SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

JANUARY 16, 2013

The Committee met at the Forum on Laboratory Accreditation, Denver CO, on Wednesday, January 16, 2013, at 8:00 am MST. 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)	Present
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)	Present
Lee Wolf, Columbia Analytical Services (Lab)	Present (by telephone)
Ken Jackson, Program Administrator	Present (by telephone)

2 – Introduction

Richard Burrows called the meeting to order at 8:00 am. The meeting's objective was to review changes made to the Chemistry Working Draft Standard (WDS) resulting from comments the committee had received and to discuss possible changes to the method detection limit (MDL) procedure referenced in 40 CFR Part 136, Appendix B.

3 – Review of Changes Made to WDS

The proposed changes, highlighted in the WDS, were presented for discussion.

There were no comments on **Sections 1.7.1, and 1.7.1.1 (c) through (h).**

Section 1.7.1.1 (h) i.

In response to a question from the floor it was confirmed that compliance with this section would require an assessor to ensure a laboratory follows what is written in its SOP. It was asked if this is a client-based quality objective for LOQ, and Richard responded yes, except some methods have that criterion. A participant was concerned that some laboratories may specify criteria that would not support good data. The attendee suggested that the limit of quantitation (LOQ) could be required to be derived from the continuing calibration verification (CCV) standard.

The committee responded that it would be working on defining language for the LOQ in the future. The committee also felt that it would not be appropriate to list criteria for all methods or analytical techniques in the standard, and no further changes were made to the text.

Action Item: The committee should decide in the future whether it would be appropriate to insert a numerical value in this section.

Discussion continued to regarding the variability of the LOQ and whether that value would be less consistent for lower LOQs. Some participants discussed requiring a positive check at the LOQ and suggested that the acceptance range for this check should be plus or minus the reporting limit (RL). Attendees also questioned what would be appropriate action when client requirements for quantitation were far above lower LOQs.

There were no comments on **Section 1.7.1.1 (h) ii.**

The following action item was decided in response to comments on **Sections 1.7.1.1 (i) and (j):**

Action Item: The committee will review the reporting sections of the standard to be sure the concerns expressed at the meeting were addressed and to ensure consistency between the reporting section and this section.

There were no comments on **Sections 1.7.1.1 (k) through (m)**.

Section 1.7.1.1 (n) is attempting to address new requirements in EPA's SW-846 Manual. The committee clarified that the calibration failure addressed in the section was only a "marginal" failure. It was stressed that some methods do not allow this, so then it would not apply, and some felt the committee should clarify that this option could only be exercised when methods allowed it.

Action Item: The committee will add language to make it clear this only applies when allowed by the method.

Others asked whether the option could be incorrectly applied to single-analyte methods. Dan suggested adding language to limit this to multi-analyte methods where marginal exceedances are allowed, but Tim cautioned that a single analyte could be run by a "multi-analyte method". Therefore, it was suggested to limit it to methods with more than 10 analytes.

After a protracted discussion, Anand explained that the intent of the section was: (i) for multi-analyte methods to allow a laboratory to proceed with calibration even if a few analytes failed the calibration; (ii) to address how much failure is allowed; (iii) to be auditable; (iv) to explain what is meant by "without further qualification"; and (v) to have a sensitivity check standard for compounds that are rarely detected anyway.

Attendees and committee members engaged in in-depth discussion. Some of the salient points expressed were:

- The meaning of "further qualification" may need to be more specifically defined.
- The committee already had discussed sensitivity checks for compounds that are not detected in samples and felt the approach in the allowance would be beneficial for precisely these types of analytes.
- For laboratories, it is a good idea to limit the number of recalibration attempts for compounds that are rarely detected in samples.
- The allowance would be applicable only to methods that detected more than ten analytes simultaneously.
- For laboratories to exercise the marginal calibration failure option, an associated sensitivity check would need to be acceptable.
- The sensitivity check refers back to the lowest level standard in the original calibration curve.
- The standard needs to define "sensitivity check".
- Including an "if and" statement can clarify that under those conditions, the standard allows reporting results for non-detected analytes.
- The bullets in the section need to be indexed to the standard's numerical convention; in this case they would 1.7.1.1. (n) i and 1.7.1.1 (n) ii. A description of the "sensitivity check" will be appended to the end of the last clause.
- The committee discussed, but was not in favor, of using a procedural (undergoing sample preparation steps) lower sensitivity check in lieu of the process described in the section.
- If an analyte not generally detected in samples were detected and that analyte also failed calibration, the laboratory would have to recalibrate and reanalyze affected samples or qualify them accordingly.

