Relevance of Measurement Traceability Standards to Environmental Testing Laboratories

(including ISO/IEC 17025, Clauses 5.6.1-5.6.2)

Florida HEALTH

ISO/IEC 17025

- “General requirements for the performance of calibration and testing laboratories”

- Are there requirements for calibration laboratories that are not applicable to environmental testing laboratories?

Clause 5.6.1

- Calibrate all equipment for tests that have a significant impact on the accuracy or validity of the test results
- Establish program for calibrating the equipment
  - Measurement standards
  - Reference materials used as measurement standards
  - Measuring and test equipment used to perform tests

Clause 5.6.2

- Clause 5.6.2.1 for Calibration Laboratories
- Clause 5.6.2.2 for Testing Laboratories

Clause 5.6.2.2

- Ensure that the equipment can provide the measurement uncertainty needed
- Design and operate a program such that measurements are traceable to national standards
- When traceability to SI not possible, establish traceability to certified reference materials, agreed methods, and/or consensus standards

Why did NELAC leave these out?

- In a word --- **UNCERTAINTY**
  - What was required?
  - When is it applicable?
  - How to calculate?
- Clients do not want it, or do not care, so why do I need to worry about uncertainty?

Was that really a good idea?

- ISO/IEC 17025 has gone through revisions to make the relevant requirements for calibration laboratories more palatable to testing laboratories
- NELAC emphasis is on ESTIMATING uncertainty where needed
  - At least have a procedure when such estimates are requested
  - But take into account all components of the measurement system

Other Accreditation Systems Use Clauses 5.6.1-5.6.2

- Department of Defence QSM
- NEFAP for FSMOs
- Signatories to the ILAC Mutual Recognition Arrangement
  - EA, APLAC, and IAAC as recognized regions
- Everyone else?

Responsibilities

- Users of ISO/IEC 17025 should use the entire standard
  - Shouldn't pick and choose
  - TNI obligations to the national member body
    - ANSI for the United States
- It is permissible to add sector-specific standards to ISO/IEC 17025, as NELAC did for environmental testing

Incentives

- Inclusion of clauses 5.6.1-5.6.2 can benefit The NELAC Institute's NELAP program
- Non-governmental accreditation bodies will almost certainly require these clauses for their accredited conformity assessment bodies (laboratories)

Vulnerabilities

- Environmental Laboratory Sector technical modules have calibration requirements

- Can these standards alone establish traceability?
- Even so, are those standards applicable to all accredited and pending fields of testing?

Vulnerabilities (continued)

- If calibration standards do not apply to everyone (e.g., titrations, residues), then is it okay to say clauses 5.6.1-5.6.2 are not applicable to a specific laboratory function, as long as it is true and it is documented?
  - It may not be true. These clauses might apply in at least some aspect of each field of accreditation.

Paint Filter Liquids

- EPA Method 9095 in the SW-846 manual
- “Free Liquids” required in EPA RCRA regulations
  - 40 CFR Parts 264.314 and 265.314
- NO measurement and test equipment required
  - Calibration requirements not applicable
- NO clear “conventional” QC requirements specified
  - What is the “spike-able” analyte?

So what NELAC standards apply?

- Initial and continuing demonstrations of capability
  - May take some creativity, as long as the procedure is documented
- Constant and consistent test conditions
  - Details nebulous in the current standard
- Method “validation”?
- SOP contents

- ISO/IEC 17025 clauses 5.6.1 and 5.6.2.2 may be the most applicable standards to the correct performance of EPA 9095
  - Correct filter mesh size
  - Controlled time period for the test
  - Specified uncertainty for yes / no results

Flash Point

- Test determines whether “waste” is a hazardous waste (Ignitability)
  - 40 CFR Part 261.21
- EPA Method 1010
  - Points to ASTM D93
- Critical points
  - Pensky-Martin closed cup tester
  - Sample filled to mark on the cup
  - Constant stirring
  - Consistent introduction of test flame
  - Accurate thermometer

What are the applicable NELAC standards?

