DOC 101 – Reading Between the Lines

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Demystifying the DOC

- Current NELAC 2003 Standard
- TNI 2009 Standard (changes only)
- Clarifications – TNI Working Draft Standard

- DOC
- Continuing/Ongoing DOC
- Method Selection
- Method Validation

Definitions

- Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)
- Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte-and matrix-specific and may be laboratory-dependent.
- Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.
- Quality Control Sample: a sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking.

Requirements

- Scattered throughout the Standard
  - 5.4.12.2.5.4 b) Administrative Records
  - 5.5.2.6 Personnel
  - 5.5.4.2 Selection of Methods
  - 5.5.4.2.2 Demonstration of Capability
  - 5.5.4.5 Validation of Methods
  - Appendix C Demonstration of Capability
    - DOC
    - Initial Test Method Evaluation
  - Appendix D
    - D.1.1.3 (Chemistry) Sample Specific Controls
    - D.2.1 (Toxicity) Positive and Negative Controls
    - D.3.3 (Microbiology) Method Evaluation
    - D.4.3 (Radiochemistry) Method Evaluation
    - D.5.3 & D.5.4 (Air) Method Evaluation & Limit of Detection
    - D.6.4 (Asbestos) Method Evaluation

Method Selection

- Method must meet the client needs must be appropriate for the environmental tests
- Use requested or mandatory methods
- Use methods in international, regional or national standards
  - Use the latest valid edition of a standard unless it is not appropriate or possible to do so.
- Client must be informed about method selection
  - Inform the client when the method proposed by the client is considered to be inappropriate or out of date.

Records Requirements

- Must be
  - Retained for each analyst
  - In personnel records
    - Continuing in Training Files
- Must use the form in Appendix C
- Allows use of Work Cells
  - Records must be in files of each analyst involved with the DOC/Continuing DOC

DOC

- Perform before Using Method in Laboratory
  - Excepted
    - Methods in use before July 1999 and
    - No significant change in instrument type, personnel or method
    - Continuing DOC must be on file
- Technical Personnel must have DOCs for all activities
- Repeat DOC if there is a change in instrument type, personnel or method.

Procedure

- Use Appendix C1 when required by method
  - For all analytes including additions
- Other approaches are acceptable if "adequate"
  - Toxicity discusses procedure
- Sources
  - QC sample from outside source
  - Standards that are prepared independently of the calibration standards
- All failed analytes must be repeated

Continuing DOC Perform Annually

- Acceptable performance of a blind sample
  - May be applied to similar test method using the sample technology
- An initial measurement system evaluation
- Another DOC
- At least 4 consecutive LCSs w/ acceptable precision and accuracy
- When all else fails
  - Analyze authentic samples
  - Results statistically indistinguishable from those of another trained analyst

Validation

- Non-standard methods, laboratory-designed/developed methods, standard methods used outside their published scope, and amplifications and modifications of standard methods

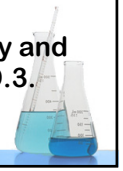
Vs

- Standard Methods

Validation

- Must be evaluated before using the method:
- LOD*
- LOQ*
- Precision and Bias
- Selectivity

* LOD and LOQ not required for Microbiology and Toxicity – Procedures outlined in D.2 and D.3.



LOD

- Applies to all analytes
- Must include all sample processing step
- Confirmed by qualitative ID of analyte(s) in QS Matrix (concentration restrictions)
- Performed on each instrument used for analysis
- Not required if
  - Spiking or QC samples are unavailable
  - or
  - Test Results are not reported to LOD
- Verify Annually

LOQ

- Applies to all analytes
- Confirm by successful analysis of analytes at concentration of 1-2 times LOQ
- Must relate to LOD
- Must be above the LOD
- Verify annually for each quality system matrix, method and analyte
  - Not required if the LOD is reevaluated or verified

Selectivity

- Use all checks specified in method
  - Mass Spec Tuning
  - 2nd Colum Confirmation
  - Inter-element Interference Checks
  - Retention time windows,
  - Blanks
  - Spectrochemical absorption or fluorescence profiles
  - Responses factors
  - Etc.

Weaknesses

- Written by chemists for chemists
- Confusion on whether a DOC applied to lab or analyst
- Muddled and outdated version of ISO 17025.
- Hard to find all the laboratory requirements

ENVIRONMENTAL
LABORATORY SECTOR
EL-V2-2009

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Methods & Method Validation

- Module 2, Section 5.4
  - 5.4.1 General
  - 5.4.2 Selection of Methods
  - 5.4.3 Laboratory Developed Methods
  - ~~5.4.4 Non-Standard Methods~~
  - ~~5.4.5 Method Validation~~

ISO 17025 language

- Modules 3-6
  - 1.4 Method Selection: ISO 5.4.4 plus
  - 1.5 Method Validation: ISO 5.4.5 plus
- Module 7 (WET) uses simplified language

