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GAS ANALYSIS PROFICIENCY TESTING SCHEMES

DESCRIPTION

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Contents

1. INTRODUCTION

The NMISA Gas Analysis Section has been delivering PT and interlaboratory comparisons (ILCs) to the air quality, pollution and emission monitoring industry over the last 13 years.

Participation in Proficiency testing (PT) schemes provides participants with an objective evaluation of the laboratory's technical competence and analytical capabilities. Regular participation in PT is a requirement of ISO/IEC 17025. PT scheme reports can also be used to demonstrate laboratory and staff competence to both clients and accreditation bodies.

Gas PT samples are prepared by NMISA and provided in cylinders containing the target measurands as listed in Table 1. Planned PTs are run annually (Table 2), as communicated in this document, and on our website. Those PTs not planned for the current year, may be arranged on request as an interlaboratory comparison (ILC) with NMISA.

Homogeneity and short-term stability of the NMISA PT gas mixtures are checked prior to shipment. After the PT deadline cylinders must be returned to NMISA for verification and completion of the confidential PT result report.

Table 1: PT scheme measurand, amount fraction range and expanded relative uncertainty (%REU) offered at gas analysis section

#	**Description**	**Amount Fraction**	**%REU**
1	Methane in nitrogen	1 to 10 % mol.mol^{-1}	0.5
2	Carbon dioxide in nitrogen	1 to 10 % mol.mol^{-1}	0.5
3	Carbon monoxide in nitrogen	100 to 1000 µmol.mol^{-1}	0.5
4	Sulphur dioxide in nitrogen	100 to 1000 µmol.mol^{-1}	0.5
5	Ozone	0 to 500 nmol.mol-1	3
6	Hydrogen sulphide in nitrogen	100 to 1000 µmol.mol^{-1}	1.5
7	Hydrogen sulphide in nitrogen	10 to 100 µmol.mol^{-1}	3
8	Sulphur dioxide in nitrogen	10 to 100 µmol.mol^{-1}	1.0
9	Nitrogen dioxide in nitrogen	10 to 100 µmol.mol^{-1}	3.6
10	Ethanol in nitrogen	50 to 550 µmol.mol^{-1}	1.0
11	Nitrogen dioxide in nitrogen	100 to 1000 µmol.mol^{-1}	2
12	Nitrogen oxide in nitrogen	10 to 100 µmol.mol^{-1}	1
13	Nitrogen oxide in nitrogen	100 to 1000 µmol.mol^{-1}	2

Note: the gas analysis laboratory offers multicomponent proficiency testing schemes on request.

2. ACCREDITATION STATUS

All NMISA PT schemes in the Gas Analysis Section are conducted in accordance with the ISO/IEC 17043 (2023) standard: conformity assessment – general requirements for proficiency testing.

3. COMMUNICATION

NMISA has appointed key personnel and management representatives responsible for delivering the PT schemes. Contact details of the PT coordinator, the logistics administrator and technical manager are given below. Where applicable alternative scheme coordinators are communicated for each PT.

Scheme coordinators

Ms Mudalo Jozela/ Ms Nompumelelo Leshabane/ Ms Portia Seemane/ Mr Mphara Mogale

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Technical manager

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4. SCHEDULE

4.1. Shipment of PT samples

Shipment of the PT samples to and from the participant will be arranged by NMISA. NMISA will notify the participant when the cylinder is ready for shipment. A participant may also opt for self-collection at NMISA premises. NMISA subcontracts the transportation of PT samples to qualified courier companies that meet safety and quality requirements. On receipt of the cylinder's participants are required to complete delivery notes and submit these to NMISA.

4.2. PT scheme schedule and registration

The cost for a single participation in planned NMISA PTs are listed in **Table 2**. Including delivery and collection. Requests for custom PT/ILC not planned in 2024/25 are priced on request. Registration is confirmed upon receipt of a Purchase Order or proof of payment received through the NMISA customer portal.