- SOP? IDOC/CDOC? QC? Calibration?
- Is the thermometer no longer just support equipment and is now a bona-fide test instrument?
- What is the test result?
  - Waste is ignitable? (yes/no)
  - The actual flash point temperature?
- ISO/IEC 17025 clauses 5.6.1-5.6.2.2 are the most applicable here as well.

Odor

- US EPA drinking water secondary inorganic contaminant
- Recommended method: SM 2150 B
- Only equipment is water-bath that maintains the test temperature, plus the thermometer to measure that temperature
- Actual measurement system: panel of laboratory analysts sniffing the sample dilutions

Applicable Requirements

- Calibration of thermometer (and sample dilution devices)
- Method blank – per NELAC
- Sample duplicate – per SM 2000-series
- Demonstration of capability – also needs a documented, creative approach
- How to control uncertainty?
  - Have more analysts available to sniff aliquots
  - Eliminate confounding variables (e.g., smokers)

TCLP and SPLP

- TCLP used to determine whether a waste is hazardous (by characteristic of Toxicity)
- 40 CFR Part 261 (RCRA)
- EPA Method 1311 required (TCLP)
  - EPA Method 1312 used for SPLP
- Specific analytes extracted from waste sample into a method-defined leachate solution under controlled conditions
  - Other “appropriate” methods used to prepare and analyze the leachate

TCLP and SPLP

- Most specified quality control requirements found in subsequent digestion, extraction, and analytical methods, not in EPA 1311 and 1312
- Successful performance of TCLP and SPLP: Follow Methods 1311 and 1312 as written
- Critical elements:
  - Composition and pH of leaching fluid
  - Tumbler rotation rate
  - Leaching period
  - Temperature during leaching
  - No leaks in Zero-Headspace Extraction vessels
  - Ratio of leaching fluid volume to sample extracted

Essential Quality Control Standards

- Positive and negative Controls
  - Not clearly defined in EPA 9095, 1010, 1311 and 1312 (except for ZHEs)
  - Method blank possible for SM 2150 B
- Repeatability and/or variability
  - Not specified in EPA 9095, 1010, 1311 and 1312 but possible to perform
  - Duplicate on 10% of samples or batch, per SM2020B
- Accuracy
  - Not specified for any of these methods, and may not be possible or feasible

Essential Quality Control (continued)

- Test method capability and range of applicability
  - Limits of Detection, Limits of Quantitation and linearity evaluations not applicable to these methods
- Appropriate Formulas
  - Raw data is the final result for EPA 9095 and 1010
  - Threshold Odor Number for SM 2150 B
  - EPA 1311 and 1312 by themselves do not produce final results
- Selectivity
  - Not applicable or possible for any of these methods

Essential Quality Control (continued)

- Reagents and standards
  - None specified for EPA 9095 and SM 2150 B
  - Standard not specified for EPA 1010 but possible to use an appropriate one (if the lab chooses to do so)
  - Extraction fluids critical for EPA 1311 and 1312
- Constant and consistent test conditions
  - Critical for EPA 9095, 1010, 1311, 1312 and SM 2150 B
  - Thus, what detailed written protocols are needed?

Measurement Traceability Standards

- Calibrate all equipment for tests and subsidiary measurements (and have established programs for this calibration)
  - Thermometers for EPA 1010, 1311, 1312 and SM 2150 B
  - pH meters for EPA 1311 and 1312
  - Test periods for EPA 9095, 1311 and 1312
- Equipment provides the uncertainty of measurement needed
  - Filter mesh size for EPA 9095
  - Tumblers and ZHEs for EPA 1311 and 1312
  - Functional equipment (+ constant stirring) for EPA 1010
  - Trained, capable analysts for SM 2150 B (and all these other methods as well)

Questions?

Thank you for your attention!

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