TNI ENVIRONMENTAL MEASUREMENT METHODS EXPERT COMMITTEE MEETING

MARCH 30, 2012

The Committee held a conference call on Friday, March 30, 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)	Present
Dan Dickinson, NYSDOH (Accreditation Body)	Absent
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, TNI administrative support staff	Present

Associate Committee Members present: Diana Shannon; Bharat Chandramouli

2 – Minutes from March 2, 2012

It was moved by John and seconded by Anand to approve the minutes of March 2, with the following amendments:

Change Francoise's affiliation to NYC DEP;
In Section 1.7.1.1 j), correct several typos;
In Section 1.7.1.1 k), amend the second sentence to read "There was inconclusive discussion on whether 'at least one'…."

All were in favor.

3 – Continued discussion of Items to Include in the Calibration Section of the Standard

Prior to the meeting, Richard had circulated **John's re-write of Section 1.7.1** (Attached). This was discussed and edited.

Anand noted section 1.7.1 applies to both the instrument level and method level of calibration. It was agreed the section reads well and no changes were suggested.

Section 1.7.1.1 (Initial Calibration) was considered next. It was discussed whether the requirements in this section apply if there are more stringent requirements in the method.

It was pointed out there is already a requirement in the quality system standard that you must follow the standard unless there are method requirements that are more stringent or are in conflict, and then the method requirements must be met. The last two sentences of the paragraph (beginning with "If more stringent standards or requirements….") already cover this. Francoise said, when the text she and Anand had written is reviewed, the Committee should make sure it is not in conflict with this language. Richard said after completing these sections, the Committee should make sure the language does not contradict anything already in the quality systems standard.

There was discussion on the first sentence reading "The initial calibration is the most recent set of valid calibration standards analyzed prior to the analytical batch." Specifically it was questioned what is meant by "valid", and if this be interpreted that some non-valid standards would not be considered; i.e., you could reject a set of standards because you felt the calibration did not work, and go back to the previous calibration curve. Nancy said the initial calibration is what you have to use to evaluate your batch, so we have to define the group of standards that meet the criteria of being the initial calibration. After further discussion it was agreed to remove the first sentence of Section 1.7.1.1, and insert a new subsection c) to read "The laboratory shall use the most recent calibration standard(s) analyzed prior to the analytical batch, unless otherwise specified by the standard." This wording would also apply to a one-point calibration. These changes would avoid having to define "initial calibration". The paragraph beginning "criteria shall be established.." would then become d).

The next section discussed was **Anand and Francoise's re-write of Section 1.7.1.1 j and k.**

In the table of subsection j) , it was agreed to change "Type of Standard Curve" to "Type of Calibration". In order to account for any curve beyond quadratic, it was agreed to change the second sentence to read "For regression type calibrations not included in the table, the number of initial calibration standards must be sufficient for at least two statistical degrees of freedom." This also would take out "linear", so it applied to all regression techniques. There was discussion on whether it should be specified that some analytical techniques require more than the minimum number of calibration standards listed in the table, but since this is method-specific it was agreed this should be discussed in the guidance document. Richard was reluctant to point to the guidance document at this time, saying the Committee should wait until the guidance document has been written and then consider coming back and having the standard point to the guidance documents. There was also discussion on whether the column on degrees of freedom should be included and it was suggested to leave it in, since analysts should be reminded to always consider degrees of freedom. It was agreed to modify the first sentence by inserting "for common calibration types' after "..the initial instrument calibration". Anand and Francoise agreed to go back and consider modifications that might further clarify this section (they might want to add "regression: after "Quadratic" in column 1 of the table).

Action item: to have the guidance document consider orders of magnitude in deciding the minimum number of standards, and keep a placeholder in this section to refer to it.

In subsection k), Anand said they wanted to make sure one-point calibrations (for threshold testing) would be covered. A question to be addressed is whether it is necessary to re-analyze a sample if it is detected at the pass/fail level on a one-point curve. Richard said it had been stated by EPA's SW846 staff that it was acceptable not to re-analyze with a multipoint curve in such a case. Richard added an editorial note to say something else may need to be added for methods like 1668. It was suggested to precede the first sentence with "Where specified in the method and for…". It was suggested to add, after the first sentence, "In this case the working range is defined by the analyte(s) that do have multi-point calibrations". However, Anand questioned the applicability of this sentence and then whether an exception should really be included in this section. Nancy questioned whether the sentence "In this case the working range.." belongs in this section or should be in the calibration range section. Anand and Francoise will work further on this section. Tim asked if pass/fail" is the correct terminology, and Anand suggested it might be "threshold testing".

Action Item: It was noted a definition for threshold testing is required.

4- Next steps

The committee should review the continuing calibration requirements prior to discussing Section 1.7.2, which will probably be discussed during the May call.

Ken will combine Richard's, John's and Anand's changes into a single document, and will circulate to the Committee.

5 – Adjournment

The meeting was adjourned at 3:30 pm EST. The next meeting will be April 6, 2012 at 2:00 pm EDT, and after that, May 4.

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

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
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	Not determined
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	Not determined
11	3/2/12	Include a paragraph in the standard that addresses a single-point calibration for P/A testing.	Committee	Not determined

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
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 modidfied further as a result of the Mrch 30 discussions.	Anand and Francoise	April 6, 2012
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	Not determined

ATTACHMENT

John's Section 1.7.1

1.7   Technical Requirements

1.7.1   Calibration

This module specifies the essential elements that shall define the procedures and documentation for initial calibration and continuing calibration verification to ensure that the data shall be of known quality for the intended use. This Standard does not specify detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not apparent which Standard is more stringent, then the requirements of the regulation or mandated method are to be followed.

Calibrations may be performed at the instrument level (analytical step only) or the method level (analytical plus preparation steps). For certain methods, such as purge & trap or head space analyses, it is not possible to not separate sample preparation from the analytical step. The elements presented in this section may be applied to either instrument or method calibrations.

1.7.1.1   Initial Calibration

The initial calibration is the most recent set of valid calibration standards analyzed prior to the analytical batch. The following items are essential elements of initial instrument calibration:

a)   the details of the initial instrument calibration procedures including calculations, integrations, acceptance criteria and associated statistics shall be included or referenced in the method SOP. When initial instrument calibration procedures are referenced in the method, then the referenced material shall be retained by the laboratory and be available for review;

Anand/Francoise's Sections 1.7.1.1 j and k

j. if a reference or mandated method does not specify the number of calibration standards, the minimum number of points for establishing the initial instrument calibration is given in the table below. For linear regression techniques
the number of initial calibration standards must be sufficient for at least two statistical degrees of
                  freedom.

Type of Standard Curve	Minimum number of calibration standards	Degrees of Freedom	
Pass/Fail Threshold Testing	1	Not Applicable	
Average Response	3	2	
Linear Regression	4	2	
Quadratic	5	2	

k. For multi-peak analytes (e.g, PCBs, technical chlordane,toxaphene), it is acceptable to perform an initial one point calibration, as long as it demonstrates that all representative peaks can be detected at the required reporting limit. Samples above the required reporting limit shall be reanalyzed and quantitated on a valid multipoint curve. Exception: Samples analyzed for pass/fail testing (threshold testing) do not need to be reanalyzed if the initial one point calibration is at the project specified presence/absence (pass/fail or threshold) level. .