SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

APRIL 26, 2013

The Committee held a conference call on Friday, April 26, 2013, at 2:00 pm EDT.

1 – Roll call

Richard Burrows, Test America (Lab)	Present
Francoise Chauvin, NYC DEP (Lab)	Absent
Brooke Connor, USGS (Other)	Present
Dan Dickinson, NYSDOH (Accreditation Body)	Present
Tim Fitzpatrick, Florida DEP (Lab)	Present
Nancy Grams, Advanced Earth Technologists, Inc. (Other)	Present
Anand Mudambi, USEPA (Other)	Present
John Phillips, Ford Motor Co., (Other)	Present
Lee Wolf, Columbia Analytical Services (Lab)	Present
Ken Jackson, Program Administrator	Present

Associate Committee Members present: Diana Shannon; Chung-Rei Mao

2 – Previous Minutes

It was moved by Tim and seconded by Anand to approve the March 28 minutes. All were in favor. The minutes were therefore approved.

3 – Method Detection Limit Procedure

The Committee worked through an edited draft of the MDL document, submitted by Chung-Rei.

Definition section; LLOQ. It had been suggested to change it to LOQ. Richard reminded the Committee this had been discussed at length during the previous call. Tim and Nancy agreed it should be named differently from LOQ, but Nancy argued that precision and accuracy are not achieved. Brooke suggested making it "acceptable precision and accuracy". Nancy said it was not sufficient to just say "reliably return results above the MDL", and wanted it spelled out that it is the point at which false negatives are not found. Nancy also thought "precision and accuracy of the method have been demonstrated" should be changed from "demonstrated" to "determined". Chung-Rei suggested "bias" would be a better word than "accuracy". Nancy also did not like "reliably". After further discussion it was agreed to change the definition to:

"The Lower Limit of Quantitation (LLOQ) is the lowest concentration above the MDL for which false negative control, precision, and bias can be demonstrated."

At this point, Dan questioned from an auditing perspective if a laboratory could answer a question of where it is controlling its false negatives. This led to discussion on whether to add more information in the definition; e.g., 98% false negative control. Nancy added that is only on the annual basis, not the quarterly one.

Scope and Application section; First paragraph.

There was discussion on "all" in the last sentence: "It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit." Dan asked if there are a lot of different clean-up procedures, do they need to do it separately for each of them? Nancy said it should be all the steps the laboratory does, not all the steps in the method. It was agreed to change the wording to:

"It is essential that all sample processing steps used by the laboratory be included in the determination of the method detection limit."

Procedure section step 1b; comment CRM5.

Referring to "The concentration value that corresponds to an instrument signal/noise in the range of 3 to 5. ", Chung-Rei said this should clarify the concentration of a substance that has gone through the entire sample preparation process; otherwise this is an instrument detection limit. On discussion, the Committee agreed to make no changes.

Procedure section step 4; comment CRM6.

The step read: "Calculate the sample standard deviation (S) of the replicate spiked blank measurements and the standard deviation of the replicate method blank measurements from all instruments". It was commented that "7 blank samples", instead of "7 method blank samples" was used in Section 3 above. It was pointed out, however, that they become method blanks after step 3.

Procedure section step 5; comment CRM7

This referred to the equation:

$$MDLs = t_{(n-1,1-\propto=0.99)}S$$

It was commented that EPA has another way to calculate MDL, based on the mean method blank + t x S, where S is the sample standard deviation of replicate spiked blank samples. It was responded that this would be MDL_b in the next section.

Procedure section step 6b (i); comment CRM8

This step stated: "If none of the method blanks give numerical results, the MDL_b does not apply". The commenter suggested, assuming negative results also count, clarifying the

"numerical results" as instrument output signals or analytical results shown on a final analytical report.

