SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

AUGUST 7, 2015

The Committee held a conference call on Friday, August 7, 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
Nancy Grams, Advanced Earth Technologists, Inc. (Other)	Absent
Anand Mudambi, USEPA (Other)	Present
John Phillips, Ford Motor Co. (Other)	Absent
Scott Siders, IL DEP (Accreditation Body)	Absent
Valerie Slaven, Teklab (Laboratory)	Present
Gary Ward, OR DPH (Accreditation Body)	Absent
Ken Jackson, Program Administrator	Present

Associate Committee Members present: Arthur Denny; Tom Dziedzic; Chuck Neslund; Diana Shannon

2 – Previous Minutes

Richard deferred consideration of the July 14 and July 24 minutes until the next call.

3 – Consideration of Comments on the Detection/QuantitationVDS

The committee considered the responses to comments Francoise and Anand had been working on. Most of these had been considered at the Chicago meeting (Minutes of July 14, 2015).

Comment 1 In response to a voter's comment, Francoise explained she had changed instances of "must" to "shall" in the standard and had reworded sentences into the active voice. She e-mailed the edited standard, with all the changes flagged with comments. Ken checked with the CSDEC draft guidelines for the preparation of standards and this confirmed "shall" is to be used rather than "must".

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." Anand's proposed response was: "*Non-Persuasive: The spikes from the intial MDL verification are needed to recalculate the MDL on an annual basis. The MDL and LOQ language has been kept separate for clarity and for ease of use of the standard by laboratories*."

Comments 4 and 5 (Not considered in Chicago) *"I definitely favor the intent and the changes being proposed but I believe language I've noted in the uploaded file will be problematic and will be frustrating to labs and ABs. I would like to see this language addressed so that the committee's hard work will lead to a revised standard that can be carried out and will be beneficial to all stakeholders. Again, I thank the committee and Richard Burrows for countless hours of dedication and commitment."*

"• 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."
The committee agreed to address these comments. Francoise and Anand recommended moving these general comments to the top of the specific comments given by this commenter. Ken pointed out irrespective of the way they are ordered on the Response-to-Comments spreadsheet, each commenter would be receiving an individualized spreadsheet with just their comments.

Comment 6 This was editorial and would be fixed.

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."* Anand had prepared the response: *"Non-Persuasive: The low level of the standard does not have quantitative meaning, unless there is specific readback criteria in the method. The use of the factor of 3 is consistent with EPA's approach with the ML."* However, there had been a protracted discussion on this point and Anand wondered if more should be supplied in the response to the commenter; e.g., the statistical basis for the 3x multiplier. Richard said he would review the past minutes for the discussion and would decide if more should be added.

Comment 8 "*1.5.2.1.1 has apparent conflicting language in the initial paragraph. "If a mandated test method or applicable regulation includes protocols for determining detection limits, they shall be followed."*

"…The determination, at a minimum, shall incorporate language addressing the following requirements:…" (For example, one of the following requirements is for "evaluation of routine method blanks" but this requirement may not be part of the procedure in a mandated method. See additional comments on this requirement, below.)" Anand's suggested response was "*Persuasive: The second clause was reworded to "The laboratory MDL procedure, unless following a mandated test method or procedure, at a minimum, shall incorporate language addressing the following requirements" (added language underlined, in italics). Note that for clarification the block of text to which the first clause belongs was moved to the introductory paragraph."* This change had been made in the standard.

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."* Francoise presented revised wording in the standard. Anand's response was "*Persuasive: This section was rewritten to add a requirement for the labs to determine and document their acceptance criteria for the false positive rate in routine method blanks as follows: "The MDL procedure shall include criteria for and evaluation of false positive rates in routine method blanks"."*

Comment 10 *1.5.2.1.2 contains errors which will render the section unenforceable. It specifies that criteria listed in 1.5.2.1 a-g must be met for ongoing verification. The reference to 1.5.2.1 is incorrect. The appropriate reference is 1.5.2.1.1, a-g. Note, however, that all sections of "a-g" will not apply to single analyses run for "ongoing" verification as described in this section. For example, "c" refers to analysis on multiple days and "f" specifies the "initial determination". This sentence, "The criteria listed in section 1.5.2.1 a-g must be met for ongoing verification", will not be enforceable, in part or in total, if it contains errors.* This was discussed at length. Richard said the on-going verification is being done over multiple days and is not just the one quarterly sample. Francoise felt it was a clarity problem in the sentence *"The criteria listed in section 1.5.2.1 a-g must be met for ongoing verification"*, and suggested making it clear it applies over the whole year. Richard suggested taking "initial" out of "f". Revised language was drafted, and the response was written that it was persuasive and the language had been edited for clarification.

