SUMMARY OF THE
TNI ENVIRONMENTAL MEASUREMENT METHODS EXPERT COMMITTEE MEETING

JULY 20, 2012

The Committee held a conference call on Friday, July 20, 2012, at 2:00 pm EDT.

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
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)	Absent
Lee Wolf, Columbia Analytical Services (Lab)	Present
Ken Jackson, TNI administrative support staff	Present

Associate Committee members present: Arthur Denny; Dianna Shannon; Gale Warren

2 – Minutes from June 22

It was moved by Anand and seconded by Tim to approve the minutes. All were in favor except Nancy who abstained.

3 – Preliminary Discussion on Detection and Quantitation

Richard had circulated a document "What We Need a Procedure To Do" (Appendix C, Adopted by Consensus on July 13, 2006 by the Federal Advisory Committee on Detection and Quantitation). He suggested going through the list of items in this document to decide on the kind of approach the Committee should take on detection and quantitation. He said the Committee should evaluate what is in the current standard to determine which of these items are already in the standard. Dan asked if we will require a specific procedure, as that would then limit the procedures a laboratory is able to do. He suggested a balance between setting requirements and allowing some flexibility. Tim suggested talking about contraints before discussing what a procedure should or should not do. Francoise asked if the Committee will have to include all 14 items in the list.

Nancy added that 40 CFR Part 136 Appendix B is still there and asked if laboratories will have to continue to comply with it in addition to the standard requirements. Richard said that speaks to some of the constraints, since Appendix B will not change. ELAB had a recent call with EPA OW, and they said they have no plans to do anything further with the MDL. We will have to continue to comply with the Part 136 MDL requirement, but

we can shore up the MDLs in some way; e.g., for metals, as well as doing the required 7 replicates, we can also calculate a detection limit based on blanks and then use whichever is more stringent (highest). So laboratories will have to follow what is in the standard and still be able to say it is an MDL compliant with 40 CFR Part 136 Appendix B. There was some discussion on whether such an MDL would be required for all matrices, but it was pointed out that most states and many clients require it, even if the EPA Office of Solid Waste does not. It would, of course, be untenable for a laboratory to have two different MDLs for the same method. Nancy suggested matrix-based MDLs, and Dan added that a laboratory may have different MDLs depending on the preparative method that is used with the determinative method. Nancy said laboratories may be resistant to this, partly because of limitations with their LIMS that only accommodates one MDL per method. Richard said laboratories could be allowed to have only one MDL per matrix-type, and at least move towards more justifiable MDLs by such as considering blanks and spreading out or adding to the replicates. The Committee will need to gauge how much of an extra burden can be placed on laboratories in this way. Tim asked if the Committee should require a specific procedure that will meet both the 40 CFR Part 136 Appendix B requirements and the TNI requirements, or should we just define requirements that are in addition to 40 CFR? Richard said the standard should just define requirements, but a guidance document could provide ways of doing it.

Nancy suggested separating detection and quantitation.; i.e., the procedure for quantitation should not be based on the MDL. Laboratories must also be able to show their precision and accuracy at the LOQ. She also stressed there must be a valid statistical approach used in evaluating the concentration levels so that laboratories can only claim valid detection and quantitation limits. Richard said, to get away from MDLs and quantitation limits that are really wrong, the Committee needs to accomplish at least three things: get blank consideration in place for MDLs; spread out the MDL replicates; and get spikes at the quantitation limit that are processed through the whole method. He also wants to correct the misunderstanding that having an analyte at the MDL does not mean it can be reliably detected. Nancy stressed that this educational aspect can be incorporated into the guidance document.

The Committee next started to go through the 14 points in "What we want a Procedure to Do".

1. **Provide an explicit estimate of bias at L_Q for limits that must be verifiable by labs at those limits.**
To be evaluated by:
a. reviewing procedure(s) and specifically identifying the quantitative limit for bias at L_Q that is tested in the pilot study.
b. requiring labs to analyze samples (spikes, blind or otherwise as appropriate) and comparing observed bias to that cited by the procedure(s).