Procedure section step 6b (ii); comment CRM9

This step stated: "If some (but not all) of the method blanks give numerical results, set the MDL_b equal to the highest method blank result." It was questioned if a laboratory had 10 method blanks available and one method blank had no numerical results, could the laboratory select 7 method blanks to calculate MDL_b according to (b) iii below, or must the laboratory set MDL_b at the highest method blank? Richard responded since you have a set of method blanks you are implying you look at that set; i.e., use them all. It was decided to leave it as it is.

Procedure section step 7a; comment CRM10

This step in referring to on-going verification stated "During any quarter in which samples are being analyzed, analyze a minimum of two spiked blanks on each instrument, in separate batches if available." The commenter asked for clarification whether this section is for an ongoing verification of MDL or LLOQ. Richard clarified it is both and it was agreed to state that.

Procedure section step 7a; comment CRM11

In response to the statement "All analytes must meet the qualitative identification criteria in the method and must return a positive numerical result that is greater than the MDL.", the following comment was made. At LLOQ, method-specified quantitation criteria shall also be met. For example, RPD between primary and secondary columns shall be < 40% for organic GC analyses. Richard responded that spikes are at the LLOQ, but results will mostly be below the LLOQ. Anand clarified that the on-going verification is just being done for the MDL, and the LLOQ is being used to do that. It was agreed that step 7 should say on-going verification of the MDL.

On Tim's suggestion, the first sentence was modified by inserting "prepare and" before "analyze a minimum.."; i.e., *"During any quarter in which samples are being analyzed, prepare and analyze a minimum of two spiked blanks on each instrument, in separate batches if available."*

There were several suggestions on the last sentence of 7a *"*If those criteria are not met, raise the spiking level and LLOQ for the analytes failing the criteria by a factor of 2 and re-establish the precision and bias for the failed analytes at the raised LLOQ." Nancy suggested saying "at least a factor of 2". Nancy also questioned the statement that the precision and bias for the failed analytes is re-established. Only the MDL is re-established. Richard agreed that part should be removed. It was then suggested adding "and repeat the MDL verification" after "at least a factor of 2". Anand commented it is misleading to say "raise the spiking level and the LLOQ", because it is not known what the LLOQ becomes.

Richard commented 7a just tells you what data to collect for use for verification in 7c and7d. Therefore, 7a should be called "on-going data collection". Also, 7b should be combined into 7a, since they are connected and might be confusing as two separate sections. Richard volunteered to re-draft this section in light of the above comments.

Steps 7c, d, and e will then become "on-going annual verification".

Procedure section step 7c; comment CRM12

There was a question about the sentence "At least once per year, re-evaluate MDL_s and MDL_b based on the most recent spiked blank and unspiked blank results using the equations in section 6 (more results are generally better, up to the most recent two years of data). " It was asked if this means pooling historical data with new data. If yes, some guidelines on pooling historical data are needed. Richard responded it is data that have been gathered over time, so it is not clear what are "old" and "new" data.

Procedure section step 7c; comment CRM13

In referring to the sentence "For tests that return numerical results for some method blanks, but not all of the time, use the 99^{th} percentile of the method blank results as the MDL_b.", it was pointed out this is inconsistent with step 6b, and there is a need to clarify that this is an option for small data sets of nonparametric distributions. Richard said the 99^{th} percentile was no longer in the sentence, and he would wordsmith it to refer back to step 6.

Nancy referred back to step 2, where the spiking level was changed to 2 – 10 times the estimated detection limit. She asked if everyone was in agreement for it being changed from 2-5 times. There was general agreement, since 2-5 times would not be feasible for poor performing analytes with low recovery. Tim asked if it should be stated what constitutes poor recovery, but Richard suggested leaving it vague until some data have been put through the procedure.

Next Steps

Richard said he would get a new draft of the document out. He asked Committee members to think how the procedure should be tested. Several people should be able to provide data sets, and a written protocol will be needed for testing with existing data.

4 – Adjournment

The meeting was adjourned at 3:40 pm EDT. The next call was scheduled on May 10, 2:00 – 3:30 EDT.

