Protocol of comparison of three national vacuum standards $3 \cdot 10^{-4}$ Pa to 1 Pa with SRG as transfer standards (EURAMET project 1405)

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1. General Information

To fulfill the requirement of the technical peer review of the Vacuum Laboratory, National Institute of Metrology Thailand (NIMT) by Dr. Karl Jousten (PTB) during 9th to 10th March 2016, NIMT should perform an inter-comparison with another NMI in the range of 10^{-6} mbar to 10^{-3} mbar (10^{-4} Pa to 10^{-1} Pa) by the end of 2017. The objective of the comparison is therefore to comply with the aforementioned requirement by the determination of the relative agreement between the absolute pressure standards of PTB and NIMT via the effective accommodation factor σ_{eff}.

Also UME announced in 2016 the need to check equivalence of its re-established static expansion system in the range 10^{-4} Pa to 1 Pa with a primary standard that took part in recent CCM.M.P-K14 in the same range.

CEM has also shown interest in checking equivalence of their static expansion system in the range 10^{-4} Pa to 1 Pa.

Since PTB took part in CCM.P-K15 which is the linking comparison for the same range, PTB were selected aside from NIMT, UME and CEM.

Scope:

A Transfer Standard (TS) which is a Spinning Rotor Gauge (SRG) shall be characterized with the pressure standards (PS) of each participating institute in the range of 300 µPa to 1 Pa. From these measurements, the accommodation factor of the SRG can be determined at each measurement point in order to compare the generated pressures of the Calibration Standards (CS).

Pilot Institute:

Physikalisch-Technische Bundesanstalt, Berlin, Germany

2. Participating Institutes

Four National Metrology Institutes (NMIs) from the APMP and EURAMET region participate in this comparison. The participating institutes along with addresses and responsible persons are listed in Table 1.

Table 1: List of participating institutes

Laboratory	Address	Responsible Persons
PTB, Germany	Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany	Dr. Karl Jousten Tel: +49-30-3481-7262 Fax: +49-30-3481-7490 karl.jousten@ptb.de
NIMT, Thailand	National Institute of Metrology (Thailand) 3/4-5 Moo 3, klong 5, Klong Luang, Pathumthani 12120, Thailand	Mr. Suwat Phanakulwijit Tel: +66-2577-5100 ext. 2106 Fax: +66-2577-3658 suwat@nimt.or.th
UME, Turkey	TUBITAK UME, TUBITAK Gebze Yerleskesi, P.K. 54,	Dr. Rifat Kangi Tel: +90 262 679 5000

	41470 Gebze/KOCAELI, Turkey	Fax: +90 262 679 5001 rifat.kangi@tubitak.gov.tr
CEM, Spain	Centro Español de Metrología C/ Alfar 2 28760 Tres Cantos, Madrid , Spain	Dr. David Herranz Dr. Nieves Medina Tel: +34 91 8074789 Fax: +34 91 8074807 dherranz@cem.minetur.es mnmedina@cem.minetur.es

3. Time Schedule

The time schedule for the measurements is given in Table 2.

Table 2: Measurement schedule

No.	Period of Measurement	NMIs
1	March, 2017	PTB
2	April, 2017	NIMT
3	May, 2017	UME
4	June, 2017	PTB
5	July, 2017	CEM

The measurement's equipment will be accompanied by an ATA carnet. Each participant is responsible for the costs of transportation to the following recipient.

4. Transfer Standards

Two spinning rotor gauges (SRG) supplied by PTB are used as a transfer standards (TS) of the comparison. The TS are kept under vacuum using an all-metal angle valve to reduce drifts during transportation. One SRG controller (MKS SRG3) supplied by UME will also be circulated.

