PROTOCOL FOR EURAMET KEY COMPARISON EURAMET.M.P-K15.2 IN GAS MEDIA IN THE RANGE FROM 0.3 mPa TO 1 Pa (ABSOLUTE MODE)

EURAMET TC-M PROJECT 1580

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1. GENERAL INFORMATION

The objective of the comparison is to assess the degree of equivalence of the absolute pressure standards of the participating laboratories in the pressure range from 10^{-4} Pa to 1 Pa, by circulating a pair of spinning rotor gauges (SRGs) as transfer standard.

This comparison was decided at the EURAMET TC-M Subcommittee Pressure meeting in October 2021 and confirmed at the EURAMET TC-M Subcommittee Pressure meeting in September 2022, following a request from INRIM, that was established for the role as pilot. PTB acts as co-pilot. METAS provides the link to the key comparison CCM.P-K15 [1].

PTB provides the transfer standard, consisting of two spinning rotor gauges, and one control electronic unit.

The comparison will be carried out following a loop scheme in which the transfer standard is characterised by the pilot laboratory before and after the set of measurements performed in each loop.

1.1 Scope

Two spinning rotor gauges (SRGs) will be characterised by means of the pressure standards of each participating institute in the range from 0.3 mPa to 1 Pa [2]. The measurements carried out by each participating laboratory will allow the determination of the accommodation factors of the SRGs at each foreseen measurement point, in order to compare the absolute standard pressures generated by each participant.

1.2 Participants

In table 1, the participants with their addresses and responsible persons are reported.

Laboratory	**Address**	**Person responsible**
CEM, Spain	Centro Español de Metrología C/Alfar nº 2. 28760 Tres Cantos (Madrid), SPAIN	Dr. David Herranz phone: +34 918074710 e-mail: dherranz@cem.es Dr. Carmen García Izquierdo phone: +34 918074789 e-mail: mcgarciaiz@cem.es
CMI, Czech Republic	CMI Okruzni 772/31 63800 Brno, Czech Republic	Dr. Martin Vičar phone: +420 734 411 406 mvicar@cmi.cz dprazak@cmi.cz
EMI, United Arab Emirates	Krypto Labs Building, Masdar City, Abu Dhabi, UAE	Mr. Brian Justice phone +971 (0)2 406 6540 e-mail: brian.justice@qcc.gov.ae

INRIM, Italy pilot	Istituto Nazionale di Ricerca Metrologica Strada delle Cacce, 91 10135 Torino - Italy	Dr. Domenico Mari phone: +39 011 3919914 e-mail: d.mari@INRIM.it
LNE, France	Laboratoire national de métrologie et d'essais 1 rue Gaston Boissier 75724 Paris Cedex 15 France Shipping address: 23 avenue Albert Bartholomé 75015 Paris France	Dr. Frédéric Boineau Phone: +33 1 40 43 38 08 e-mail: frederic.boineau@lne.fr
METAS, Switzerland	METAS Office federal de métrologie Lindenweg 50 3003 Bern-Wabern	Dr. Christian Wüthrich Christian Phone: +41 58 387 04 23 e-mail: christian.wuethrich@metas.ch
PTB, Germany	Physikalisch-Technische Bundesanstalt Abbestr. 2-12 10587 Berlin - Germany	Dr. Matthias Bernien phone: +49 30 3481 7230 e-mail: matthias.bernien@ptb.de
RISE, Sweden	RISE Research Institutes of Sweden Brinellgatan 4 50462 Borås, Sweden	Mr. David Szabo phone: +4610 516 5586 e-mail: david.szabo@ri.se

Table 1 – List of Participants

Table 2 shows the applied methods and the status of the participant's standards at the time of the comparison.

