BAM

EURACHEM WORKSHOP ON
Validation / Traceability / Measurement Uncertainty
Challenges for the 21st Century's analysts

Current discussion within EA and ILAC on accreditors' policy on traceability

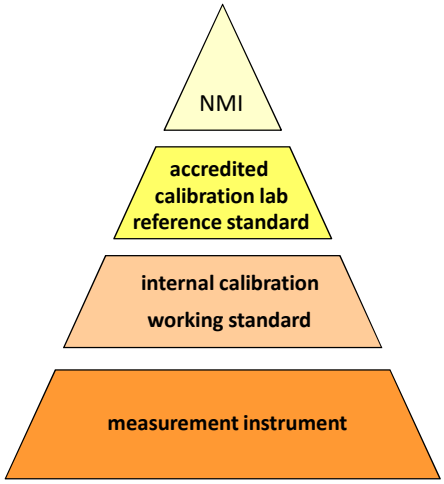
Manfred Golze
manfred.golze@bam.de

BAM

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.

VIM 3



A reference could be e.g. a
- measurement unit,
- measurement procedure,
- measurement standard.

BAM

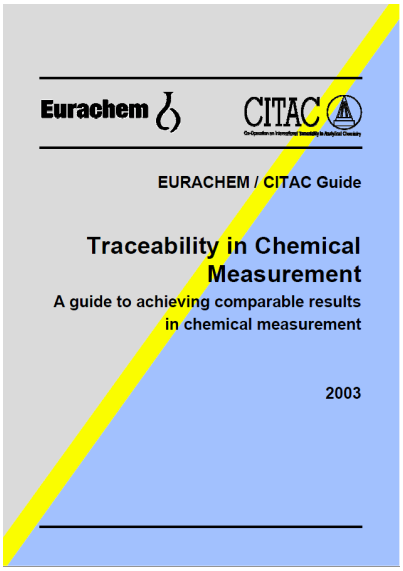
Traceability in Chemistry

Equation for the measurand y:

$$y = f(x_1, x_2, ..., x_m)\big|_{x_{m+1}, x_{m+2}, ..., x_n}$$

The variables $x_1,...,x_m$ are input quantities from which y is calculated.

$X_{m+1},...,x_n$ describe the conditions under which the measurement is performed.



Validation / Traceability / Measurement Uncertainty

BAM

Traceability of the relevant physical quantities

Some of the input variables or of the test conditions might be physical quantities as e.g.

- mass,
- volume,
- time,
- pressure,
- temperature,

whose traceability could be ensured using the well established metrological infrastructure.

Validation / Traceability / Measurement Uncertainty

Requirements of ISO/IEC 17025 (I)

5.6 Measurement traceability

5.6.1 General

All equipment used for tests and/or calibrations, including equipment for subsidiary measurements (e.g. for environmental conditions) having a significant effect on the accuracy or validity of the result of the test, calibration or sampling shall be calibrated before being put into service. The laboratory shall have an established programme and procedure for the calibration of its equipment.

Requirements of ISO/IEC 17025 (II)

5.6.2.1.1

[…] When using external calibration services, traceability of measurement shall be assured by the use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification.

Requirements of ISO/IEC 17025 (III)

5.6.2.2.1

For testing laboratories, the requirements given in 5.6.2.1 apply for measuring and test equipment with measuring functions used, unless it has been established that the associated contribution from the calibration contributes little to the total uncertainty of the test result. When this situation arises, the laboratory shall ensure that the equipment used can provide the uncertainty of measurement needed.

Interpretation by the accreditors

Currently valid interpretation documents:

- EA 4/07 rev 01:1995 *Traceability of measuring and test equipment to national standards*,
- ILAC P10:2002 *ILAC Policy on Traceability of Measurement Results*.

ILAC P10 is currently under revision. Recently a new draft failed to get the approval by the ILAC members in the ballot (67% votes in favour instead of 75% which are necessary for approval).

