Calibration of measuring instruments of low absolute pressures

A I Antsukova, V N Gorobei, A B Liubomirov, A A Pimenova and A A Chernyshenko
D. I. Mendeleev Institute for Metrology (VNIIM), 190005, St. Petersburg, Russia

E-mail: a.i.antsukova@vniim.ru, V.N.Gorobey@vniim.ru, a.b.lubomirov@vniim.ru, a.a.pimenova@vniim.ru, vacuum@vniim.ru

Abstract. The article presents some sections of the method of calibration of measuring instruments of low absolute pressures (vacuum), used in the research department of state standards in the field of pressure measurement of D. I. Mendeleev Institute for Metrology (VNIIM), the design and composition of the reference SI used in installations of the 1st and 2nd categories are described, the procedure for determining the basic metrological characteristics and evaluation of calibration results is shown.

1. Introduction
Currently, the procedure of calibration of measuring instruments (hereinafter referred to as MI) of low absolute pressures is increasingly in demand.

Calibration of MI – a set of operations that establish a relationship between the value obtained by this MI and the corresponding value determined by the standard in order to determine the metrological characteristics of the MI [1].

According to the Federal Law "On ensuring the unity of measurements" MI, not intended for use in the field of state regulation of ensuring the unity of measurements, may be voluntarily subjected to calibration. MI calibration is performed using the standards of units of quantities traceable to the state primary standards of the relevant units of quantities, and in the absence of the relevant state primary standards of units of quantities – to the national standards of units of quantities of foreign states (chapter 4, article 18 of the Law [2]).

It should be noted that the results of calibration of MI can be used in the verification of MI including in the field of state regulation to ensure the unity of measurements.

2. Calibration tools
In the research department of state standards in the field of pressure measurements (NIO 231) of D. I. Mendeleev Institute for Metrology (VNIIM) (hereinafter – VNIIM) for verification and calibration of vacuum gauges reference installations are applied: the reference vacuum pressure reduction installation VOU-1 of 1th grade and the vacuum reference 2nd grade installation UVE-3, traceable to the national primary special standard unit of pressure to areas of low absolute pressure in the range of 1·10^{-6} to 1·10^3 Pa (GET 49-2016).
Reference MI in installations of the 1st and 2nd grades receive unit size in accordance with the Testing scheme from the GET 49-2016 and provide high precision of measurement results during the playback of the unit and the transfer of its size.

Design and composition of the reference MI used in the 1st and 2nd grade installations provide the constancy of the size of the unit of pressure in time. At the same time, the transmission of the unit size over the entire measurement range is provided by two reference MI mutually overlapping the measurement range from $1 \times 10^{-3}$ to 1 Pa.

The composition and mutual arrangement of the main elements of the reference units of the 1st and 2nd grade installations must comply with the schemes given in the passport of the units and the attached operational documentation (hereinafter – OD). The measuring chamber shall be designed so that the gas distribution in the measuring volume is sufficiently uniform in space and stable in time. Measuring chambers of installations shall be in the form of spheres or cylinders with a diameter of not less than 0.1 m and the ratio of length to diameter 0.7–1.5. The volume of the measuring chamber shall be not less than 10 times greater than the total volume of all the MI (transducers) attached to it, but not less than $2 \times 10^{-3}$ m$^3$. The material of the calibration chamber shall be selected so that the residual pressure limit $P_0$, determined by the speed of the pump $q_v$ and the total degassing rate $Q_{out}$, in the calibration chamber (no leakage) is low enough to perform the calibration. The reference installation of the 1st and 2nd grades have the metrological characteristics given in table 1.

