SUMMARY OF THE
TNI ENVIRONMENTAL MEASUREMENT METHODS EXPERT COMMITTEE MEETING

JANUARY 6, 2012

The Committee held a conference call on Friday, January 6, 2012, at 2:00 pm EDT.

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. (Other)	Absent
Anand Mudambi, USEPA (Other)	Present
John Phillips, Ford Motor Co., (Other)	Present
Lee Wolf, Columbia Analytical Services (Lab)	Absent
Ken Jackson, TNI administrative support staff	Present

The following Associate Committee member was also present: Francoise Chauvin (NYC DOH).

2 – Minutes from December 2, 2011

It was moved by John and seconded by Anand to approve the minutes. All were in favor.

3 – Discussion of input received on non-consensus items

a. Richard had circulated a discussion paper on a quantification limit control standard and calibration verification standards in each analytical batch (Attachment 1). He opened the discussion by asking what should be done about methods needing multiple standards. John said determinations of multiple analytes need multiple concentrations of various analytes in the mixture, so it may be necessary to run several sets of the mixtures. Richard added there may be 3 mixtures for method 8270, requiring 3 CCVs, but not all have the same QL, making it harder to cover every analyte in a mix with one CCV. That may necessitate running a lot of CCVs. Dan cautioned that methods are becoming very broad and inclusive, so it may take far too long to consider all QC requirements. He suggested maybe the number of analytes include in a test should be limited, but Richard said that is beyond the committee's scope. John said perhaps some reporting limits could be adjusted upwards so they are all the same. Tim said perhaps LCS's designed to check quantitation limits should be limited to a suite of surrogate analytes. It was agreed that the LCS should include all steps of the analysis including clean-up. John wondered what to do if the QC fails, since the method may not be good enough for it to pass if it is re-run, and perhaps the data need to be qualified. Anand considered it would be unreasonable to ask labs to collect additional data at this time, and Richard suggested

periodic analyses to gather data for performance testing. Tim pointed out that the 2003 NELAC standard requires varying the concentration of the LCS over the range of quantitation. That requirement was dropped, presumably because QC limits in methods were intended to represent DQOs at the mid-range of concentrations, and not at the QL. Richard said method 8000, not yet published, may require a quantification limit control standard quarterly, and he will see what it says. Although additional work will be needed, perhaps the method 8000 requirements will be palatable.

b. Richard had previously circulated item #6 to the committee, stating that currently there is nothing in the TNI standard regarding multi-component analytes. This results in varying interpretations for different labs and assessments, resulting in a non-level playing field, and even data rejection due to inadequate communication of the specific requirements for a given project. He presented 3 options:

(1) Require a full multipoint calibration for every analyte, and require a CCV for every analyte. There may be 5 or 6 different PCB mixes, technical chlordane, toxaphene and 2 different individual pesticide mixes in a Pest/PCB analysis. This means 50 or more analytical runs to establish an initial calibration, and up to 10 CCVs per 10 samples.

(2) Require a full multi point for the individual pesticides, technical chlordane and toxaphene, and for 1016/1260, allow single points for the other PCBs unless there is a hit in a sample for a single point PCB, then require re-analysis on a multipoint curve.

(3) Same as #2, but allow quantitation of the PCBs from single points (except 1016/1260). Also allow just the single component pesticides, and 1016/1260 as CCVs, unless there is a hit.

