EL-V1M4-2009

ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 4: Quality Systems for Chemical Testing

Voting Draft Standard
December 2011

P.O. Box 2439
Weatherford, TX 76086
817-598-1624
www.nelac-institute.org

This page intentionally left blank.

PREFACE

This Standard is the result of many hours of effort by those volunteers on The NELAC Institute (TNI) Quality Systems Committee. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the voting process.

This Standard supplements Module 2, Quality Systems General Requirements, and may be used by any organization that wishes to implement a program for the accreditation of environmental laboratories.

This page intentionally left blank.

VOLUME 1, MODULE 4

Quality Systems for Chemical Testing

Table of Contents

This page intentionally left blank.

VOLUME 1, MODULE 4

Quality Systems for Chemical Testing

1.0 CHEMICAL TESTING

1.3.1 Additional Terms and Definitions

~~**Physical Parameter:** a measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical or biological components.~~

Reserved

1.3.2 Exclusions and Exceptions

Reserved

1.4 Method Selection

Refer to Volume 1 Module 2 Sections 5.4.2, 5.4.3 and 5.4.4.

~~A reference method is a method issued by an organization generally recognized as competent to do so. (When ISO refers to a standard method, that term is equivalent to reference method). When a laboratory is required to analyze a parameter by a specified method due to a regulatory requirement, the parameter/method combination is recognized as a reference method.~~

~~If there is not a regulatory requirement for the parameter/method combination, the parameter/ method combination need not be validated under 1.5.1b) as a non-reference method if it can be analyzed by another similar reference method of the same matrix and technology.~~ The inclusion of the ~~parameter~~analyte in the method shall meet all required calibration requirements and the quality control requirements of the method to which the ~~parameter~~analyte is being added. If no QC exists in the method, the laboratory shall adhere to the requirements outlined in ~~the~~ a ~~similar~~ reference method of the same technology (when available). For example, when adding acetone to Method 624, the calibration and QC requirements shall follow Method 624. A method that meets ~~these above~~ requirements shall be identified in such a way so that there is no confusion that the method analyte list has been modified.

~~When it is necessary to use methods not covered by reference methods, these shall be subject to agreement with the client and shall include a clear specification of the client's requirements and the purpose of the environmental test. The method developed shall have been validated appropriately before use.~~

1.5 Method Validation

1.5.1 Validation of Methods

Prior to acceptance and institution of any method for which data will be reported, all methods shall be validated.

~~a) The laboratory shall validate reference methods via the procedures specified in Sections 1.5.12 and 1.5.2Refer to Volume 1 Module 2, Section 5.4.5.~~

a) The laboratory shall validate reference methods via the procedures specified in Sections 1.5.2 and 1.5.3. For reference methods, the procedures outlined in 1.6 can satisfy the requirements of 1.5.23.

Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Double underline, Highlight
Formatted: Indent: Left: 0.63"
Formatted: Double underline
Formatted: Highlight
Formatted: Double strikethrough, Highlight
Formatted: Highlight

b) For all methods, except reference methods, the validation must comply with Volume 1, Module 2, Sections 5.4.5.1, 5.4.5.2, and 5.4.5.3. This validation must include the minimum requirements outlined in Sections 1.5.2, 1.5.3 and 1.5.4. of this module.

~~The laboratory shall validate non-reference methods, laboratory-designed/developed methods, reference methods used outside their published scope, and amplifications and modifications of reference methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application. The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use. In the absence of other specifications, the minimum requirements for method validation are given in Sections 1.5.2, 1.5.3 and 1.5.4.~~

1.5.2 Limit of Detection and Limit of Quantitation (However Named)

Procedures used for determining limits of detection and quantitation shall be documented. Documentation shall include the quality system matrix type. All supporting data shall be retained.

1.5.2.1 Limit of Detection (LOD)

If the laboratory is not reporting a value below the Limit of Quantitation, a Limit of Detection study is not required, unless specified by the method.

An LOD study is not required for physical parameters, for any analyte for which spiking solutions are not practicable or for any method that does not use a calibration curve (e.g., residues, specific conductance, chlorophyll, or titrimetric determinations ~~etc.~~).

The laboratory shall utilize a method that provides an LOD that is appropriate and relevant for the intended use of the data. If a mandated method or regulation includes ~~protocols~~ procedures for determining detection limits, these shall be followed. The laboratory shall document how LODs were derived from the determinations. If the protocol for determining the LOD is not specified, the selection of the procedure shall reflect instrument limitations and the intended application of the method.

