Traceability in Chemical Analysis – Are we ready to deliver?

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Metrological Traceability

Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty

Today's topics:

- Relevance
- Identity
- Traceability 'chain'
- Reference points

*ISO Guide 99 (2007)*
Relevance: What to measure?

'customer' question

↓

information need

↓

measurement task

Example: Health-status marker

whole molecule? (identity, number…)

↓

activity/reactivity

part(s) of the molecule? (identity…)

Definition of decision-relevant measurand – targeted property ‘structural’, ‘functional’, other?
Problem Definition

- sampling
- conservation, sub-sampling
- analytical sample preparation
- analyte identification
- quantification (quantity value attribution)
- data evaluation
- assessment

Focus of this talk

Starting point in this process for the traceability 'chain'?
Measurement in chemistry

- Sample
  - Weighing, milling
  - Extraction
  - Clean-up
  - Quantification (e.g., by LC-FLD)

Traceability ‘chain’?

Relevance?

DON?
Challenge for measurements in chemistry
(bio/life sciences, material science,…):

What is on the 'balance'?

How much is on the 'balance'?
Traceability 'split' into

- **Identity (measurand)**
  - Operationally defined
  - 'Structurally' defined

- **Quantity value (number and unit)**
  - Reference: SI
  - Reference: Artefact (quantity value embedded in)
Quality Assurance of identification

Analytical sample

Sample preparation

Analyte identification & Quantification (attribution of quantity value)

Data evaluation

CRMs for qualitative analysis
- chemical identity

Selectivity?

Classification?

5' −ctttggcccaTGAGGCTGG−3'

5' −ctttggcccaCGAGGCTGG−3'
Taxonomic identification of authorised probiotic feed additives or food pathogens

**CRM:** Genomic DNA agarose inserts for Pulsed Field Gel Electrophoresis (PFGE):

- *Bacillus licheniformis* DSM 5749 (IRMM-311)
- *Bacillus subtilis* DSM 5750 (IRMM-312)

**CRM:** Lyophilised gDNA as positive control for diagnostic PCR

- *Listeria monocytogenes* (IRMM-447)
- *Campylobacter jejuni* (IRMM-448)
- *Escherichia coli* O157 (IRMM-449)
Analytical process & identity

\[ x \pm U \]

Absorption measurement

Vaporisation

Me-Hg

Extraction

Demethylation

Reduction

Amalgamation

Measurement

Transformations known?

Transformation yield?

Transformation reproducibility?

Losses / interferences?

Hg\(^0\)

Hg(Au)

Hg\(^0\)(air)

Hg\(^0\)(sol)
Establishing traceability

- Sample
- Weighing, milling
- Extraction
- Clean-up
- Quantification e.g., by LC-FLD

Traceability ‘chain’?
Establishing traceability

Further challenges:

- availability of appropriate calibrants
- known purity of calibrants
- often measurand ≠ measured quantity
  ⇒ knowing the correlations for 'surrogates'
- operationally defined identity of measurand
Establishing harmonised operationally defined measurands

Reference Methods

Reference Laboratories

GM content as measured by PCR method…

C-reactive protein in human serum via method…

Parameter (‘quantity’) definition

Reference Material

‘quantity’

often relevant for parts of procedures

Traceability anchor

essential for functional properties!
Which reference? for operationally defined measurands

INTERNATIONALLY AGREED & ACCEPTED

Primary calibrator

Definition of (SI) unit

Primary reference measurement procedure

Secondary reference measurement procedure

Laboratories

Working calibrator

Laboratories measurement procedure

Routine sample

RESULT

3D plots? (identities)
How to realise the reference points?

Reference measurement procedure

Certified Reference Material
Dienestrol in bovine urine as obtained by enzymatic deconjugation, clean-up and subsequent chromatography in combination with mass spectrometry.

**Traceability**

**Identity**
- Operationally defined
- 'Structurally' defined

**Quantity value**
- Mass fraction: $(5.5 \pm 1.4) \mu g/kg$

**Reference:** SI

**Reference:** Artefact
✓ Chemical 'measurements' require calibration of procedures (not only of instruments)

✓ Most procedures for chemical measurements contain operationally defined steps (relevant for stated 'identity')

✓ A 'primary realisation of the unit' requires for chemical measurements more than a 'value realisation'

✓ However, pragmatic approaches exist for anchor points (references) in the traceability chains → multitude of CRMs

✓ Adequate uncertainty estimations, needed for traceability claims, are still very challenging for many labs
Are we ready to deliver?

- Concepts & guidance documents for relevant measurement tasks
- Tracability tools such as CRMs
- Competent assessors ('calibrated')
- Teaching & training
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Traceability in Chemical Measurement

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