2. **Provide an explicit estimate of precision at L_Q for limits that must be verifiable by labs at those limits.**

To be evaluated by:
a. reviewing procedure(s) and specifically identifying the quantitative limit for precision at L_Q that is tested in the pilot study.
b. requiring labs to analyze samples (spikes, blind or otherwise as appropriate) and comparing observed precision to that cited by the procedure(s).
See Appendix for specific MQOs adopted by the Committee for the pilot study

This means it must be verified that the experimental quantitation limit is reasonable. This can be done by running some spikes at that limit and measuring the precision and accuracy. Nancy and Richard stressed that measurement of bias and precision must be required. Tim said the goal is to set limits on bias and precision so that if it is felt the limits are too wide, or the bias is too low, you would choose a higher limit of quantitation.

3. **Provide an explicit false positive rate for L_C.**
To be evaluated by:
a. reviewing procedure(s) and specifically identifying the false positive error rate predicted for each limit that is tested in the pilot study.
b. comparing the false positive rate of lab blanks at the estimated levels of L_C to those predicted by the procedure(s).

Richard explained this as follows. If 1% is the predicted false positive rate, the data will either show that the blanks have no hits (i.e., the false positive rate is zero), or they will have rates that may be well above 1%. It depends on the degree of censoring that is in the method. The point is measuring what you are actually getting and then changing the estimate if you are not getting what you think you should be. Tim said that is what some smaller waste-water laboratories thought too difficult to accomplish, because they do not have the database to examine long-term data. Perhaps a simpler way should be found to accomplish this. Nancy wondered why they could not do this by plotting a control chart and observing the rate of false positives.

4. **Provide an explicit false negative rate at L_C for the true value at L_D or L_Q that must be observed in labs at L_C for the estimated values of L_D or L_Q.**
To be evaluated by:
a. reviewing procedure(s) and specifically identifying the false negative error rate predicted for L_D/L_Q that is tested in the pilot study.
b. comparing the false negative rate of results obtained by analyzing samples spiked at the L_D/L_Q concentration to those predicted by the procedure(s).

Richard said this means if the true value is at the detection limit or the quantitation limit then you should be getting results above your MDL for those spikes, and you should be getting results of zero for non-detects. To demonstrate this at the quantitation limit, you can use those same spikes you are using to get your bias and precision statements at the quantitation limit. The detection limit is more difficult. He said he believes the difference between the quantitation limit and the MDL is so small it is unrealistic to try to put another value in between. We should say that the quantitation limit has to meet the

requirements for the detection limit and we will call L_Q our L_D. Nancy added many people confuse MDL with the true detection limit L_D. She suggested the guidance document should explain the difference between the two terms..

5. **Provide that qualitative identification criteria defined in the analytical method are met at the determined detection and quantitation limits.**
To be evaluated by:
a. requiring that all method qualitative identification criteria be satisfied in order for detection to occur.
b. requiring revision of L_Q or L_D if all spikes at L_Q or L_D are not detected.

Richard believes this needs more flushing out. If your true value is at the quantitation limit, then your result should meet all of the analytical method identification criteria. That result may go down to a value close to the MDL. However, if the true value is at the MDL, you don't necessarily meet the identification criteria. Tim has seen laboratories with 2 orders of magnitude between their reported detection limit and their quantitation limit, so how can we be sure they could meet qualitative identification criteria for a true value at the quantitation limit and satisfy it at the detection limit. A difference of two orders of magnitude is unrealistic, suggesting the LOQ is arbitrarily chosen. Richard suggested saying if your LOQ (the level at which you have run spikes) is within some multiple of your MDL then getting results from spikes above the MDL verifies you have freedom from false negatives. However, if your LOQ is 2 orders of magnitude higher, some MDL verification samples must be run that are (say) 3 times the MDL. Nancy suggested a way should be found to drive MDL levels higher when a laboratory has made them unnecessarily low; e.g., using qualitative identification criteria.

4 – Next Steps

The Committee may continue to go through these steps if there is time available at the Washington DC meeting.

5 – Adjournment

The meeting was adjourned at 3:30 pm EST. The next meeting will be at the Environmental measurement Symposium in Washington DC.

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)	Ongoing
6	2/17/12	Review Volume 1 Module 4 of the 2009 standard to identify any inconsistencies with the new language	All Committee Members	Not determined
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	6/18/12
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