# SUMMARY OF THE TNI ENVIRONMENTAL MEASUREMENT METHODS EXPERT COMMITTEE MEETING

### **MARCH 9, 2011**

The Committee held a conference call on Wednesday, March 9, 2011, at 1:00 pm EST.

#### 1 - Roll call

Richard Burrows, Test America (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.	Present	
(Other)		
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	

In addition, the following Associate Committee Members were present: Justin Brown; Arthur Denny; Bernie Wilk

### 2 – Approval of Minutes from February 3, 2011

The committee had approved these minutes by e-mail, with 5 Members voting in favor and 1 abstention. Action items were reviewed and updated.

## 3 – Voting Procedures

The decision-making rules provisionally accepted at the Savannah meeting were revisited. The following was proposed by Richard and seconded by Anand;

This was approved unanimously. Following discussion on the voting rules for decisions not covered in the previously approved decision-making rules, the following was proposed as a decision-making rule by John and seconded by Lee:

"For all other decisions, two-thirds of the Committee must vote and simple majority prevails".

The voting rule for approval of standards at any stage was changed to be consistent with SOP 2-100 (Procedures Governing Standards Development); i.e., a two-thirds favorable majority vote of the Committee Members is required for passage.

<sup>&</sup>quot;A quorum will be a majority of Committee Members".

Ken was charged with making these changes, adding the language for a quorum, and adding language to allow electronic voting by Committee Members. The modified decision-making rules are attached.

### 4 – Discussion of Calibration Procedures Workgroup document

Tim described the document (attached), and asked members to send him any additional items that should be added.

It was suggested that, for consistency, a method must have a defined calibration type. Then the standard should outline the options available within that type. This would avoid people changing calibration procedures during the operation of a method.

Nancy had a question regarding the scope of the committee and in expected work products: is the intent to provide guidance where flexibility allows selection of calibration approach, or is it only to address 'allowable' improvements to prescriptive calibration requirements? The discussion that followed indicated that relative to prescriptive methods, the committee can only say what analysts may do in addition to the prescriptive requirement, since they must at least perform the calibration procedures mandated by the method but many prescriptive methods only prescribe the basics of calibration. Where there is flexibility - from completely to filling in between prescription - the discussion suggested that guidance is within the scope of the committee as regards calibration.

It was agreed the Calibration Procedures Workgroup should expand its document by providing current calibration practices and showing how they could be modified to fix any problems. Anand volunteered to put this into tabular form and circulate to the entire committee. Brooke will send ideas to Anand on adding a criterion for "intercept". For putting everything together including the other workgroup's material, Nancy proposed to use the framework described in Vanatta and Coleman's 40+ part series on Statistics in Analytical Chemistry that appeared in *American Laboratory*. Nancy will work on an outline of the higher level framework similar to Vanatta and Coleman's approach.

### 5- Discussion of Calibration Assessment Workgroup document.

Richard will organize a conference call to discuss the talking points raised so far. He will then expand on the current document (attached). It was suggested the term "reasonable person" should not be used in the document, and it would be better to use DQOs as a basis.

**6-Adjournment.** The meeting was adjourned at 2:30 pm EST

# LIST OF ACTION ITEMS TO BE COMPLETED

Item Date To be				
No.	Proposed	Action	Assigned to:	Completed by:
1	10/26/10 (by Steering Committee)	Investigate availability of data on EPA study on the single laboratory DL QL Procedure v2.4.	Ken/Anand	Complete
2	1/7/11	Prepare condensed agenda for Savannah meeting and send to Jerry Parr	Ken	Complete
3	2/3/11	Ken will post powerpoint presentations, the committee charter, and future meeting schedule on the website	Ken	Complete
4	2/3/11	A group will brainstorm calibration procedures	Anand, Tim, Lee and John	Ongoing
5	2/3/11	A group will brainstorm calibration assessment	Richard, Nancy, Brooke and Arthur Denny	Ongoing
6	3/9/11	The amended decision- making rules will be posted on the website following approval by the committee	Ken	3/23/11
7	3/9/11	The Calibration Procedures document will be expanded and put into tabular form	Anand/Brooke	3/23/11
8	3/9/11	An outline will be prepared of the higher level framework similar to Vanatta and Coleman's approach	Nancy	3/23/11
9	3/9/11	The Calibration Assessment Workgroup will hold a conference call, and the document will be expanded	Richard	3/30/11

# **Environmental Measurement Methods Expert Committee Conference call 3/9/11**

### **AGENDA**

- 1. Roll call
- 2. Minutes approval
- 3. Voting procedures
- 4. Discussion of Calibration procedures workgroup document
- 5. (if time is available) Discussion of calibration assessment workgroup document

# Decision-Making Rules for Environmental Measurement Methods Expert Committee Operations; March 9, 2011

A quorum is a majority of Committee Members present.

