CCQM-GAWG guidance document for purity analysis and CMC claims

1. Scope

- This document describes the current CCQM-GAWG practice for demonstrating capabilities for purity analysis to ensure that the evidence and these services meet the traceability requirements of the CIPM-MRA.
- Institutes have their own in-house capabilities for purity analysis that are indirectly assessed through successful participation in key comparisons as purity analysis is a critical step in the preparation process of gas reference materials (national primary standards).
- As stakeholders tighten their quality requirements for measurement, impurities in the gas mixture, in some cases, will have a noticeable impact on the final uncertainty of the target component of gas reference materials.
- This document supplements and extends the guidance in the "CCQM-GAWG strategy for comparisons and CMC claims", as an aid for the establishment and review of CMCs.

2. Current practice and recommendations for improvement

2.1. Current practice in the CCQM-GAWG

- **Primary realisation and gravimetric preparation**: the CCQM-GAWG requires metrologically traceable methods (e.g. ISO 6142, ISO 6144, ISO 6145; see also ISO 14167) including in-house purity analysis capabilities for the primary realisation of national primary standards in gas metrology to be applied.
- **Key comparisons** are used to indirectly assess purity analysis capabilities since impurities in the source gases and their uncertainties are determined in a metrologically traceable way and reported in key comparison reports.
- **Purity analysis CMCs for underpinning measurements of impurities in source gases**: For services to customers, institutes need to develop their own purity analysis capabilities and maintain their purity analysis CMCs for underpinning measurements of impurities in source gases. These services can include the calculation or measurement of the content of the most abundant component.
- **The statement of the source of traceability in CMC claims**: If institutes perform their own primary realisation with their in-house purity analysis capability, they can state themselves as the source of traceability in CMC claims with supporting evidence, i.e.

    a) key comparison results
    b) fully described methods for purity analysis, including instrumentation, calibration, and models used to calculate the content of impurities, and, where applicable the content of the most abundant component,
    c) performance evaluation of the method described under b),
    d) results of the method described under b),
    e) a description of the metrological traceability,
    f) a comprehensive uncertainty evaluation in accordance with the guidance of the Guide to the Expression of Uncertainty in Measurement and its supplements.

Unless there is a key comparison which covers the purity analysis offered as a service directly, supporting evidence is required. It is not required that this supporting evidence is in the form of a peer-reviewed article in a scientific journal.

The CCQM-GAWG extrapolation scheme is not applicable to purity analysis, unless the submitter of the CMC can demonstrate otherwise. The rationale behind this statement is simple: only in rare cases, the same instrumentation including calibration standards and methods is used and performance is obtained that is relevant to the purity analysis. Such cases should be indicated by the submitter (if applicable). The said extrapolation scheme has not been designed nor validated for this purpose.

2.2. Recommendations for improvement

- **Impact of impurities on key comparison results**: there are some areas where purity analysis impacts the uncertainty significantly and the purity analysis capability is a barrier to improve concordance (i.e., metrological compatibility). For example, the World Meteorological Organisation (WMO), an important global stakeholder of the CCQM-GAWG, has set data quality objectives for greenhouse gases (such as CO_2, CH_4 and N_2O) for measurements of well-mixed background air in the Global Atmospheric Watch (GAW) network, which are challenging even for National Metrology Institutes. In CCQM-K82 (methane in air at ambient level, 1800 nmol mol^{-1}), the amount fraction of methane in the matrix source gases is a significant contributor to the final uncertainty of the amount fraction of methane in the gas reference materials.
- **CCQM-GAWG approach for purity analysis**: Institutes are expected to quantify impurities and account for the contribution to the final uncertainty of the amount fraction of the target analyte, in accordance with international standards (e.g., ISO 19229). All impurity measurement capabilities for which a CMC is being claimed will be considered to be significant (as defined in ISO 19229) and will require SI traceable purity analysis. Indicative purity analysis is not acceptable for the establishment of purity analysis CMCs. The latter also applies for data included to calculate the amount fraction of the most abundant component.
- **Using key comparisons to claim CMCs for purity analysis**: In order to maximise the utilisation of key comparison results and assist the CMC review process, it is important to identify which key comparisons can be used as evidence, develop protocols to evaluate institutes' purity analysis capabilities, and develop CMC guidance documents for purity analysis CMC claims even at the planning stage.
- **Meeting stakeholder needs for underpinning impurities in source gases:** It is important to underpin the measurements of impurities in source gases due to recent advances in measurement techniques and methods and stringent stakeholder requirements for a better understanding of subtle changes in science and improving manufacturing infrastructure. The CCQM-GAWG proposes new initiatives on meeting stakeholder requirements for purity analysis (e.g., common matrix gases, hydrogen fuels, gases for advanced manufacturing) and analytical challenges (e.g., O_2 and Ar).

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References

[1] Calibration and measurement capabilities in the context of the CIPM MRA (CIPM MRA-G-13)

*In the context of the CIPM MRA and ILAC Arrangement the common definition is: **a CMC is a calibration and measurement capability available to customers under normal conditions**.*

1. *as published in the BIPM key comparison database (KCDB) of the CIPM MRA; or*
2. *as described in the laboratory's scope of accreditation granted by a signatory to the ILAC Arrangement*

[2] Overview and implementation of the CIPM MRA (CIPM MRA-P-11)

*An institute participating in the CIPM MRA shall establish its **metrological traceability route** to the International System of Units (SI) via one of the following routes:*

1. *A primary realization or representation of the unit of measurement concerned. In this case, traceability shall be declared to **its own demonstrable realization of the SI**;*
2. *Another participant having relevant CMCs with appropriate measurement uncertainty published in the KCDB or through calibration and measurement services offered by the BIPM. In this case, traceability shall be declared through the laboratory providing the service.*

***Note 1**: In order for a primary realization or representation of the unit of measurement to be considered valid, it shall be approved by the relevant Consultative Committee.*

***Note 2**: The institute shall make available a full assessment of the measurement uncertainty budget and the traceability route for its measurement activity when submitting CMCs for intra-regional and JCRB review.*

***Note 3**: An institute may use measurement services provided by laboratories accredited by a signatory to the ILAC Arrangement for auxiliary influence quantities that are not part of the main traceability path to the SI and can be shown to make only a minor contribution to the total combined measurement uncertainty of the CMC.*

***Note 4**: Traceability route 1 includes institutes using CRMs or high-purity primary chemical reference materials that have been value-assigned by applying their own measurement capabilities as described and recognized within published CMCs.*

Task Group members

Adriaan VAN DER VEEN, Takuya SHIMOSAKA, Bernhard NIEDERHAUSER, Jennifer CARNEY, Robert WIELGOSZ, Paul BREWER, Sangil LEE