V1M2-M6

IDOC & Continuing DOC

- Modules 3-7
  - 1.61 – IDOC
  - 1.62 – Continuing DOC

V1M3-M6

- 5.4.12.2.5.4 b) Administrative Records
- 5.5.2.6 Personnel
- 5.5.4.2 Selection of Methods
- 5.5.4.2.2 Demonstration of Capability
- 5.5.4.5 Validation of Methods
- Appendix C Demonstration of Capability
  - DOC
  - Initial Test Method Evaluation
- Appendix D
  - D.1.1.3 (Chemistry) Sample Specific Controls
  - D.2.1 (Toxicity) Positive and Negative Controls
  - D.3.3 (Microbiology) Method Evaluation
  - D.4.3 (Radiochemistry) Method Evaluation
  - D.5.3 & D.5.4 (Air) Method Evaluation & Limit of Detection
  - D.6.4 (Asbestos) Method Evaluation

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Technical Modules KEY ELEMENTS

Consistent in all Technical Modules

- 1.4 Method Selection
- 1.5 Method Validation
- 1.6 Demonstration of Capability
- 1.7 Technical Requirements
  - Calibration
  - Quality Control
  - Data Acceptance/Rejection
  - Sample Handling

V1M3-M7

Where's Air? (D.5)

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It's in V1M4 Chemical

- EL-V1M2-ISO-2009
  - Quality Systems General Requirements
- EL-V1M3-2009
  - Quality Systems for Asbestos Testing (D.6)
- EL-V1M4-2009
  - Quality Systems for Chemical Testing (D.1)
- EL-V1M5-2009
  - Quality Systems for Microbiological Testing (D.3)
- EL-V1M6-2009
  - Quality Systems for Radiochemical Testing (D.4)
- EL-V1M7-2009
  - Quality Systems for Toxicity Testing (D.2)

Demonstration of Capability

- NELAC 5.5.2.6 and Appendix C
- Not in Module 2
- DOC is contained in Modules 3-7 and varies based on the scientific discipline
- Note: Work Cells eliminated entirely

V1M2

Reference Method

- A reference method is a method issued by an organization generally recognized as competent to do so.
- 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.
- ISO "standard method" = TNI "reference method"

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1.4 Method Selection

- Language from ISO 5.4.4 (use of non-standard methods) with some twists

Module 2, Section 5.4
5.4.1 General
5.4.2 Selection of Methods

V1M3-M6

ISO AND TNI

ISO 5.4.4

TNI 1.4

- 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….

V1M4

1.4 Method Selection Adding Analytes

- Becomes a reference method if:
  - Analyte/Method is identified in a different reference method
  - Reference method is applicable to same matrix and technology

V1M3-M6

1.4 Adding Analytes

- Parameter must meet all QC requirements in method
- If no QC in method, must meet QC in "the similar" method
- Method must be identified as modified

V1M3-M6

Adding Analytes – QC Requirements - Acetone

624	8260
ICAL RSD: 35%	ICAL RSD: 30%
CCAL:	CCAL: 20%
MIN RF:	MIN RF: 0.1
BFB Recovery:	BFB Recovery: 86-118

So, if you follow the QC requirements of Method 624 and 8260, then Acetone by 624 can be considered a Reference Method

Remember to report 624 as a modified method

1.4 Non-Reference Methods

ISO 5.4.4 (not in 2009)

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

TNI 1.4

- 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

- Retains 2 sections from ISO 17025
  - Definition of validation (except M4)
  - All methods require some level of validation (except M7)
- Adds language about PT samples
- Does not contain ISO section on assessing data for intended use
- Each module has additional details on validation

V1M3-M7

Validating Reference Methods

ISO 17025

TNI 1.5.1

- LOD, LOQ & Precision & Bias for Chemistry*
- MDA & Precision & Bias for Radiochemistry*
- Asbestos and Micro – implied IDOC

*Precision & Bias could be determined by an IDOC

V1M4

Non-Reference Methods

- Includes:
  - non-reference methods
  - laboratory-designed/developed methods
  - reference methods used outside their published scope
  - amplifications and modifications of reference methods to confirm that the methods are fit for the intended use.

Validating Non-Reference Methods

ISO 5.4.5.2

- The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use....

TNI 1.5.1

- b) 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....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.

V1M4

1.5 Method Validation

- Evaluation of:
  - 1.5.2 LOD (if reporting to LOD) and LOQ
  - 1.5.3 Precision and bias
  - 1.5.4 Selectivity (not required for reference methods)

V1M4

1.5.2 limit of Detection

- Combination of NELAC C.3.1 and D.1.2.1
- Differences:
  - Qualitative replace by “detection”

V1M4

1.5.2 Limit of Quantitation

- Combination of NELAC C.3.2 and D.1.2.2
- Differences:
  - Removed: "must have procedures to relate LOD to LOQ"

V1M4

1.5.3 Precision & Bias

Reference Methods

- Initial DOC, or
- Alternate procedure

Non-Reference Methods

- Evaluate precision and bias across the analytical range
  - e.g., Triplicates analyzed at multiple concentrations
  - EPA Tier 1, 2, or 3 ATP procedure
- Same as NELAC C.3.3(b)

V1M4

ISO AND TNI

ISO 5.4.5.3

- The range and accuracy of the values obtainable from validated methods (e.g. detection limit, selectivity, linearity, limit of repeatability, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample), as assessed for the intended use, shall be relevant to the customers' needs.