All sample-processing and analysis steps of the analytical method shall be included in the determination or validation of the LOD.

a) When required, the laboratory shall determine or verify the LOD for the method for each target analyte of concern in the quality system matrices.

b) The LOD shall be initially determined for the analytes of interest in each method in a quality system matrix in which there are neither target analytes nor interferences at a concentration that would impact the results or the LOD shall be performed in the quality system matrix of interest.

c) An LOD study shall be performed each time there is a change in the method that affects how the test is performed, ~~or when~~ a change in ~~instrumentation occurs that affects the~~ instrument sensitivity ~~of the analysis~~ or a change in instrument.

d) The LOD, if required, shall be verified annually for each quality system matrix, technology, and analyte.

e) The validity of the LOD shall be verified by analyzing a QC sample prepared in the quality system matrix and containing the analyte of interest at a concentration of no more than 3x the LOD for single analyte tests and 4X the LOD for multiple analyte tests. The LOD is confirmed by either i) detection of an instrument signal greater than 3x the instrument noise level or ii) a response that is distinguishable from the blank. The LOD verification must meet all qualitative identification criteria appropriate for the method, such as

Formatted: Highlight
Formatted: AAA-Level2, Indent: Left: 0", First line: 0", Tab stops: 0.63", Left + Not at 0.63"
Formatted: Highlight
Formatted: Indent: Left: 0", Hanging: 0.63"
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Not Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Not Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Normal, Don't adjust space between Latin and Asian text, Don't adjust space between Asian text and numbers, Tab stops: Not at 0.63" + 1.38" + 1.75"
Formatted: Highlight
Formatted: Font: (Default) Arial, 10 pt, Highlight

ion ratios for GC/MS and peak pattern for PCB. This verification shall be performed on every instrument that is to be used for analysis of samples and reporting of data. The validity of the LOD shall be verified as part of the LOD determination process. ~~The validity of the LOD shall be verified by detection (a value above zero) of the analyte(s) in a QC sample in each quality system matrix. This QC sample shall contain the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests. This verification shall be performed on every instrument that is to be used for analysis of samples and reporting of data. The validity of the LOD shall be verified as part of the LOD determination process. This verification shall be done prior to the use of the LOD for the sample analysis.~~

~~c) An LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature.~~

~~d~~c) ~~The LOD shall be initially determined for the compound~~analytes of interest ~~in each method in a quality system matrix in which there are neither target analytes nor interferences at a concentration that would impact the results or the LOD shall be performed in the quality system matrix of interest.~~

~~e~~d) ~~An LOD shall be performed each time there is a change in the method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.~~

~~f~~e) ~~The LOD, if required, shall be verified annually for each quality system matrix, technology, and analyte.~~

1.5.2.2 Limit of Quantitation (LOQ)

The LOQ must be ~~established~~ for each analyte in a reported test. A determination of an LOQ is not required for physical parameters, for any ~~component~~ analyte for which spiking solutions are not available or for any test that does not use a calibration curve (e.g., ~~residues,~~ specific conductance, chlorophyll, ~~or~~ titrimetric determinations~~, etc.~~). While an LOQ determination may not be required, some methods or regulations require reporting to a specific level or restrict reporting values below a certain level (e.g., BOD and residues).

~~When required, t~~The laboratory shall ~~establish~~ the LOQ by:

- using test conditions or instrument restrictions (e.g., sample volume, accuracy of balance, method QC requirements) or
- by a study using spiked samples (when required). ~~If spiking samples is not an option, or the laboratory shall determine an appropriate LOQ~~ or ~~,~~ as the basis

a) All sample-processing and analysis steps of the analytical method shall be included in the determination of the LOQ.

~~b) The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not available or otherwise inappropriate (e.g., pH).~~

~~e~~b) The LOQ shall be verified annually for each quality system matrix, technology, and analyte. Such verification shall be performed on every instrument that is to be used for analysis of samples and reporting of data unless ~~However, the annual LOQ verification is not required if~~ the LOD was determined or verified annually on that instrument.

c) The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory-established method acceptance criteria or client data quality objectives for accuracy.

d~~e~~) When an LOD is determined or verified by the laboratory, the LOQ shall be above the LOD.

Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Indent: Left: 0", Hanging: 0.63"
Formatted: Strikethrough, Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Indent: Left: 0.75", Bulleted + Level: 1 + Aligned at: 0.28" + Indent at: 0.53", Tab stops: 1", Left
Formatted: Not Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Double underline
Formatted: Highlight
Formatted: Highlight

~~e)~~ ~~The LOQ shall be verified annually for each quality system matrix, technology, and analyte. However, the annual LOQ verification is not required if the LOD was determined or verified annually on that instrument.~~

1

1.6 Demonstration of Capability (DOC)

1.6.1 General

a) ~~Prior to~~ An individual who performs any activity involved with preparation and/or analysis of samples must have constant, close supervision until ~~acceptance and institution of any method for which data will be reported,~~ a satisfactory initial DOC is ~~required~~ completed (see Section 1.6.2).

b) Thereafter, ongoing DOC (Section 1.6.3), as per the quality control requirements in Section 1.7.3 (such as laboratory control samples) is required.

c) In cases where ~~a laboratory analyzes~~an individual has prepared and/or analyzed samples using a method that has been in use by the laboratory for at least one year prior to applying for accreditation, and there have been no significant changes in instrument type, ~~personnel~~ or method, the ongoing DOC shall be acceptable as an initial DOC. The laboratory shall have records on file to demonstrate that an initial DOC is not required.

