SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

NOVEMBER 22, 2013

The Committee held a conference call on Friday, November 22, 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
Mandi Edwards, Envirochem (Lab)	Absent
Tim Fitzpatrick, Florida DEP (Lab)	Present
Andrew Friedrich, Chevron (Lab)	Present
Nancy Grams, Advanced Earth Technologists, Inc. (Other)	Absent
Anand Mudambi, USEPA (Other)	Absent
John Phillips, Ford Motor Co., (Other)	Present
Scott Siders, IL DEP (AB)	Absent
Gary Ward, OR DPH (AB)	Present
Ken Jackson, Program Administrator	Present

Associate Committee members present: Lynn Boysen; Arthur Denny

2 – Previous Minutes

Ken had amended the draft minutes of November 1, and had circulated them. It was moved by John and seconded by Andrew to approve them. All were in favor and the minutes were approved.

3 – Calibration Voting Draft Standard

The committee had conducted considerable e-mail discussion on **Section 1.7.1.1 k)**, and Richard had then sent out the following language for consideration:

"when test procedures are employed that specify calibration with a single calibration standard and a zero point (blank or zero, however specified by the method), the following shall occur:

i. The zero point and single calibration standard within the linear range shall be analyzed at least daily and used to establish the ~~slope of the~~ calibration.

ii. To verify adequate sensitivity a standard shall be analyzed at or below the lowest concentration for which quantitative data are to be reported without qualifiers. This standard shall be analyzed prior to sample analysis with each calibration and shall meet the criteria established by the method. If no criteria exist the laboratory shall specify criteria in the SOP.

iii. Some methods allow data within the linear range of the instrument, but above the daily calibration, to be reported without qualification. For these methods, the upper reporting limit must be established through analysis of a series of standards. The upper reporting limit is equal to the concentration of the highest standard meeting the method limits for accuracy. Linearity must be established annually and checked at least quarterly with a standard at the top of the linear working range, or at the frequency defined by the method. Samples with results above the linear range must be diluted, or the over-range results qualified as estimated values."

In subsection iii John was concerned that, in meeting the method limits for accuracy, the intent should be the ability to achieve the requirements for precision and recovery/ bias. After some discussion, it was agreed to defer this until quantification was discussed.

In subsection ii, Tim said the standard expects the method criteria to be met at the LOQ or lowest standard being run, but most methods only publish recovery criteria at higher levels (e.g., the mid-point). On Richard's suggestion "and shall meet the QL criteria established by the method" was added.

Francoise had a comment on **section 1.7.1.1 d) v**: "Only one entire concentration level may be removed or replaced from the interior of the calibration curve. If replaced, the replacement level must be analyzed within 24 hours of the initial level and prior to sample analysis." She pointed out that replacement was also discussed in subsection ii of the same section, and suggested moving the text to a new paragraph. Brooke had also suggested putting everything related to replacing a standard together in a separate subsection and Francoise volunteered to do this.

Francoise identified a typographical error in **Section 1.7.1.1 n)** (should be "based").

In **section 1.7.2 c)**, "The concentration of the calibration verification standard shall be equal to or less than the highest level in the calibration", Francoise pointed out it should state "..less than half the highest level..".

On Dan's suggestion **Section 1.7.2 d) iii** "a calibration verification (second source calibration verification) that passes the continuing calibration verification criteria may be used in place of a continuing calibration verification standard." was reworded to read "a second source calibration verification that passes the continuing calibration…".

Brooke had made several comments on **Section 1.7.1.1 d)**. In subsection iii she suggested moving this section to be ii instead of iii. That way, the subject of individual analytes are grouped together in i and ii, rather than separated by a section about multiple

analytes. In subsection ii Brooke suggested saying "at the low AND/OR high end of the calibration" rather than just "the low or high end". Also in subsection ii, she said the second clause of the same paragraph states that "only one level may be replaced". She didn't remember the committee was differentiating between 'remove' and 'replace' as different cases and would have missed the distinction. She suggested making the second part a new section, since it is a different subject, and make it: "iv) Only one level may be replaced (rather than simple removal) from the low and/or high end of the calibration." Francoise would be re-writing this section and she agreed to take Brooke's comments into consideration.

Brooke felt that Sections **1.7.1.1 i) and j)** were a little confusing with all the terms flying around. She suggested trying to keep the acceptance criteria for the initial calibration and the new concept of relative error more separate by adding the word "additionally" before **j)**. This would more clearly separate the two criteria. The change was made.

Several more clarification changes were made on Brooke's suggestion. In **Section 1.7.1.1 j)**, the wording "The relative standard deviation (RSD) from an average response factor (RF) calibration is a sufficient measure of relative error." was changed to "For calibrations using an average response factor, the relative standard deviation (RSD) may be used for the measure of the relative error." In **Section 1.7.1.1.l)**, "(e.g. 1016/1260)" was changed to "(e.g. a mixture of 1016 and 1260)". The acronyms ICV and CCV were inserted respectively after "initial calibration" in **Section 1.7.1.1 m**), and after the title "Continuing Calibration Verification in **Section 1.7.2**.

