Quality Management in Analytical Laboratories According to ISO / IEC 17025

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Introduction

The International Standard ISO / IEC 17025 “General requirements for the competence of testing and calibration laboratories” was published in 1999, replacing both EN 45001 and ISO Guide 25.

After the end of the transition period for its implementation, that was fixed by ILAC, the International Laboratory Accreditation Co-operation, to be 31 December 2002, it is now the standard used for the accreditation of laboratories worldwide.
The standard defines the requirements that testing and calibration laboratories have to meet if they wish to demonstrate that

- they operate a quality system,
- are technically competent,
- are able to generate technically valid results.
Introduction

ISO / IEC 17025 contains two parts on
- management requirements,
- technical requirements.

Quality management

Documentation and records are necessary
• to define objectives and policies,
• to ensure a constant level of performance,
• to enable traceability concerning the process results (trackability).

Document hierarchy:
• Quality manual,
• Documents for general procedures,
• Standard operation procedures.
Other important elements of a quality system are e.g.:  

- Training of staff,
- Audits  
  systematic and independent examination whether objectives are achieved and procedures implemented effectively
- Management reviews  
  activity to determine the suitability, adequacy and effectiveness of the quality system with regard to objectives and policies
- Corrective and preventive actions  
  actions to eliminate the cause of a detected or potential nonconformity

Aim ⇒ continuous improvement
Quality management

PDCA methodology

**Plan:** establish the objectives and processes necessary to deliver results in accordance with customer requirements and the organisation’s policies

**Do:** implement the processes

**Check:** monitor and measure processes against policies, objectives and requirements for the product and report the results

**Act:** take actions to continually improve process performance

Plan → Do → Check → Act → Plan
R & D and non-routine activities

Also in an R&D environment principles for quality and valid results should be met, e.g.

- fulfilment of agreed requirements,
- competent staff,
- use of adequate equipment,
- well defined quality assurance procedures,
- comparability with results achieved elsewhere,
- independent assessment of technical performance.

Reference: EURACHEM / CITAC Guide on Quality assurance of research and development and non-routine analysis
According to ISO/IEC 17025 the results of measurements and tests should, if possible, be traceable to the International System of Units (SI).

A result is traceable to an SI unit, if it is related by an unbroken chain of comparisons – each with stated uncertainty – with a primary realisation of this unit.
Testing means the determination of one or more characteristics of the item under test to a specified procedure. In general, a quantitative test result will depend on the test procedure applied.

**Example:** Surface analysis with techniques with different surface sensitivity.

As a consequence the concept of traceability has to be generalised for chemical analysis and testing. The test procedure and the conditions, that have to be met, must be taken into account.

See EURACHEM/CITAC Guide on Traceability in chemical measurement (www.eurachem.ul.pt)
Traceability established by means of a RM

BAM developed a certified RM with a multilayer pattern. It can be used to determine various instrumental parameters for SIMS, e.g. for the calibration of a length measurement.

In an interlaboratory comparison 12 of 16 participants measured the length of the calibration distance (964 ± 35 nm, k=2) within an accuracy of ± 4%.

Reference: M. Senoner, BAM Report on an inter-laboratory comparison, July 2003
Measurement uncertainty

According to ISO / IEC 17025 testing and calibration laboratories shall have and apply procedures for estimating uncertainty of measurement. All relevant uncertainty components shall be taken into account.

A measurement uncertainty statement should be reported together with the result if

- it is relevant to the validity or application of the test result,
- a client’s instruction requires so,
- uncertainty affects compliance with a specification limit.
Measurement uncertainty is defined as

“parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand.”

\[ y = m \pm u \]
The Guide to the expression of uncertainty in measurement (GUM) is the master document for measurement uncertainty.

In the GUM the following basic principles are laid down:

- all relevant uncertainty components,
- correction for systematic deviations,
- components of type A (statistical techniques) and type B (other sources) treated in the same way,
- all components expressed as standard deviations,
- combination of components according error propagation theory (summation of variances),
- expanded uncertainty by multiplication with a coverage factor (often $k = 2$).
The analytical approach to evaluate MU

1. Specification of measurand and mathematical model defining the relation between measurand and input quantities
2. Determination of input quantities
3. Quantification of standard uncertainties of all relevant components
4. Identification of co-variances (if input quantities are correlated)
5. Calculation of measurement result from input quantities (2)
6. Calculation of combined uncertainty
7. Calculation of expanded uncertainty
8. Reporting of measurement result (5) together with its uncertainty
Uncertainty budget

An example for an uncertainty budget obtained by this method is dealing with the uncertainty in measurement of overlayer thickness of thermally oxidized silicon using x-ray photoelectron spectroscopy. A combined relative standard uncertainty of ± 15% was evaluated and the uncertainty of the effective attenuation length was identified as the major source of uncertainty.

Use of method performance data

- **Accuracy**
  - **Trueness**
    - Precise and true
    - Precise but not true
  - **Precision**
    - Not precise but true
    - Not precise not true
Use of method performance data

- **Precision**
  - **Repeatability conditions**
    - same measurement procedure
    - identical items or samples
    - same laboratory
    - same operator
    - same equipment
    - within short time interval
  - **Reproducibility conditions**
    - same measurement procedure
    - different laboratories
    - different operators
    - different equipment

Cases between these extremes: **Intermediate conditions**

Or if all measurements from the same lab: **Within laboratory reproducibility**
Use of method performance data

1. Specification of measurand

2. Quantification of reproducibility within laboratory
   A: from control samples
   B: by including steps not covered by the control samples

3. Quantification of systematic components (e.g. by use of CRM, interlaboratory comparisons or recovery tests)

4. Conversion of components into standard uncertainties

5. Calculation of combined standard uncertainty

6. Calculation of expanded uncertainty

Method performance data from an ILC

BAM Interlaboratory comparison on the measurement of heights of steps in the nm-range using Atomic Force Microscopy (AFM)

Results according to ISO 5725 e.g.

Reference value: 81.0 nm
Median: 79.4 nm
Bias: -1.6 nm
Repeatability: 2.05 nm
Reproducibility: 5.01 nm

There are indications that bias and reproducibility could be considerably reduced by improved calibration of the AFMs.

Method validation

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

According to ISO/IEC 17025 the laboratory shall validate non-standard methods and standard methods used outside their intended scope by e.g.

- calibration using reference standards or reference materials,
- comparison of results achieved with other methods,
- interlaboratory comparisons,
- systematic assessment of the factors influencing the result,
- assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience.
Method validation

Example: Interlaboratory comparison for validation
Within a VAMAS project an ILC on the evaluation of static charge stabilisation and determination methods in XPS on non-conducting samples was performed.
In XPS a charge correction is necessary for correct binding energy determination.

In the ILC 27 participating laboratories determined binding energies with reference to Au 4f\textsubscript{7/2} binding energy of 84.00 eV using three types of non-conducting samples with a deposit of 15 nm gold particles.
Results:  
repeatability  \approx 0.05 \text{ eV}
reproducibility  > 0.15 \text{ eV}

Conclusions

- ISO/IEC 17025 is the International Standard on the competence of testing and calibration laboratories.
- In the standard technical aspects as traceability, measurement uncertainty and validation are strongly emphasised.
- Consequently reference materials, reference procedures and interlaboratory comparisons are of great importance for the laboratory community.
- Since January 2003 ISO / IEC 17025 is used for accreditation of laboratories worldwide.
Acknowledgements

The support by my colleagues from BAM:

Thomas Gross,
Mathias Senoner,
Wolfgang Unger,

who provided the material for the examples given in this presentation, is gratefully acknowledged.