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The NELAC Institute Environmental Laboratory Sector - Management and Technical Requirements for Laboratories Module 6: Quality Systems for Radiochemical Testing

Changes from 2009/2012 Standard

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Background

- The laboratory community is familiar with "stable" chemistry.
  - Environmental testing for inorganic and organic, and radiochemical parameters, have evolved separately
  - As a result, some protocols and concepts are less applicable to radiochemistry.

Regulatory Background

- Only one formalized program
  - EPA Laboratory Certification for Water
    - Narrow and specific set of parameters for water (SDWA and CWA)
    - Methods based on 1950s -1980s EPA lab procedures
      - Expanded to include DOE, USGS, ASTM and SM
    - Technology has changed
    - Instruments and even reagents not always available
    - Little or no performance/validation data
    - Peculiar biases specific to SDWA and CWA compliance
    - Quality requirements sparse *(to put it politely…)*

What are Some Key Differences?

- Isotopic measurements of radionuclides
  - Chemical separations with yield tracers
    - Minimizes bias – except gross alpha/beta, total U, and tritium
- Lab-developed, performance-based methods
  - SDWA/CWA compliance testing is the exception.
- Measurements relative to background
- Reported
  - "As measured" - positive, negative or zero
  - Not censored against IDL, MDL, RL!
- The measurement uncertainty is calculated and reported together with each result

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Multi-Agency Radiological Laboratory Analytical Protocols Manual

FDA USGS NIST

July 2004

"MARLAP"

- Cross-agency document developed and approved by eight federal agencies:
  - EPA, DOD, DOE, DHS, NRC, FDA, USGS, NIST
    - Part I for project planners / managers of radioanalytical projects
    - Parts II and III address technical topics

Very worthwhile !!!

Radiochemistry Expert Committee (REC)

- Formed in 2012
  - Consists of 10 radiochemists
  - Extensive experience in operation and oversight of state, federal, and commercial laboratories
  - Reviewed and updated TNI Standard Module 6:
    - Working Draft Standard posted for comment last summer
    - "Modified WDS" posted for comment in December 2014.
  - The Voting Draft Standard was successfully balloted in April 2015.

Terms and Definitions

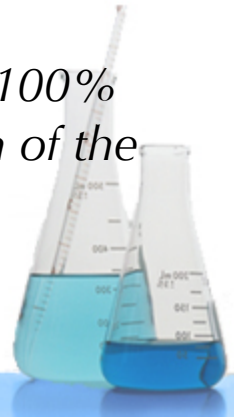
Definitions specific to Module 6 add clarity

For example:

critical value: *value to which a measurement result is compared to make a detection decision (also known as critical level or decision level)*

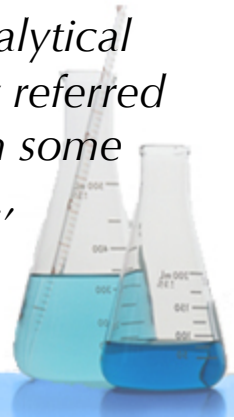
Note: The critical value is designed to give a specified low probability, α, of false detection in an analyte-free sample, which implies that a result that exceeds the critical value gives high confidence $(1 - \alpha)$ that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.

detection limit (DL) for Safe Drinking Water Act (SDWA) compliance:
Laboratories that analyze drinking-water compliance samples for SDWA must use methods that provide sufficient detection capability to meet the detection limit (DL) requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25(c) as the radionuclide concentration which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).



Uncertainty is Clearly Defined

- **Measurement Uncertainty:** *parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.*
- **Standard Uncertainty**: *an estimate of the measurement uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).*
- **Expanded Uncertainty**: *the product of the standard uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand. (c.f. Standard Uncertainty)*
- **Counting Uncertainty**: *the component of measurement uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts) (after MARLAP). Older references sometimes refer to this parameter as Counting Error or Count Error. (c.f., Total Uncertainty).*
- **Total Uncertainty**: *an estimate of the measurement uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar terms. (c.f., Counting Uncertainty).*



Exclusions and Exceptions

- Module 6 is applicable to measurements used to monitor radioactivity or determine compliance with regulations pertaining to radioactivity.
- The laboratory may fall back on Module 4 specifications when technique-specific requirements or QA/QC are not addressed by Module 6.
  - For example, calibrations, calibration verifications, and detection statistic determinations, and method-specific quality control for (radio)isotopic determinations by ICP-MS.

Validation of Methods

- Method performance data for all methods must be characterized and published in the lab SOP (e.g., scope)
  - May be drawn from published validation data, historical QC results, or method validation;
  - Must address detection capability, precision, bias, measurement uncertainty, and selectivity *(consistent with published guidelines such as MARLAP, FEM, EUROCHEM)*
- Validation must span range of sample activity expected, including zero activity

Demonstration of Capability (DOC)

- Analyze four samples ***and four blanks***
  - Blanks added since results are not censored
    - "Absolute bias" (i.e., bias at zero activity can compromise low-activity results)
- Otherwise, this section is essentially unchanged

Technical Requirements

Reorganized and clarified to address the calibration life-cycle

1) Set-up of instrumentation (1.7.1.1)

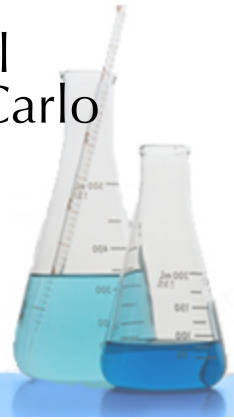
2) Initial calibration for method (1.7.1.2)

3) Calibration verification - true verification of method-specific calibrations (1.7.1.3)

4) Establish and perform instrument QC (1.7.1.4) (eliminates misleading term "CCV")

(see ASTM D7282-Standard Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements)

- The section reiterates the need for physical calibration of instruments against traceable reference materials but opens the door to and addresses requirements for validating mathematical or statistical *corrections* based on mathematical techniques such as Monte Carlo simulations.