Principles in draft ILAC P10

Equipment or reference standards of an accredited laboratory shall be calibrated by:

- a National Metrology Institute (NMI) or Designated Institute (DI) signatory of the CIPM MRA,
- an accredited calibration laboratory whose service is suitable for the intended need and is covered by the ILAC MRA,
- another organisation whose service meets the relevant criteria for traceability in ISO/IEC 17025 and whose certificate includes statements concerning measurement uncertainty and traceability.

The CIPM MRA

The Mutual Recognition Arrangement of the International Committee for Weights and Measures (CIPM MRA) was signed in 1999 by the directors of NMIs from 38 countries. It aims at:

- establishing the degree of equivalence of national measurement standards maintained by NMIs and DIs,
- providing for the mutual recognition of calibration and measurement certificates issued by NMIs and DIs,
- thereby providing governments and other parties with a secure technical foundation for wider agreements related to international trade, commerce, and regulatory affairs.

Cited according to A. Wallard, Metrology Principles and Organisation, Springer Handbook of Metrology and Testing, 2011

The CIPM MRA

The MRA is based on the following pillars:

- the so-called key comparisons between NMIs or DIs organised by the Consultative Committees (CCs) of the CIPM or by Regional Metrology Organisations such as EURAMET,
- the database of the Calibration and Measurement Capabilities (CMCs) which are usually confirmed by key comparisons (http://kcdb.bipm.org),
- the commitment of the NMIs and DIs to establish and maintain quality systems according to ISO/IEC 17025 (and ISO Guide 34, if relevant).

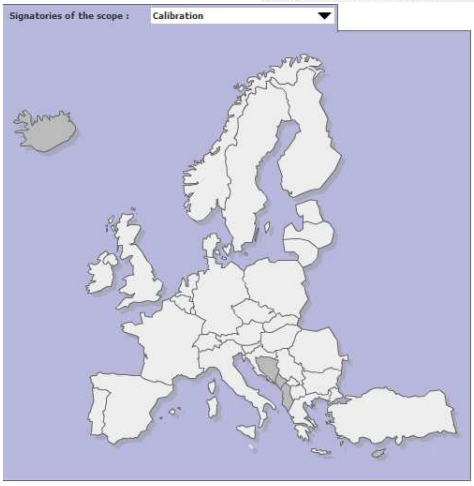
Validation / Traceability / Measurement Uncertainty

EA MLA Calibration

The EA MLA for calibration is part of the ILAC MRA. Signatories are all EU and EFTA countries (besides Cyprus, Iceland and Liechtenstein).

Additionally:

ISRAC (Israel),
HAA (Croatia),
ATS (Serbia),
IARM (Macedonia),
TUNAC (Tunisia),
TURKAK (Turkey),
NAAU (Ukraine).



Source: http://www.european-accreditation.org/content/mla/scopes.htm

Validation / Traceability / Measurement Uncertainty

The (controversial) third option

Other organisations include

- NMIs and DIs offering services outside the CIPM MRA,
- accredited calibration laboratories performing calibrations outside the scope of their accreditation,
- non accredited organisations.

The EA Advisory Board discussed during its past meeting a position paper submitted by the CAB college which advocated this option as a necessary complement, because the other two options might not be technically or economically feasible in all cases.

Intermezzo: Hypothesis testing

Thesis 1:

An accredited organisation is competent to perform specified conformity assessment activities!

☺

Thesis 2:

A non accredited organisation is incompetent!

☹

BAM

Some reasons for the third option

- The requirement is beyond the standard ISO/IEC 17025.
- Often not all services offered by an accredited organisation are covered by the accreditation scope.
- Calibration of sophisticated equipment is often not offered by commercial calibration laboratories but only by the producer of this equipment.
- There are other evaluation schemes besides accreditation.
- Internal calibration.

BAM

Other references or evaluation schemes

It is conceded in ISO/IEC 17025 that traceability to the SI is not possible in all cases.

Other references or evaluation schemes are established e.g. by

- World Health Organisation (WHO),
- Joint Committee for Traceability in Laboratory Medicine (JCTLM),
- International Atomic Energy Agency (IAEA),
- International Organisation for Legal Metrology (OIML).