He emphasized the huge difference in required effort between #1 and #3, but in his opinion not much difference in data quality. Tim said an extra option would be a single point for all multicomponent analytes unless a detect above the QL was registered. John favored option 2. Richard said for multicomponent analytes, maybe calibrate with a single point at the reporting limit and then re-calibrate if detected. Brooke said PCBs are a very poor method so are we really going to improve on it? It was agreed to discuss these options further in Sarasota.

c. Previously circulated item #7 asked why quantitation from the CCV should be allowed. Richard said, assuming an average RF calibration is being used, the question comes down to:

What is the relative amount of variability and drift in the short term (the initial calibration) vs. the long term (the ongoing sample analysis). If the long term is significantly greater (and we know it is) then quantitation based on the CCV will be overall more accurate because it reflects the response of the instrument at the time. If an

analyte has 20%D in the CCV, is it going to be more accurate to measure from the CCV or the initial calibration? The counter argument is that someone made an error in adding the CCV standard and that is why it is different from the ICAL. However, Richard thought it is much more common that the instrument has just drifted. Quantitation from the CCV could only be used for average RF calibrations, so he thought perhaps it is not worth arguing over. He would be prepared to let it drop, although he is convinced that it could (somewhat) improve accuracy of quantitation.

Tim asked if quantitation from the CCV should be allowed for all models, except methods that do not allow it (SW846). Richard said only for an average type calibration. John suggested a more accurate quantitation is obtained from the initial calibration in some cases, but in other cases quantitation from a CCV may be more accurate. Dan said the name "CCV" needs to be changed if it is to be used for quantitation, and he considered it flawed to allow a single point when multipoint calibration had been required previously. This will also be discussed further in Sarasota.

d. Nancy had previously circulated an e-mail message on multipoint calibration with replication. She suggested there should be a statement in the standard with a definition of when it is applicable, with limiting scope, and including a reference to a guidance document for minimum requirements. Standards would be prepared at a series of concentrations and analyzed in replicate at each concentration to fully characterize the dose-response relationship. This would be for a new method, a major modification of an existing prescriptive method, a major analyte, or a method using a flexible approach. Reasons would be:

- to scientifically determine the appropriate model for calibration over the range of interest;
- to evaluate the limitations of a specific model (e.g. where linear model ends);
- to determine the precision and thus the uncertainty in the calibration and calibration model;
- to select and determine confidence in one model of calibration over other models;
- to produce scientifically and legally defensible documentation of method choices as regards calibration;
- to establish a sound basis for the range, calibration model, calibrants, n, and calibration QC;
- to estimate the best-possible quantitation limit (for use in initial QL determination); and
- to estimate the best-possible detection limit (for use in initial DL determination).

Richard questioned what you are going to do with the data gathered. Tim and Anand felt it should not be a standard requirement, and maybe it could be in guidance. Richard said if it was done, it could not then be applied to all instruments in the laboratory. Tim said maybe this should just be addressed in method validation, and Richard said if that is done it should just be guidance. It was agreed that Nancy should be asked to provide more justification and more detail on what would be done when the data were gathered.

e. Item 10 concerned spacing of calibration standards. Dan had e-mailed that he agreed the standard shouldn't be prescriptive with regard to spacing. However, since the existing standard is mute on the topic, it probably doesn't get discussed during on-site assessments and may be overlooked as a means of quality improvement at some laboratories. Assessors do see data that could be better had the analyst considered spacing. He argued that most experienced analysts do have a sense of the expected range for most samples. They should, and probably do consider that information in creating the calibration. For those that don't, however, he considered it a good idea to talk to them about it and a have a standard reference to back it up, if necessary. Therefore, he advocated for a sentence or two in the standard that addresses this in a flexible and practical manner. Dan stressed it needs to be auditable, and it may be enough to just say it must be considered. He will circulate some suggested wording.

f. Item 12, intercept test, had been circulated by Francoise (Attachment 2). She said this documented what she does in her laboratory. Richard thought this might be covered in other items.

At the close of this discussion, Richard asked people to expand on their justifications, and especially anyone opposed should say why. The pros and cons will then be considered at the Sarasota meeting. Brooke asked if training in calibration should be required of analysts.

4 – Committee Appointments

This item was discussed in a closed session restricted to Committee Members.

It was proposed by Richard and seconded by Tim that Francoise Chauvin should be appointed as a Committee Member. All were in favor.