**Table 1. Reference calibration tools and their calibration capabilities.**

| Name | Pressure range | Uncertainty, error |
|------|----------------|--------------------|
| Reference vacuum pressure reduction installation VOU-1 of 1th grade | $1 \times 10^{-7}$–$1 \times 10^{-3}$ Pa | Limits of permissible relative error of not more than $\pm(7$–$3)$ %. Total standard uncertainty in the range: $1 \times 10^{-7}$–$1 \times 10^{-2}$ Pa: 3.43 %; $1 \times 10^{-2}$–$1 \times 10^{-1}$ Pa: 0.88 %. |
| Vacuum reference 2nd grade installation UVE-3 | $1 \times 10^{-5}$–$1 \times 10^{-3}$ Pa | Limits of permissible relative error in the range: $1 \times 10^{-5}$–$1 \times 10^{-3}$ Pa: 15 %; $1 \times 10^{-3}$–$1 \times 10^{-1}$ Pa: 10 %. Combined standard uncertainty 4.3 %. |

**Table 2. Auxiliary MI.**

Name of the auxiliary MI and technical devices

1. Measuring chamber with valves and vacuum fittings, for vacuum pump and turbomolecular pump.
2. Control MI: convection sensor vacuum gauge of thermal modification CVM211 or any resistance vacuum gauge APG.
3. The device of heating of the measuring chamber on the basis of a two-core heating cable with switching equipment.
4. Manual vacuum leak valve with the function of the valve to provide a gas inlet into the vacuum chamber of the type GW-J2.
5. Thermometer-hygrometer IVA-6N-D.

To reduce the uneven distribution of molecules (pressure) due to sorption, self-pumping of MI, degassing, etc., the pipeline connecting the measuring chamber, reference and calibrated MI, should be as short as possible and have an area not less than the area of the flow section of the inlet flange of MI. In cases where the calibrated MI has a significant thermal load on the measuring chamber, the length of the pipeline can be increased to reduce the effect of thermal conductivity. Care should be taken to ensure that the simultaneous use of reference and calibrated MI does not lead to significant mutual influence and does not affect the stability of their work.
The location of the reference installation in the room shall not give rise to air flows resulting in significant cooling or heating of the ambient air flow, calibrated and reference MI.

3. Determination of basic metrological characteristics

During determining the metrological characteristics – range (transformation) pressure and the main relative error of measurement (transformation) of the calibrated MI the following operation should be performed.

Attach the calibrated vacuum meter (transducer) to the reference vacuum installation. The orientation of the vacuum gauge (transducer) in space must take into account the instructions in the corresponding OD. When performing MI calibration operations, you must strictly follow the manufacturer's instructions, unless otherwise specified.

Reference and calibrated MI with the dependence of the readings on the gas density in the measuring chamber should have correction factors used to obtain significant results. This is especially important for MI with an ionization converter, with a sensing element using the effect of vacuum thermal conductivity, and for some resonance frequency devices, where the vibrating element is located directly in the working environment. The correction factor for the various gases is given in OD on MI.

Pump out the measuring chamber of the reference vacuum measuring unit to the limit of the residual pressure $P_o$ associated with the lower limit $p_{\text{min}}$ range of measurement (conversion) calibrated MI with the ratio:

$$P_o = \alpha p_{\text{min}}, \quad (1)$$

where $\alpha$ – coefficient which value is selected from table 3.

**Table 3.** The value of the coefficient $\alpha$ during the calibration of MI.

| Maximum permissible basic relative error of the calibrated MI, % | Value of the coefficient $\alpha$ during the calibration of MI |
|---------------------------------------------------------------|----------------------------------------------------------|
| $\pm(4–6)$                                              | $< 0.01$                                          |
| $\pm(6–15)$                                             | 0.02                                              |
| $\pm(15–30)$                                           | 0.03                                              |
| $\pm(30–50)$                                           | 0.05                                              |
| $> |\pm30|$, and at pressures less than $10^{-6}$ Pa        | 0.1                                               |

If necessary, the measuring chamber and the calibrated MI should be warmed up. Methods and modes of heating should be specified in the OD of the reference installation and calibrated MI.

Vacuum meters (transducers) must be switched on when the pressure in the measuring chamber reaches the operating conditions of the corresponding MI (after decontamination by heating during the cooling phase). Vacuum meters (transducers) and their measuring units must be warmed up and stabilized. The stabilization time depends on the type of vacuum gauge and the required uncertainty.

Some types of vacuum meters require "degassing" at a certain pressure, which must be provided during the stabilization period.

If vacuum meters (transducers) are exposed to vacuum by means of a shut-off valve, it should only be opened when the pressure in the calibration chamber is below the expected pressure value in the vacuum meter (transducer).

