Auditing Essential Quality Control Elements LCS/MS

THE NELAC INSTITUTE
ASSESSMENT FORUM
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What's in a Name?

Laboratory Control Sample (LCS) –NELAC & OSW (SW-846)

Laboratory Fortified Blank (LFB)- EPA Methods mainly DW

Blank Spike (BS) – Labs

QC Check Sample – Labs and WW (600 methods).

Control Sample - Labs

During an assessment it can take about 15-30 min. alone to figure out the definitions used in the lab!

TNI Draft Interim Standard

V1 M4 (Chemistry)1.7.3.2.1

Purpose: The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps.

Any affected samples associated with an "out of control" LCS shall be reprocessed for re-analysis or the results reported with appropriate data qualifying codes.

TNI Draft Interim Standard

V1 M4 (Chemistry)1.7.3.2.2

Frequency: The LCS shall be analyzed at a minimum of 1 per preparation batch.

Except for analytes that can't be spiked (pH, odor, etc.)

If no separate preparation method (e.g. volatiles in water) the batch is defined as environmental samples analyzed together with the same method, personnel, same lots of reagents, ≤20 enviro samples, not including batch QC samples (MB, LCS/LCSD, MS/MSD).

TNI Draft Interim Standard

V1 M4 (Chemistry)1.7.3.2.3

Matrix spike may be used in place of LCS control as long as the acceptance criteria are as stringent as LCS.

LCS may consist of a media containing known and verified concentrations of analytes or as Certified Reference Material (CRM)

TNI Draft Interim Standards

V1 M4 (Chemistry) 1.7.3.2.3

Spiked Analytes: Components to be spiked shall be as specified by the mandated test method or other regulatory requirement (SDW/CWA) or as requested by the client.

For interfering analytes (e.g. toxaphene, PCBS) the spike shall be chosen that represents the chemistries and elution patterns of the components to be reported.

If not specified …

- i. For methods with 1-10 targets, spike all
- ii. For methods with 11-20 targets, spike at least 10 or 80%, whichever is greater
- iii. For methods with >20 targets, spike at least 16

All targeted components in the spike mixture over a 2-year period.

Matrix Spike

V1 M4 (Chemistry)1.7.3.3.1

Matrix Spike; Matrix Spike Duplicates

- Matrix-specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method.

- The information from these controls is sample/matrix specific and would not normally be used to determine the validity of the entire batch.

- Frequency and Spiking Components same as LCS.

Corrective Action “out of control” LCS

Samples considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.

This includes any allowable marginal exceedance as described in:

V1 M4 (Chemistry)1.7.4.2

- i. When the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes. Otherwise the samples affected shall be reprocessed and re-analyzed; or

- ii. when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. Otherwise the samples affected by the unacceptable positive control shall be reprocessed and reanalyzed.

Marginal Exceedences for LCS

V1 M4 (Chemistry) 1.7.4.2.b

Allowable ME. If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control, therefore corrective action may not be necessary. Data still reported with appropriate data qualifying codes.

# of Analytes in LCS	Allowed as ME
> 90	5
71– 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

Spike Analytes Dilemma

Dilemma: A subcontracted lab uses a short list in spiking a Solid Waste GCMS method while the original lab spikes all compounds;

Possible Solutions:

Some OSW methods do allow spiking with a short list, however,

a) if the samples are suspected to contain certain analytes, then they should be included in the mix.

b) A subcontracted laboratory should be instructed by the original lab or data user as to what analytes are to be spiked.

DW and CWA methods do require spiking LCS/MS with all reported compounds.

EPA DW Labcert Manual 5th Edition

Dilemma: Chapter IV, Section 7.2.12 states that "Laboratories should run a LFB at their MRL every analysis day" (Section 7.2.6 states to run it routinely at the MRL).

What is the acceptance criteria for an LFB at the MRL?

Possible Solutions:

a) Specify the LFB as an MRL LFB and establish historical limits or commonly seen default limit of ± 50% recovery.

b) Or maintain the method specified recovery criteria if the analyte at the laboratory's MRL can be easily recovered.

Second Source Requirement for the LCS/MS

Dilemma: SM 20th ed., 1020B states to analyze an externally supplied standard and it is sometimes specified in the SW-846 methods that the LCS/MS be a second source, while DW methods often specify the QCS (second source check) to be analyzed when a new calibration curve is generated or at least quarterly.

Possible Solutions:

a) It is often noted that laboratories will use a second source for the LCS/MS, but not recommended by the accrediting authorities unless the method specifies it.

The purpose of the LCS/MS is not to verify the curve, but rather the preparation process, the second source should be only analyzed after calibration or daily if lab chooses to.

b) Either way, the laboratory would have to meet the LCS criteria whether it is second source or not.

MS vs. MSD Recovery

Dilemma: The MS recovery is out of the method acceptance criteria, but the MSD is acceptable and the RSD is also within the acceptance criteria. Is the batch acceptable based on the MSD recovery?

Possible Solutions:

a) The recovery of the MSD should also be reported, and picking and choosing should not be made to make QC criteria acceptable.

b) The laboratory should be qualifying the original sample as not having met the MS recovery criteria for one of the duplicates, but could specify that the LCS recovery and precision was met, indicating a possible matrix interference.

MS/MSD Frequency

Dilemma: The method specifies an MS at a 10% frequency of samples and duplicates are either not specified or at a 5% frequency of samples (often seen in DW methods). Can an MS/MSD at a 5% frequency satisfy the 10% analysis frequency?

Possible Solutions:

a) It is often noted that laboratories elect to run an MS/MSD in lieu of a sample/sample dup so a precision measurement can be calculated from a detected value (SW-846, 8000C, 9.5).

b) If the lab specifies in their SOP or QAP that they are running an MS/MSD at a 5% frequency rather than a 10% frequency, then the recovery would have to be calculated for both MS and MSD and qualified if either is out.

Matrix Spike Frequency

Dilemma: The method specifies an MS at a set frequency of samples but the sampler would be required to provide the extra sample due to full volume preparation (e.g. 1664), which they do not do.

Possible Solutions:

a) The laboratory can show the documentation proving that they have contacted the client to meet the QC requirements

b) The lab can qualify the data as not meeting the QC frequency criteria due sample submittal limitations.

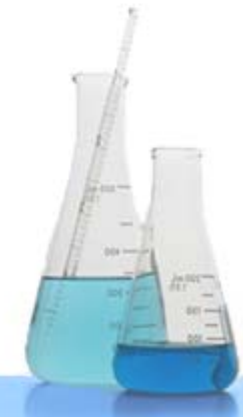
Typical Analytical Run

- Initial Calibration (frequency per method)
  - QCS – second source check (recommend daily)
  - ICB
  - ICV – same source
  - MRL check
  - 9 field samples
  - LRB
  - LCS (LFB) preferably same source as Cal standards
  - Original unspiked sample
  - MS (LFM) preferably same source as LCS
  - MSD (LFMD)
  - CCV – same source
  - And so on……

References

- TNI Draft Interim Version of the Chemical Testing Requirements (Module 4) June 15, 2007
- EPA Manual for the Certification of Laboratories Analyzing Drinking Water, 5th ed.
- OSW SW-846 Chapter One, Revision 1
- Standard Methods, 20th ed., SM1020B

- http://www.epa.gov/sw-846/faqs_qc.htm



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