterly verification, there is no assurance that the method sensitivity has not changed dramatically.

Comment 15 This addressed clause 1.5.2.1.2. "*Hopefully, you agree to move item g) from 1.5.2.1.1 to this section. If you do the last sentence should be updated so that "….1.5.2.1. a-f must be met…." to remove item g) from that list. I understand that the ongoing verification points back to the criteria in 1.5.2.1.1.d & .e for the spike (d) and the method blank (e). My concern is that the standard does not address what criteria to use to evaluate the method blank in the ongoing verification because 1.5.2.1.1.e simply states an evaluation must occur as part of the initial MDL determination. In the ongoing verification phase, there is an MDL with which to test the method blank value. I am suggesting for the ongoing verification that you add criteria for the method blank result. I think the committee views the MDL determination to be similar to Currie's LC so that the MDL value will protect against false positives. The criteria for the ongoing verification of the method blank should be that it is less than (<) the MDL. The spike sample seems unnecessary with respect to the false positive criteria.*
Consider adding the following text or something similar: The MDL is considered verified when the following criteria are met: a.) The method blank result is less than (<) the MDL" Richard suggested the committee might need to re-list items relative to ongoing verification. He asked Committee Members to think and come up with suggested language.

Comment 16 This addressed clause 1.5.2.2.1. *"This procedure for verification of the LOQ consumes excessive resources and provides little or no value."* The committee disagreed and would rule this non-persuasive. Richard said if the procedure is not followed, you do not know what the precision and bias are.

Comment 17 This addressed clause 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.*

If it is the intent of this Committee that LOQ must be independently done in EACH FoA matrix in the particular matrix for each accredited analyte and test method (i.e., analyte MUST be spiked into an actual biological tissue, extracted, and analyzed to evaluate a BT LOQ), then please ignore this comment." Richard said he would contact the commenter for clarification of the question and for examples where this happens.

Comment 18 This addressed clause 1.5.2.2.1. *"In the note under item a) consider adding advice to perform method blanks, as well, to comport with the EPA MDL procedure and 1.5.2.1.1.e."* Language changes may have resolved that comment.

The following two comments addressed the same issue in clause 1.5.2.2

Comment 20 "*As with the requirement for the quarterly verification of the LOD, the quarterly verifications of the LOQ consumes excessive resources and provides little or no value. As a DOD assessor, conversations with labs about this issue unanimously indicates it requires a lot of time for compliance and provides no value."*

Comment 21 *"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)."*

Again, if it is the intent of this Committee that LOQ must be independently verified in EACH FoA matrix in the particular matrix for each accredited analyte and test method (e.g., Lead and all other EPA 6010 analytes actually spiked into a base-matrix soil, digested, and analyzed to verify the soil LOQs for each Metal), then please ignore this comment." Richard believed these concerns may already have been covered with "..each quarter in which samples are being analyzed.", but he would check with one of the commenters.

Comment 23 This addressed clause 1.5.2.2.2.b. *Lacks clarity.*
• There is an instruction to "tabulate" all results but there is no instruction about what to do with this tabulation.
• There is a "minimum of 7 samples <u>required"</u> but no provision for what happens if there are less. For

example, if a laboratory failed to collect some of the quarterly verification data during that period and does not have 7 samples, what happens then? [This will likely happen! I'd expect this quarterly process to be part of a learning curve for laboratories, therefore laboratories and evaluators need to know what do if 7 data points representing evenly distributed periodic sampling over a 2 year period are not available.]

• The second paragraph of this section refers to a LOQ value (which is an apparent result of the "tabulated" data?) but there is no instruction about where this value came from. If it is calculated from the tabulated data, this instruction (and what calculation is done) needs to be clearly communicated.

The committee had already dealt with some of this. It was agreed it is not appropriate for an assessor to tell a laboratory what its corrective action should be. Such "consulting" is not allowed in ISO 17025. However, Cathy Westerman suggested language could be added to state what a laboratory should do if it has not run 7 replicates in a year.

At this point the committee stopped working through the list of comments, but Richard asked the audience if anyone wanted to discuss any other specific comment. Hearing none, the committee spent the next hour working on possible language.

An issue was then raised that had been brought up with the Proficiency Test (PT) Expert Committee. It seemed the ISO Guide 33 second source standard Adopted by PT was in conflict with the Quality Systems standard. The issue revolved around the definition of "lot", which is used in Volume 1 Module 4, but is not defined. On discussion it was suggested the Chemistry Committee add the ISO definition of "lot" to its definition section. In response to a question from Paul Junio, it was agreed this also belongs in Module 2, and the Quality Systems Committee would put it through as a Voting Draft Standard. This should happen quickly under the revised SOP 2-100 standards development procedure.

5 – Next Steps

Richard said the committee would resolve the persuasive comments by modifying the standard, and would then submit it as an Interim Standard.

The calibration standard already developed and the detection/quantitation standard discussed during the current session would complete the committee's work on the 2015 standard. Richard asked for topics the committee might consider for future standards development. Suggested were non-standard methods, the performance approach, and procedures for re-validation of expired standards.

6 – Adjournment

The meeting was adjourned at 4:30 pm CDT.