Laboratory	**Standard**	**Definition**	**Traceability**
CEM, Spain	Continuous expansion system; static expansion system	Primary	Independent
CMI, Czech Republic	Continuous expansion system	Primary	Independent
EMI, United Arab Emirates	Leybold CS7 Vacuum Calibration System	Secondary	CMI, Czech Republic
INRIM, Italy pilot	Continuous expansion system; static expansion system	Primary	Independent
LNE	Continuous expansion system; comparison with SRG and CDG	Primary	Independent
METAS, Switzerland	Static expansion system	Primary	Independent
PTB, Germany	Continuous expansion system; static expansion system	Primary	Independent
RISE, Sweden	Static expansion system	Primary	Independent

Table 2. Applied methods and status of the participant's standards

2. TRANSFER STANDARD

The transfer standards consist of a pair of SRGs kept under vacuum using an all-metal valve. The rotor of the SRGs is immobilised by a spring during the transport and kept under vacuum:

a stainless-steel spring is mounted on the plate of the valve to immobilise the ball once the valve is closed, during the transportation of the transfer standard.
The valve should be opened as far as it stops and then closed by a quarter of a turn clockwise. It should be noted that when the valve is open, the spring is far enough from the ball to avoid spurious drag eventually due to an electromagnetic coupling between the ball and the spring through the magnetic field of the ball. The specifications of the transfer standard are listed in the table 3; some of these specifications have not been obtained by means of measurements: in such case they are conventional values to be used in order to determine the accommodation coefficient.

Transfer Standard	**SRG1**	**SRG2**
SRG ID	7	8
Valve part number (Varian)	9515014	9515014
Valve Serial Number	80492	81023
Volume, valve open	18.5 cm^3 (u= 1.0 cm^3)	18.5 cm^3 (u= 1.0 cm^3)
Rotor diameter (nominal)	4.762E-3 m	4.762E-3 m
Rotor density (nominal)	7.715E3 kg/m^3	7.715E3 kg/m^3
Rotation frequency (provided readout unit)	min: 405 Hz max: 415 Hz	min: 405 Hz max: 415 Hz

Table 3. Specifications of the transfer standards

One electronic readout unit and one measuring head will be circulated with the transfer standard. The provided readout unit works at rotation frequency between 405 Hz and 415 Hz. The participating laboratories can use this readout unit or their own unit for one SRG, but will need to supply their own unit for the other SRG. The following list (table 4) shows the items that will circulate:

Object	**Type/Serial number**
SRGs mounted on all metal valves	see Table 3
Measuring Head (MKS)	SRG SH700/91882 G
Electronic unit (MKS)	SRG-2-IEEE488/20546
Torque wrench (Tonichi)	1200QL-N/01937B

Table 4. Packing list

3. TIME SCHEDULE

The transfer standards will circulate for the comparison in accordance with the schedule in the Table 5. The laboratory that ships the transfer standard must contact the ship-to laboratory and the pilot laboratory prior to the shipment to make sure that the transfer standard can be received without problem. The receiving laboratory shall inspect the material immediately upon arrival: if there is any damage that could prevent the measurements, this must be communicated to the pilot laboratory, and the pilot lab will advise on the next course of action. The equipment will be accompanied by an ATA carnet where necessary. Each lab is responsible for its own costs of the measurements, the costs of transportation to the next participant, and any custom charges within its country as well as any damage that may occur within its country.

1	*INRIM / Italy*	*27 February 2023 - 14 April 2023*
2	METAS / Switzerland	17 April 2023 - 19 May 2023
3	PTB / Germany	22 May 2023 - 23 June 2023
4	*INRIM / Italy*	*26 June 2023 - 28 July 2023*
5	CMI / Czech Republic	31 July 2023 - 01 September 2023
6	RISE / Sweden	04 September 2023 - 06 October 2023
7	LNE / France	09 October 2023 - 10 November 2023
9	*INRIM / Italy*	*13 November 2023 - 15 December 2023*
10	CEM / Spain	08 January 2024 - 09 February 2024
11	EMI / United Arab Emirates	12 February 2024 - 15 March 2024
12	*INRIM / Italy*	*18 March 2024 – 19 April 2024*

Table 5. Time schedule

4. MEASUREMENTS

4.1 Operating conditions

The temperature of the system shall be monitored and the mean temperature should be (22±2) °C. The measurement can be performed at the rotational speed of the ball in the range 405 Hz to 440 Hz, depending on the used readout unit.

4.2 Pressure medium

Nitrogen gas with at least 99.99% purity.