After receiving the gas pressure in the measuring chamber below or equal to the limit residual pressure $P_o$ with the help of a leaker, the gas supply to the measuring chamber is regulated. In the pressure range from $p_{\text{min}}$ to $P_o$ ($P_{\text{min}}$ and $P_{\text{max}}$ – lower and upper limits of the measuring range given in the passport of the calibrated MI) discretely set the pressure values at the selected calibration points, placing them in order of increasing pressure with an intensity of at least five points within each decade of the pressure range. The calibration is produced in the whole range of measurements. The definition
of the metrological characteristics of the calibrated MI is produced by a direct comparison with the readings of the reference MI of the reference installation.

During calibration, at least three series of independent measurements are taken over the entire range. Calibration of vacuum meters that do not affect the pressure and composition of the residual gas (for example, thermal and deformation vacuum meters) at pressures above 1 Pa is allowed to be made in static mode, i.e. without continuous pumping of the chamber, at the time of establishing the required calibration pressure in the measuring chamber. In other cases, the calibration should be performed in dynamic mode, i.e. with continuous pumping of the camera.

The static method means that the valve connecting the vacuum system to the measuring chamber is closed and the gas is fed into the chamber until the required pressure value of the calibration point is reached. The dynamic equilibrium method is provided when the inlet valve connecting the measuring chamber and the vacuum system is fully open or partially closed. The gas is fed into the measuring chamber until the required pressure value of the calibration point is reached.

After establishing a constant pressure at the calibration point for 1 minute, fixed by the immutability of the reference vacuum meter readings, it is necessary to register both the reference and calibrated MI readings. The measurements were carried out in accordance with the instructions contained in OD to them. When calibrating a multichannel vacuum meter, read the readings on all channels in series. When calibrating a vacuum meter (transducer), the metrological characteristics of which are normalized by the analog output, the readings are counted by the corresponding controller connected to the vacuum meter (transducer), or by connecting a digital multimeter to the analog output.

In calibration, affecting the composition and pressure of the residual gas (e.g., ionization vacuum gauges and magnetdischarge in complex with closed probes), before the measurement should be controlled the gas dynamic balance in the volume of the converter in accordance with OD on the reference vacuum installation.

After completion of the measurements at the desired control pressure value, perform the pumping of the vacuum system of the reference installation, to ensure that in the process of calibration, there have been no unacceptable leaks, significant adsorption of impurities to the surface of the camera or failure of the pumping system, etc. System should reach a limiting residual pressure for a period of not more than 10 minutes. If it takes more than 10 minutes for the system to be pumped out, the vacuum system must be brought into working order, reconfigured (for example, leak test, pump test, purge, warm-up), and then the calibration must be repeated.

4. Processing of the calibration results

On the basis of the records of measurements made in each series of measurements for each value of the pressure calibration point it is necessary to establish the value of the relative error of the calibrated MI. The calculation is based on the formula:

$$\Delta_{\text{rel}(i)j} = \frac{P_{(i)j} - P_{(o)j}}{P_{(o)j}}$$  \hspace{1cm} (2)

where $\Delta_{\text{rel}(i)j}$ – relative error of the calibrated MI in the $i$-th pressure calibration point in the $j$-th measurement series; $P_{(o)j}$ – readings of standard instruments in the $i$-th calibration point pressure; $P_{(i)j}$ – readings of the calibrated MI at the $i$-th pressure calibration point; $j = 1...n$, where $n$ – number of independent measurement series.

The average value of the relative error of the calibrated MI at the $i$-th pressure calibration point is determined:

$$\overline{\Delta_{\text{rel}(i)}} = \frac{1}{3} \sum_{j=1}^{n} \Delta_{\text{rel}(i)j}$$  \hspace{1cm} (3)

The experimental standard deviation of the measurement result at the $i$-th calibration point is calculated by the formula:

$$S_{\text{rel}}^{2}(p_{i}) = \frac{1}{n-1} \sum_{j=1}^{n} (\Delta_{\text{rel}(i)j} - \overline{\Delta_{\text{rel}(i)}})^{2}$$  \hspace{1cm} (4)

where $S_{\text{rel}}(p_{i})$ – is the mean square value of the measurement result at the $i$-th point.
The relative standard uncertainty of type A at each calibration point is determined by the formula:

\[ u_{\text{rel}(i)A} = \sqrt{\sum_{j=1}^{n} \left( \frac{\Delta_{\text{rel}(i)j} - \Delta_{\text{rel}(i)j}}{j(j-1)} \right)^2} \]  \hspace{1cm} (5)

If, during the first two series of measurements, the readings of the calibrated MI at the calibration point remain constant (i.e., the calibrated MI is sufficiently coarse, with low sensitivity), then the third series of measurements is impractical.

