EURAMET project no. 1498

Protocol for pilot study, comparison of 100 µmol/mol hydrogen chloride in nitrogen gas

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Summary

This pilot study is aimed at preparing participants to participate in the key comparison CCQM-KXXX[1] to be conducted under the CCQM. Furthermore, it is aimed at investigating the level of comparability of laboratories' hydrogen chloride in nitrogen gas analytical capabilities at a level of 100 µmol/mol.

Certified reference gas mixtures will be provided by either the Korea Research Institute of Standard and Science (KRISS) and the Physikalisch-Technische Bundesanstalt (PTB). The comparison will be based on two cylinders, each of them analyzed for its hydrogen chloride amount-of-substance fraction by PTB and KRISS.

The report will be written by PTB and agreed with KRISS to subsequently get approved by the EURAMET TC-MC SCGA. The comparison is considered to present a Track C challenge in the CCQM nomenclature.

[1] GAWG19-44: Overview of Current Key Comparisons (https://www.bipm.org/wg/CCQM/GAWG/Restricted/October_2019/GAWG19-44-Overview_of_current_CCQM-GAWG_key_comparisons.pdf)

Purpose and scope

The pilot study between the Physikalisch-Technische Bundesanstalt (PTB) and the Korea Research Institute of Standard and Science (KRISS) will provide a comparison of standards and capabilities for the measurement of 100 µmol/mol hydrogen chloride (HCl) in nitrogen gas (N_2). The participants will verify two HCl certified reference gas mixtures (CRMs) at 100 µmol/mol levels using their own standards. To verify the CRMs, the PTB will use the optical gas standard and the KRISS will use gravimetrically prepared primary reference gas mixtures. The applicability of the optical gas standard will be confirmed by this comparison and diversify standards for validation of HCl.

Measurand, quantities and units

The measurand is the amount-of-substance fraction of HCl in N_2, with measurement results being expressed in mol mol^{-1} (or one of its multiples mmol mol^{-1}, µmol mol^{-1} or nmol mol^{-1}).

Preparation of CRMs

CRMs will be prepared by KRISS, and PTB at a level of 100 µmol/mol HCl in N_2. Both CRMs will be traceable to the SI, either based on own CMCs (KRISS) or certified commercial mixture (PTB). Mixtures are in cylinders with a volume equal or greater than 5 L, with total pressure in the range 100 bar to 150 bar and fitted with a suitable cylinder valve which conforms to one of the standards of DIN.

Verification of stability of CRMs

Participants are recommended to verify the stability of the mixtures before and after shipment due to their own procedures. Typically, the verification of the mixtures will be performed soon after their preparation or purchase. Further investigations of the mixtures may be undertaken over the following months to check the stability of the mixtures. The measurement schedule envisages that the KRISS CRM may be prepared in February 2020 and returned to KRISS in June 2020 after analysis at PTB. PTB will certify its mixture and assign the amount-of-substance fraction to the KRISS CRM in between February and May 2020 and subsequently ship both mixtures to KRISS in June 2020. While PTB has both mixtures in house, they will perform stability checks.

Transport of cylinders

Transport of cylinders to PTB and to KRISS, back to PTB respectively, will be on the individual laboratory's budget, including customs clearance in Germany and Korea.

Measurement schedule

The comparison will be organized by PTB following the schedule below:

February 2020	Shipment of the KRISS CRM from KRISS to PTB
February 2020 – May 2020	First analysis of the KRISS CRM and PTB CRM at PTB
June 2020	Shipment of both CRMs from PTB to KRISS
June 2020 - September 2020	Analysis of both CRMs at KRISS
September 2020	Shipment of PTB CRM from KRISS to PTB

September 2020 – November 2020	Second analysis of PTB CRM at PTB
November 2020	KRISS results submitted to PTB and vice versa,
November 2020	PTB to prepare draft A report, discussion on results with KRISS
December 2020 – February 2021	Finalizing report and discussing results with TC-MC SCGA

Reporting of Results

The PTB is responsible for the preparation of the comparison report. The report has to include the following information where participants are to provide feedback on:

- Instrumentation: analyzers, devices, materials, environment, etc., used.
- Analysis procedure: calibration, analyte handling, number of measurements.
- Uncertainty evaluation: GUM-compliant uncertainty budget based on a model equation (influence quantities, distribution)

How far the light shines statement

This comparison might be used to evidence CMC claims for HCl in N_2 gas in the range of 50 µmol/mol to 200 µmol/mol HCl. In particular, this comparison shall be used as preparation for a participation in the anticipated Key Comparison CCQM-KXXX on HCl in N_2 to be organized in 2021.[2]

[2] GAWG19-44: Overview of Current Key Comparisons (https://www.bipm.org/wg/CCQM/GAWG/Restricted/October_2019/GAWG19-44-Overview_of_current_CCQM-GAWG_key_comparisons.pdf)