Table 2 Planned PT schemes and ILCs schedule for 2024/2025

	Description	Amount Fraction	Registration deadline	Dispatch date	Result submission deadline	Collection date	Final Report	Cost (Rands) 0% VAT
1.	**Methane in nitrogen**	1 to 10 % mol.mol^{-1}	5 July 24	15 Oct 24	12 Nov 24	15 Nov 24	24 Jan 25	R 9 700
2.	**Carbon dioxide in nitrogen**	1 to 10 % mol.mol^{-1}	5 July 24	29 Oct 24	26 Nov 24	29 Nov 24	31 Jan 25	R 9 700
3.	**Carbon monoxide in nitrogen**	100 to 1000 µmol.mol^{-1}	5 July 24	15 Oct 24	12 Nov 24	15 Nov 24	24 Jan 25	R 9 700
4.	**Sulphur dioxide in nitrogen**	100 to 1000 µmol.mol^{-1}	5 July 24	15 Oct 24	12 Nov 24	15 Nov 24	24 Jan 25	R 9 700
5.	**Sulphur dioxide in nitrogen**	10 to 100 µmol.mol^{-1}	5 July 24	29 Oct 24	26 Nov 24	29 Nov 24	31 Jan 25	R 9 700
6.	**Hydrogen sulphide in nitrogen**	10 to 100 µmol.mol^{-1}	5 July 24	29 Oct 24	26 Nov 24	29 Nov 24	31 Jan 25	R 11 000
7.	**Ethanol in nitrogen**	10 to 100 µmol.mol^{-1}	5 July 24	15 Oct 24	12 Nov 24	15 Nov 24	24 Jan 25	R 11 000

	Description	Amount Fraction	Registration deadline	Dispatch date	Result submission deadline	Collection date	Final Report	Cost (Rands) 0% VAT
8.	**Hydrogen sulphide in nitrogen**	100 to 1000 µmol.mol^{-1}	5 July 24	31 Jan 25	28 Feb 25	03 Mar 25	25 Mar 25	R11 000

Please note that the ozone ILCs will be scheduled between October to December upon request (Cost: R 9 700).

5. PT SCHEME MIXTURES

5.1. Preparation method and homogeneity assessment

Cylinders containing gas mixtures of the measurand with similar nominal amount fractions will be prepared gravimetrically by NMISA according to ISO 6142-1 (2015). The mixtures will be verified against NMISA primary standard gas mixture (PSGM). The pressure in a cylinder will be approximately 9.0 MPa at the time of dispatch; a cylinder of 10 or 5 dm^3 nominal volume may be used.

5.2. Stability of mixtures

Stability is defined as the ability of a reference material, when stored under specified conditions, to maintain a stated property value within specified limits for specified period. The stability study of gas mixtures aims to determine the degree of instability of gas mixtures after preparation or to confirm the stability. Before the PT sample is sent to the participant the short-term stability of the cylinder is assessed by verifying the mixtures for a minimum of three measurements over a month duration. The samples are verified when they are returned to NMISA after the PT deadline. According to our historical data our PT samples are stable for a minimum of twelve months.

6. REPORTING

6.1. Result submission

An electronic result report template is provided to participants. Participants are required to not make any modifications to the given template. The completed measurement result report must be submitted by the indicated deadline.

Participants will be given a month to complete their measurements after the PT sample distribution date. Results received after the deadline will not be processed or included in the report. In case of foreseeable delays, participants are requested to report such delays to the scheme coordinator in advance, with the expected result submission date. New deadlines, if extension request is granted, will be communicated to all participants.

After the measurements are completed, the participant should return the cylinder with a sufficient amount of the gas in the cylinder (> 50 bar) to the NMISA for re-verification analysis.

6.2. Measurement results

Laboratories will be required to submit results for at least three independent measurements taken from each cylinder, which is obtained under repeatability conditions with at least three independent calibrations, e.g., calibration − > measurement − > calibration − > measurement − > calibration − > measurement. This is required for proper statistical analysis of the reported data.

