The preliminary results of the regional key comparison COOMET.M.P-K15 in the pressure range from 0.3 mPa to 0.9 Pa

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Abstract. Within the COOMET region a regional key comparison (COOMET.M.P-K15) was carried out in order to compare national vacuum standards in the pressure range from 0.3 mPa to 0.9 Pa. The participants were D.I. Mendeleiev Institute for Metrology VNIIM (Russia) and TÜBİTAK Ulusal Metroloji Enstitüsü UME (Turkey) as pilot laboratory. The measurements were carried out from May 2017 to November 2018. Two spinning rotor gauges were used as transfer standards. The effective accommodation coefficients of the rotors had to be determined at eight target points between 0.3 mPa and 0.9 Pa. The uncertainty of the generated pressure in the calibration standard was reported as part of the calibration report by each laboratory. All additional uncertainties that were related to the transfer standard were evaluated by the pilot laboratory in order to have a uniform uncertainty analysis for both participants and in order to emphasize the importance of the reported uncertainty of the generated pressure. Preliminary results have been presented in this paper.

1. Introduction

This comparison is based on the visit of specialists from D.I. Mendeleev Institute of Metrology of the National Institute of Metrology of Turkey (UME) from September 28 to October 2, 2015. The purpose of the comparison is to confirm the required level of accuracy for pressure measurements and to ensure the uniformity of low absolute pressure measurements in the participating countries.

The objective of the comparison is therefore to comply with the requirement by the determination of the relative agreement between the absolute pressure standards of UME and VNIIM via the effective accommodation factor $\sigma_{\text{eff}}$ using transfer standard spinning rotor gauge (SRG).

Two spinning rotor gauges (SRG) were characterised with the pressure standards of each participating institute in the range from 0.3 mPa to 0.9 Pa. From these measurements, the accommodation factor of the SRG was determined at each measurement point by which the generated pressures in the standards could be compared.

2. Participating laboratories and their standards

The UME’s pressure standard (MSSE1) is based on the static expansion method (also called serial expansion method), whereby the range is extended to the lower pressures by multiple-expansion type. The apparatus consists of 6 vessels that provide a pressure reduction by a factor of about $10^8$ in the main calibration vessel after four-step expansion and of 17 platinum resistance thermometers.
mounted on the vessels that are used for temperature effects correction. The MSSE1 system is used to measure medium and high vacuum within the range of $1 \cdot 10^{-4}$ Pa to $1 \cdot 10^{3}$ Pa absolute pressure. It consists of six known volumes, three small volumes of nominally 0.15 L, 0.15 L and 0.7 L and three expansion chambers, 15 L, 15 L and 72 L nominally. With this primary standard the Boyle–Marriott law is applied: A known small volume of gas at a known relatively high pressure is expanded into a previously evacuated larger volume (under isothermal conditions). This generates a pressure drop which depends on the initial and final volumes’ ratio. The expanded ($k = 2$) relative uncertainties of pressures generated by this new standard range from $2.1 \cdot 10^{-3}$ at $9 \cdot 10^{-4}$ Pa to $9.5 \cdot 10^{-4}$ at $1 \cdot 10^{3}$ Pa. The system is described in more detail in reference [1].

**Table 1.** List of participants in alphabetic order and the standards used for the calibration of the transfer standards.

| Laboratory | Standard | Character of standard | Traceable to: | CMC listed |
|------------|----------|-----------------------|---------------|------------|
| TÜBİTAK Ulusal Metroloji Enstitüsü (UME), Turkey | Static expansion system | Primary | Independent | Yes |
| D.I. Mendeleyev Institute for Metrology VNIIM (Russia) | 1. Compensation type membrane-capacitance manometer | Primary | Independent | Yes |
| | 2. Dynamic expansion system | Primary | Independent | Yes |

The VNIIM’s pressure standard (GET 49-2016) is based on the two methods: continuous expansion method and on the method of compensation of pressure by capacitance diaphragm manometer. Continuous expansion system is used to measure medium and high vacuum within the range of $1 \cdot 10^{-4}$ Pa to $1 \cdot 10^{-2}$ Pa absolute pressure. The VNIIM’s continuous expansion system is based on the expansion of pure gas, which is pumped through a fixed conductance. The effective pumping speed is computed from the values of calculated conductance and the ratio of the conductance to the pumping speed is periodically measured. The expanded ($k = 2$) relative uncertainties of pressures generated by this system range from $6 \cdot 10^{-6}$ to $5 \cdot 10^{-4}$.

