

**Summary of Technical Assistance  
Detection and Quantitation Guidance Document Sub-Committee Meeting  
September 4, 2009**

1. Roll Call: Subcommittee chair, Richard Burrows called the meeting to order at 1:00 PM EDT. Subcommittee members present included Richard Burrows, Brooke Connor, Prabha Acharya, and Rock Vitale. Carol Batterton was present as TNI staff support.
2. Approval of minutes: Minutes from the last meeting (8-21-09) were reviewed. The minutes were approved by acclamation.
3. Homework: Richard Burrows had prepared the suggested acceptance criteria below for ICP and ICP/MS. Richard asked for discussion on these proposals:

Options for acceptance criteria for the LOD spike for methods such as ICP and ICPMS

1. **Result is  $\geq$  mean of method blanks**

Pros – Simple, and easy to pass

Cons – Half of the analytes would pass this criterion for an individual sample even if the analyst forgot to spike the sample – i.e. ran a method blank by mistake. This does not seem reasonable.

- Requires lab to collect their method blank data and calculate the average

The consensus of those on the call was that this was not a good option.

2. **Result is  $\geq$  mean of method blanks + Students t times standard deviation of method blanks**

Pros – Technically rigorous, ensures that the result is above the distribution of the method blanks, i.e. is capable of being distinguished from a blank.

- Consistent with DoD (DoD uses 3 standard deviations, which is Student's t for approx 7 or 8 replicates)

Cons – Requires that lab collect their method blank results and calculate the average and standard deviation

- If the lab is not adjusting their LOD for method blank results, then the LOD may be so low that a standard to give a result above the method blank distribution might be outside of the 1-4X NELAC allowed spiking concentration. This could also be considered a pro, since if that is the case the LOD is much lower than can be justified.

Comments on this option included:

The criteria must show that there is a reasonable belief that the analyte is in the sample.

Negative values should be omitted. The consequence of doing this is a higher mean and lower standard deviation.

If negative values are excluded, distribution could be a lower number.

Consensus of those present was that this option is a good approach.

3. **Three times the noise level**

Pros – Sounds simple, at least

Cons – No straightforward way to measure the signal to noise for these instruments

Comments on this option included:

This option sounds simple, but really is not.

No way to measure the noise level.

No support for this approach.

Consensus option for acceptance criteria for the LOD verification spike for methods such as GC/MS

Results from spikes at the LOD must meet qualitative identification criteria for mass spectrometry. A good indication that qualitative identification criteria are met is that the normal lab processing used for routine analysis works, i.e. all analytes are detected without employing extra steps. The qualifier ions normally used for compound identification should be present. Note that for analytes that exhibit generally low recovery, spiking towards the upper end of the acceptable range (1-4X the LOD) is advisable. Since some GC/MS analytes exhibit low recovery, the results from some of the LOD verification spikes may be below the LOD.

Options for acceptance criteria for the LOD verification spike for general chemistry methods based on colorimetry or spectrophotometry

Richard asked for suggestions for options for general chemistry colorimetric methods. Suggestions and comments included:

The analyst should get a result with the spike that is greater than the LOD.

Spike at 2-3X the LOD, if you see above the LOD, then it is good verification for colorimetric methods.

Are titrations different?

Options for acceptance criteria for the LOD verification spike for GC and LC methods

Suggestions and options for these methods included:

These are multi-component analyses, but there is not fundamental difference from the above. Would be a calibrated number.

If running two columns, both would need to return a result > than the LOD.

How many times would it have to be run? If only once, what happens if the it fails?

Should suggest corrective action failure in the guidance. Could say to run spike 3-5 X

5. Next Meeting: The subcommittee will meet via teleconference on Friday, September 17, 2009, at 1:00 PM ET. The call in number and agenda will be sent prior to the call.

Home work: Richard asked Brooke Connor to incorporate what the subcommittee had discussed so far in to her PowerPoint presentation for the next meeting. Also, Richard asked each member to review the NELAC standard for LOQ and be prepared to provide thoughts about approaches for LOQ verification.