Table 3: Specifications of the transfer standards

Transfer Standard #1	Spinning Rotor Gauge (SRG)
Manufacturer	MKS
Part Number	SRG1
Serial Number	9
Ball Diameter (Nominal)	4.762 mm
Ball Density (Nominal)	7715 kg/m³
Rotation Frequency	Min: 430 Hz Max: 440 Hz
Valve Manufacturer	Varian
Valve Serial Number	535T
Volume, Valve Open	18.5cm³
Transfer Standard #2	Spinning Rotor Gauge (SRG)
Manufacturer	MKS
Part Number	SRG2
Serial Number	12
Ball Diameter (Nominal)	4.762 mm
Ball Density (Nominal)	7715 kg/m³
Rotation Frequency	Min: 430 Hz Max: 440 Hz

Valve Manufacturer	Varian
Valve Serial Number	83845
Volume, Valve Open	18.5cm³

5. Comparison Measurements

Pressure Medium

Nitrogen gas with 99.99% purity shall be for the calibration.

Operating Conditions

The temperature of the system shall be monitored and the mean temperature should be 23±2 °C. The measurement shall be performed at the rotational speed of the ball between 430-440 Hz (The factory default value).

Measurand

The accommodation coefficient of the ball is the measurand of the comparison. The measurand will be calculated from the following equation [1,4]:

$$\sigma = \left[-\frac{\dot{\omega}}{\omega} - RD(\omega)\right]\frac{\pi\, d_b\, \rho_b}{20\, p}\overline{c} \qquad (1)$$

where, the average molecular speed, $\overline{c}$ is calculated from:

$$\overline{c} = \sqrt{\frac{8\,R\,T}{\pi\, m}} \qquad (2)$$

and

p	is the generated pressure,
RD	is the residual drag at zero pressure due to temperature drift and eddy currents in the ball and in the surrounding metal materials,
ω	is the rotational speed of the ball,
$\dot{\omega}$	is the rate of change of rotational speed of the ball,
d_b	is the nominal diameter of the ball,
R	is the gas constant ($R = 8.314472$ J mol^{-1} K^{-1}, $U(R) = 0.000014$ J mol^{-1} K^{-1}),
ρ_b	is the nominal density of the ball,
m	is the molar mass of the gas (Nitrogen),
T	is the gas temperature,
$-\frac{\dot{\omega}}{\omega}$	is the relative retardation of the sphere per unit time or the deceleration rate (DCR).

The accommodation coefficient's behavior is depended on the gas flow regime as follow:

Flow Regime	Behavior	Trend	Procedure
The Molecular Flow	Constant	-	-
The Continuum Flow	Depend on Pressure	Inverse to Pressure	Linear Fit: σ vs p

For the target points **≤ 3·10^{-2} Pa**, where the flow around the ball is the molecular flow, the accommodation coefficient is independent of pressure. The value of the target pressure p_{st} could be used as the nominal target pressure without any problem.

For the target points > **3·10^{-2} Pa**, where the flow around the ball is the continuum flow, the accommodation coefficient is significantly dependent on the pressure. Therefore, each laboratory shall calculate a linear fit function for three data points at **9·10^{-2} Pa, 0.3 Pa, and 1 Pa** of the accommodation coefficients σ versus the target pressures p_{st}. 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.

Residual Drag Determination

Residual Drag $RD(\omega)$, which is a function of rotational speed of the sphere, is required in the calculation of equation (1). To determine $RD(\omega)$, there are two methods as follow:

Method 1: Determination of $RD(\omega)$ before or after the calibration

The approximation equation of $RD(\omega)$ as a function of the rotational speed ω could be found from the plot of RD versus ω. To do the plot, the measurements of RD shall be performed under 10^{-6} Pa over the entire range of ω. This method requires a high temperature stability of the laboratory over a long period of time due to the dependence of RD on temperature drift.

Method 2: Determination of $RD(\omega)$ during the calibration

The $RD(\omega)$ could be determined during the course of the calibrations by pumping down the vacuum system to residual pressure conditions after each target pressure point so that the residual drag could be measured immediately before and after the measurement.

In both cases a linear square fit had to be applied to obtain the function $RD(\omega)$.

Method 2 is the preferable option and is recommended to be used to check the data obtained from Method 1.