In this case, the standard uncertainty due to the calibrated MI is calculated by the formula:

\[ u_{\text{rel}(i)A} = \frac{0.5 \theta_i}{\sqrt{3} P_i} \]  \hspace{1cm} (6)

where \( \theta_i \) – resolution of the calibrated MI (division price).

The standard type B uncertainty includes as one of the components the uncertainty of pressure reproduction in the \( i \)-th measurement point. The relative standard uncertainty of the pressure reproduction at the \( i \)-th calibration point is determined by the total standard uncertainty of the reference installation used (VOU-1 or UVE-3) and is quantified on the basis of data obtained from their certification as working standards \( U_B \) installations is given in table 1 as the total standard relative uncertainty.

The relative standard uncertainty of type B is defined as:

\[ u_B^2 = \left( \frac{\partial \Delta_{\text{rel}(i)j}}{\partial P_{oi}} \right)^2 \left( \frac{P_i}{P_{oi}} \right)^2 \cdot u^2(P_{oi}) \]  \hspace{1cm} (7)

The relative total standard uncertainty is calculated by the formula:

\[ u_{ci}(p) = \sqrt{u_B^2 + u_{\text{rel}(i)A}^2} \]  \hspace{1cm} (8)

The relative extended uncertainty, based on the normal law of distribution of measurements and the confidence probability of 0.95% corresponding to the coverage factor \( k = 2 \), is obtained by the formula:

\[ U_{\text{prel}(i)} = 2 \cdot u_{ci}(p) \]  \hspace{1cm} (9)

| Table 4. The uncertainty budget of the calibrated MI. |
|---------------------------------|------------------|------------------|------------------|------------------|------------------|
| Value in the \( i \)-th point | Mean value of rel. error | Rel. st. uncert. type A | Rel. st. uncert. type B | Rel. total st. uncert. | Rel. exp. uncert. |
| \( i_1 \) | \( \Delta_{\text{rel}1} \) | \( u_{\text{rel}(i)A} \) | \( u_B \) | \( u_C \) | \( u_{\text{rel}(i)} \), \( k=2 \) |
| \( i_2 \) | \( \Delta_{\text{rel}2} \) | \( u_{\text{rel}(i)A} \) | \( u_B \) | \( u_C \) | \( u_{\text{rel}(i)} \) |
| \( i_3 \) | \( \Delta_{\text{rel}3} \) | \( u_{\text{rel}(i)A} \) | \( u_B \) | \( u_C \) | \( u_{\text{rel}(i)} \) |
| \( \vdots \) | \( \vdots \) | \( \vdots \) | \( \vdots \) | \( \vdots \) | \( \vdots \) |
| \( i_{n-1} \) | \( \Delta_{\text{rel}(n-1)} \) | \( u_{\text{rel}(i)A} \) | \( u_B \) | \( u_C \) | \( u_{\text{rel}(n-1)} \) |
| \( i_n \) | \( \Delta_{\text{rel}n} \) | \( u_{\text{rel}(i)A} \) | \( u_B \) | \( u_C \) | \( u_{\text{rel}(n)} \) |

5. Conclusion
According to the results of calibration, a calibration protocol and (or) a calibration certificate are issued. The calibration certificate is a document confirming the metrological characteristics of the MI. According to the results of MI calibration, if there is information about compliance with the
mandatory metrological requirements for the standard in accordance with the state (local) verification scheme, this MI can be certified as a standard.

References
[1] RMG 29-2013 GSI. Metrology. Basic terms and definitions.
[2] Federal Law "About ensuring unity of measurements" 26.06.2008 No. 102-FZ.