To generate absolute pressure in the medium and high vacuum range within of $1 \cdot 10^{-2}$ Pa to 10 Pa compensation type capacitance diaphragm manometer system has been used. Compensation principle is based on balancing between the pressure force and electrostatic force acting on the plates of flat capacitor. The expanded ($k = 2$) relative uncertainties of pressures generated by this standard range from $5 \cdot 10^{-4}$ to 0.14. The system is described in more detail in reference [2].

3. **Transfer standards**

Two spinning rotor gauges (SRG) supplied by UME and VNIIM were used as transfer standards (TS) in the comparison. One SRG controller (MKS SRG-2CE) supplied by UME was also circulated. UME SRG finger was sealed with a special all metal valve which had two functions:

1. To seal the rotor in the finger so that it could be transported under vacuum.
2. To fix the rotor during transportation so that the surface would not be changed due to milling and friction effects of the rolling ball.

Transfer Standards (TS) which are Spinning Rotor Gauges (SRG) were characterized with the pressure standards (PS) of each participating institute in the range of 0.3 mPa to 0.9 Pa, also with measuring flange SRG-BF with thimble, mounting rails and retaining screw for measuring head.
From these measurements, the accommodation factors of the SRG were determined at each measurement point in order to compare the generated pressures of the Calibration Standards (CS). The appearance of the transfer standards are shown in Figure 1.

4. Calibration constant
The value to be calibrated by each laboratory $j$ for each pressure and for each rotor $i$ was the effective accommodation coefficient $\sigma_{ij}$ [3], often called $\sigma_{eff}$, which is mainly determined by the tangential momentum accommodation coefficient of the gas molecules to the rotor, and partly by the energy accommodation factor [3] and additionally by using nominal values for diameter and density of the rotors instead of the real ones.

$\sigma_{ij}$ was determined by the following equation:

$$\sigma_{ij} = \sqrt{\frac{8kT_j}{\pi m}} \cdot \frac{\pi d_i \rho_i}{20p_{stj}} \left( \frac{\omega}{\omega_0} - RD_i(\omega) \right). \tag{1}$$

Herein $p_{stj}$ is the generated pressure in the standard, $T_j$ the temperature of gas in the calibration chamber, $d_i$ and $\rho_i$ are the (nominal) diameter and density of the rotor $i$, $m$ is the molecular mass of nitrogen, $(\dot{\omega}/\omega_0)$, also called DCR, is the relative deceleration rate of the rotor frequency $\omega$, and $RD$ is a pressure independent residual drag, caused by eddy current losses in the surrounding metal structures and the rotor itself.

$RD$ is generally a function of $\omega$, $RD(\omega)$, so that it was required that, whenever $\sigma_{ij}$ was determined, the value of $\omega$ also had to be measured in order to subtract the correct $RD(\omega)$ in Eq. (1).

For measuring $RD(\omega)$ mentioned below option offered by the comparison protocol was used.

Before starting the calibrations the rotor frequency dependence of the residual drag (in unit DCR = s$^{-1}$) was measured over a long period of time. The rotor frequency had to cover the full range that may occur during a calibration, that is normally $\omega/2\pi = 430$ s$^{-1} \ldots 440$ s$^{-1}$. For this, the residual pressure in the standard had to be below 10$^{-6}$ Pa.

The determination of $RD(\omega)$ was considered as part of the calibration, because it affects its accuracy, and was the responsibility of each laboratory.

It is well known [3] that in the molecular regime up to about 3·10$^{-2}$ Pa $\sigma_{eff}$ is pressure independent. For this reason, it was clear, a priori, that any pressure dependencies are likely to be due to measurement errors or problems of the calibration standard.

5. Calibration procedure and results to be reported
The following calibration procedure was agreed upon before the comparison: Each laboratory was to calibrate the two SRGs at the following 8 nominal target pressures $p_t$ for nitrogen pressure in ascending order: 0.3 mPa, 0.9 mPa, 3·10$^{-3}$ Pa, 9·10$^{-3}$ Pa, 3·10$^{-2}$ Pa, 9·10$^{-2}$ Pa, 0.3 Pa, 0.9 Pa.

