Comparison of nitrogen dioxide gas standards – Optical and gravimetric method

Pilot Study Protocol

Summary

Nitrogen oxides (NOx) emissions have been regulated by both road and non-road sectors [1] [2]. Nitrogen dioxide (NO_2) is one of the species of NOx and an important pollutant which has negative effects on air quality and human health [3] [4].

In this bilateral pilot study, to evaluate the level of comparability of a newly developed optical NO_2 standard and a classical reference material based NO_2 standard, the amount fractions of NO_2 in gas mixtures are analyzed by the participants – Physikalisch-Technische Bundesanstalt (PTB) and Korea Research Institute of Standards and Science (KRISS). Gas mixtures are provided at around 10 µmol mol^{-1} level NO_2 in N_2.

The report will be written by PTB and agreed by KRISS to get approved by the EURAMET TC-MC Subcommittee Gas Analysis. This pilot study results can be used to prepare and support the participants for potential participation in future comparisons.

Purpose and scope

The aim of this pilot study is, firstly, to evaluate the level of comparability of a newly developed optical NO_2 standard by PTB and a classical reference material based NO_2 standard by KRISS at 10 µmol/mol level. Secondly, to prepare and support the participants for potential participation in future comparisons (e.g. BIPM.QM-K6 regarding 10 µmol/mol NO_2 in N_2).

Quantities and units

The measurand is the amount fraction of nitrogen dioxide in nitrogen. The measurement results being expressed in mol mol^{-1} and its multiples.

Participants

The pilot study is conducted by two laboratories as a bilateral comparison.

- **Coordinator:** Physikalisch-Technische Bundesanstalt (PTB)

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- **Participating partner:** Korea Research Institute of Standards and Science (KRISS)

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Schedule

September 2025	Preparation and analysis of gas mixtures A and B at KRISS
	Analysis of a gas mixture C at PTB
October 2025	Shipment of the gas mixtures A and B from KRISS to PTB
November – December 2025	Analysis of the gas mixtures A, B and C at PTB
January 2026	Shipment of the gas mixtures A, B, and C from PTB to KRISS
February – March 2026	Analysis of the gas mixtures A, B and C at KRISS
April 2026	Shipment of the gas mixture C from KRISS to PTB
May 2026	Analysis of the gas mixture C at PTB
June 2026	Results submission from KRISS to PTB
July 2026	Preparation and distribution of Draft A report by PTB
August 2026	Distribution of Draft B report

Gas mixture preparation and measurement

Two gas mixtures are prepared and provided by KRISS, and one gas mixture is provided by PTB. Gas mixtures are in > 5 L volume cylinders with total pressure in the range of 10-15 MPa. The participants perform the first measurement as soon as they prepare or receive the gas mixture(s). The measurements will be made at the participating laboratories within a time period of one year.

Transport of gas mixtures

Transport of gas mixtures in cylinders between participants, including customs clearance, is carried out at the expense of each laboratory. The participants should ensure that there is enough gas (> 5 MPa) before shipment to the next laboratory.

Reporting results

PTB is responsible for writing the report. The report includes the following information:

- Instrumentation
- Analysis procedure
- Measurement results
- GUM compliant uncertainty evaluation

‘How far the light shines’ statement

The results of this pilot study can be used to support CMC claims for preparative and analytical capabilities for NO_2 in N_2 mixtures in the range from 10 -1000 µmol mol^{-1}.

References

[1] European Commission, Commission Regulation (EU) 2023/443 of 8 February 2023 amending Regulation (EU) 2017/1151 as regards the emission type approval procedures for light passenger and commercial vehicles, https://eur-lex.europa.eu/eli/reg/2023/443/oj

[2] Revised MARPOL Annex VI: Regulations for the Prevention of Air Pollution from Ships and Nox Technical Code 2008. (2009). ISBN: 978-9280142433.

[3] European Environment Agency, González Ortiz, A., Guerreiro, C. and Soares, J., Air quality in Europe – 2020 report, Publications Office, 2020, https://data.europa.eu/doi/10.2800/786656.

[4] World Health Organization, WHO global air quality guidelines: particulate matter (PM2.5 and PM10), ozone, nitrogen dioxide, sulfur dioxide and carbon monoxide, 2021, https://iris.who.int/handle/10665/345329

Report form

EURAMET Project No. 1704: Comparison of nitrogen dioxide gas standards - Optical and gravimetric method

- **Laboratory:**
- **Contact:** Name, Telephone number, E-mail

Results

Measurement result (Cylinder number: xxx)

Analyte	Amount fraction ($\mu mol\ mol^{-1}$)	Expanded uncertainty ($\mu mol\ mol^{-1}$)	Coverage factor, *k*
NO_2			

Description	Date (dd/mm/yyyy)	NO_2 amount fraction ($\mu mol\ mol^{-1}$)	Standard deviation (% relative)	Number of measurements
Preparation				
Measurement 1				
Measurement 2				
Measurement 3				
Measurement #				

Calibration standards

Please provide a description of the calibration standards used for measurement.

- Cylinder type/treatment applied
- Preparation date
- Purity analysis
- Method of preparation
- Verification procedure
- Stability testing (if possible)

Analytical method

Please provide a description of the analytical method used.

- Details on the instrumentation
- Analytical condition and procedure

Uncertainty evaluation

Please provide a description of the measurement uncertainty evaluation.