SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

AUGUST 4 AND 5, 2014

The Committee met at the Environmental Measurement Symposium, Washington DC on August 4 and 5. Chair Richard Burrows led the meeting.

1 – Roll call

Richard Burrows, Test America (Lab)	Present
Francoise Chauvin, NYC DEP (Lab)	Present
Brooke Connor, USGS (Other)	Absent
Dan Dickinson, NYSDOH (Accreditation Body)	Present
Mandi Edwards, Envirochem (Lab)	Present by telephone
Tim Fitzpatrick, Florida DEP (Lab)	Present
JD Gentry, ESC (Lab)	Present
Nancy Grams, Advanced Earth Technologists, Inc. (Other)	Present (Tuesday session only)
Anand Mudambi, USEPA (Other)	Present
John Phillips, Ford Motor Co., (Other)	Present
Scott Siders, IL DEP (AB)	Present
Gary Ward, OR DPH (AB)	Present
Ken Jackson, Program Administrator	Present

2 – Introductions

The Monday morning session was from 9:00 to 12:00 EDT. Richard welcomed the participants and the Committee Members introduced themselves. Richard outlined the agenda, saying the Committee would consider comments received on the Calibration Interim Standard on Monday morning (Aug. 4), and would have a working session on Tuesday morning (Aug. 5) to consider quantitation limits for the proposed working draft standard on detection and quantitation.

3 – Comments received on the Calibration Interim Standard

1.7.1

The committee considered if the sentence "Calibration requirements for analytical support equipment are specified in Module 2' should be removed. Dan said it framed the clauses that followed and Tim added it adds context. There were no opinions from the floor and the committee decided to leave it in. There had been a request to remove the second paragraph. Richard felt it was not essential; it was just there to clarify instrument vs. method calibration. Jerry Parr commented that clarification was needed, because you do not calibrate methods. Dan felt there were precedents (instrument detection limit and method detection limit). Anand moved to remove all except the first sentence to get rid of the examples that LASEC did not like. Tim seconded the motion and all were in favor.

1.7.1.1

It had been asked why "instrument calibration" had been changed to "calibration". Richard felt there was no telling reason to put it back in. The phrase "appropriate data qualifiers" had been questioned, since EPA drinking water requirements do not allow qualification. However, Richard wanted to leave it in, pointing out there is already a statement that regulations supersede the standard. Judy Morgan said LASEC was concerned that it should be made clear where drinking water regulations diverge from TNI. Anand replied that the committee had decided not to keep putting in everywhere "unless not allowed by regulation". Judy responded that most laboratories have policies for dealing with exceptions, so it does not belong in a standard, and Roger Kenton added the standard requires laboratories to have procedures to handle exceptions. Richard thought if it was taken out it could precipitate Standards Interpretation Requests to ask what you need to do if you cannot reanalyze. Jack Farrell suggested referring to a laboratory's policies and procedures for dealing with non-conformances, and Bob DiRienzo said it should refer to V1M2 clause 4.9.1 in the 2009 standard. It was moved and seconded to do as Bob suggested, and all were in favor.

1.7.1.1.c

A commenter had said the standard should say how old a calibration may be. However, Richard said that would be a new requirement that could not be dealt with at this time. On Jerry Parr's suggestion, it was tabled until the next revision of the standard.

1.7.1.1.d

The committee had now received a comment that this section on removal and replacement of standards was "overdone", after initially responding to a comment to be more specific. Judy Morgan said this was not in the 2009 standard and no Standards Interpretation requests had been received. She thought it was now confusing. Richard responded there were a lot of comments at the Working Draft Standard stage that laboratories could do what they wanted to remove standards. However, Judy felt that laboratories would not do other than what was now stated in the standard and the Accreditation Bodies could keep control of it. Richard countered in that case it would not be auditable. Judy thought the language was confusing and could not be audited consistently as written. Jerry Parr suggested looking at the DoD Quality System Manual and think about re-drafting the language. Fred McLean added that DoD says anything rejected other than the high or low standard must be justified, and it has not been an issue that it might be un-auditable. The committee agreed to review and re-draft after looking at the DoD language. However, June Flowers liked the language, saying it is now very clear when it was never clear before.

1.7.1.1.d (ii) b

This clause states the replacement levels are analyzed within 24 hours of the initial levels, but it had been commented this was too long. John said it was there because a run may be left overnight. Deborah Gaynor suggested it is only important that instrument calibration has not changed during the period. Richard said the committee would note that.

1.7.1.1.d (ii) c

The standard stated for multi-analyte methods, replacement of individual points from any interior concentration levels of the calibration curve is not permitted. A commenter had suggested clarifying you need to run again if that happens. The committee rejected this.