It was proposed by Richard and seconded by Anand to reappoint Dan and Tim to 3 year terms. All were in favor.

5 – Adjourment

The meeting adjourned at 3:30 pm. The next meeting will be in Sarasota.

Attachment 1

A QUANTIFICATION LIMIT CONTROL STANDARD AND CALIBRATION VERIFICATION STANDARDS IN EACH ANALYTICAL BATCH

WHO:

Laboratories environmental reporting data

WHAT/WHERE:

A requirement in the Standard for a laboratory control standard (LCS) at the lower Quantification Limit to be included in every analytical batch when data is reported, with a reference to a guidance document for minimum requirements.

A section in the guidance document describing the minimum requirements and documentation requirements.

Besides the lower QL LCS a calibration verification standards should be run periodically to verify the calibration stability across the entire quantitative range of the method. The recommendation is one verification standard every batch, cycling the concentration across the calibration range.

WHEN:

Run one QL LCS every batch, plus one calibration verification standard (at varying concentrations) every batch, whenever reporting environmental analytical data.

WHY:

To verify that a control sample spiked at a concentration equal to the quantification limit can be determined with acceptable measurement bias.

To verify that the entire concentration range of the calibration curve hss acceptable precision relative to the initial calibration.

Performance at the low end of the calibration curve has the greatest likelihood of showing deterioration in method performance and the greatest opportunity to be impacted by interferences. An LCS at the QL can serve other purposes including verification of the QL and DL, especially when the analyte is reported as ND or below the QL. Therefore it is proposed that a QL LCS (blank spike at the quantification level run through the entire method), be run in every analytical batch.

A calibration verification standard at any concentration in the laboratory's quantitative range should be acceptable for showing that the system remains in control. However it is wise to verify the entire quantification range (not just the midpoint of the calibration) with the verification standard. Therefore it is proposed that one verification standard (calibration standard only and not run through entire method) be analyzed with each batch and that the concentration of the standard is cycled from batch to batch.

Attachment 2

INTERCEPT TEST

WHO:
Accredited Laboratories

WHAT/WHERE:
Statement in the standard

- ✓ Perform an intercept test (relative bias at the Limit of Quantitation) for each new calibration:
  - Divide the y intercept by the slope of the calibration = I
- ✓ Acceptance criteria: absolute value of I to be within laboratory established acceptance criteria or client data quality objectives for allowed uncertainty at the LOQ.

Statement in the guidance document

- ✓ Perform an intercept test for each new calibration (same as in the standard): Divide the y intercept by the slope of the calibration = I
- ✓ Acceptance criteria such as: The absolute value of I should be no more than 1/3 of the minimum reporting limit (these are our current criteria).

WHEN:
For each new calibration

WHY:
To routinely verify method performance at the low end of the calibration range.

To provide more confidence in the quality of data in the vicinity of the LOQ - Impact on data collected for compliance evaluation when LOQ is a reporting limit.

Most currently approved methods impose some requirements on the overall goodness of the fit (usually the goodness of fit for the highest concentration points dominate the assessment) and performance requirements regarding precision and accuracy (usually in the middle of the calibration range). Requirements regarding sensitivity (and precision and accuracy at the low end of the calibration range) are usually not required for routine work. Though, currently, LOQ is to be verified on an annual basis, instrument drift and other possible causes may cause method performance to degrade over time.

CONTEXT:
The intercept test could also be part of the approach for improving data quality for non-detects (item # 2 in the "consolidated list of items to include in the standard").

This proposal is also related to item # 4 in the "consolidated list of items to include in the standard". There is value in both checks, as they have a different "slant":

- ✓ The result from a check at the quantitation limit in each analytical batch will be affected by the inherently lower precision in this region of the curve, and by the calibration equation used (appropriate or not at the low range).
- ✓ The intercept test allows an upfront assessment of whether this particular calibration is appropriate for concentrations at the low end of the calibration range; the test is performed prior to any time/effort being spent on sample preparation.