The following language was then agreed upon:

" For those methods with more than 10 analytes… [needs expanding] (i) the calibration criterion fails marginally (by a maximum of an additional 10% RSD/E or 0.01 correlation coefficient/ coefficient of determination) and (ii) a successful calibration sensitivity check determination has been performed. Non-detect sample results may be reported without qualification for initial calibration failure. The demonstration of sensitivity shall be the successful detection of the analyte(s) in the lowest calibration standard (at or below the Limit of Quantitation) and meeting all identification criteria specified in the method or the SOP".

Section 1.7.2 (d) iii

The committee clarified that this section pertains to the first analytical batch analyzed after a full initial calibration or the batch that contains a second source calibration verification standard (CCV). The discussion thus centered on understanding that a CCV need not be analyzed after completing a full calibration.

Highlights of the discussion included:

- Using a second source standard as a CCV is not a good practice and is greatly discouraged.
- The standard should state that when methods do not require a CCV, a CCV is still required unless the method itself, as for example "color", is not amenable to this type of verification.
- Attendants pondered whether an LCS could be used in place of a CCV or an initial calibration verification (ICV) standard (second source) if the LCS were prepared from a second source, but the committee did not conclusively address the issue. The committee did agree that LCSs could be used instead of CCVs when calibration standards were procedural and the source of the LCSs and the CCVs were the same as that used for the calibration standards.

The committee considered various changes on the language content and placement to make this section more clear. Finally, the existing 1.7.2 (d) iii, was replaced with the following:

"iii. An ICV (second source calibration verification) may be used in place of a continuing calibration verification immediately after an initial calibration.

iv A LCS may be used in place of a continuing calibration verification for methods where the calibration goes through the same process as the LCS (using the CCV limits)."

The committee agreed to review the section for editorial soundness and to ensure the clause captures the committee's intent accurately.

The committee clarified that when an LCS is legitimately used as a CCV, the limits of acceptance for the LCS would be those of the CCV, not the LCS.

Section 1.7.2 (f)

A written comment had been received on this section, which is unchanged from the existing language. It had been questioned if it was correct to run a second CCV if the first one fails and to then accept the second CCV. Lee commented that, if this is the right thing to do, it still needs to be clarified because it has been interpreted inconsistently. After some discussion it was agreed to remove the wording that allows a second CCV if the first one fails.

The committee added to 1.7.2 (f) iii that when a CCV failed low, results could be reported without qualification if the appropriate sensitivity check had been successfully performed.

The committee clarified that the sensitivity check was not the same as the low level CCV. The sensitivity check is not required to be analyzed, but when a laboratory analyzes it and it passes, it can be used to allow reporting unqualified results even when a CCV fails.

4 – Review of Changes Made to the MDL Procedure

The committee presented justification for its decision to modify the exiting MDL protocol instead of proposing an alternative sensitivity parameter. The applicability of the MDL is so pervasive that proposing another detection limit would be confusing and would require computing two different sensitivity measures. Consequently, the committee decided to propose changes to 40 CFR Part 136 Appendix B and submit them to EPA to facilitate changing the MDL protocol to be more accurate and robust.

The committee briefed attendees on the following changes it was considering to the MDL procedure:

MDL Definitions Section

- "Minimum analytical result" replaces "minimum concentration".
- "Concentration distinguishable from a method blank" replaces "concentration greater than zero".

Scope and Application

- Added non-applicability of MDL to certain "non-standard" methods.
- Added a statement about an on-going verification check of MDL.
- Added language addressing the non-quantitative region of data between the MDL and the LOQ.

Section 1

- The method blank can be used to make an MDL estimate.
- Eliminated reagent blank interference discussion.
- Added that sample preservatives need to be included in the replicates used to determine the MDL.

Section 4

- Replicates are to be analyzed in three different batches run on separate days.
- Removed allowance for blank subtraction.
- Added allowance of deriving the MDL from multiple instruments.

Section 7

- Added calculation for the quantity MDL_b, to determine a numerical result from method blank detects. The MDL itself becomes the greater of the two quantities: the traditional MDL or the MDL_b.
- Added a quarterly verification of the MDL.
- Included an annual recalculation of the MDL using a verification spike and a method blank.
- Removed the optional reiterative procedure to verify the MDL estimate.
- Incorporated method blank data when blanks provide numerical results.
- Added verification of MDL instead of a having to perform new MDL determinations annually.

