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 references used. To be specific we here use the wording metrological traceability. The current VIM 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

1Metrology is the science of measurement

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 °C, 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.

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

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SI system

Illustrations by Douglas Hasbun