CITAC

Co-Operation on International Traceability in Analytical Chemistry

Traceability in Chemical Measurement

Executive Summary

Traceability[1][1] is a key element in the mutual recognition of testing results. This explains the renewed emphasis on this topic particularly in ISO 17025.

For chemical measurements this involves the need for stated references and a clear uncertainty statement, which should be derived from an uncertainty budget with due regard to the fact that several references, such as amount of substance, mass, volume, time, temperature are generally involved in a single analytical procedure contributing distinct, but different portions to the overall uncertainty.

This uncertainty budget must not only take into account the uncertainties of all the references used in connection with the analytical procedure, but also the uncertainties from the operation of the laboratory procedure as documented in the validation report. The uncertainty from the measurement procedure is frequently much larger than the uncertainties carried by the references.

[1] Traceability is the property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

Background: why traceability

In today´s global society comparable results are needed in order to avoid duplicating measurements which cost time and money. The need for mutual recognition -- the ability to directly and transparently compare results -- explains the emphasis on traceability in ISO 17025 [2]. Comparable results can only be achieved by anchoring them to a common base. In other words we need results traceable to a common base preferably to one with worldwide recognition.

The overall merits of producing and dealing with traceable results of measurement have clearly been acknowledged by the signatories of the Meter Convention whose primary raison-d´être is in fact traceability [3]. It is therefore a central question how this traceability of results is best achieved in chemical measurement. This has to be seen in the light of the two key elements that must be in place for producing traceable values:

- stated and/or internationally agreed suitable references and
- an uncertainty statement for the measurement according to the principles of GUM [4]

whose key role is to enable us to judge the "fitness for purpose" of a result.

Of course, another central question regarding fitness is whether a particular type of measurement is a suitable one for the purpose at all. This is, however, a matter of professional judgement addressed by the choice of an appropriate method.

Merits and Added Value of Traceability for Laboratories and Customers

The value of traceability for laboratories and customers are in many instances closely related to each other. It has to do with the immediate recognition that an accurate value can only be claimed within the limits of the boundaries indicated in the statement on uncertainty. This helps to avoid over-interpretation of the data and gives a clear view on the limits of validity. Failures in traceability potentially undermine the trust in the professional integrity of analytical chemists. Embarrassing results from these failures could be avoided by paying more attention to the nature and limitations of the traceability of references and of the measurement process itself.

When uncertainty is estimated according to GUM it is given as an interval around the result of the measurement and it is fairly straightforward to decide one or more of the following:

a) Is the upper or lower limit of this uncertainty statement close to a statutory or legal limit, or does it reach beyond such a limit ?

b) How much overlap is between the uncertainty statement of similar measurements on the same or another sample ?

c) Do the intervals expressing uncertainty of measurements from different laboratories on the same sample overlap ?

For most purposes it is less important to have a particularly minute uncertainty, but more pertinent to have a good estimate of the uncertainty for answering questions just as the ones mentioned above.

These and similar questions are important in the self-assessment of a laboratory, benchmarking and establishment of confidence in the working relationship with a customer.

Technical Elements of Traceability

A laboratory finds itself typically at the end of the traceability chain. Therefore, in order to produce traceable results it must be able to rely on all the references necessary in the measurement process, as well as on method validation [5]. A prerequisite for supplying traceable results to the customers is therefore that the values of all references are themselves traceable to stated references and are accompanied by a reliable uncertainty statement. The technical expertise of the laboratory as established by accreditation then must ensure the proper use and handling of these references and of the samples. This is generally a matter of training and expertise. It is particularly useful if there is expertise in the development and adaptation of analytical procedures, as this is much needed in the obligatory validation procedure.

Determination of amount of substance often requires measurements of different properties, for example: sample mass, on a balance compared to a mass reference; analyte identity by comparison to a reference, perhaps using a spectrometer and a database of known compounds; and analyte quantitation by comparison to a different reference, perhaps a reference material. Each property of the result should be traceable, and each may contribute uncertainty to the reported result. Thus, claims of traceability of a result must include not only a description of the references and uncertainty budgets for comparison to them, but also a description of the scope of traceability.

In most cases in analytical chemistry one faces the situation that the contribution of uncertainties of the references to measurement uncertainty is small relative to those contributions that come from the measurement process itself. Under such circumstances the results can only be improved by improving the analytical procedure.

Laboratories are urged to concentrate on the measurement process they are operating. This involves a thorough validation process leading to valid results including a realistic statement of measurement uncertainty that also duly accounts for the uncertainty of the relevant references.

If validation is exercised with due regard to traceability it must provide sufficient information for the subsequent estimation of measurement uncertainty. In this manner, a traceability chain is established as part of validation.

Traceability of Values Carried by Reference Materials

The one key to traceability that must be supplied from outside the laboratory is the traceability of values carried by references, especially by certified reference materials. As these values are also established by measurements, the same features required from analytical laboratories also apply for producers of reference materials. Additionally information on the stability and homogeneity of the reference material in form of an expiration date or by equivalent means is required.

The producers of reference materials must be aware that the values they supply are invariably an indispensable link in the traceability chain. They must implement all procedures necessary to provide evidence internally and externally (e.g. by peer review, laboratory intercomparison studies, etc.) that they have met the conditions required for obtaining traceable results at all times.

Conclusions

- Traceability of results and reference values is a central issue in modern laboratory operation. It is not an end in itself, but serves the purpose of achieving a reliable result.
- Traceability of results can only be claimed if results are accompanied by an uncertainty statement based on traceability of all references, chemical and physical, as well as on procedural contributions to uncertainty.
- A result must be "fit for purpose", thus estimation of measurement uncertainty from uncertainties of references and procedures is added value for laboratories and simple when guidelines are followed.

References

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