

**SUMMARY OF THE
TNI ENVIRONMENTAL MEASUREMENT METHODS EXPERT COMMITTEE
MEETING**

APRIL 1, 2011

The Committee held a conference call on Friday, April 1, 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)	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	Absent

In addition, the following Associate Committee Members were present: Bernie Wilk

2 – Approval of Minutes from March 9, 2011

The committee had approved these minutes by e-mail, with 6 Members voting in favor. Action items were reviewed and updated.

3 – Discussion regarding “Minimum Practices for Scientifically Valid Calibration Procedures and Assessment” document: Parts A and B Sections 1, 2, 3 and 4

The group discussed the document (attached) which was drafted by the Calibration Procedures and Calibration Assessment workgroups (last version sent by Tim on March 31 to the committee).

The committee agreed that the Sections 1, 2, 3, and 4 (Introduction, Background, Scope, and Definitions sections) could be combined for the Calibration Procedures and Calibration Assessment parts of the document. The committee also agreed that there would be a general reference section as well as separate reference section for the calibration procedures and calibration assessment parts.

Brooke asked whether this document would be guidance or a standard and who would be the audience.

Richard stated that this document would be guidance initially for the committee and then for TNI and EPA. The first complete draft would be for internal use in order to identify

issues and to make the case to TNI (as a presentation at NEMC conference in August) for going forward with a guidance document.

The discussion then focused on how the committee would go about fleshing out the document (who will do what).

Anand agreed to put section numbers on the document for easier reference.

The following members agreed to write the first draft (outline) of the section noted below:

Section 1: Introduction: Brooke

Comment – should be concise.

Section 2: Background: Richard

Comment – note current weaknesses in calibration.

Section 3: Scope: The committee decided to leave writing the scope till other parts of the document were fleshed out.

Discussion – use the EPA TNI grant document as the basis?

Section 4: Definitions: John

Discussion – Richard asked John to use definitions from the EPA Glossary of Terms whenever possible. John said he would first work on the terms needed and then compare them to the EPA Glossary. He would focus on calibration terms not detection or quantitation.

The following points were raised – whether Sections 1 through 4 should include calibration for microbiological and radiological methods as well as isotope dilution.

It was agreed that these sections should focus on chemistry methods and keep placeholders for the radiological methods (the committee would need to request assistance from radiological method experts).

Anand stated that the committee should focus on the majority of chemistry method commonly used in the environmental arena. Tim said the committee would need to bring additional experts to address issues with less commonly used methods.

It was agreed that the focus should be on the following calibration types and issues:

Internal standard

External standard

Linear and non-linear

Weighted and unweighted

Method of Standard Additions

Differences between Organic and Inorganic calibrations

4 – Discussion regarding “Minimum Practices for Scientifically Valid Calibration Procedures and Assessment” document: Part A Calibration Procedures Sections 5 and 6

The discussion continued regarding Sections 5 and 6 which would be kept separate for calibration procedures and calibration assessment. The following members agreed to draft an outline of the sections noted below.

Section 5a: Implementation of Current Methods (Items i to iv): Tim with Lee’s assistance.

Discussion: Tim will flesh out issues with short term vs. long term response drift. Lee brought up the use of second source standards but felt it was more of an assessment issue. Tim thought it could be addressed in both the procedures and assessment sections and will do so in the procedures section for published methods. Richard also asked Tim to consider the resource implications for short term and long term calibrations. Brooke asked for an alternative techniques section under Calibration Procedures similar to the one written for Calibration Assessment. An example would be low range and high range curves for the same analyte. Tim stated that some analyte responses are curved and better calibrations are obtained with quadratic curves. It was agreed to put a placeholder for non-linear responses which would include tests such as micro and ELISA.

Section 5b: Laboratory Developed Methods: Will be developed after Section 5a is drafted.

5 – Discussion regarding “Minimum Practices for Scientifically Valid Calibration Procedures and Assessment” document: Part B Calibration Assessment Sections 5 and 6

The following members agreed to draft an outline of the sections noted below.

Section 5a: Current Methods Practices: Richard with assistance from Brooke.

Section 5b: Alternative Techniques Assessment:

Relative Standard Error: Richard

Replications Outline: Nancy

6- Due Dates and Next Call

The due dates for all action items will be Friday April 29, 2011. The next committee conference call will take place on Friday May 6, 2011 at 100 PM ET.

7 - Adjournment. The meeting was adjourned at 2:10 pm ET.

LIST OF ACTION ITEMS

Item No.	Date Proposed	Action	Assigned to:	To be Completed by:
1	2/3/11	A group will brainstorm calibration procedures	Anand, Tim, Lee and John	Complete
2	2/3/11	A group will brainstorm calibration assessment	Richard, Nancy, Brooke and Arthur Denny	Complete
3	3/9/11	The amended decision-making rules will be posted on the website following approval by the committee	Ken	Complete
4	3/9/11	The Calibration Procedures document will be expanded and put into tabular form	Anand/Brooke	Complete
5	3/9/11	An outline will be prepared of the higher level framework similar to Vanatta and Coleman's approach	Nancy	Complete
6	3/9/11	The Calibration Assessment Workgroup will hold a conference call, and the document will be expanded	Richard	Complete
7	4/1/11	Minimum Practices Section 1 Introduction	Brooke	4/29/11
8	4/1/11	Minimum Practices Section 2 Background	Richard	4/29/11
9	4/1/11	Minimum Practices Section 4 Definitions	John	4/29/11
10	4/1/11	Minimum Practices Calibration Procedures Section 5a	Tim with Lee's assistance	4/29/11
11	4/1/11	Minimum Practices Calibration Assessment Section 5a	Richard with Brooke's assistance	4/29/11
12	4/1/11	Minimum Practices Calibration Assessment Section 5b Relative Standard Error	Richard	4/29/11
13	4/1/11	Minimum Practices Calibration Assessment Section 5b Replications Outline	Nancy	4/29/11

