SUMMARY OF THE
TNI CHEMISTRY EXPERT COMMITTEE MEETING

MAY 30, 2014

The Committee held a conference call on Friday, May 30, 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)	Present
Dan Dickinson, NYSDOH (Accreditation Body)	Present
Mandi Edwards, Envirochem (Lab)	Present
Tim Fitzpatrick, Florida DEP (Lab)	Absent
JD Gentry, ESC (Lab)	Present
Nancy Grams, Advanced Earth Technologists, Inc. (Other)	Present
Anand Mudambi, USEPA (Other)	Present
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; Gail Warren.

2 – Quantitation Limits

As requested by Richard during the previous call, Committee members had sent in quantitation limit proposals. Richard asked each person in turn to address their proposals.

Dan Dickinson. *"The limit for quantitation is 34% RSD. Any analyte with greater than 34% RSD at the level spiked is non-quantitative. Lab shall generate a statement of precision and bias for each analyte based on the LOQ spikes."* Dan stressed there must be no limits at the end of the study that go below zero. He did not include anything on recovery limits. He said "all results must exceed the LOD" is as much as we can say about recovery, because there are so many methods and biases out there that give different recoveries, and many analytes would fail. There may be cases where 34% RSD is very difficult to achieve, so the laboratory would need a higher reporting level, or decide that analyte is not quantitative. He added that many analytes that are not detected are routinely non-detects anyway. Dan also considered it important to have an initial precision and bias in order to tie into the next section (1.5.3) on the evaluation of precision and bias. In response to a question from Nancy, Dan confirmed the RSD would be calculated without rejection of outliers.

John Phillips. *"The limits for quantitation are 30% RSD and 50-150% recovery (average?)."* In addressing both precision and bias (recovery), John reminded everyone there is no recovery correction on environmental analytical data, so data are always biased unless good-performing methods are available. He presented a series of graphs showing the true limit of detection falls around 34% RSD. Richard added if the LOQ must be at least the true limit of detection, it must be at least 34% RSD, but laboratoriess will generally choose it to be higher than that. John showed good performance is about 90-110 % recovery and 10% RSD. He showed mathematically that quantitation could be expressed as <30% RSD and 50-130% recovery. Nancy asked what should be done if an analyte is a poor performer that fails these criteria. John said it should be flagged as an estimate if a number is reported or it should be reported as non-quantifiable.

Nancy Grams. *"Calculate the confidence interval for results from quantitation limit spikes. Limits still to be discussed, but would be something like: The 95% confidence interval must be within the range 10%-199% recovery. Some allowance for multi-analyte methods (ef 90% within the single analyte limit, somewhat wider limit for the other 10%). Failing analytes are identified as poor performers or LOQ repeated at higher level."* Nancy sought advice from technical experts, bearing in mind that she wanted the minimum requirements of what quantitation should be. Her approach was the use of significant digits as the baseline for quantitation. Detection could be defined as the point of zero significant digits. Quantitation is the point at which you have at least one sure significant digit; e.g., for a value of 56, the 5 digit always appears when replicates are run. If the second digit appears in the range of 4 – 8, this is said to be a part of a significant digit. Nancy said 5% RSD would be needed to be sure of 1 full significant digit (i.e., quantifiable), but most analytical measurements are at slightly less than 1 full significant digit down to zero significant digits at the LOD. A value of 50% RSD would equate to zero significant digits. Based on this she presented statistical calculations to support her recommendation.

Francoise Chauvin. ***"****Fixed limits for accuracy (eg 50-150%) and precision. Analyte/method combinations that do not meet the limits are considered non-quantitative."* Francoise said she had a much simpler proposal reflecting her strong preference to report all data with uncertainty. Data users may not be familiar with categories of compounds that are poor performers. Therefore, a mechanism is needed to alert the data user that the compound has a very high RSD or low recovery. She believed LOQ should have fixed limits and not limits based on RSD only.

Mandi Edwards. *"LOQ spike results must be within the range detected to 150% recovery."* She agreed fixed limits are needed, and said she had nothing to add over what John had done.

Tim Fitzpatrick. *"LOQ spikes must have greater than zero recovery."* Tim was unable to be on the call, but he had sent an e-mail supporting Dan's position.

JD Gentry. *"Most analytes mass 50-150% for average recovery, but some poor performers need 40-160%. Some very poor performers (mostly in the semivolatiles MS methods) need 10-200% and even that may be too tight for one or two analytes."* He presented his approach from a laboratory perspective, saying his laboratory had this criterion in place for about 18 months. The Department of Defense

requires a laboratory to evaluate its LOQ data even if they do not have set criteria for it. Going to 40-160% recovery covers 96-98% of the compounds. JD was concerned with moving below 50%, since certain compounds perform lower, and there would be a concern that some laboratories might not inform clients of poor performers.

Richard opened the discussion of these proposals by showing Department of Defense LCS recovery results (mean and standard deviation). Method 8270 showed, for mid-level spikes, that for most analytes continuous liquid-liquid extraction brings them within the sort of limits people had been suggesting. However, separatory funnel preparation resulted in low recoveries for phenolics. From the presentations discussed on this conference call, he said a consensus was emerging that the precision limit can be in the 30% range. As long as labs are using reasonable levels for their LOQs not much would fail that. He said a 50% recovery criterion makes him more nervous, because some very common analyte/method combinations would not meet that criterion. Nancy said there is no scientific foundation for specifically requiring (say) 50% recovery. The approach she described allows a low recovery but there is a bound on how wide the confidence interval can be. It does not say a laboratory cannot have 10 or 12% recovery, but it must have to meet some criteria that say it can actually get there. She added as long as you have good precision you can manage bias. Richard pointed to Dan's presentation, saying an option is not to have a recovery limit, but just have a limit on precision in combination with the other requirement that on a sample-by-sample basis you have to get results that are above your MDL. Dan added at the end of the day the laboratory would need to calculate its confidence interval for those data. Richard said, when thinking about the implementation phase the 30 or 34% RSD requirement can be scientifically defended. He added it can also be said you are actually detecting anything you are saying is quantifiable. This will include results that are not very good, but it should be remembered it is the lower bound and most results going to be better than that. He emphasized this would be something that could be implemented as scientifically valid, making it more likely to be acceptable.

In closing, Richard asked everyone to consider if they had been swayed by any discussion, and to decide which proposal they were leaning towards. This would form the basis of a discussion during the next conference call on June 13.

3 – Adjournment

The meeting was adjourned at 3:30 pm EDT.