Procedure to Ensure the Accommodation Coefficient Factor "Stability"

The stability of the measurand which is the accommodation coefficient is important in the measurement. Therefore, the following procedures shall be followed:

- The rotor shall be transported under vacuum

- The SRG will not be baked during the comaprison
- The rotor shall not be exposed to a strong magnetic field
- The rotor shall be in rotation only when needed for calibration
- SRG should start rotation 12 hours before the measurement

Calibration Procedure

The calibration is performed on a SRG at the following 9 nominal target pressures p_t in ascending 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, 0.3 Pa, 1 Pa.

A tolerance of ± 10% in hitting the nominal pressure is accepted for $p_t < 9 \cdot 10^{-2}$ Pa, and ± 5% for $p_t \geq 9 \cdot 10^{-2}$ Pa.

Each target pressure has to be generated 3 times. This means that after a measurement at the target point, the system is pumped down to residual pressure conditions and the same point re-generated. In total 8·3 = 24 points are to be measured in this way and considered as one calibration sequence. It is required that this calibration sequence be repeated at least once on another day.

The readings of each of the SRGs were to be sampled in the following manner:

Target Pressure Points (Pa)	Interval (s)	Number of Measurements at Each Target Point
$3 \cdot 10^{-4}$, $9 \cdot 10^{-4}$, and $3 \cdot 10^{-3}$	30* or 20**	5
$9 \cdot 10^{-3}$ Pa, $3 \cdot 10^{-2}$, $9 \cdot 10^{-2}$	30*or 20**	3
.3 and 1	10*or 20**	3

The interval time of PTB/UME/CEM(*) and NIMT(**) (NIMT's standard cannot be adjusted during the calibrations)

Thereby a stable and known pressure $p_{std,i}$ has to be established in the standard of institute i near each target pressure p_t of the SRG (over the interval indicated in the table above). The reading pressure of the TS, p_i can be determined from the measure DCR value.

Comparison Parameter

The effective accommodation coefficient σ_{eff} which is used as a comparison parameter will be calculated from the following equation:

$$\sigma_{\text{eff},i} = \frac{p_i}{p_{\text{std},i}} \tag{3}$$

where,

p_i is the reading pressure of institute i which is calculated from DCR value,
$p_{\text{std},i}$ is the pressure generated in the national standard of institute i.

6. Report of Results

A template will be provided. Each participant shall report the results of the calibration to PTB within 4 weeks after the measurements were completed.

Pressure values shall be given in Pascal (Pa) with 5 significant digits. The uncertainties shall be given as one standard uncertainty (k = 1), as according to GUM.

7. Evaluation of the calibration results

Since σ_{eff} is pressure dependent for > **3·10^-2 Pa**, which may affect the comparison accuracy when the target pressures are not achieve exactly, a linear fit through the 9 measurement points above $3 \cdot 10^{-2}$ Pa shall be used to calculate σ_i at the exact target pressures in the following manner:

$$\sigma_i = (\sigma_i)_{det} + (p_t - p_{std,i})m \qquad (4)$$

where,

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

8. Relevant Uncertainty

According to GUM, all uncertainties should be given for the coverage factor k = 1 as one sigma standard uncertainties.

The uncertainty of the generated pressure $p_{std,i}$ has to be determined by the particpants at the time of calibration “including” the uncertainties of Type A (random uncertainties).

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

9. References

[1] Karl Jousten, Mercede Bergoglio, Anita Calcatelli, Jean-Noel Durocher, John Greenwood, Rifat Kangi, Jean-Claude Legras, Carmen Matilla, Janez Setina: Results of the regional key comparison Euromet.M.P-K1.b in the pressure range from $3 \cdot 10^{-4}$ Pa to 1 Pa, Technical Supplement to Metrologia, 2005, 42, Tech. Suppl. 07001.

[2] Christian Wüthrich, Karl Jousten: Protocol for the CCM comparison CCM.M.P-K14 for absolute pressures in the range from 100 µPa to 1 Pa, 2009

[3] Karl Jousten: Protocol for the Euromet comparison phase B in the range 10^{-4} Pa up to 1 Pa (Euromet Project 442), 2000

[4] Karl Jousten: Handbook of Vacuum Technology, John Wiley & Sons, 2008