LIST OF ACTION ITEMS TO BE COMPLETED

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
1	1/31/12	Add a definition of Reporting Limit or Quantitation limit to the standard.	Committee	Defer to quantitation sections
2	1/31/12	Continue to consider the concept of routine low-level QC in the standard.	Committee	Ongoing
3	1/31/12	Review Sections 1.5 and 1.6 of the 2009 standard's chemistry module to determine if current calibration requirements are adequate.	Committee	Not determined
4	1/31/12	Spacing of calibration standards will be considered for the guidance document.	Committee	Ongoing
5	2/17/12	Draft language for items in the calibration standard	Richard (Items 1 and 2) Anand (Item 3) Nancy (Item 5) Anand and Francoise (Item 6) Tim (Item 11)	Complete
6	2/17/12	Review Volume 1 Module 4 of the 2009 standard to identify any inconsistencies with the new language	All Committee Members	Complete
7	3/2/12	Add 1-2 sentences under the header 1.7.1 to explain that method is also included in calibration.	John	Complete
8	3/2/12	Clean up the parts of Section 1.7.1 referring to initial calibration and the parts referring to continuing calibration.	Committee	Complete

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
9	3/2/12	Add criteria for rejection of calibration standards to the guidance document.	Committee	Not determined
10	3/2/12	Add to the guidance document discussion of analysts using the most recent calibration rather than choosing which of 2 or more curves to use.	Committee	Complete (done in the standard)
11	3/2/12	Include a paragraph in the standard that addresses a single-point calibration for P/A testing.	Committee	Complete
12	3/30/12	Check the language does not contradict the existing standard regarding meeting method requirements vs. standard requirements for calibration.	Committee	Not determined
13	3/30/12	Sections 1.7.1.1 j and k will be modified further as a result of the March 30 discussions.	Anand and Francoise	Complete
14	3/30/12	Have the guidance document consider orders of magnitude in deciding the minimum number of standards, and keep a placeholder in Section 1.7.1 to refer to it.	Committee	Not determined
15	3/30/12	Add a definition for threshold testing	Committee	Not determined
16	3/30/12	Richard's, John's and Anand's March 30 changes will be incorporated into a single document.	Ken	Complete
17	5/4/12	Add to the guidance document that Section 1.7.1.1 (g) requirements should also be applicable	Committee	Not determined

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
		for average response, when you evaluate with the RSD, and that is numerically the same value as the RSE.		
18	5/4/12	Discuss in the guidance document how to check quarterly (ref. Section 1.7.1.1 (j) (i).	Committee	Not determined
19	6/1/12	Bullet points will be drafted for a proposed PowerPoint presentation	Brooke, Richard, Tim, Francoise, Anand	Complete
20	6/1/12	Bullet points will be drafted for a slide that will describe the items to be discussed in the guidance document.	John	Complete
21	7/20/12	Explain in the guidance document the difference between MDL and the true detection limit.	Committee	Not determined
22	10/5/12	A note will be appended to the draft language of Section 1.7.1.1 n until the CCV language has been written.	Anand	Complete
23	11/2/12	For the MDL document, language will be drafted in the scope to limit the use.	John	Complete
24	11/2/12	In the Scope and Application section of the edited MDL document, the sentence "To accomplish this, the procedure was made device- or instrument-independent." Will be re-worked.	John	Complete
25	11/30/12	A letter will be drafted to the EPA OW, asking what kind of stakeholder composition they want	John	12/14/12

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
		ELAB to put together for reviewing the modified MDL procedure.		
26	2/1/13	In the calibration standard Sections 1.7.1.1 (h) i and 1.71.1 (k) i, revisit the suggestion to replace LOQ with “lowest concentration for which quantitative data are to be reported”if LOQ is re-defined.	Committee	Not determined
27	2/15/13	Check on travel funding for face-to-face meeting	Ken	3/1/13