~~For the initial DOC, appropriate records as discussed in Section 1.6.2 shall be completed.~~

~~An initial DOC shall be completed each time there is a change in instrument type, personnel, or method.~~

d) All demonstrations shall be documented. All data applicable to the demonstration shall be retained and readily available at the laboratory.

1.6.2 Initial DOC

An individual must successfully perform A~~a~~n initial DOC ~~shall be conducted~~ prior to using any method (see 1.6.1 a) above), and ~~at~~ any time there is a change in instrument type, ~~personnel~~ or method or any time that a method has not been performed by the ~~laboratory or~~ analyst in a twelve (12) month period.

1.6.2.1 The laboratory shall document each initial DOC in a manner such that the following information is readily available for each affected employee:

c) analyte(s), class of analyte(s)~~, or measured parameter(s);~~ ;

1.6.2.2 If the method or regulation does not specify an initial DOC, the following procedure is acceptable. It is the responsibility of the laboratory to document that other approaches to initial DOC are adequate.

d) Compare the information from (c) above to the corresponding acceptance criteria for precision and accuracy in the method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all ~~parameter~~analytes meet the acceptance criteria, the analysis of actual samples may begin. If any one of the ~~parameter~~analytes does not meet the acceptance criteria, the performance is unacceptable for that ~~parameter~~analyte.

Formatted: Highlight
Formatted: Tab stops: 1", Left
Formatted: Highlight
Formatted: Highlight
Formatted: Tab stops: 1", Left
Formatted: Tab stops: 1", Left
Formatted: Highlight
Formatted: Highlight
Formatted: Tab stops: 1", Left
Formatted: Highlight
Formatted: Tab stops: 1", Left
Formatted: Tab stops: 1", Left
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight

e) When one or more of the tested ~~parameter~~analytes fail at least one of the acceptance criteria, the analyst shall proceed according to i) or ii) below.

i. Locate and correct the source of the problem and repeat the test for all ~~parameter~~analytes of interest beginning with b) above.

ii. Beginning with b) above, repeat the test for all ~~parameter~~analytes that failed to meet criteria.

f) Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all ~~compound~~analytes of interest beginning with b).

1.6.3 Ongoing DOC

1.6.3.1 The laboratory shall have a documented procedure describing ongoing DOC that includes how the laboratory intends to identify data associated with ongoing DOCs. The analyst(s) shall demonstrate on-going capability by routinely meeting the quality control requirements of the method, laboratory SOP, client specifications, and/or this Standard. If the method has not been performed by the analyst in a twelve (12) month period, an Initial DOC (1.6.2) shall be performed. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.

1.6.3.2 This on-going demonstration may be one of the following:

a) acceptable performance of a blind sample (single blind to the analyst) ;

~~Note: S~~or successful analysis of a blind performance sample on a similar method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624 or 5030/8260); ~~uccessful analysis of a blind performance sample on a similar method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624 or 5030/8260) would only require documentation for one of the tests.~~

d) a documented process of reviewing QC samples performed by an analyst or groups of analysts relative to the quality control requirements of the method, laboratory SOP, client specifications, and/or this Standard. ~~review using QC samples. QC samples can be reviewed~~ This review can be used to identify patterns for individuals or groups of analysts and determine if corrective action or retraining is necessary;

1.7 Technical Requirements

1.7.2 Continuing Calibration

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification.

b) Calibration shall be verified for each ~~compound~~analyte, element, or other discrete chemical species, except for multi-component analytes such as aroclors, chlordane, total petroleum hydrocarbons, or toxaphene, where a representative chemical, related substance or mixture can be used.

e) Criteria for the acceptance of a continuing instrument calibration verification shall be established. If the continuing instrument calibration verification results obtained are outside the established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, ~~and analysis of a second consecutive (immediate)~~

Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Highlight
Formatted: Font: (Default) Arial, 10 pt
Formatted: Font: (Default) Arial, 10 pt, Highlight

~~calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. T~~the laboratory ~~shall~~ must demonstrate acceptable performance after additional corrective action measures with two consecutive calibration verifications, or a new initial instrument calibration shall be performed. If the laboratory has not verified calibration, sample analyses may not occur until the analytical system is calibrated or calibration verified. If samples are analyzed using a system on which the calibration has not yet been verified the results shall be flagged. Data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

Formatted: Font: (Default) Arial, 10 pt

1.7.5 Sample Handling

b) The laboratory shall implement procedures for checking sample preservation using readily available techniques, such as pH or chlorine, prior to or during sample preparation or analysis. An exception is allowed for volatile organic ~~compound~~analyte analyses; chemical preservation may be checked after analysis.