SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

MARCH 15, 2013

The Committee held a conference call on Friday, March 15, 2013, at 2:00 pm EDT.

1 – Roll call

Richard Burrows, Test America (Lab)	Present
Francoise Chauvin, NYC DEP (Lab)	Present
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)	Absent
Ken Jackson, Program Administrator	Present

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

2 – Previous Minutes

In the draft minutes of March 1, 2013, action item 25 was changed to "complete". With this change it was moved by John and seconded by Tim to approve the March 1 minutes. All were in favor. The minutes were therefore approved.

3 – Calibration Voting Draft Standard

Further changes were made to the draft document.

1.7.1.1 e). The last sentence was changed to "For calibrations not listed below, the number of initial calibration standards must result in at least two statistical degrees of freedom."

1.7.1.1 j) (i). A change was made to "x'_i = Measured concentration of the calibration standard."

1.7.1.1 j) (ii). Two changes were made:

"measurement of the Relative Standard Error (RSE). The RSE shall be less than or equal to the maximum specified in the method. If no criterion is specified in the method, the maximum allowable RSE shall be specified in the laboratory SOP. RSE is calculated by re-fitting the calibration data back to the model, using the following equation:"; and

"x_i = True value of the calibration level i."

1.7.1.1 n) was modified to: "a successful calibration sensitivity check determination as described below has been performed;"

1.7.2 d) (i) was changed to: " if an internal standard is used, calibration verification shall be performed at the beginning of each analytical batch, and at the frequency defined in the method;"

With the above changes in place, it was moved by Anand and seconded by John to send this document out of committee as a Voting Draft Standard. All were in favor. Ken was tasked with arranging for the document to be sent out for membership vote.

4 – Method Detection Limit Procedure

Section 3. It was proposed to add a new sentence to say the spiking concentration will become the minimum level; i.e.., the lowest level at which precision and accuracy data would be available. There was some discussion over the term "minimum level". Nancy was concerned it may be something that is used differently in some regulations, and "minimum reporting level" might be better. John remarked that minimum level is normally 3 times the MDL, but it has been defined in different ways and this could be an opportunity to fix the definition. John and Tim thought "quantitation limit" might be better and Richard suggested putting in "minimum level" and then having "quantitation limit" as a fallback if the EPA Office of water doesn't like it. (There are some methods that talk about "minimum level"). After further discussion, however, it was agreed to change it to "Limit of Quantitation". Another change prompted by Dan was to say the spiking concentration "is assumed to be" rather than "will become". The agreed wording was "The spiking concentration is assumed to be the limit of quantitation .." This change would also be reflected in the definition section.

Section 7a. Richard suggested removing the third sentence ("At least 90% of the analytes.."). He preferred to just say all analytes must meet the qualitative identification criteria in the method. Then, in the yearly re-evaluation, talk about what to do if some proportion of the spike sample results are less than the MDL. A long discussion on qualitative identification criteria followed. Tim was concerned that qualitative identification criteria may not always be possible; e.g., for metals by Method 200.7. Richard asked if it should say for methods that don't have qualitative identification criteria, the result has to be greater than the MDL. Brooke said this talks only about false negatives by determining whether the spike is detected, and false positives with blanks are not addressed. She said maybe the background levels should be looked at to verify the false positive rate is not exceeded. Tim said in the process of setting the MDL blanks are being looked at, which would reflect any false positives; you are looking at the variability of your spikes and setting your MDL to be the higher of the two. Therefore,

if background contributes a significant positive bias the blanks will define the detection limit, which will be the point at which you are 99% confident you are not seeing a false positive. Richard said a situation in which you are likely to fail that is more likely to be an organic method because of the low bias, and for those methods you are probably not seeing anything in your blanks, so assessing the blanks won't make any difference. Nancy pointed to the scenario of an organic method where the blanks are always zero and there are no qualitative identification criteria. She asked if an alternative criterion should be the MDL or a positive result. Richard suggested a gc method with just a peak might have the identification criterion of a positive numerical result. Tim added there are some organic methods in which the blank is not always "not detected" and there are no straightforward qualitative identification criteria. If the peak falls in a retention time window it is assumed to be the analyte. Richard suggested the statement "all methods must meet the qualitative identification criteria in the method and must return a positive numerical result" would work for the gc methods. Richard said maybe in Section 6 there should be something about the quantitation limit where you do your first evaluation. Perhaps spikes should have to give results greater than the calculated MDL_b, and otherwise you must start over with a higher spike. After some discussion it was decided on a footnote about methods with low recoveries; e.g., if the recovery is less than (say) 30%, then the spiking level must be more than 5 times the MDL. Richard inserted a note in Section 6 that a section on evaluation of the LOQ with the initial determination needs to be added.

Nancy pointed to the first sentence of the section, saying 2-5 times the MDL is too low for methods with poor bias. Rich suggested changing it to "the spike level is at the limit of quantitation".

Section 7b. Nancy said, if a statistical outlier test is included, there must be a maximum number allowed to be removed from the data set. Brooke added when there are intermittent blank problems you may be removing the actual false positives in your data set, even though they are infrequent. This led to questioning whether outlier testing should be done at all. Brooke suggested just leaving this out; i.e., don't say you cannot do it, but don't say you can do so either. Richard said you may need to be able to take out data points in some cases, such as where someone accidentally labeled an LCS as a blank. Richard agreed to put in a note how it would apply to (say) Method 200.7 in what constitutes qualitative identification criteria.

5 – Adjournment

The meeting was adjourned at 3:30 pm EST. The next call was scheduled on March 28 at 2:00 pm 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
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	Committee	Complete (done in the

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
		analysts using the most recent calibration rather than choosing which of 2 or more curves to use.		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 for average response, when you evaluate with the RSD, and that is numerically the same value as the RSE.	Committee	Not determined

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
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 ELAB to put together for reviewing the modified MDL procedure.	John	12/14/12
26	2/1/13	In the calibration standard	Committee	Not determined

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
		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.		
27	2/15/13	Check on travel funding for face-to-face meeting	Ken	3/1/13