SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

MAY 16, 2014

The Committee held a conference call on Friday, May 16, 2014, at 2:00 pm EDT. 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)	Absent
Dan Dickinson, NYSDOH (Accreditation Body)	Present
Mandi Edwards, Envirochem (Lab)	Present
Tim Fitzpatrick, Florida DEP (Lab)	Present
JD Gentry, ESC (Lab)	Present
Nancy Grams, Advanced Earth Technologists, Inc. (Other)	Present
Anand Mudambi, USEPA (Other)	Absent
John Phillips, Ford Motor Co., (Other)	Present
Scott Siders, IL DEP (AB)	Absent
Gary Ward, OR DPH (AB)	Absent
Ken Jackson, Program Administrator	Absent

Associate Committee members present: Arthur Denny; Reed Jeffery; Dixie Marlin; Diana Shannon;

2 – Previous Minutes

It was moved by Francoise and seconded by Mandi to approve the minutes of April 25. All were in favor except Tim who abstained.

3 – Quantitation Limits

Discussed was Richard's attached draft of Section 1.5.2.2 (Limit of Quantitation) that he had circulated for comments. He said they would go through the comments in order.

In response to a question from Brooke, Richard said the LOQ is the level at which you have to qualify your results as being outside the calibration range. He said it was agreed earlier that a laboratory could choose whatever it wanted as its LOQ, but then had to verify it. It was agreed this interpretation of LOQ was consistent with the existing TNI definition of LOQ (in V1M2). Nancy suggested the first section should be parallel to the MDL language, stating what you should think about when you do your initial selection of the concentration. Nancy said precision and accuracy need to be known. Richard said he would add a section saying how a laboratory will will choose its initial LOQ, and this could include a client project requirement, some multiple of its LOD, or some signal-to-noise ratio etc.

1.5.2.2.a. Chung-Rei had asked about addressing drying and grinding (e.g., Method 8330B for soils), pointing out that the more volatile compounds will be lost during drying. Richard said it is very unusual for people to spike incremental samples before the grinding and then sub-sampling , partly because loss of analyte from a spike is more than from the native material. Nancy wondered if there should be something on matrix-based LOQ, and Richard said he would make a note of it. Richard noted that the language is already a part of the current standard. He thought they could say they do not think incremental sampling is a part of the analytical method. Dan said he would not expect a laboratory to go into that much detail, but if they had a high-efficiency extraction technique and a low-efficiency extraction technique, he would expect an LOQ for each. John suggested adding "post sub-sampling" after "method". Richard suggested adding in parentheses "including sample preservation but not sub-sampling". Nancy suggested saying you have to do the preservation and the standard is going to talk about matrix elsewhere. Also in this section, in response to a comment from Brooke, it was agreed to strike "determination" and to put in "verification".

1.5.2.2.b. The committee agreed to delete "LOQ study" and to put in "LOQ verification and LOQ". It was pointed out that some analyses (e.g., pH) do not need an LOQ.

1.5.2.2.c. In the first sentence, Nancy did not like "claimed LOQ", and suggested just saying it was the LOQ established above. Richard said the language in 1.5.2.2.c.1 was mostly taken from the MDL language. Francoise wanted to make it clear about independent preparation of the 7 QC samples, and John suggested "separately prepare and analyze". Richard said he would keep the language similar to the MDL language. In 1.5.2.2.c.1.(ii) it was questioned what should be done if an LOD is not determined. It was agreed to strike the language "or two times the MDL if a LOD is not determined".

Referring to 1.5.2.2.c.1.(iii), Richard said his laboratories would have quite a few of the better-performing analytes failing this criterion. For spikes at the LOQ, for some analytes the calculated limits of mean +/- 3 SD were more than 20% wider than the LCS limits. Nancy was concerned if a laboratory generated its own LCS control limits, then adding 20% would mean a laboratory with wide LCS limits would have very wide limits while a laboratory with tighter LCS limits would be penalized. She suggested having a different criterion for this first section than the on-going section. A laboratory may not have run the method before and hence may not have the LCS data. She said this section needed to talk about the method LCS criterion, and then what criterion a laboratory should use if there was no method LCS criterion. She added the on-going section should then talk about the laboratory's own LCS criterion and not the method criterion. Nancy thought for initial verification, all analytes should meet the 20% limits, but the method criterion should apply for LCS since laboratory LCS data may not exist. She added for the method LCS data all 7 samples would have to satisfy LCS +/- 20% (except the LCS cannot go below zero). Tim suggested telling the laboratory they have to define limits for their LCS if not mandated by the method, but they must use mandated LCS limits if they are in the method . Nancy asked how to instruct a laboratory to set its LCS control limits if it has never used the method before. Richard said for such a case he would add language based on demonstration of capability in Section 1.6.2.2 .d. (i.e., use what is in the method or generate your own if it is not. Dan thought, for initial verification, there should be a statement on precision and bias. He said if it does not meet a certain RSD or recovery then it is not a quantitative analyte, and it might then be possible to de-link the LCS and drop the LCS +/- 20% criterion. As a solution to the problem of analytes with different performances, Arthur suggested limits such as 10% RSD for ICP/ICPMS, 20% RSD for volatiles, and 30% RSD for semi-volatiles. Nancy added that state or client criteria should be added on top of this. It was also

pointed out that a comprehensive list of limits in excess of the examples given by Arthur would be needed. Since time was running out, Richard stopped the discussion and asked the call participants to let him know what they thought the acceptance criteria for the initial LOQ study should be. Then on the next call they would go over the suggested options.

