

Facilitation Team's Suggested Questions for Verification Discussion

Definitions

Verification:

Validation:

Confirmation:

Do we need verification? Initial and/or Ongoing?

If verification is needed, why? What are the rationales on why we need verification?

What components/key aspects on verification do we want to see included?

Authorship and Discussion by: **David Kimbrough**

Verification & Remediation

The FACDQ has proposed the establishment of nationally promulgated QLs and DLs that would be required for reporting purposes by permittees to CWA primacy programs. The FACDQ has also proposed that permittees be required to use laboratories that verify that they are capable of producing results that meet certain MQOs at the QL and DL. For the discussion it is important to distinguish between a laboratory's observed QL and DL from the estimated QL and DL. Many statistical procedures are used to estimate at what concentration a particular analytical method will have a QL or DL (e.g. the USEPA's MDL procedure). These procedures for estimating QLs and DLs are used because actually determining the observed QL and DL is often impractical. The observed DL would be a concentration at which the MQO for unspiked blanks is met, such as 1% false positive rate. An observed QL would be a concentration at which spiked blanks will have a reported value plus or minus some percentage of the spiked value or a %RSD less than some value. One of the FACDQ's tasks is to recommend a specific procedure that individual laboratories can use to estimate their own laboratory specific DL and / or QL. However, these estimates may or may not accurately predict the actual concentration where a particular observed QL or DL occurs. Further, laboratory conditions often change quickly and an estimated QL or DL, even if accurate at the time of determination, may soon become inaccurate.

Currently USEPA methods may require that the Method Detection Limit (MDL) be determined only at the time of the Initial Demonstration of Capability (IDC), or in a few cases on an annual or semi-annual basis. However, the MDL is estimated in a procedure that usually occurs on a single day separate from routine analysis. Several of the proposed alternative procedures to replace the MDL use data collected from routine analysis to provide an on-going estimate of the DL or QL to incorporate changing conditions. However, this does not address whether the estimated DL or QL is within some acceptable range of the observed DL or QL.

Verification could then have two elements: one is the determination that the estimated DL and QL are less than the nationally promulgated QLs and DLs, and the other that the estimated DL and QL are in fact acceptable estimates of the observed DL and QL. The first is quite simple and straight-forward. The second is less immediately obvious. There are several policy options that need to be determined.

- 1) Will the QL and DL both be verified, or just one or the other?
- 2) What will be the frequency of verification? It could be done:
 - a. On a time based approach (daily, weekly, monthly, quarterly, annually)
 - b. On a certain number of batches (every batch, every 5th batch, etc).
 - c. After a certain number of samples (After every 20th or 100th or some other number irrespective of the number batches or time period).
 - d. Based on instrument run time (after some many hours of operation, irrespective of amount of time since the last determination, how many samples or batches were run).

- 3) Will verification be determined using spiked blanks, unspiked blanks, or something else?
- 4) Examples for the DL might include, running an unspiked blank at a fixed frequency:
 - a. The false positive could be determined on an on-going basis. It would need to be less than a targeted rate (e.g. 0, 0.5, 1.0, 2.0, percent)
 - b. Value of the unspiked blank must be less than the DL.
- 5) Examples for the QL might include:
 - a. Running a spiked blank with a concentration less than or equal to the QL at a fixed frequency.
 - b. The measured value could be within a fixed percentage of the spiked value on a batch-by-batch basis.
 - c. The running average of the last number of spiked blanks could be within a fixed percentage of the spiked value.
 - d. The target value could be based on the running average of the last number of spiked blanks rather than the spiked value.
 - e. The acceptance limits could be three standard deviations of the average rather than a fixed percentage.
- 6) What happens if verification fails? This really depends of the frequency of verification and the form. If no spiked or unspiked blanks are run in a batch, no remediation is possible. These are just a few possibilities.
 - a. Batch by Batch Verification.
 - i. Unspiked blank has a value greater than QL? No results may be reported from that batch above the QL.
 - ii. Unspiked blank has a value greater than DL but less than QL? No results may be reported from that batch as DNQ.
 - iii. Spiked blank at the QL gets a recovery that is too low. No results may be reported from that batch as ND or <QL.
 - b. The FP rate of unspiked blanks is too high? Raise the DL, if it does not exceed the nationally promulgated. Are corrective actions required to lower the DL? What if the FP is zero?
 - c. Average spiked blank recovery is too high or too low? Raise the QL , if it does not exceed the nationally promulgated QL.

<u>Procedure</u>	<u>Verification</u>	<u>Test</u>
Detection Limits		
USEPA MDL	None	
ACIL Uncensored	Annual Re-Estimation based on LRBs	Annual F-test of variance of old % RSD of Old LRBs vs. New LRBs
ACIL Censored	None	
CG	Determination of 1% FPR	Check LRB FPR < 2%
EBMUD	Batch by batch	FNQC +/-50%
Hubaux-Vos	None	
ASTM IDE	None	
Quantitation Limits		
USEPA ML	None	
ACIL Uncensored	Quarterly Analysis of LFB	Annual Determination of %RSD of LFBs, if >20%
ACIL Censored	Quarterly Analysis by LFB and	Presence / absence of LFB.
CG	Batch by batch	Mean spike recovery +/-50%
LC-MRL	Batch by batch	MRL Check +/-50%
ASTM IQE	None	

NOTE: On-going re-estimation and verification can be added to any of these procedures.