How to apply "Quality" in nuclear analytical chemistry: an illustration.

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The term "Quality"

- Totality of characteristics of an entity to bear on its ability to satisfy stated and implied needs
  > EN ISO 8402
- In the lab: the benefit your customer has from your test result
- The quality system is the organisation you put up in order to deliver results that satisfy these specific requirements
ISO 9000 standards set the basic rules for quality systems - from design, through manufacturing to delivery - whatever product or service. In fact a set of ‘good practice’ rules for manufacturing a product or delivering a service to achieve customer satisfaction by preventing deviations in all phases of the process. Not a technical standard!

By analogy with the standard ISO 9000, a standard is developed specifically for routine testing laboratories. EN 45001 or ISO guide 25, but, ISO 17025 is coming which guarantees full relevant compliance with ISO 9001.
Accreditation vs. Certification

(situation in Belgium...)

- Accreditation according to ISO 17025 or EN 45001 involves the assessment and periodic audit of the adequacy of the quality system by a third party "an Accreditation Body."

- An accredited lab satisfies the lab standard which lays down the quality assurance requirement and the technical competence; the accreditation guarantees also that the result is assured (within the measurement uncertainty that you define yourself).


What’s the difference?

(situation in Belgium...)

- When you are looking e.g. for a competent calibration lab you will find those that are certified and those that are accredited;

- The certified lab will guarantee you that the calibration will be carried out conform to a quality system and will be well documented, but it does not imply that the lab has the technical competence to perform such a calibration (qualified personnel, traceable instruments, calculated uncertainty, ...).
Illustration of an accredited method:
Isotopic Analysis + Isotopic Dilution of Pu by TIMS

Full description of the method in working instructions
(from reception of the sample up to final result).
- the so feared paperwork...

Validation study:
the 'business card' of your analysis

- validation of analytical instruments and procedures in order to proof the
technical competence and (as a result) the claimed "quality".
- validation parameters: repeatability, reproducibility, accuracy,
specificity, detection limits, linearity, sensitivity,...
- other tests in this validation context:
  > comparison of the results of both our TIMS instruments
  > comparison of the results of both our qualified technicians
  > regular quality checks of all labware
    (balances, pipettes, volumetric flasks, ...)
Accuracy in MS: mass bias

- Accuracy is tested by measuring Certified Reference Materials (CRM's).
- In MS, mass bias induces differences in measured isotopic ratio's versus certified isotopic ratio's. The linear law to correct for this mass discrimination states:

\[ \frac{R_{tr}}{R_{meas}} = 1 + \Delta m \cdot B \]

with:
- \( R \): ratio of (isotope \( m_1 \) / isotope \( m_2 \))
- \( tr \): true, \( meas \): measured
- \( \Delta m \): difference in atomic mass units \(- m_1 - m_2 \)
- \( B \): mass bias per atomic mass unit

- The mass bias routinely applied, is based on the measured ratio's (Pu-240/Pu-239) and (Pu-242/Pu-239) of CRM NBS-947 (n=37).
- Result: \( B = 0.00100 \pm 0.00022 \) (1s)

Validation of TIMS for the isotopic analysis of U and Pu (SCK-CEN document MT.RA.BN/901)

"The uncertainty on a measurement of the isotopic composition of an element by TIMS is estimated at the 95% confidence level (2s) to be:

- \( \pm 10\% \) at the abundancy level of \( 0.003 - 0.01\% \)
- \( \pm 5\% \) at the abundancy level of \( 0.01 - 0.05\% \)
- \( \pm 2\% \) at the abundancy level of \( 0.05 - 0.5\% \)
- \( \pm 0.5\% \) at the abundancy level of \( 0.5 - 3\% \)
- \( \pm 0.25\% \) at the abundancy level of \( 3 - 10\% \)
- \( \pm 0.1-0.2\% \) at the abundancy level of \( > 10\% \)"
Quality Control: Control Chart TIMS
measurement of an isotopic reference standard (e.g. NBS947) on each barrel; control of \( \text{Pu}^{(240/239)} \)\text{measured} / \( \text{Pu}^{(240/239)} \)\text{certified}.

Traceability of results back to recognised standards

e.g. the spike used in Isotopic Dilution measurements:

- the making of the spike solution is documented
  (composition is certified; concentration is theoretically known and experimentally checked)
- the management of the spike solution is documented
- the concentration is regularly checked by spiking with another CRM.

Other examples:

- standard weights to calibrate balances (even in hotcell)
- calibration of thermometers
- ...
Same 'philosophy' applied to burnup determination (1):

- Burnup FIMA (Fissions per Initial Metal Atom)

\[
\text{at.} \% FIMA = \frac{\sum (\Delta N)}{\sum (No)} \cdot 100 = \frac{\sum (\Delta N)}{\sum (Ne) + \sum (\Delta N)} \cdot 100
\]

\[\sum (\Delta N) = \text{number of heavy atoms fissioned (}= \text{fissions})\]
\[\sum (No) = \text{number of heavy atoms initially present}\]
\[\sum (Ne) = \text{number of heavy atoms at end of irradiation}\]

Same 'philosophy' applied to burnup determination (2):

- \(\sum (\Delta N) = 100 \cdot \text{#at (Nd-148) / MWFY (Nd-148)}\)
  - #at (Nd-148): total number of Nd-148 nuclide
  - determined by TIMS (IA+ID)
  - traceable to certified standard

- MWFY (Nd-148): Mean Weight Fission Yield of Nd-148
  - is calculated from literature data (fission yields)
  - measurement of Nd-148/Nd-150 ratio (TIMS IA)
  - measurement of Pu-241/Pu-239 ratio (TIMS IA)
  - (assumption: all fissions are from U-235, Pu-239 and Pu-241)
Same ‘philosophy’ applied to burnup determination (3):

\[ \Sigma(\Delta Ne) = \text{at (U)}_{EOL} + \text{at (Pu)}_{EOL} + \text{at (TPu)}_{EOL} \]

- \text{at (U)}_{EOL}: total number of U-atoms at end of irradiation
  - determined by TIMS IA+ID
  - traceable to certified standard
- \text{at (Pu)}_{EOL}: total number of Pu-atoms at end of irradiation
  - determined by TIMS IA+ID
  - traceable to certified standard
- \text{at (TPu)}_{EOL}: total number of Np + Am + Cm atoms at end of irradiation
  - determined by TIMS, \alpha- and/or \gamma-spectrometry
  - all results traceable to certified standards

Same ‘philosophy’ applied to burnup determination (4):

With uncertainties of

0.3 - 0.5 % for \( \Sigma(\text{Ne}) \) and
2 - 4 % for \( \Sigma(\Delta N) \)

an overall uncertainty in the burnup determination can be estimated to be 2.5 - 4 %.

Remark: main contribution comes from the MWFY...