A tolerance of $\pm 10\%$ in hitting the nominal pressure was accepted for $p_t < 9\cdot 10^{-2}$ Pa and $\pm 5\%$ for 9·10$^{-2}$ Pa, 0.3 Pa, 0.9 Pa. Each target pressure had to be generated 2 times. This meant that after a
measurement at the target point, the system was pumped down to residual pressure conditions and the
same point re-generated. In total 8·2 = 16 points were measured in this way and were considered as
one calibration sequence. It was 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:
- 5 repeat points at 30 s intervals for the target points 3·10^{-3} Pa, 9·10^{-2} Pa, and 3·10^{-1} Pa.
- 3 repeat points at 30 s intervals for 9·10^{-2} Pa, 3·10^{-2} Pa, 9·10^{-5} Pa.
- 3 repeat points at 10 s intervals for 0.3 Pa and 0.9 Pa.

For two reasons it was agreed that no bake-out should be performed with the rotors:
1) Bake-out is a time consuming factor and would make it impossible to have a period of one
month for each laboratory.
2) \( \sigma_{eff} \) may change after a bake-out.

Since \( \sigma_{eff} \) is pressure dependent for \( \rho > 3·10^{-2} \) Pa, which may make the comparison inaccurate,
when the target pressures are not hit exactly, it was agreed that a linear fit line through the 6 points
\( \sigma_i(p_{stj} \approx 3·10^{-2} \) Pa), \( \sigma_i(p_{stj} \approx 0.3 \) Pa), and \( \sigma_i(p_{stj} \approx 0.9 \) Pa) would be used to calculate \( \sigma_{ij} \) at the exact
target pressures in the following manner:

\[
\sigma_{ij} = (\sigma_i)_{det} + (p_i - p_{stj}) \cdot m_i, \tag{2}
\]

\( p_i \) are the nominal target pressures 0.09 Pa, 0.3 Pa, 0.9 Pa, \( p_{stj} \) the generated pressures close to
\( p_i \), \( (\sigma_i)_{det} \) the values determined by the calibration at \( p_{stj} \), and \( m_i \) the slope of the fit line for rotor \( i \).

At the end of this calibration procedure, for each generated \( p_{stj} \) near the respective target point and
for each rotor \( i \) and for each of the calibration sequences a value for \( \sigma_{ij} \) existed and was reported to the
pilot laboratory. With the value of \( \sigma_{ij} \) each laboratory reported the standard uncertainty \( u(p_{stj}) \) of \( p_{stj} \).

6. Reported results of the laboratories
Since each laboratory carried out 2 calibration sequences it was possible to check if significant
changes could be observed between the \( \sigma_{ij} \) for the different sequences. Such a change could be
important for the comparison for two reasons:
1) If instability in the calibration constant of a transfer standard occurred during the calibrations in
a single laboratory, it could be taken into account by normalization. If only one SRG showed a change
in value it is highly probable that such instability was detected.
2) If the primary standard showed instability, it is highly probable that both \( \sigma_{ij} \) and \( \sigma_{2j} \) would show
a shift of same size in the same direction. This could help the participant to improve their standard.

Fortunately, no such changes of the calibration constant of the transfer standards were found during
the 3 pairs of calibration sequences. Therefore the mean value of all data for a single rotor and single
target pressure could be taken for data reduction:

\[
\sigma_{ij} = \frac{1}{n} \sum_{k=1}^{n} \sigma_{ijk}, \quad n = 6 \tag{3}
\]

**Table 2.** The mean values \( \sigma_{ij} \) of the reported results for Rotor 1 and the uncertainties
as calculated from Eq. (4).

