Use of Reference Materials in the Accreditation Framework

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ANSI - ASQ National Accreditation Board

- Laboratories – ISO/IEC 17025
- Inspection Bodies –
  - ISO/IEC 17020
- RMPs – ISO Guide 34
- PT Providers – ISO/IEC 17043
- Product Certifiers –
  - ISO Guide 65 (w/ANSI)
- Government Programs:
  - DoD ELAP, EPA Energy Star, CPSC Toy Safety, NRC, NST IPV6, US Navy
- Training Programs

- Accreditation for ISO/IEC 17025 forensic test laboratories and ISO/IEC 17020 forensic test agencies
- Academic Programs
- Workshops and Training

- Certification Bodies – ISO/IEC 17021
- Accreditation for Management System Certification Bodies:
  - ISO 9001 (QMS)
  - ISO 14001 (EMS)
  - TS 16949 (US Automotive)

  etc.

Overview

- Related Standards
- Definitions of RMs
- Types of RMs
- Data Supporting RMs
- Use of RMs

RM Related Standards

- ISO Guide 30
  - Vocabulary
- ISO Guide 31
  - Contents of certificates and labels
- ISO Guide 32
  - Calibration in analytical chemistry and use of certified reference materials

RM Related Standards

- ISO Guide 33
  - Uses of certified reference materials
- ISO Guide 34
  - General requirements for the competence of reference material producers
- ISO Guide 35
  - Reference materials -- General and statistical principles for certification

RM Related Standards

- ISO Draft Guide 80
- Guidance for in-house preparation of reference materials for quality control

Definitions

- **Reference Material (RM)**
  material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process
- **Certified Reference Material (CRM)**
  RM characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

In-house Quality Control Materials

- Not a new class of RMs
- Materials used routinely to assess the precision of test procedures.
  - in-house reference materials
  - quality control materials
  - check samples

Data behind RMs and CRMs

- Assessing of:
  - Homogeneity
  - Stability
  - Characterization of RM
- Establishing of:
  - Uncertainty
  - Metrological traceability
    - Comparison to higher in the metrological traceability hierarchy
      - Identity (measurand)
      - Quantity

Homogeneity

- Homogeneity - uniform in composition or character –
  - *Within-bottle Homogeneity*
    - Checks product for stratification or precipitation
  - *Between-bottle Homogeneity*
    - Samples multiple containers from each lot to check for homogeneity

Homogeneity

- Units analyzed in random order, not 'as bottled'
  - Separate analytical drift from bottling trends
  - ANOVA
  - Determines the contribution to the combined uncertainty from possible inhomogeneity

Homogeneity

- Assists in the assessment of the statement of minimum weight on the certificate

Stability

- Not reactive during normal use
- Retains properties
  - In expected timescale
  - In the presence of expected conditions of application
- Unstable material
  - corrode, decompose, polymerize, burn or explode under the 'normal' conditions

Stability

- Prior information
  - Use data from related materials
  - Use published and/or readily available information
- New stability studies
  - Accelerated testing
  - Long-term testing
  - Determines the value of the contribution to the combined uncertainty for instability

Characterization

- Single primary method in one laboratory
  - Cost effective if methodology and equipment is readily available
- Two or more independent methods in one or more laboratory
  - Requires detailed uncertainty information for methods
- Consensus certification
  - Multiple laboratory study using competent laboratories
    - Sometimes free choice of method
    - Sometimes method specified

Uncertainty

- Calculated from the standard uncertainties associated with:
  - Homogeneity assessment
  - Characterization measurements
  - Possible long-term instability
  - Other contributions
- Contributions are combined and expanded to give a 95% confidence interval

Uncertainty

NMI (1º Std)

CRMs

RMs

In-house QCMs

Decreasing Uncertainty

Metrological Traceability

- Common reference point
  - SI
  - NMI material or higher level RM in the metrological traceability hierarchy
    - Primary Standard
  - Applies to:
    - assessment of homogeneity and stability
      assignment of values in characterization

Metrological Traceability

Measurement Method	Traceability
Primary Method	SI
Method of Known Bias	SI/International Standard
Independent method(s)	Results of Specified Method(s)
Inter laboratory Comparison	Results of Specified Method(s)

Use of Reference Materials

- CRMs
- RMs
- In-house QCMs

Accreditation Body Policies

- Accredited laboratories to use, where available and appropriate, RM for the verification/validation of critical steps and processes in their methods
- Laboratories to ensure that RM they purchase are obtained from a competent producer of reference materials

RM Hierarchy

- NMI materials (i.e. SRMs)
- CRMs
- RMs
- In-house QCMs

CRM use

- Establish Traceability
- Measurement Uncertainty
- Method Validation
- Method Verification (Correct for use) (RM)
- Calibration (RM)

QCM use

- Matrix matching
  - suitable for ongoing quality control
- Suitable day-to-day RM to complement a commercially available CRM
- No suitable CRM exists

QCM use

- Application does not require a material having the full characteristics of a CRM
  - Traceability and uncertainty
    - Method development

QCM use

- Preparation of Control Charts
- Comparison of Results (Overtime)
- Method Development
- Instrument Performance Checks
- Repeatability and reproducibility studies
- Check Sample
- Operator Variability
- Influence of Environmental Conditions

Uses of Reference Materials

CRM

- Method validation
- Accuracy
- Conformity check

RM

- Method reproducibility / comparisons

QCM

- Basic research /
- Development of methods
- Ongoing Verification

In Conclusion

- Choice of RM dependent upon:
  - Availability
  - Appropriateness
    - Degree of Characterization for intended use
  - Competence of Supplier

Questions?

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