Changes in Instrument Performance Checks

- Semiconductor gamma-ray detectors
  - Twice weekly for continuously operating detector (consistent with ANSI N42.15);
  - Day of use for non-continuously operating detector;
- Solid-state scintillation detectors used for non-spectrometric measurements (e.g., silicon detectors):
  - Day of use
- Exceptions added to facilitate long sample counts (similar to current) and radiation measurement batches
  - Requires bracketing counts on manual counters;
  - Limits run duration to 7 days on automated sample changers

Changes with Background Measurements

- Previous requirements confused *contamination control* with *control of backgrounds*
  - Provided sparse guidance to labs and auditors
  - Effective oversight difficult
  - Bias at low activities
  - Elevated rates of detection decision errors common
- The update differentiates between:
  - subtraction backgrounds;
  - short-term background checks
  - contamination controls
- The update
  - specifies frequencies and functional requirements
  - recognizes approaches for determining and controlling backgrounds *already in use at labs*

Radiation Measurements Batch (RMB)

- Preparation Batch still applicable to almost all testing
  - Most tests require physical or chemical preparation that affects the outcome of the test
- RMB added to address QC for non-destructive tests

  An RMB is composed of one (1) to twenty (20) environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameters, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections) and the maximum time between the start of processing of the first and last samples in an RMB is fourteen (14) calendar days.

- Analytical Batch: not generally used for radchem tests

Negative Control
Method Blank

- Laboratory shall have procedures to determine when a method blank result is different from zero
  - Compare result to CSU
- Blanks evaluated for long term trends / bias
- No subtraction of batch method blank
  - However, may correct all results for average historical activity of method blanks to address demonstrated bias
  - Must account for additional uncertainty

Positive Control
Laboratory Control Sample

- Overall concept unchanged
  - One per Preparation Batch
    - *Minimum* spike concentration based on the relative uncertainty of acceptance criteria
  - One per Radiation Measurement Batch (RMB)
    - Pre-prepared LCSs may be used since there is no prep for Radiation Measurement Batches

Positive Control Laboratory Control Sample

- Must include all radionuclides being determined except:
  - Gross activity measurements may use an appropriate surrogate (e.g. ^{230}Th for gross α)
  - Alpha spectrometry measurements
    - When multiple radionuclides with similar characteristics are determined simultaneously only one analyte/isotope needs to be included
  - Gamma-ray spectrometry
    - When using energy/efficiency calibration curve
      - Must check low-energy and high-energy gamma
      - May rely on a radionuclide of similar energy

Sample Specific QC Measures

- Matrix Spikes
  - One per preparation batch
    - Components consistent with those of the LCS
    - Not required for non-destructive methods (e.g., gamma spec)
    - Not required for methods with tracers or carriers
- Duplicate
  - One per batch
    - For Radiation Measurement Batches a 2nd count on a different detector is required when multiple detectors used
- Tracers/Carriers
  - Essentially unchanged.

No Special Handling of QC Samples

- Systematic preference of detectors, equipment, or glassware for QC samples not allowable

Reagent Quality, Water Quality, and Checks

- Essentially unchanged except:
  - Where there is no known provider of a traceable standard, the lab will use reference standards from a national metrology institute (NMI), e.g. NIST;
  - Reference standards may also be obtained from an ISO/ IEC Guide 34 accredited reference material provider, or an ANSI N42.227 reference material manufacturer;
  - The laboratory must verify standards before use

Data Acceptance / Rejection Criteria

- Renamed "Data Evaluation and Reporting"
  - Evaluation of tracers and carriers discussed *(entirely missing from 2009 Standard)*
  - Allows reporting qualified results if activity measured in samples greater than 5 times activity found in blank

Reporting

- Results reported as measured,
  - The activity reference date must be reported
  - Includes estimate of uncertainty (BIPM *Guide to the Expression of Uncertainty in Measurement (GUM), MARLAP, or equivalent approaches);*
  - Uncensored (e.g., against detection limit or reporting limit)
- Project- or client-specified reporting requirements may take precedence over the requirements of the standard with regard to uncertainty reporting
  - For example, compliance with SDWA, other regulations, or contracts, labs may require reporting counting uncertainty;
  - All other measurements are to be reported with total/ combined uncertainty

Sample Handling

- The laboratory must:
  - Verify that samples have been preserved as required by regulation/method/ contract, or in the quality management system.
  - Document timing, methods used, acceptance range, or other conditions indicating acceptable preservation.

Our Next Steps?

- Assessment Checklist
- Training for assessors
- Training for laboratories

Thank you!!!

Any Questions?