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

Starting-points of ISO 9000

- 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!

For testing laboratories

- 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)

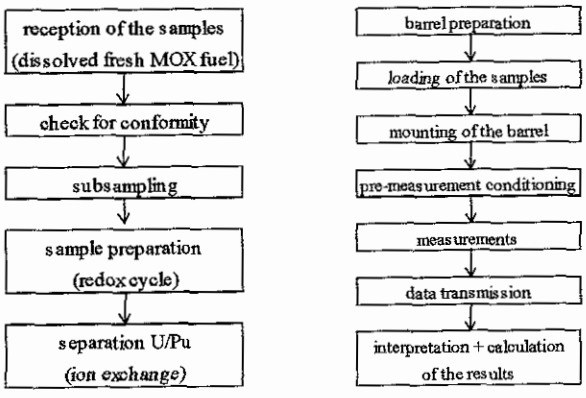
What's the difference?

(situation in **Belgium**...)

- When you are looking e.g. for a competent calibrationlab 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 :

$$R_{tr} / R_{meas} = 1 + \Delta m \cdot B$$

with : R = ratio of (isotope m_1 / isotope m_2)
tr = true, meas = measured
Δm = 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 +/- 0.00022 (1s)

p 9

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

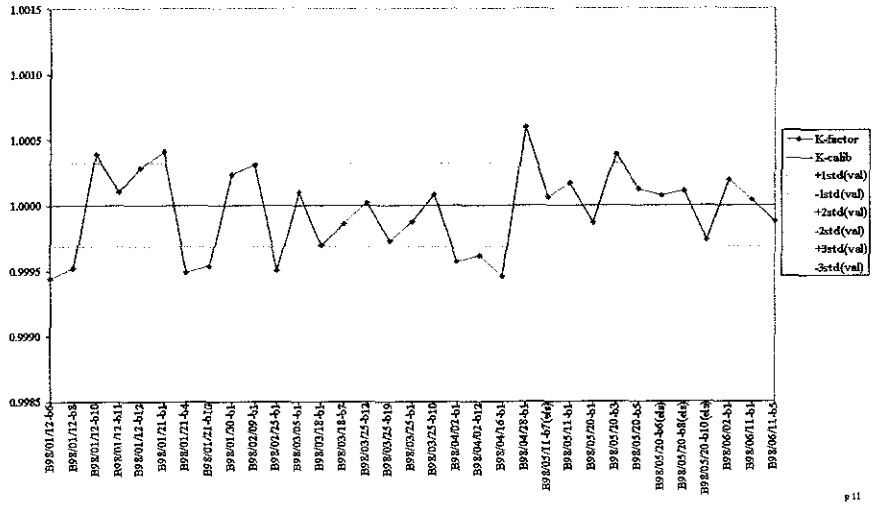
"the uncertainty on a measurement of the isotopic composition of an element by TIMS is estimated at the 95% confidence level (2s) to be :

± 10 %	at the abundancy level of	0.003 - 0.01 %
± 5 %	at the abundancy level of	0.01 - 0.05 %
± 2 %	at the abundancy level of	0.05 - 0.5 %
± 0.5 %	at the abundancy level of	0.5 - 3 %
± 0.25 %	at the abundancy level of	3 - 10 %
± 0.1-0.2 %	at the abundancy level of	> 10%"

p 10

Quality Control : Control Chart TIMS

measurement of an isotopic reference standard (e.g. NBS947) on each barrel; control of $Pu(240/239)_{measured}$ / $Pu(240/239)_{certified}$



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)

$$at.\%FIMA = \frac{\Sigma(\Delta N)}{\Sigma(No)} \cdot 100 = \frac{\Sigma(\Delta N)}{\Sigma(Ne) + \Sigma(\Delta N)} \cdot 100$$

$\Sigma(\Delta N) =$ *number of heavy atoms fissioned (= fissions)*

$\Sigma(No) =$ *number of heavy atoms initially present*

$\Sigma(Ne) =$ *number of heavy atoms at end of irradiation*

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

- Σ(ΔN) = 100 . #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):

- $\Sigma(\Delta Ne) = \#at\ (U)_{EOL} + \#at\ (Pu)_{EOL} + \#at\ (TPu)_{EOL}$
  - ➢ $\#at\ (U)_{EOL}$: total number of U-atoms at end of irradiation
    - ♦determined by TIMS IA+ID
    - ♦traceable to certified standard
  - ➢ $\#at\ (Pu)_{EOL}$: total number of Pu-atoms at end of irradiation
    - ♦determined by TIMS IA+ID
    - ♦traceable to certified standard
  - ➢ $\#at\ (TPu)_{EOL}$: total number of Np + Am + Cm atoms at end of irradiation
    - ♦determined by TIMS, α- and/or γ-spectrometry
    - ♦all results traceable to certified standards

Same 'philosophy' applied to burnup determination (4):

With uncertainties of

0.3 - 0.5 % for $\Sigma(Ne)$ 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...