On the reliability of calibration gases – Laboratory accreditation according to ISO/IEC 17025

Most analytical methods currently used in practice are, in principle, comparison methods. This means that the analyzer must be calibrated before quantitative measurements are possible. In the case of gas analysis, this is generally done by measuring a "zero gas" as well as one or more calibration gases with well characterised composition. Therefore, the final analytical results critically depend on the reliability of the calibration gases used.

Production of calibration gases

With the **manometric method** of filling gas cylinders, the partial pressures of the components are added in accordance with Raoult's Law. This procedure involves measuring the pressure increase in the cylinder during and after the addition of each component of the mixture at a defined temperature. The production tolerance mainly depends on the accuracy of the pressure gauges and temperature measurement. The advantage of this method is its high flexibility: all mixture types can be produced as long as the partial pressure reaches a measurable value. The disadvantage of this method is its systematically higher process uncertainty.

A subsequent analysis of the individual cylinder according to **ISO 6143** (Gas analysis - Comparison methods for determining and checking the composition of calibration gas mixtures) generally allows for a much more accurate determination of the actual value of the component. That is why, in this case, the analytic values and their uncertainties are certified.

In order to realise the full potential of manometric filling, a reliable method for the calculation of the dosage of various components at different pressures is required. For this purpose, Messer uses state of the art equations of state, implemented in a proprietary software package.

Laboratory Mitry-Mory, France

With the **gravimetric method** according to **ISO 6142** (Gas analysis - Preparation of calibration gas mixtures - Gravimetric method), the individual components are weighed in. The mass contents are determined directly, which can then be converted into substance contents (molar fractions). The weighing process is one of the most accurate physical measuring processes known. That is why high precision gas mixtures can be produced by this method. Quantitative control analyses do not, as a rule, achieve the same levels of accuracy. They are used, however, to verify the process parameters. Certificates are issued for the value determined by gravimetric weighing and its uncertainty.

Filling stand in Lenzburg, Switzerland

Traceability

As stated before, most of the analytical methods we use in our production are **comparison methods**. Appropriate measures must be taken to ensure that the results achieved can be traced back by direct comparison to recognised standards. With quantitative **gravimetric mixture production**, this is done by calibrating the scales used with certified weight standards. This means that the results obtained with these scales, i.e. the quantities of gravimetrically produced gas mixtures, can be traced back directly to the national mass standard of the relevant producer country.

Standard weights in Lenzburg, Switzerland

Analytical quantitative analysis is based on the calibration gases used in our laboratories. Only high-precision, gravimetrically produced gas mixtures are used for this, so the results can be traced back to the mass standard again. If possible, these mixtures are also confirmed by means of direct comparison with standard reference material (SRM) from metrological institutes (e.g. **NIST** (National Institute of Standards and Technology, USA), **VSL** (Van Swinden Laboratorium, Netherlands), **BAM** (Bundesanstalt für Materialforschung und –prüfung, Germany), **LNE** (Laboratoire National de Métrologie et d'Essais, France), **METAS** (Metrologie und Akkreditierung Schweiz, Switzerland) or **NPL** (National Physical Laboratory, UK). If quantitative analysis is per- formed by means of a direct comparison with standard reference material, the products are regarded as directly traceable to that standard.

In any case the uncertainty has to be estimated taking into account all significant contributions

The main factors influencing the uncertainty of a given gas mixture are listed below.

Manometric production:

- uncertainties of standards used
- uncertainty of analytical comparison measurement

Gravimetric production:

- the masses of individual components as determined by weighing
- the overall purities of all components of the gas mixture as well as their specific impurities
- the influence of atmospheric conditions on buoyancy, such as temperature, air pressure and air humidity
- mass increase or mass loss of cylinders during the weighing process due to handling
- analysis of the mixture

For calibration gases it is common to state the "expanded uncertainty" (U=k*s, s: standard deviation) with the coverage factor k=2.

All relevant information on the cylinder and on the production and analytical methods used, e.g. the target and actual composition as well as the uncertainties are stated on the certificate of analysis according to **ISO 6141** (Gas analysis - Requirements for certificates for calibration gases and gas mixtures).

In many cases, conversions of gas mixture composition data from one unit to another unit are required (e.g. ppm(v/v) to ppm (mole/mole) or mg/m^3). For this purpose, Messer uses software based on the standard **ISO 14912** (Gas analysis – Conversion of gas mixture composition data).
In addition to the certificate of analysis for calibration gas mixtures from an accredited laboratory the calibration certificate acc. to **ISO/IEC 17025** is issued.

Accreditation according to ISO/IEC 17025

To continuously assure the highest quality of calibration gases produced, a strict quality management system is mandatory. Besides the general quality management system according to **ISO/EN 9001 et seq**. for laboratories, the more comprehensive **ISO/IEC 17025** (General requirements for the competence of testing and calibration laboratories) should be applied. The latter standard also covers the specific requirements of a laboratory.

Calibration Certificate acc. to ISO/IEC 17025

The goal is the reliability of measuring results, which is based on:

- application of standard operating procedures (SOP) for all methods, including the validation of methods and a detailed estimation of measurement uncertainty
- running a proper system for the monitoring of inspection, measurement and test equipment
- traceability of measurement results to SI units or recognised standard reference materials
- participation in national and international round robin tests
- adequate continuing education and training of employees

The "accreditation" of a laboratory is defined as the confirmation by a third party formally stating that an accredited laboratory has the competence to perform certain conformity evaluation tasks according to **ISO/IEC 17025**.

Such conformity evaluations can be performed relative to testing or calibration. The difference between them is that a testing laboratory is approved for the testing of measurement devices or materials; in addition to that, a calibration laboratory is also authorised to produce and certify internationally recognised calibration gases traceable to international standards.

Accreditation means the formal recognition of the technical and organisational competence of an authority to execute a specific service as described in the scope of accreditation. Competence is the key to transparency, confidence and comparability.

Messer has received accreditation for several laboratories all over Europe:

- Messer Schweiz AG, Lenzburg, Switzerland (calibration laboratory)
- Messer France, Mitry-Mory, France (calibration laboratory)
- Messer Hungarogáz, Budapest, Hungary (calibration laboratory)
- Messer Benelux, Machelen, Belgium (calibration laboratory)
- Messer Tehnogas, Belgrade, Serbia (testing laboratory)

The different scopes can be found in the Internet under:

- http://www.seco.admin.ch/sas/
- http://www.cofrac.fr/cofrac_en.htm
- http://www.nat.hu/
- http://economie.fgov.be/organization_market/belac/intro_en.asp
- http://www.ats.org.yu

Certificates of accreditation

Based on these laboratories Messer is able to ensure and continuously improve the quality of accredited (and non-accredited) calibration gas mixtures. External and inter-company round robin tests help to ensure that the compositions and uncertainties of gas mixtures produced at any Messer location are comparable and traceable according to the requirements of our customers throughout Europe.

Service and support

Not only production and analysis of the calibration gas mixtures influence the quality of these mixtures: storage and handling can also have significant effects. The right choice of the gas supply system is as crucial as the careful withdrawal of the calibration gas mixture. Our customer consultants will be happy to help you choose the optimal solution for your specific requirements. We look forward to hearing from you!

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