ANSI-ASQ National Accreditation Board

ANAB

A Primer on Reference Materials

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Overview

- Related Standards
- Definitions and 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 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

Definitions

- **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

In-house Quality Control Materials

- SW846 Examples
  - 8260 & 8270
    - Stock solutions may be prepared from pure standard materials…
  - 6010 & 6020
    - Stock solutions may be prepared from ultra-high purity grade chemicals or metals (99.99% pure or greater)

Production concepts of RMs

- ISO Guide 30
- Production Batch (lot)
  - Produced in single manufacturing cycle
  - Intended to have uniform:
    - Character
    - Quality

CRMs

- Characterized by metrologically valid procedure
  - Homogeneity
  - Stability
- Certificate
  - Value of spcified property
  - Associated uncertainty
  - Statement of traceability

Data behind CRMs

- Assessing of:
  - Homogeneity
  - Stability
  - Characterization of RM
- Establishing of:
  - Uncertainty
  - Metrological traceability
    - oIdentity (measurand)
    - oQuantity

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 sample 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 (Primary Std)

CRMs (2ndary Std)

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)

Metrological Traceability

- Achieved by
  - Unbroken chain of comparison, with uncertainties
  - When not possible
    - Correlation of results with other stated values
    - By exhaustive evaluation of the measurement process or comparison with accepted CRMs

Metrological Traceability

- Appendix A (Guide 34)
  - Chemical composition
  - Physical response of instrument after
    - oSampling
    - oDissolution or extraction
    - oSeparation by chromatography
    - oTraditional wet-chemistry

Metrological Traceability

- Appendix A (Guide 34)
  - Chemical composition
  - Any or all of these may constitute links in the metrological chain of the final result

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

QSM 5 1.7.1.1

- The use of a standard from a second lot obtained from the same manufacturer **(independently prepared from different source materials)** is acceptable for use as a second source standard.

- Is this necessary understanding the international concept of CRMs?

CRMs

- Characterized by metrologically valid procedure
  - Homogeneity
  - Stability
- Certificate
  - Value of spcified property
  - Associated uncertainty
  - Statement of traceability

In Conclusion

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

Questions?

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