The committee gave an overview of the efforts leading to its current approach to modifying the MDL procedure. A committee authorized under the Federal Advisory Committee Act (FACA) met and deliberated for two and half years and devised an alternative procedure to replace the MDL. EPA felt that the procedure was too complicated and different from the existing MDL protocol and suggested a redefined process anchored in the existing MDL would be more viable. This led to discarding the notion of having a completely different protocol and instead creating a procedure that would improve what laboratories use now. This new protocol may not be perfect, but it would be a measurable improvement over current practices.

The committee envisions including the revised MDL protocol as part of an upcoming EPA methods update rule. For this to happen, a draft of the protocol has to be completed this summer. If that were the case, stakeholder review would happen between September and October 2013 and the final version published sometime in 2014. EPA has asked the Environmental Laboratory Advisory Board (ELAB) to form a stakeholder group to review the committee's protocol.

The protocol would only technically apply to methods published by EPA, but in practice, it is expected it would be adopted by reference by other method developers such as Standard Methods. The protocol developed would not apply to certain tests such as total dissolved solids (TDS), total suspended solids (TSS), alkalinity, acidity, and conductivity.

The committee proceeded to discuss ways of evaluating method blank data and in particular, what to do when the results of a method blank were always zero or non-numerical. Some analytical instruments can give negative numerical results. It is also the case that not all blank results reported as zero are true zeroes. Minimum area reject settings and smoothing algorithms in chromatography can result in false zeroes.

The committee agreed to add definitions for numerical and non-numerical results to the revised MDL protocol.

The committee discussed the proper use of method blank results when numerical and non-numerical results were obtained for the same method or application. The committee proposed that in those situations, when at least 80% of method blank results available were numerical, using numerical non-zero results to calculate the standard deviation (SD) and mean to derive the MDL_b, as long as a minimum of seven numerical non-zero results were available, was allowable. This approach assumes all data obey a normal distribution. The committee also thought that if more than seven numerical non-zero results were available, only results generated during the most recent year should be used.

The committee understood the distinction between the case of an absolutely initial determination of an MDL and that of one where sufficient data had been previously generated to determine a valid MDL. For the latter case, a laboratory could opt to use the MDL verification procedures included in the draft.

The committee considered, without clear resolution, what circumstances might lead to determine an MDL in the same manner as required for an absolutely initial determination. Among those factors evaluated were major changes to instrumentation, and performing a method new to the laboratory. The committee felt that dealing with the transition phase between using the established and the new MDL protocols could be addressed in the new procedure's verification section. The committee clarified that the initial procedure in the new MDL protocol would not apply when laboratories had already established MDLs by the existing 40 CFR Part 136 Appendix B procedure and suggested changes to Section 1 to address this.

The committee agreed that the verification section of the proposed MDL protocol would specify when laboratories had to start at the initial determination step of the protocol and would not be able to use its verification step. The committee also agreed to discuss what would constitute a significant change requiring employing the initial determination step of the protocol.

The committee also agreed that when method blanks and spiked samples were used to calculate an MDL both, the method blank and its corresponding spiked samples, had to be analyzed on the same day under identical circumstances. In other words, method blanks and spiked samples had to be paired daily, but several paired sets would be required to arrive at an MDL.

Members in attendance asked about how to address the case when less than 80% of the method blank results were numerical non-zeroes. The committee clarified that in this case, the highest numerical result would be used as the MDL_b.

The committee proceeded to discuss a possible definition for spike level and felt it needed to specify the spike concentration in the sample used to verify an existing MDL. The committee also felt it would need to address how to proceed when the verification procedure failed to validate the continued applicability of an existing MDL. The committee agreed conceptually that after an MDL had been established every subsequent year at least an MDL had to be verified or recalculated, implying also that the MDL_b would also be verified or recalculated.

The committee noted that it would need to clarify the rounding rules applicable to MDL determinations.

Next Steps

The committee will re-write the section dealing with the initial MDL determination for clarity and to reflect the discussion at this meeting. Francoise and Anand agreed to provide a discussion draft for discussion in February.

The committee will meet by teleconference on February 1, 2013 at 2:00 PM ET.

The committee will review the terms of its current members.

The committee will merge existing versions of the MDL document to incorporate previous discussions and those of this meeting.

5 – Adjournment

The committee concluded its meeting at 5:05 pm MST.