Comparison APMP.QM-K90
Formaldehyde in nitrogen at 2 µmol/mol

Protocol and Report Forms

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Background

The comparison of formaldehyde in nitrogen at 2 μmol/mol was done as CCQM-K90 to evaluate the measurement capability of NMIs/DIs.[1)] Since NMIs/DIs in the APMP region requested a comparison for measurement capability of formaldehyde in nitrogen at a few μmol/mol, the comparison of APMP.QM-K90 coordinated by NMIJ and KRISS was proposed and approved in the APMP TCQM meeting on Nov, 2017.

This document describes the protocol of the key comparison APMP.QM-K90 for formaldehyde in nitrogen gas mixture at the nominal amount fraction of 2 μmol/mol.

Measurand, Quantities and Units

The measurand is the amount fraction of formaldehyde in nitrogen. The measurement results will be expressed in mol/mol or one of its multiples.

Transfer Standards

KRISS will prepare the mixtures of formaldehyde in nitrogen in 30-L high pressure cylinders with 13 MPa. The nominal amount fraction of formaldehyde in the mixtures is 2 μmol/mol and source material of formaldehyde is paraformaldehyde. The number of mixtures to be prepared is six. NMIJ will verify the stability of the mixtures in three months (see “Stability Testing” section). The mixtures showing the best stability will be selected as the transfer standards. Number of the other mixtures not to be selected should be two or three and the mixtures will be used to monitor the stability in NMIJ.

Each participating laboratory will be provided with one cylinder of the transfer standard. Nominal pressure at the shipment to the participants is 10 MPa. Type of the cylinder valves is CGA 350 SS threads right male. The participants should return the cylinder with pressure larger than 8MPa.

Participants can request additional information on the cylinders using the registration form annexed to this protocol.

Stability Testing of Transfer Standards

NMIJ will analyse the gas mixtures of formaldehyde by the same method used in CCQM-K90 to test the stability and determine the amount fractions before shipment to the participants. Gas mixtures showing the best stability will be selected as transfer standards to be sent to participating institutes.

NMIJ will also analyse the transfer standards and other gas mixtures after the transfer standards will be returned to NMIJ.

Transport of the Cylinders

It is better that import procedures for cylinders is done by a lab to receive the cylinders because the labs can process the procedures for the import more smoothly. In this comparison, the responsibility of cylinder transport is as follows.

Transport of the cylinders will be arranged by laboratory which receives the cylinders. Namely, transport of the cylinders from KRISS to NMIJ will be arranged by NMIJ. Transport of the cylinders from NMIJ to participants will be arranged by participants. Transport of the cylinders from the participants to NMIJ will be arranged by NMIJ. After this comparison is finalized, shipping of the cylinders to KRSS is responsible to KRISS.

The participants are also responsible for returning the cylinders promptly after completing the measurements. Failure to meet the deadline to return the cylinder may result in a participant not having its results included in the comparison report. The participants must return the cylinders to NMIJ no later than **30th June, 2019**.

Reporting

Participants are expected to report the following information in the Report Form of APMP.QM-K90:

1) the value and uncertainty of the formaldehyde amount fraction in the transfer standard measured by the partipant
2) the date when the measurements were performed.
3) a description of the gas analysis procedure of the participants
4) a complete uncertainty budget; if necessary, with explanation of the way to calculate the uncertainty of the reported value.

Key Comparison Reference Values

The capability of the measurement and the dynamic standards systems at NMIJ was shown in CCQM-K90. This will allow NMIJ to propose Key Comparison Reference Value based either on combined results from participants and/or based on its own measurement capabilities.

Each transfer standard will have its own reference value and associated expanded uncertainty. An expression for the degree of equivalence between each participating institute and the KCRV will be calculated by NMIJ from the comparison results and measurement uncertainties submitted by participating laboratories. The calculation method for the KCRV will be supposed to be similar to that used in CCQM-K90.

Supported claims

The claims supported by this comparison are supposed to be the same as that of CCQM-K90.