Members absent during a voting session of a conference call or a face-to-face meeting of the committee will, in all cases, have the opportunity to vote by e-mail.

Type of Decision	Decision-Making Rule
Meeting dates, times	Person-in-charge decides after discussion
Meeting adjournment	Person-in-charge decides after all business is conducted or allotted time expires
Meeting minutes approval	Request for approval by email to all committee members – changes approved if needed from email. No Vote
Meeting cancellations	Person-in-charge decides
Addition of Committee members	Two-thirds of committee must vote and simple majority prevails
Removal of Expert Committee Members	Person-in-charge decides after discussion
Approval of Standards – any stage	A two-thirds favorable majority vote of the Committee Members is required for passage.
Creation of a new subcommittee	Simple vote of attendees
Election of Committee Chair	Two-thirds of committee must vote and simple majority prevails
All other decisions	Two-thirds of committee must vote and simple majority prevails

# Calibration Procedures Workgroup (for Chemicals) - Draft

#### **Members:**

Timothy Fitzpatrick, Florida DEP Anand Mudambi, US EPA John Phillips, Ford Motor Co Lee Wolf, Columbia Analytical Services

### **Points to Consider**

Calibration Range – At what points are the lower and upper calibration ranges reached? Should some methods only be conducted within a linear range? What about methods that are inherently non-linear; should there be minimum requirements for the change in detector response per unit of concentration?

Calibration Concentrations – should they be evenly distributed throughout the curve or weighted for the upper or lower end? Should the strategy change for non-linear (nth order) calibration? Should there be criteria for minimum spacing (e.g., such that uncertainty ranges of signals do not overlap)? When should blanks be run? When should calibrations be forced through zero?

How many replicates per concentration should be performed?

- Is this dependent upon concentration?
- Is this dependent upon type of curve?
- Is this dependent upon the use of the data?

Order of calibration standards in an analysis run – Might there be situations where it would be advantageous to disperse standards throughout the run so long as all samples are bracketed by CCVs and an ICV is evaluated first during signal processing. This procedure might be applicable to techniques where signal processing is conducted after the analysis is complete (e.g., chromatography and a few other techniques) and the order of signal processing is irrelevant. In this case the calibration would only be applicable to samples in the run. In most cases calibrations are part of an event which consist of running all standards in one run and establishing a calibration curve (prior to running samples).

Should the response factor derived from CCVs be considered for quantification for some test methods?

Use of second source standards to verify calibrations.

Minimum number of standards per type of curve fit.

Choosing a calibration type (including internal or external) based on analyte characteristics.

Choosing a calibration type (including internal or external) based on technology types.

Is commercially available instrument (or instrument compatible) software available for processing the calibration data using all calibration models? Many are not.

Criteria for discarding calibration points.

Frequency of calibration if not specified by the method.

Frequency and criteria for continuing calibration verification if not specified by the method.

Calibration procedures where analytes are anticipated to be non-detected or non-quantifiable.

Bias Correction (NOAA methods require surrogate correction of results).

In what cases should standards be prepared in the same manner as samples?

### **Calibration Assessment Workgroup**

Members: Nancy Grams Dan Dickinson Brooke Connor Richard Burrows

## Characteristics of a good tool for Calibration Assessment

Prohibit the use of calibrations that a "reasonable person" would consider unsuitable for environmental analysis

Allow the use of calibrations that a "reasonable person" would consider suitable for environmental analysis

The term 'reasonable person' may be difficult to define. I think it would be better to discuss the use of calibrations suitable for expected detector characterisitics and response. It can happen that a higher order curve fit will mask an underlying detector issue in cases where such a detector is designed for linear response. It could include situations where a detector range or other operating parameter is not optimized resulting in calibration range that is not suited to the calibration type selected.

Allow easy comparison of the suitability of different curve types

Be useful for all calibration curve types

Be straightforward to calculate and review

Allow the use of calibration criteria currently contained in EPA methods Do not rely on R<sup>2</sup>. Instead use at least one other more pertinent measure for the calibration model chosen.

I assume we are talking about an individual curve created for a specific data set – not how to assess which calibration to choose for this method overall, right??

I think we need to assume that most calculations are going to be done in the instrument software. I don't think many labs will agree to download their calibration data into a spreadsheet and assess it separately. Or will they??

Do we even know how the different analytical instruments treat calibration statistics? Are there differences in approach or computation?

A good tool for calibration assessment is to calibrate the calibrants against the curve and get the expected result within +/-X%.

All the analysts I ever met thought they were the most reasonable people in the world.

Even more reasonable than those who disagree with their calibration practices.

I don't know which sub-group should tackle this question, but, we aren't suggesting that an analyst can change the calibration model day-by-day, are we? Or are we?