4 – August meeting in Washington DC

Richard reminded Committee Members to let Ken and Jerry know if they would need travel assistance. It was suggested the assigned half-day session would not be enough and Richard said he would ask Jerry for an additional half-day session.

5 – Next Call

The next call was scheduled for Friday, May 30 from 2:00-3:30 P.M. Eastern time.

6 – Adjournment

The call was adjourned at 3:30 pm Eastern

1.5.2.2 Limit of Quantitation (LOQ)[CBF1]

a) All sample-processing [CRM2]and analysis steps of the analytical method shall be included in the determination [CBF3]of the LOQ.

b) The LOQ study[CBF4] is not required for any component or property for which spiking solutions or quality control samples are not available or otherwise inappropriate (e.g., pH).

c) The validity of the LOQ shall be verified. At a minimum verification includes:

1) Initial Verification[CRM5]

Prepare and analyze a minimum of 7 QC samples containing the analytes of concern at or below the claimed LOQ[1], spread amongst a minimum of three separate preparation and analysis batches. If the same LOQ will be used for multiple instruments, a minimum of 2 replicates must be analyzed on each instrument. The quantitation limit is verified if the following criteria are met:

i) All results must meet the qualitative identification criteria in the method (for example, recognizable spectra, signal to noise, and presence of qualifier ions).

ii) All results must exceed the LOD, if a LOD has been determined, or two times the MDL if a LOD is not determined[CRM6]

iii) For each analyte, 90%[CBF7] of the recoveries of the initial LOQ verification must be within the LCS control limits, widened by +/- 20%. (For example, if the LCS limits are 70 – 130%, the LOQ limits are 50 – 150%).[CBF8] If the lower LCS control limit is 20% or less, the analyte is considered non-quantitative and must be qualified as estimated value.

iv) If the criteria for the initial verification of the LOQ are not met, correct any problems and repeat the test and/or repeat the test at a higher spiking level (which will result in a higher LOQ).[CBF9]

2) Continuing verification

Prepare and analyze a minimum of two QC samples spiked at or below the LOQ [CBF10]on each instrument during any quarter in which samples are being analyzed.

At least once per year evaluate the results of the ongoing verification samples[2]. To increase the statistical power of the precision and bias estimates (2iv and 2v below), include all of the previous 2 years LOQ verification sample results along with the new LOQ verification results. Include the initial LOQ QC samples if within two years.[CBF11] The annual verification is successful if:

i) All results must meet the qualitative identification criteria in the method (for example, recognizable spectra, signal to noise, presence of qualifier ions).

ii) At least 80% of results exceed the LOD, if a LOD has been determined, or two times the LOD if a LOD is not determined

iii) For each analyte, 80% of the recoveries of the continuing LOQ verification must be within the LCS control limits, widened by +/- 20%. (For example, if the LCS control limits are 70 – 130%, the LOQ limits are 50 – 150%). If the lower LCS limit is 20% or less, the analyte is considered non-quantitative and must be qualified as estimated value.[CBF12]

iv) Generate a statement of precision and bias for each analyte based on the LOQ spikes, expressed as mean recovery +/- 3 times the standard deviation of the results.

v) Calculate confidence intervals for the mean and standard deviation.[CRM13]

vi) The statement of precision and bias of the LOQ, and their confidence intervals, must be available to clients and regulatory agencies upon request.

vii) If the annual verification is not successful, i.e., one or more of the criteria are not met, then required corrective action includes correction of sample preparation and/or instrument determination problems followed by a new initial LOQ verification, or increasing the LOQ followed by a new LOQ determination and initial LOQ verification. All affected clients between the failed continuing LOQ verification and the last passed continuing LOQ verification or the initial LOQ verification must be notified the problem and potential adversed impacts on data quality and usability.

d)

The LOQ shall be verified annually for each quality system matrix, technology, and analyte.

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[1] If a LOD has not been determined, the LOQ must be at least 2 times higher than the MDL

[2] If gross failures in the LOQ verification samples are observed, for example, failure to qualitatively detect the analytes, corrective action must be taken immediately. Corrective action may include correction of instrument determination or sample preparation problems followed by analysis of at least two LOQ level QC samples or increasing the LOQ and repeating the initial LOQ verification.