Participants

By now the following APMP NMIs/DIs expressed interest in participation in this key comparison.

Participants : CMS/ITRI, KRISS(coordinating laboratory), NMIJ(coordinating laboratory)

Schedule

Date	Event
Feb, 2018	Draft protocol of this comparison
March, 2018	Agreement of Protocol of this comparison
May 31st, 2018	Registration of participants
June to Aug, 2018	Preparation of mixtures
Sept, 2018	Shipment of the cylinders from KRISS to NMIJ
Sept to March, 2019	Stability test at NMIJ
April 1st, 2019	Dispatch of the cylinders to Participants
June 30th, 2019	Return the cylinders to NMIJ
June 30th, 2019	Deadline of submission of measurement report to the coordinating laboratory
July to Oct, 2019	Verification & stability test at NMIJ
Oct to Nov, 2019	Return the cylinders to KRISS
Dec, 2019	Draft A report
Feb, 2020	Draft B report

Coordinators

Coordinating Labs are NMIJ and KRISS.

Contact persons of each Labs are as follows. The registration and report should be sent to Takuya Shimosaka (t-shimosaka@aist.go.jp), NMIJ.

NMIJ:

Coordinator

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References

1) Final report of CCQM-K90: Metrologia, 2017, 54, Tech. Suppl., 08029.

2) N.Aoki et al., Analyst, 2013, Analyst, 138, 6930-6937.

3) N. Aoki et al., Accred. Qual. Assur., 2018, Accepted.

4) N. Panda, et al, Accred. Qual. Assur., 2016, 21(4), 295-304.

Registration Form of APMP.QM-K90

Formaldehyde in nitrogen, 2 µmol/mol

If laboratories wish to participate in the key comparison of APMP.QM-K90, this form should be completed sent **before 1 May 2018** to

Takuya Shimosaka,
National Metrology Institute of Japan (NMIJ), National Institute of Advanced Industrial Science and Technology (AIST)
AIST Central-3, Umezono1-1-1, Ibaraki, 305-8563, Japan
Phone +81 29 861 6851
E-mail t-shimosaka@aist.go.jp

Participating Institute Information:

Institute :

Address :

Contact person :

Telephone :

Fax :

e-mail :

Transfer standard shipment prepration:

Please describe below any additional information required to prepare the shipment.

Report Form of APMP.QM-K90

Please complete and return the form by email to t-shimosaka@aist.go.jp

Comparison Coordinator : Dr. Takuya Shimosaka
National Metrology Institute of Japan (NMIJ), National Institute of Advanced Industrial Science and Technology (AIST)
AIST Central-3, Umezono1-1-1, Ibaraki, 305-8563, Japan
Phone +81 29 861 6851
E-mail t-shimosaka@aist.go.jp

Participant Information:

Institute :

Address :

Contact Person :

Telephone :

Email :

Transfer Standard Information:

Date of Reception of the transfer cylinder :

ID Number of cylinder :

Cylinder pressure as received :

Cylinder pressure before shipment to NMIJ :

Results of measurements:

Final Result

Date of Analysis (DD/MM/YYYY)	**Formaldehyde amount fraction : *x*(HCHCO) / mol mol^{-1}**	**Expanded uncertainty *U*(*x*(HCHO)) / µmol mol^{-1}**	**Coverage Factor**

Measurement results in each day

	Date of Analysis (DD/MM/YYYY)	**Formaldehyde amount fraction : x(HCHCO) / mol mol^{-1}**	**Expanded uncertainty U(x(HCHO)) / µmol mol^{-1}**	**Coverage Factor**
1				
2				
3				
		Add lines if necessary.		

Description of measurements

Please provide below a description of the measurements performed, including the description of national/working standards, analytical instrument(s), handling of the transfer standard, and the calibration procedure followed to deduce the formaldehyde mole fraction in the transfer standard.

Uncertainty budget

Please provide below the uncertainty budget used to calculate the uncertainty associated with the measurement of the formaldehyde mole fraction.

Authors