World Health Organization

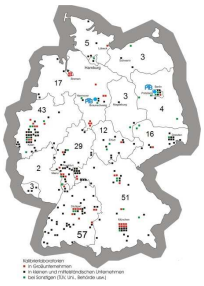
JCTLM

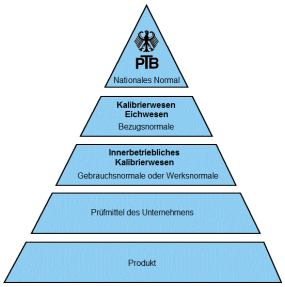
JCTLM Database
Laboratory medicine and *in vitro* diagnostics

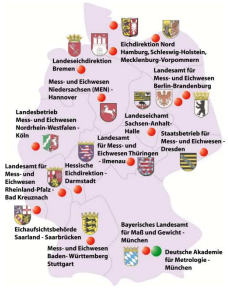
OIML

Legal metrology - a German example

In Germany a peer evaluation system is going to be established for the German verification authorities. The German Accreditation Body (DAkkS) will accept this system.







Requirements for the 3rd option

As appropriate evidence for claimed traceability Annex A of revised final draft ILAC P10 lists:

- records of calibration method validation,
- procedures for estimation of uncertainty calculations,
- documentation for traceability of measurements,
- documentation for assuring the quality of calibration results,
- documentation for competence of staff,
- audits of the calibration laboratory.

Content of calibration certificates

According to clauses 5.10.2 and 5.10.4 of ISO/IEC 17025 a calibration certificate shall include among others the following information:

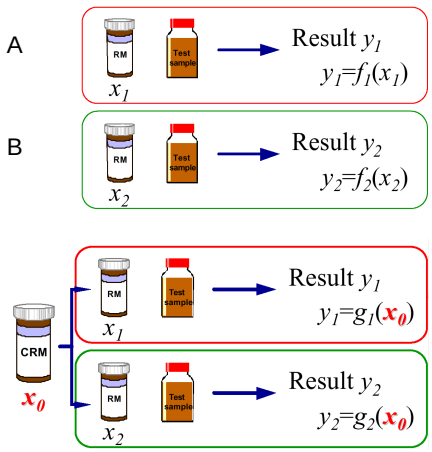
- an unambiguous identification of the item(s) calibrated,
- identification of the method used,
- the environmental conditions, if relevant,
- the calibration results and their uncertainty,
- a statement concerning traceability.

Internal calibrations

A laboratory might decide to calibrate some of its equipment internally.

For modern analytical equipment (e.g. GC or ICP) internal calibration is predominant since the calibration is often performed in the same run as the analysis of the test samples. For this purpose adequate calibration materials (pure substances, certified reference materials) are used.

The competent performance of these internal calibrations can easily be assessed during accreditation or surveillance visits.



Reference: EURACHEM / CITAC Guide 2003

ILAC policy concerning CRMs

According to the policy defined in ILAC P10 (revised 2nd final draft) CRMs are accepted as validly traceable, if

- included in the BIPM database,
- produced by an accredited RM producer within its scope of accreditation.

As the majority of the CRMs are produced by non accredited producers, these CRMs are considered as critical consumables and the laboratory shall demonstrate that they are suitable for their intended use.

Conclusions (I)

- For an accredited laboratory certainly it is the preferred and easiest way to use calibration services provided by a NMI, DI or accredited calibration laboratory.
- But the third way, using calibration services offered by other organisations, is nevertheless a necessary option.
- Using this option shifts the burden of proof concerning the competence of the other organisation to the accredited laboratory.

Conclusions (II)

- Internal calibrations should be assessed during the regular accreditation or surveillance visits.
- Accreditation of RM producers is so far not required, but helpful.
- CRMs of non accredited producers are considered as critical consumables. The laboratory shall demonstrate that they are suitable for their intended use.

BAM

Thank you for your attention.

Validation / Traceability / Measurement Uncertainty