4.3 Measurand

The measurand of the comparison is the effective accommodation factor of the rotor. The accommodation factor [3-7] is determined during the comparison according to the following equation:

$$\sigma = \left(\frac{-\dot{\omega}}{\omega} - RD(\omega)\right)\frac{\pi d\rho}{20 p_{st}}\sqrt{\frac{8RT}{\pi m}} \quad (1)$$

Where

p_{st}	generated pressure
RD	residual drag at “zero pressure” due to temperature drift and eddy currents in the rotor and in the surrounding metal materials
ω	rotational speed of the rotor
$\dot{\omega}$	rate of change of rotational speed of rotor
d	nominal diameter of the rotor
R	gas constant (R=8.314463 J mol^{-1} K^{-1})
ρ	nominal density of the rotor
m	molar mass of the gas
T	temperature
$\frac{\dot{\omega}}{\omega}$	relative retardation of the sphere per unit time or deceleration rate (DCR).

The effective accommodation coefficient is determined using the nominal rotor diameter and rotor density given in the description of the transfer standard. The accommodation coefficient is known to be constant in the molecular regime (target pressure points ≤ $3\cdot10^{-2}$ Pa) and decreases as the pressure increases in the continuum regime (target pressure points > $3\cdot10^{-2}$ Pa).

For the target points > 0.03 Pa, σ is significantly dependent on pressure, therefore each laboratory shall calculate a linear fit function of the effective accommodation coefficient σ versus the target pressures, for the three data points at 0.09 Pa, 0.3 Pa and 1 Pa. The slope of the fit will allow each participant to calculate an accommodation coefficient for the common value of a pressure, even if the target pressures were not exactly matched during the calibrations.

The measurement shall be performed with a rotational speed between 405 Hz and 440 Hz.

4.4 Residual Drag determination

The RD (residual drag) and its uncertainty is an important influence factor that has to be determined carefully, especially for measurements performed at low pressure.

Two methods are possible to measure the residual drag, and the participants are free to choose the one that is the most adapted to their instrumentation. For each method, the uncertainty of the measurement shall be estimated.

Technique 1: Determination of *RD(ω)* as a function of rotation speed before or after the calibration.

In this technique the rotor is brought into rotation and the deceleration is measured under vacuum at near or less than $1 \cdot 10^{-6}$ Pa. The measurement is repeated throughout the range of rotation speed. A linear fit of the residual drag versus the rotational speed is carried out and used to correct the measured drag at the target pressures. For each pressure measurement, the relative deceleration rate (DCR) and the rotational frequency must be recorded (please refer to the manuals of the controller). This technique requires a high temperature stability of the laboratory over a long period of time due to the dependence of RD on temperature drift.

Technique 2: Determination of *RD* during the calibration.

The residual drag *RD* is measured immediately before and after the measurement of the deceleration when the rotor is exposed to the pressure and the mean of the two values is used for calculation of the indicated pressure. The *RD* could be determined during the course of the calibrations by pumping down the vacuum system to residual pressure conditions after each target pressure point. Please note that *RD* is changing because of its frequency dependence (some 10E-10 DCR per Hz). Special care must be taken that there is no acceleration of the rotor during the recording of values. Such accelerations are indicated with the letter “M” if the internal printer is used. This technique is preferable because the residual drag is a function of the temperature and this influence parameter is expected to remain stable during the small time span needed for the two measurements of the deceleration.

4.5 Care of the transfer standard

A few important aspects have to be considered in order to perform a stable and reproducible measurement and to ensure the stability of the transfer standard for all the time of the comparison:

- Vacuum conditions: the rotors must be transported under vacuum conditions; the all-metal valves associated to each SRG shall be opened only when the pressure in the calibration chamber is less than 0.1 mPa.
- Do not bake the SRGs: the SRGs cannot be baked during the comparison.
- Rotation: the rotor shall be in rotation only when it is necessary; the rotation shall start at least 12 hours before the beginning of the measurements.
- Magnetic fields: do not expose the rotors to a strong magnetic field.
- Closing torque of the valves of the SRGs: 6 Nm
- Integrity of the transfer standard: Do not modify the SRGs. Do not disassemble the thimbles from the valves of the SRGs

- Use an UPS (uninterrupted power supply): it is recommended to use an UPS between the mains and the controller, as a power failure can crash the rotor and change the accommodation factor.