1.7.1.1.e

Some commenters did not like degrees of freedom being in the standard, and some wanted the minimum number of standards in the table to be more. John said a polynomial curve needs to consider degrees of freedom, but he felt it could be taken out of the table, and a reference to degrees of freedom could be included. Aaren Alger said Pennsylvania regulations require 6 standards for a quadratic fit, and Judy Morgan added that is so for most Accreditation Bodies (ABs). Aaren suggested taking degrees of freedom out of the table, since degrees of freedom is just a statement of how the committee decided on a specific number of standards. Scott thought it would help if a definition of degrees of freedom were included (or a note). Richard said a minimum number of standards are specified, so nothing prevents anyone requiring more standards. Anand moved to leave the number of standards as stated, because there is no statistical rationale for increasing the number. He added the ABs can always require more. Tim seconded the motion, and all Committee members were in favor. In regard to footnote *b*, Lem Walker had asked if the standard addresses systems where the calibration is already pre-defined in the instrument; i.e., if you still have to verify that calibration. Richard said that would be in the calibration verification section. Aaren had commented that footnote *b* should not tell a laboratory what it cannot do, so Richard asked if that footnote could be removed. Scott said it could not, because that could lead to a finding; e.g., the laboratory might need to buy a new instrument. Aaren added the standard should not say a laboratory can use an instrument that will not let it meet the standard, and suggested a qualifier should be used instead. Scott questioned if the footnote was removed, whether additional language would then be needed in the initial calibration verification. Richard said the committee would need to think about this more before reaching a decision.

1.7.1.1.h

The statement "..unless otherwise required by regulation, method, or program." was language originally in the standard, but it had been suggested to change it. Since the committee was unable to make any change at this point, the comment was tabled until the next revision cycle.

1.7.1.1.i

The statement "The criteria used shall be appropriate to the calibration technique employed." was also questioned, but is also original language so it was also tabled.

1.7.1.1.j

A request to remove "additionally" was agreed to. A commenter thought the concept of Relative Error was confusing, but Richard said it was very important to the standard. He added that Relative Standard Error (RSE) is in the latest version of Method 8000. June Flowers thought it would be made clear if the Relative Standard deviation (RSD) equation was also listed. Judy Morgan said LASEC wanted a criterion for the RSE; i.e., a threshold on what is reasonable. She said it is not auditable as it is. Jim Todaro thought a laboratory criterion might not be acceptable. Anand said the committee could only

put in a maximum (e.g., 35%), but then that would be too high for ICP and ICPMS. Richard suggested the next revision of the standard could have numerical criteria, since data would then have been accumulated. It was asked why Relative Error was used only for the middle and lowest standards, and Richard responded that would catch most problems without requiring more work. Ed Askew suggested a way was needed to evaluate what is a reasonable RSE for data users.

At this point, the meeting adjourned at 12:00 noon EDT.

The Tuesday session was from 9:00 am to 12:00 EDT. Richard again welcomed the participants and the Committee Members introduced themselves. Richard explained that a Working Draft Standard for Limit of Quantitation (LOQ) was a work in progress. He presented an initial draft of a standard, explaining that a laboratory would be allowed to choose its own LOQ and then go through a procedure to verify it. Richard described the draft procedure.

Comments were solicited on the Initial Verification. Tim said the LOD must be increased if there is sporadic contamination, and it may even be brought into the quantifiable range. John said a floor will be set for the LOQ which may be around L_D. He added that false negatives need to be controlled. With small data sets one exceedance will mean the LOQ must be increased. It was explained if there are 700 data points from 7 samples, there is a chance the LOD will be exceeded even if it is quantifiable. The criteria are (1) you can have a certain number of exceedances for multi-analyte methods; and (2) if you get a second hit from the same compound you will need to reject it. Scott Hoatson asked, if a result is less than the LOD, should it be used in the precision and accuracy calculations. Nancy replied that if it is a single analyte method, it is clear you want precision and accuracy data out of that analyte, but if it is a list used for screening with many compounds not detected, allowances are made for flexibility. She said her biggest concern is the consequences of failure, and there is a bigger likelihood of failures in later years. That means tighter limits are needed for initial verification and then more flexibility should be allowed with on-going verification. Paul Junio was concerned the process needs to simple enough for a wastewater treatment plant operator running phosphorous. Richard said it is made clear you can just select an LOQ and you do not have to calculate it. This standard is about verification only. Bob DiRienzo was concerned that data at the LOQ were being subjected to statistics. He thought it could not be done for such variable data at these low levels, and suggested consulting a statistician. Scott said a laboratory can decide how high to make its LOQ, then a simple procedure is doable, but said not to use a point below the LOD. Nancy disagreed, saying you cannot just exclude data. She added she has a problem with data being below the MDL and suggested having a minimum distance between the LOD and LOQ (e.g., LOQ = 3.14 x LOD, and it is not an LOQ if you are getting false negatives. Dan said the concentration needs to be increased until all 7 samples are above the LOD. However, Richard said if the MDL data are used, there will be some results below the LOD. Francoise said it is being made easy for a laboratory, but questioned if it was so for the data user. JD thought this approach could be limited if a laboratory has set the LOQ it needs for its clients.

Continuing Calibration was discussed with John presenting his analysis of data (described in the minutes of July 25, 2014) that was used to study various quantitation limit concepts. This led to a protracted discussion on how to decide the most appropriate criteria. John said he would look at more data sets. Richard said the data need to be in the range a laboratory will use for its LOD and LOQ limits. Richard showed a table of lowest expected result (LER) with 99% confidence. The aim was to change spike

level, recovery, and RSD to get a LER more than twice the MDL. He said the criterion that all results must be greater than the MDL will be very difficult to meet.

5 – Adjournment

The meeting was adjourned at 12:00 Noon EDT.