TNI ???

Demonstration of Capability (DOC)

nelac

- 5.5.2.6 "demonstrated capability"
- 5.5.2.6 "continued proficiency"
- 5.5.4.2.2 "demonstration of method capability
- 5.5.4.2.2 "continuing demonstration of method performance"
- Appendix C "DOC"

TNI

- 1.6.2 Initial DOC
- 1.6.3 Continuing DOC

Demonstration of Capability: A procedure to establish the ability of the **analyst** to generate analytical results of acceptable accuracy and precision.

LCAT

1.6.1 DOC: General

- Initial DOC is required for all methods and analysts
  - Except for methods in place for one year before applying for accreditation
- Initial DOC required if change in instrument, method or personnel
- Ongoing DOC is required
- Retain records

V1M4

1.6.2 Initial DOC

- Prior to using method
- Change in instrument type, personnel or method
- If method not performed by an analyst within 12 months

V1M4

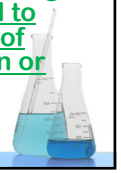
1.6.2 Initial DOC

- 4 replicates is one option but not required
- Form in NELAC Appendix C deleted but requirements for documentation remain:
  - a) analyst(s);
  - b) matrix;
  - c) analyte(s);
  - d) identification of method(s) performed;
  - e) identification of laboratory-specific SOP;
  - f) date(s) of analysis; and
  - g) summary of analyses.
- Not required to be in personnel file

V1M4

1.6.3 On-Going DOC

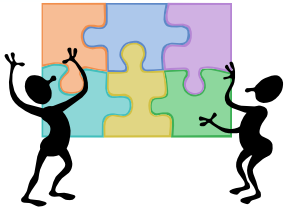
- Requirements are the same
- Options from NELAC 5.5.2.6 still allowed:
  - Single-blind sample
  - Initial DOC
  - 4 LCSs
  - Real-world sample analysis modified to require predefined acceptance criteria (not statistically indistinguishable)
- Another option added:
  - A documented process of analyst review using QC samples. QC samples can be reviewed to identify patterns for individuals or groups of analysts and determine if corrective action or retraining is necessary.



V1M4

LOOKING AHEAD – WORKING DRAFT STANDARD

SIMPLIFY AND CONSOLIDATE



EL-V1M2

- Define:
  - Reference Method
    - Exclude from Method Validation
  - Analyte
  - Parameter
- Insert ISO Language
  - 5.4.4 Non-Standard Methods
  - 5.4.5 Method Validation

EL-V1M3-7

- 1.4 – Method Selection
  - Refer to V1M2, Sections 5.4.2, 5.4.3 and 5.4.4
- 1.5 – Method Validation
  - Reference method validation is DOC (additional requirements for Chemistry and Radiochemistry)
  - Non-Reference Methods
    - Refer to V1M2 Sections 5.4.5

Simply Said:

- Reference Method Validation is a DOC (+ minimal requirements)
- Non-Reference Method Validation is more extensive

Standards for:

- •Method Validation
- •DOC
- •IDOC

PERFORMING DOCS

nelac DOCs

- Initial DOC: Classic 4 replicates
- Continuing DOC
  - Acceptable performance of a blind sample (single blind to the analyst). Applicable to other tests using the same technology.
  - An initial measurement system evaluation or another demonstration of capability
  - At least four consecutive laboratory control samples with acceptable levels of precision and accuracy.
  - Analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

WHAT DO YOU DO?

IDOC

- Dependent on Technology
  - Asbestos – Classic 4
  - Chemistry – Classic 4
  - Microbiology – Classic 4 with different calculations
  - Radiochemistry – Classic 4
  - Toxicity – Run test; QC must meet test methods

Ongoing DOC Options

	Asb	Chem	Micro	Rad	Tox
Acceptable performance of a blind sample	X	X	X	X	
Another initial DOC	X	X		X	X
At least four (4) consecutive laboratory control samples with acceptable levels of precision and accuracy	X	X	X	X	
A documented process of analyst review using quality control (QC) samples.	X	X	X	X	X
Analysis of sample fortified with known quantity of organism			X		
Analysis of one sample in duplicate			X		
Analysis of real-world samples with results within predefined acceptance criteria	X	X	X		X

DO THE NEW OPTIONS PROVIDE FLEXIBILITY?
WILL YOU USE THEM?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Thank You!
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