**Environmental Measurement Methods Expert Committee
Conference call 4/1/11**

AGENDA

1. Roll call
2. Minutes approval
3. Discussion of “Minimum Practices Required for Scientifically Valid Calibration Procedures and Assessment” workgroup document

Minimum Practices Required for Scientifically Valid Calibration Procedures and Assessment

1. Introduction: What is Calibration?
2. Background: Why this guide(s)/procedure(s) needed/worth the time
3. Scope: Provide guidance on how to best fill in the gaps on prescriptive method and plan, produce technically (or scientifically) appropriate calibrations for the use(s) and evaluate the performance of said calibrations against goals/objectives (where allowable).
4. Definitions: [Nancy Grams: I think we should start putting terms in here as we start to use them, both definitions and 'use' terms that we could like to standardize between different authors (e.g., bias vs. accuracy).

Possible resources for Definitions;

Part 24 of the Coleman & Vanatta – Analytical Laboratory series is a Glossary

R. Gibbons and D. Coleman, "Statistical Methods for Detection and Quantification of Environmental Contamination", John Wiley & Sons, New York, 2001 – Glossary of Measurement Terminology pg 323

R. Calcutt and R. Body, "Statistics for Analytical Chemists", Chapman & Hall, London, 1983 – May have definitions perhaps Ken could check his copy

Meir and Zund, "Statistical Methods in Analytical Chemistry", John Wiley & Sons, New York, 1993 – May have definitions perhaps Tim could check his copy

EPA Environmental Measurement Glossary of Terms

(http://www.epa.gov/fem/pdfs/Env_Measurement_Glossary_Final_Jan_2010.pdf)

A. Calibration Procedures

Designing a Calibration (from Lee Wolf)

- a. Implementation of published methods
 - i. Evaluate current practices and potential weaknesses
 1. Initial calibration
 2. Ongoing calibration verification
 - ii. Technology and instrumentation considerations
 - iii. Analyte considerations
 1. Multi-response analytes
 2. Analytes not commonly found in samples (and/or analytes not detected in a specific sample)

- iv. Allowed flexibility and restrictions/limitations for routine use
- v. Recommended calibration practices

b. Alternative Procedures

c. Laboratory developed methods (aka in-house methods, lab SOP, etc)

- i. Appropriate technique for the analyte
- ii. Validation of calibration model(s) to be applied
- iii. Define restrictions/limitations for routine use
- iv. Allowed flexibility for routine use
- v. Recommended calibration practices

6. Points to Consider:

Calibration Range

Calibration Concentrations

Second Source Standards for verification

Minimum number of standards per type of curve fit

Weighting

Selection of curve fit type

Criteria for disregarding calibration points

Frequency of calibration if not specified by the method

Calibration procedures for analytes anticipated to be non-detected or non-quantifiable

Y-intercept

The workgroup will probably want to put bounds around or make general recommendations for all items under "Designing a Calibration", before we start the evaluation procedure.

B. Calibration Assessment

5.

a. Current methods

Evaluate current practices and potential weaknesses

Technology and instrumentation considerations and limitations.

Detected vs. not detected analytes

b. Alternative techniques

Strengths and weaknesses of alternative techniques

Relative Standard Error

Replications Outline

Assessment of errors at each calibration standard

Verifying calibration points (e.g., when one standard and one blank is used for the initial curve)

Recommended practices

6. Points to consider

- We need numerical criteria.
- Need to allow easy comparison of alternative fits
- Prefer to use existing numerical assessment criteria in EPA methods if possible
- The RSE may be the best tool we have for assessing the quality of a calibration curve.
- We are deleting all references to "reasonable persons".
- We probably care more about relative error than absolute error.
- We probably care as much about a 20% error at the low end as we care about a 20% error at the high end.
- It will be difficult to come up with criteria for justifying a quadratic curve.
- Perhaps a decision tree will be needed to determine if a quadratic curve is justified.
- We should keep chemistry in the equation of evaluating curves. Don't use a quadratic to forego maintenance.
- Maybe comparing 2 curve types can be accomplished by determining which one has less error.
- We can't limit what kind of curves people use. (??)
- We maybe need to define how many calibrants are required per curve type.
- The two major tools we use currently are the average response factor and the correlation coefficient.
- We should apply the thought process of "what do we need a calibration testing tool to do" to these existing tools.
- Instrument software must adapt to our recommendations. They have been amenable to change in the past so long as it is very clear what the need will be.
- The RSE will be one tool that will provide a fair assessment across all curves. We can't compare an average RF to a correlation coefficient fairly.
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- We do recommend quantifying the calibrants against the curve created with them, and to set acceptance limits for that. It may be analyte specific (within reason) for large analyte suites.
- Our tools must assess the quality of the tools written into regulatory methods. A new version of 8000D will assess RSE against RSD criterion in the method.
- Cubic = bad
- We are aware that different manufacturers may use slightly different algorithms to calculate statistics. We will deal with this problem as best we can.
- Change calibration model while still using same calibrants? = bad