

Puget Sound Naval Shipyard Test Laboratory F608

See attached file.

WAC 173-50-040 definitions

“Laboratory Control Sample” or “LCS” - An aliquot of analyte-free water or analyte-free solid (e.g., Ottawa sand, anhydrous sodium sulfate, or other purified solid) to which known amounts of the method analytes are added.

This definition of a LCS is far too limited. Positive or bias process control samples can also be (are preferred to be) matrix-matched or related reference materials, either certified or not. (Control samples are not used to establish result traceability therefore they need not be certified.)

“Limit of Quantitation” or “LOQ” - The lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve.

For many processes, LOQs cannot be based solely on a low calibration point, especially for indirect measurement processes where samples are transformed (digested or extracted) prior to measurement. Detection and quantitation limits are established based on sensitivity studies which help estimate the lowest concentration where measurement quantitative results can be reported with some level of certainty.

Where samples are transformed prior to measurement, the LOQ has to be verified by running a LOQ verification sample through the entire process of digestion/extraction and measurement. The measurement process is only half the process. The LOQ for the process is established at a level that can support the uncertainty of the entire process. Merely using the low calibration point to establish the LOQ is erroneous.

LOQs also have to produce a quantitative result with known precision and bias.

Suggest incorporating part of the DOD QSM definition:

“Limits of Quantitation (LOQ) (Clarification): The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.”

*“For DoD/DOE, at a minimum, the LOQ shall be verified quarterly. However, not all possible combinations of preparation and cleanup techniques are required to have LOQ verifications. If LOQ verifications are not performed on all combinations, the laboratory must base the LOQ verifications on the worst case **basis (preparation method with all applicable cleanup steps).**”*

“Proficiency testing (PT)” - Evaluation of the results from the analysis of samples, the true values of which are known to the supplier of the samples but unknown to the laboratory conducting the analyses. PT samples are provided by a source external to the environmental laboratory.

Include that PTs providers are to be ISO/IEC 17043 accredited.

"Quality control (QC)" - The routine application of statistically based procedures to evaluate and control the accuracy of analytical results.

Quality control is process control. QC measurements do not "control the accuracy of analytical results". QC measurements are made to ensure that process bias and/or precision is in statistical control. Results are not controlled. Processes are controlled. Quality assurance programs help ensure the accuracy of analytical results.

WAC 173-50-061 Required QC

It seems that a better place to document this section, containing very specific technical requirements, would be the WA DOE accreditation program, Publication No. 10-03-048, and not publish as part of the State law.

(3) Each calibration point must have its value recalculated against the calibration curve. Unless specified in the method, each calibration point must have its percent error meet the calibration verification acceptance limits from the method; with the exception of the lowest point which is 50-150%.

Specifying that every calibration point must have its percent error calculated does not allow this to be applied universally. Suggest using the TNI EL-V1M4-2017-Rev2.2 requirement that requires percent recovery calculated at two values; One near the mid-point and one at the sample reporting level.

In addition, some analytical measurement techniques use an average response factors (e.g., PCBs) for which a relative standard error, not percent (relative) error, is calculated. Need to accommodate both error calculations.

Suggest using the National Environmental Laboratories standard, TNI EL-V1M4-2017-Rev2.2, section 1.7.1.1(k)

- k) the laboratory shall use and document a measure of relative error in the calibration;*
- i. for calibrations evaluated using an average response factor, the determination of the relative standard deviation (RSD) is the measure of the relative error;*
 - ii. for calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory shall evaluate relative error by either:*
 - a. measurement of the Relative Error (%RE)*

This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level. The Relative Error at both of these levels shall meet the criteria specified in the method. If no criterion for the lowest calibration level is specified in the method, the criterion and the procedure for deriving the criterion shall be specified in the laboratory SOP.

or,

- b. measurement of the Relative Standard Error (%RSE)*

The RSE shall meet the criterion specified in the method. If no criterion is specified in the method, the maximum allowable RSE shall be numerically identical to the requirement for RSD in the method. If there is no specification for RSE or RSD in the method, then the RSE shall be specified in the laboratory SOP.

WAC 173-55-069 Data and record traceability

(d) Document that all temperature-based equipment such as a refrigerator, oven, or incubator is both within control and checked manually as required by the relevant method;
(2) When records are hand-written, they must be in indelible ink and comply with the relevant method requirements. Incubator temperatures must be handwritten and include the date and time of reading, temperature, and technician's initials. (3) When records are kept electronically, they must be populated at the time of record, using a fully traceable and secure format. Use of continuous data-loggers is not an acceptable substitute for method-required temperature checks.

Automatic data logging is preferred to produce records of test environmental conditions including temperature. Manual recording is not preferred and seems dated. There must be outdated method requirements to manual record? Regardless, again, something so specific seems to be better documented in Publication No. 10-03-048, not State law.

WAC 173-50-070 Proficiency Testing

(7) When two approved PT providers are available for a parameter, the laboratory must analyze and pass a PT to gain or maintain accreditation.

Two acceptable PT samples from an approved (17043 accredited) prior to, and to maintain accreditation = yes. Intent of what is written is not understood.

WAC 173-50-080 On-site Audit

The laboratory must undergo an on-site audit by the department to assess critical elements and areas of recommended practices.

Third-party accreditation by an ISO 17011 accreditation provider should not require on-site assessment by WA DOE. Save resources. Onsite should be required only for direct accreditation by WA DOE. This aligns with WAC-173-170 (5).

WAC 173-5-090 Evaluation and Issuance of certificate

- Assess the importance of each finding; and

Is the intent that "finding" is a "negative finding"; a deficiency? Findings can be positive, neutral or negative. "Deficiency" is a negative finding and would match language in (2.a) and WAC 173-50-110 sections.