6.3. Measurement uncertainty

The reported amount fraction of the measurand should have uncertainty estimates. The measurement uncertainty should be evaluated according to the GUM (ISO/IEC GUIDE 98-3:2008). For example,

- method of evaluation (type A or Type B)
- (assumed) probability distribution)
- standard uncertainties and sensitivity coefficients

The participant will be responsible for the calibration of their equipment according to the routine procedures applied in their own laboratory. To ensure a proper evaluation of the data, it is necessary that the calibration method, as well as how the calibration mixtures are prepared, should be reported to the NMISA. This information is needed for the evaluation of the preparation facilities as an integral part of this comparison.

7. STORAGE AND HANDLING OF PT SAMPLES

Store the cylinder away from direct heat between temperatures of 10 to 40 °C in a well-ventilated area. More guidance on the use of calibration gas mixtures can be found in ISO 16664 (Gas Analysis – Handling of calibration gases and gas mixtures – Guidelines). PT measurements must be completed in a similar manner to routine measurements to allow accurate evaluation of participant measurement capabilities that are representative of daily operational performance.

8. SAFETY

The cylinder should be handled with care and by experienced personnel in a laboratory environment suitably equipped for the safe handling of gases materials.

9. PERFORMANCE EVALUATION

9.1. PT scheme reference value

The amount-of-substance fraction (in µmol.mol^{-1}) for each individual mixture obtained from the weighing process and the purity verification of the parent gases will be provided by NMISA. The NMISA metrologically traceable gravimetric preparation value will be used as the PT scheme assigned reference value.

The uncertainty associated with the reference value is calculated by combining gravimetric uncertainty and verification uncertainty. The PT scheme uncertainty for each measurand will be calculated from the percentage relative expanded uncertainty. The percentage relative expanded uncertainty (%REU) is different for each measurand based on the NMISA's calibration and measurement capabilities for each component and amount fraction and are listed in **Table 1**.

9.2. Evaluation using En-score

The E_n numbers or normalised errors are used to determine the conformance and non-conformance of participating laboratories in proficiency schemes where the uncertainty in the measurement result from both the laboratory reported value and reference value are compared using the E_n equation below:

$$E_n = \frac{x_{lab} - x_{Ref}}{\sqrt{U_{lab}^2 + U_{Ref}^2}} \quad (1)$$

Where:

$E_n = the\ E_n\ score$

$x_{lab} = participant\ laboratory\ result$

$x_{Ref} = reference\ value$

$U_{lab} = is\ the\ expanded\ uncertainty\ of\ a\ participant's\ results\ (x)$

$U_{Ref} = is\ the\ expanded\ uncertainty\ of\ the\ reference\ laboratory's\ assigned\ value\ (x)$

I. |En| ≤ 1.0 the score indicates "satisfactory" performance.
II. |En| > 1.0 the score indicates "unsatisfactory" performance.

10. PT REPORT

An electronic confidential PT report will be provided within 1 month of the PT deadline. Where needed alternative timeframes may be communicated to participants. Unique codes will be allocated to each participant in the report to ensure confidentiality of the results.

11. APPEALS

Participants will be given an opportunity to review the draft report within 2 weeks for transcription errors. Thereafter the final report is issued. Participants who wish to appeal their results may direct their queries to the Scheme Coordinator. If necessary, the appeal may be escalated to the technical manager. Where applicable a revised report may be issued.

12. REFERENCES

ISO/IEC 17025:2017 *General requirements for the competence of testing and calibration laboratories*

ISO/IEC 17043:2023 *Conformity assessment - General requirements for proficiency testing*

Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM : 2008)

ISO 6142-1 *Gas analysis - Preparation of calibration gas mixtures - Part 1: Gravimetric method for Class I mixtures*

Eurachem guide: Selection, use and interpretation of proficiency testing (PT) schemes. 2011. 2nd Edition.