The committee next returned to the specific voters' comments.

Roger Kenton, Section 1.7.1.1 k). His comment was *Deletion of ". . . the following shall occur for instrument technology (such as ICP or ICP/MS) . . . " and addition of "when test procedures are employed that specify calibration with a single calibration standard and a zero point . . . " The new language seems to indicate that a quantitative result cannot be reported without qualification for ICP or ICP/MS methods that utilize multiple calibration levels if the highest calibration standard is exceeded even if a linearity check sample is analyzed and meets acceptance criteria.The standard should clearly indicate that linearity check standards (if a linearity study has been previously performed) can be used to report quantitative values without qualification for multiple calibration level ICP and ICP/MS methods.If the use of an ICP or ICP/MS linearity check sample is acceptable for a single point calibration and a zero point, then the use of an ICP or ICP/MS linearity check sample should be acceptable too. (I understand that this change may affect other technologies.) I thank the Committee for all of their efforts and for considering my comments. I recognize that the standard revision process can be challenging.* This language had already been clarified.

Pam Varner and Greg Jones, Section 1.7.1.1 k) i had both made the identical comment *1. The statement "Prior to calibration .... " would infer that linear dynamic range determination is performed before the analytical system is calibrated which is incorrect. Calibration must be performed prior to LDR detennination. 2. The procedure appears to*

require a multipoint calibration with standards all the way up to the upper linear range to be used continuously with only reslope required on a routine basis. First, the multi point calibration described would be used primarily for ICP-OES and ICP-MS systems. These systems have very wide linear dynamic ranges (several orders of magnitude) and including the high concentration standards in the actual calibration function would greatly bias the low end of the calibration ranges. The concentration levels of the standards would also mandate that individual or "short list" standard mixes be prepared due to the levels of dissolved solids and interelement effects that would be sufficient to bias raw values. This would result in a significant increase in the number of individual standards that would need to be prepared and analyzed for no improvement in the linear dynamic range definition. 3. Use of the new process as written would result in LDR determined using calibrations not consistent with that used for routine sample analysis. The system should be calibrated with the single standard and zero point exactly as it would be for routine analysis with LDR evaluation based on this function. Current industry standard, manufacturer's recommended and method specified means for LDR determination with single point calibrations are sufficient. This had been re-worded to make it clearer that you are doing a calibration first.

Pam Varner and Greg Jones, Section 1.7.1.1 k) ii had also made the identical comment *Resloping of a previously constructed calibration function using a single standard has long been forbidden in most environmental reference methods. This practice will result in poorer accuracy than the current daily, single point calibration allowed in Method 6010c.* Richard clarified that nothing is being done to adjust the previously created calibration function; a new calibration is created each day.

Aaren Alger, Section 1.7.1.1 k) i commented *Section 1.7.1.1.k.i does not include any acceptance criterion to determine if linearity and verification of the standard at the top of the linear range. It just requires that the verification occur*. Richard responded that in general specific criteria are not included in the standard; method requirements are used.

Gary Ward, Section 1.7.1.1 k) iii commented *This allows reporting below the LOQ without qualification and with criteria decided by the lab. The lab can set the criteria at any value to make everything acceptable.* The committee had already commented "See language for LOQ 1.5.2.2c".

Roger Kenton, Section not specified, said *This section removes the allowance to use a linearity check sample to extend the calibration range for highly linear techniques such as ICP. Depending upon the project (especially for waste testing), little is gained by cutting an ICP sample and re-running if a linearity check sample was performed and acceptance criteria were met. (See discusion for Section 1.7.1.1.k below too.).* This was a misinterpretation of the language and it had been clarified.

Michelle Wade, Section 1.7.1.1, 1st paragraph questioned *Does it matter if it's the most current initial calibration - because reading this the way it is written does not even imply that. So is it now acceptable for a laboratory to utilize a passing calibration from a month ago, since the calibration ran this morning failed? I see where it's at below - but a*

lab isn't going to read that far. The language had been clarified to state most recent calibration.

This concluded the consideration of all comments. Ken volunteered to draft the Response-to-Comments document. The committee would then need to vote on each comment to rule it persuasive or non-persuasive.

4 – ELAB Input on the Revised MDL Procedure

Richard said the committee must decide whether to change anything as a result of the comments or whether it would be sufficient to provide explanations to ELAB. First, however, Richard suggested the committee should review a document John had prepared to compare the old and new language. They should also review the presentation Richard gave to EPA explaining why the changes were deemed necessary. Richard asked the committee to review both documents in detail before the next meeting. The committee would need to respond not only to ELAB's comments, but those expected from EPA and perhaps other stakeholder groups. John added that the committee might also look at the 2003 comments EPA received as a result of proposed revisions to the MDL. He felt the revised procedure already addressed most of those comments. Also, he said EPA had asked for the rationale for some of the numbers used in the procedure, and the committee should prepare to respond to that. John said he would send out the comment summary he had prepared.

The next call was scheduled for December 20, 2:00 – 3:30 pm Eastern Time.

5 – Adjournment

The call was adjourned at 3:05 pm EST.