| \( p_i \) Pa | UME 1 | VNIIM | UME 2 |
|-------------|-------|-------|-------|
|              | \( \sigma_i \) | \( u(\sigma_i) \) | \( \sigma_i \) | \( u(\sigma_i) \) | \( \sigma_i \) | \( u(\sigma_i) \) |
| 3.00E-04    | 0.9783 | 0.0124 | 0.9754 | 0.0688 | 0.9714 | 0.0120 |
| 9.00E-04    | 0.9798 | 0.0095 | 0.9627 | 0.0521 | 0.9643 | 0.0073 |
| 3.00E-03    | 0.9769 | 0.0069 | 1.0142 | 0.0276 | 0.9716 | 0.0052 |
| 9.00E-03    | 0.9744 | 0.0035 | 1.0976 | 0.0290 | 0.9721 | 0.0029 |
| 3.00E-02    | 0.9731 | 0.0029 | 0.9652 | 0.0298 | 0.9722 | 0.0028 |
| 9.00E-02    | 0.9725 | 0.0026 | 0.9672 | 0.0272 | 0.9715 | 0.0024 |
| 3.00E-01    | 0.9688 | 0.0030 | 0.9627 | 0.0175 | 0.9667 | 0.0025 |
| 9.00E-01    | 0.9566 | 0.0017 | 0.9546 | 0.0069 | 0.9568 | 0.0017 |
The results reported by each laboratory and the corresponding uncertainties according to Eq. (4) are shown in the following tables and figures.

\begin{equation}
 u_{\sigma_{ij}}^2 = \frac{n-1}{n} s_{\sigma_{ij}}^2 + \left( \frac{\delta \sigma_{ij}}{\delta R D_i} \right)^2 u_{R D_i}^2 + \left( \frac{\delta \sigma_{ij}}{\delta T_j} \right)^2 u_{T_j}^2 + \left( \frac{\delta \sigma_{ij}}{\delta \rho_{stj}} \right)^2 u_{\rho_{stj}}^2
\end{equation}

where \( s_{\sigma_{ij}}^2 \) is the square of the standard deviation of the mean of the repeat measurements \( \sigma_{ijk} \) and where we understand that all standard uncertainties \( u \) are due to systematic effects which do not contribute to the scatter of \( \sigma_{ij} \).

![Figure 2. Graphical presentation of the results of Rotor 1 (table 2).](image)

7. Stability of transfer standards
In order to monitor the transport stability of the calibration constant of the two rotors during the course of the comparison, the mean values of \( \sigma_1 \) and \( \sigma_2 \) between \( 9 \cdot 10^{-4} \) Pa and \( 3 \cdot 10^{-2} \) Pa of the pilot lab were calculated. Table 3 shows the results. Rotor 3 was the check standard that did not travel.

Table 3. The mean values of \( \sigma_1 \) and \( \sigma_2 \) between \( 9 \cdot 10^{-4} \) Pa and \( 3 \cdot 10^{-2} \) Pa for the two UME calibrations and the respective scatter (standard deviation \( s \)) of the mean. Rotor 3 with the value \( \sigma_3 \) did not travel and served as check standard for UME.

|       | \( \sigma_1 \) | \( s (\sigma_1) \) | \( \sigma_2 \) | \( s (\sigma_2) \) | \( \sigma_3 \) | \( s (\sigma_3) \) |
|-------|----------------|------------------|----------------|------------------|--------------|------------------|
| UME4  | 0.9765         | 0.003            | 0.9846         | 0.003            | 0.9668       | 0.003            |
| UME7  | 0.9731         | 0.002            | 0.9868         | 0.004            | 0.9649       | 0.002            |

Since the measured changes between UME1 and UME2 are within the standard deviation of each mean value, it is reasonable to assume that both effective accommodation coefficients did not changed during this bilateral comparison.

8. Conclusions
In the present comparison, the degree of equivalence between pressures generated by the vacuum primary standards of VNIIM and UME was tested. Preliminary results have been presented in this
paper. Ongoing works on the uncertainty calculations will be completed in near future.

Both standards were fully equivalent in the most part of the pressure range except the range between $3 \times 10^{-3}$ Pa and $9 \times 10^{-3}$ Pa. As a cause of non-equivalence in the specified range VNIIM identified a small leakage in the orifice flange that has been observed and confirmed immediately after having finished of the measurements.

References

[1] Kangi R, Ongun B and Elkatmis A 2004 *Metrologia* **41** 251–6
[2] Gorobei V N, Izrailov E K, Kuvandykov R E, Fomin D M and Chernyshenko A A 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **387**
[3] Fremerey J K 1982 *Vacuum* **32** 685–90