4.6 Calibration procedure

The calibration of the SRGs will be performed at the following 8 values of nominal target pressure of nitrogen in this order:

$3 \cdot 10^{-4}$ Pa, $9 \cdot 10^{-4}$ Pa, $3 \cdot 10^{-3}$ Pa, $9 \cdot 10^{-3}$ Pa, $3 \cdot 10^{-2}$ Pa, $9 \cdot 10^{-2}$ Pa, $3 \cdot 10^{-1}$ Pa, 1 Pa.

The pressure should be within ±10% of the nominal pressure for pressures lower ≤ $3 \cdot 10^{-2}$ Pa and it should be within ±5% for higher pressures.

At each target point in each calibration sequence, the accommodation coefficient has to be measured three times. Therefore, each calibration sequence is composed by $8 \cdot 3 = 24$ points.

If possible, it is recommended that both SRGs are calibrated at the same time.

The calibration sequence will be repeated at least once, making a total of at least 48 measurement points for one SRG.

The temperature of the system shall be monitored and should be (22±2) °C.

The readings of the SRGs shall be acquired according to the following sampling intervals and number of repetitions:

Target pressures	Sampling interval	Number of measurements at each target pressure
$3 \cdot 10^{-4}$ Pa, $9 \cdot 10^{-4}$ Pa, $3 \cdot 10^{-3}$ Pa	30 s	10
$9 \cdot 10^{-3}$ Pa, $3 \cdot 10^{-2}$ Pa, $9 \cdot 10^{-2}$ Pa	30 s	5
0.3 Pa, 1 Pa	10 s	5

At the end of the measurement the ball has to be properly decelerated in order to avoid any change of the accommodation due to rubbing of the ball on the wall of the tube. The deceleration procedure is described in the manual of the controller used. In the case of the circulated SRG-2 controller press the “STOP” button and wait until the red LED turns on. The valve can be closed while the SRG is still under a vacuum better than $1 \cdot 10^{-5}$ Pa.

5. COLLECTION OF DATA AND REPORT OF RESULTS

The pilot laboratory will provide a template to make the communication of the results and relevant information uniform.

All pressure values shall be given in the unit of Pa.

All pressure values shall be given with 5 significant digits, and absolute uncertainties should cover the value's last digit.

Uncertainties shall be given as standard uncertainties (*k*=1), according to GUM.

The participants shall communicate the results of the calibration to INRIM within 6 weeks after the measurements have been completed.

Beside the results and information provided through the distributed template, each participant shall send the following additional information to the pilot laboratory:

- A short description of your standard (if it is published, send the publication) including a description how the measurements were performed (positions of gauges, which option was used for offset measurement etc.).
- Date of receipt of the transfer standard set in your institution.
- Notes of inspection of the package (i.e. damage and completeness).
- Dates and times when the SRG valve was open.

6. EVALUATION OF THE CALIBRATION RESULTS AND GENERATING A COMMON REFERENCE VALUE

The effective accommodation factor σ is pressure dependent for > $3\cdot10^{-2}$ Pa: this dependency may affect the comparison accuracy, when the target pressures are not achieved exactly. To take into account this effect, a linear fit through the 9 measurement pressure points above $3\cdot10^{-2}$ Pa shall be used to calculate σ_i at the exact target pressures, according to:

$$\sigma_i = (\sigma_i)_{det} + (p_t - p_{st,i})m \qquad (2)$$

Where:

- p_t are the nominal target pressures at 0.09 Pa, 0.3 Pa and 1 Pa;
- $p_{st,i}$ are the generated pressures of institute i near each target pressure p_t;
- $(\sigma_i)_{det}$ are the values determined by the calibration at $p_{st,i}$.

7. RELEVANT UNCERTAINTIES

All uncertainties shall be provided as (one-sigma) standard uncertainties as outlined in the GUM. The reading of an SRG depends on the temperature determination, the determination of the offset RD, a possible frequency dependence of the offset, a possible drift of the offset value between its determination and the time of calibration, and the type A uncertainty of the measurement results. The uncertainty of the temperature shall be reported to the pilot lab. In most cases, however, the uncertainty due to temperature is relatively small.

The uncertainty of the calculated generated pressure $p_{st,i}$ has to be determined by each lab at the time of calibration.

The uncertainty of σ_i will be determined from the uncertainty of T, RD and $p_{st,i}$ at the time of calibration.

8. REFERENCES

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