Eurachem

A FOCUS FOR ANALYTICAL CHEMISTRY IN EUROPE

CITAC

Co-Operation on International Traceability in Analytical Chemistry

Metrological Traceability of Analytical Results

In order for results to be comparable we need traceability.

In this leaflet we would like to point out to analytical laboratories what traceability is and how a lab can demonstrate traceability of its results using an example of a mercury determination.

About traceability in 19th century

Once a dairymaid bought one kilo of flour from the local shop. Next day she returned to sell one kilo of butter to the shopkeeper. He then complained that 50 grams of the kilo were missing.

— *Oh that is odd,* the dairymaid said, *to get the correct weight I used the kilo of flour you sold to me yesterday to weigh the butter!*

Traceability today

Traceability is the ability to trace e.g. the ancestors of a person or the origin of a product. The word can be traced back to latin tractus = drawn or the verb trahere = to draw.

Traceability can refer to the documentation i.e. sampling procedure, laboratory, analyst, method etc but as in ISO/IEC 17025 we are dealing with traceability of measurement results. That is measurement results have to be traceable to the metrological[1] references **used**. To be specific we here use the wording ***metrological* traceability**. The current VIM[2] definition of metrological **traceability** is:

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[2]

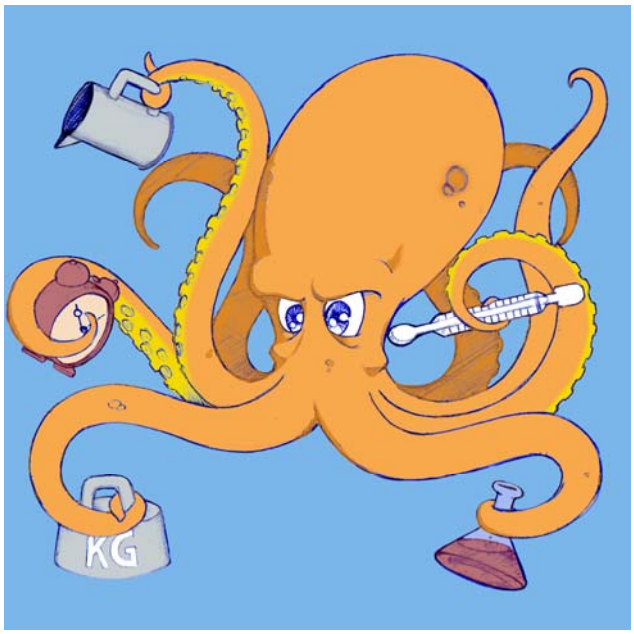
[1]Metrology is the science of measurement

[2]International vocabulary of metrology — Basic and general concepts and associated terms (VIM), ISO/IEC Guide 99, 2007

The references

Ideally the **references** should be values of national and international standards expressed in SI units. The traceability is realised through chains of **calibrations**. For temperature and many other physical quantities e.g. mass and time the traceability is relatively easily established.

In addition, in chemistry the working standards are substances with defined purity, solutions of pure substances and matrix reference materials.



An analyst not knowing the traceability of his/her result for volume, time, mass, calibration solution and temperature.

Example of traceability to the SI - Temperature

The temperature of a sample can be traced back through a **chain of calibrations** to the **reference,** an SI value of temperature at 0 °C.

Example - Mercury in tuna fish

A measurement result (mass fraction) of mercury in a tuna fish sample is 4.03 ± 0.11 mg /kg. The result is reported as total Hg on dry weight basis (105 ºC, 12 h determined on a separate sample portion) and the measurement uncertainty is reported with a 95 % level of confidence (k=2).