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."* The response would be *"Non persuasive - MDL has to reflect the entire method as performed, including performance of equipment/instruments used."*

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:"Or "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."
Francoise described the proposed language changes, and the committee agreed with Anand's proposed response *"Persuasive: the following chnages have been made to the standard:*
1) The 3rd sentence in Sectino 1.5.2.1.1 was converted to a note.
2) The sentence was reworded to include the words "MDL Procedure" for clarity.
3) The word "must" was changed to "shall" - except in c) and d), which were reworded.
4) The section was moved to the ongoing verification of the MDL section, 1.5.2.1.2.
5) The term "that can be reasonably expected to change the detection limit" was clarified and the new language is: "other than routine maintenance and the change can be expected to elevate the detection limit"."

Comment 13 *"The language here invites an interpretation request. Is the intent that laboratories be allowed to determine the MDL in ANY quality system matrix? If not, then the phrase should be "quality system matrix of interest." Also, "of interest" is open to interpretation and should be better defined. It would be more useful to tie any requirement to accredited analytes, methods, and matrices."* Francoise had changed "a quality system matrix" to "quality system matrix of interest". This would be ruled persuasive, and the response would note that requirements only apply to quality system matrix of interest.

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. The proposed response said: "*Without the quarterly verification, there is no check 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"
Francoise commented for labs that do not report results below the LOQ, "false positive" can be interpreted as "<LOQ". She raised the issue because she felt it was ambiguous. She suggested clarifying the language to read: "the MDL procedure shall include criteria for and evaluation of false positive rates (i.e., results > MDL) in routine method blanks". This generated a protracted discussion. Richard was concerned that, from the perspective of laboratories not reporting below the LOQ, they are not reporting a false positive unless it is above the LOQ. Francoise argued that this is the initial determination of the MDL that all laboratories have to do. Richard said you would still be making them measure the false positive rates between the MDL and the LOQ that they would not otherwise have to do. Anand agreed Francoise's proposed addition should not go in, because it could be confusing, generating standards interpretation requests. The decision was made to leave the language as it was. It was agreed the response should state "persuasive", because the committee had fixed the two major issues. The response should also state that the text had been modified accordingly.

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 agreed this would be non-persuasive, and agreed with Anand's response: "*Without the quarterly LOQ verification, the mean and the standard deviation of the recovery of the method at the LOQ cannot be determined at least once per year."*

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." The response would state: *"Persuasive: The language in Section 1.5.2.2 now reads "An LOQ is required for each quality system matrix of interest…." which is consistent with language in other parts of the TNI standard."*

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."* The response would state: *"Non-Persuasive: The MDL and LOQ language has been kept separate for clarity and for ease of use of the standard by laboratories."*

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 secondary 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 decided an appropriate response would be: *"Non persuasive: If spiking level is such that accurate quantitation is obtained, MDL calculated will be more than 3X lower than quantitation level, since standard deviation will be low."*

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."* The agreed response was: *"Non-persuasive - Without the quarterly LOQ verification, the mean and the standard deviation of the recovery of the method at the LOQ cannot be determined at least once per year."*

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." The committee agreed on the response: *"Persuasive: Instruction clarified by language added to 1.5.2.3: "If no analysis was performed in a given year the verification of the MDL/LOQ is not required, but a new initial MDL/LOQ verification shall be performed prior to analysis of client samples"".*

Comment 22 *"1.5.2.2.2 Ongoing Verification of the LOQ. 1) The first sentence reads very much like the LOD verification. I do not think that the LOQ verification should be the same thing as the LOD verification. Instead, the LOQ verification sample should be spiked at the LOQ level (3x MDL). 2) At 1.5.2.2.2 a, the only requirement on verifying the LOQ is that it is above zero and it meets the method qualitative identification criteria. I think because of the required 3x factor from the MDL the committee views the LOQ determination to be similar to Currie's LD, which protects against false negatives. So criteria to be above zero is not appropriate because above zero includes levels less than the MDL; the false negative zone. The real test here is whether the LOQ level is still above the MDL. So test the LOQ value, per my comment above, at 3xMDL and have criteria that it must have a result above the MDL. It does guard against setting the LOQ too low."* The committee felt it could not provide everything the commenter had asked for, and the agreed response was: *"Persuasive: LOQ verification states that LOQ spiking level must be at or below the LOQ. Ideally, it would be good to have all LOQ verification samples return results above MDL, but existence of analytes with low recovery make this problematical. Therefore we did not require all results to be above MDL, but did add a requirement for accuracy."*

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. Francoise said language had been modified such that it should completely satisfy this comment. It was agreed the response would be: *"Persuasive: Editorial comment for clarity. The word "include" has been changed to "record"."*

This completed consideration of all the comments. At the next meeting the committee would vote on moving the document to an Interim Standard. Ken reminded the committee they would need to vote to accept all the responses. Then he would take care of finalizing the Response-to-Comments spreadsheet, sending out the individual comments, and editing the standard to produce an Interim Standard. Richard referred to the meeting in Chicago between the Accreditation Council, LASEC, and CSDEC, when additional issues had been raised on the Calibration Final Standard. Although substantive changes could no longer be made, it seemed editorial changes would satisfy the concerns, and also at the next meeting the committee would need to vote on them.

4 – Adjournment

The meeting was adjourned at 3:30 pm EDT. The next call would be on August 21, 2015