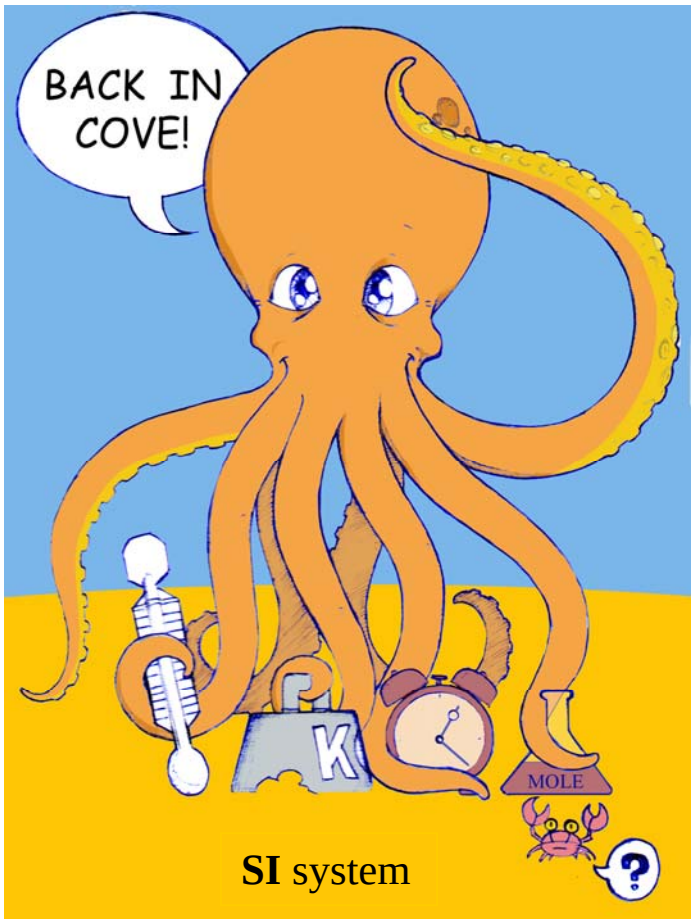
In this case mercury was determined with a mercury analyzer (AAS cold vapour) after a microwave digestion.

The samples are weighed on a balance with a calibration certificate relating the weight measured to the SI unit kg.

The acid digest is diluted in a volumetric flask where the manufacturer supplies the traceability of the volume of the flask to a national standard.

The calibration curve was made using a CRM (Certified Reference Material), a mercury standard with a certificate stating a quantity value of 0.998± 0.005 mg/kg (k=2) and with traceability to pure mercury.

The method is validated using an appropriate matrix CRM with a total mercury concentration of 1.97 ± 0.04 mg/kg (k=2). This validation is a check on the method performance.



*An analyst **knowing** the traceability of his/her result for volume, time, mass, calibration solution and temperature.*

Illustrations by Douglas Hasbun

Traceability shown by the laboratory

The evidence required by the laboratory to demonstrate traceability for the mercury result is shown below:

1. concentration of the Hg solution- a certificate of the CRM solution
2. mass of sample – calibration certificate of the balance
3. volume of volumetric flask – calibration certificate of the manufacturer
4. drying temperature – calibration of oven
5. digestion conditions – check according to specifications
6. drying time - ordinary clock or stopwatch

Point 1 would need special attention to assure the quality and traceability for the calibration standard.

Traceability for points 2, 3 and 6 is easily achieved with adequate uncertainty using commercial equipment.

Points 4 and 5 need additional attention by the lab.

The use of the matrix CRM in the method validation is vital but not part of the traceability, since it is not used for calibration. If this CRM is used for recovery correction it should be part of the traceability. However, the uncertainty associated with this matrix CRM may have to be included in the uncertainty budget.

Validation – Traceability
Measurement Uncertainty - Quality Control

- Method **validation** demonstrates that the method (set of conditions) used in this laboratory at a certain time was fit for purpose and all significant effects were identified.
- Calibration of critical equipment completes the metrological **traceability** chains.
- **Measurement uncertainty** is estimated from the method validation and the traceability.
- **Quality control** (internal and external) assures that the measurement results (including uncertainty) are of the same quality as at the time of validation.

Conclusion

The **traceability of a measurement result** refers to **metrological traceability** as defined by VIM. It relates the result to SI units or other agreed standards/references.

Traceability is essential for comparability of analytical results. It is a requirement of ISO/IEC 17025.

Traceability is easily achieved following good laboratory practice.

Further reading

Eurachem/CITAC guide on Traceability www.eurachem.com.

VAM guide – Meeting the Traceability Requirements of ISO17025 www.vam.org.uk

Traceability of Measuring and Test Equipment to National Standards, EAL-G12 www.european-accreditation.org