ACHIEVEMENTS OF HIGH-VACUUM METROLOGY

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The basic metrological problem in the sphere of vacuum measurements consists of finding methods for precision measurements of low absolute pressures and concentrations of molecules under conditions of high and ultrahigh vacuum, and the production of appropriate reference measuring equipment. The solution of this problem will provide uniform vacuum measurements in our country.

Modern vacuum technology possesses sufficient means for evacuating gas media to the point when almost all the molecules are removed from a given volume. However, it is impossible to obtain quantitative characteristics of a gas medium which contains only a few molecules per cubic centimeter, since appropriate measuring equipment is lacking. Instruments based on the ionization of gas and the measurement of discharges or pure ionic currents provide only relative indications and require for absolute measurements calibration by absolute methods. The search for such methods preoccupies at present many vacuum research institutes and laboratories of the world. This problem is being dealt with in the vacuum measurements laboratory of the D. I. Mendeleev All-Union Scientific Research Institute of Metrology (VNIIM).

Basic instruments for calibrating vacuum-measuring equipment, which consists of mercury compression manometers, have been studied at the VNIIM since the beginning of the fifties. Sets of reference instruments consisting of single and double capillary compression manometers [1-8] have been produced. They cover measurement ranges of $10^3$ to $10^{-6}$ N/m² ($10^{-10}$ to $10^{-9}$ mm Hg) with a root-mean-square error amounting to 1-3% of the measured pressure. The external appearance of one of the reference compression manometers is shown in Fig. 1.

The compression manometers use capillaries with ground channels. The specially developed techniques for grinding the channels and measuring their diameters provide capillaries whose diameters have random deviations from their mean values not exceeding 2 μ [4].

The following measurement equation has been obtained for a double capillary compression manometer:

$$P = \frac{Cg\rho}{f} \left( \frac{h_c - h_{m1} - \Delta_1}{h_0 - h_{m1} - \Delta_2} \right),$$

(1)

where $C = f/V$ is a constant, $f$ is the cross-sectional area of the capillary, $V$ is the initial compression volume, $g$ is the acceleration due to gravity at the place of measurement, $\rho$ is the density of mercury at the measurement temperature, $h_c$ and $h_m$ are the mercury levels in the comparison and measurement capillaries respectively, $h_0$ is the level of the measuring capillary's sealed end, $\Delta_1$ and $\Delta_2$ are corrections.

Correction $\Delta_1$ accounts for the difference of the mercury depression in the measuring and the comparison capillaries, which occurs despite their equal diameters and is due to the interaction of mercury with a glass surface. This interaction has as yet been insufficiently studied. Measurement practice indicates that the difference in the depression of mercury in the capillaries made of the same piece of tubing can attain 1 mm:

$$\Delta_1 = \delta_{m1} - \delta_c,$$

(2)

where $\delta_{m1}$ and $\delta_c$ are the mercury depressions in the measuring and comparison capillaries respectively.

Correction $\Delta_2$ accounts for inaccuracies in evaluating the final compression volume as the volume of a cylinder between the apex of the capillary's sealed end and the mercury meniscus:

$$\Delta_2 = \frac{v_0 - v_m}{f},$$

(3)

where $v_0$ is the volume between the surface of the capillary channel's sealed end and an imaginary cylinder whose base passes through the upper point of the sealed surface and whose side surface consists of the continuation of the capillary channel walls, $v_m$ is the volume bounded by the plane passing through the apex of the meniscus, by the
walls of the capillary, and by the surface of the mercury meniscus. Corrections $\Delta_1$ and $\Delta_2$ can be determined experimentally.

An analysis of the possible errors of compression manometers indicates that the total systematic error due to inaccurate computations of constant $C$, to the depression phenomena, and to the differences in the shapes of the meniscus and of the measuring capillary's sealed end can amount to no more than 1% of the measured pressure. The main source of random errors consists of the dispersion of mercury depressions in the capillaries [5] which obviously cannot be compensated by raising precision in evaluating the mercury levels. However, for appropriately selected constructional dimensions of manometers with a given measurement range, the root-mean-square value of random errors can be reduced to fractions of one percent. An exception is provided only by the lower measurement limit at which the above errors rise sharply.

In the range of low pressures a single-capillary compression manometer is used in which the mercury level is evaluated only in the measuring capillary and in a wide tube parallel to it. The following measuring equation is used for a single-capillary manometer:

$$p = Cg_H (H - h_{\text{in}} - \Delta_1) (h_0 - h_{\text{in}} - \Delta_2).$$

Since this manometer is used for measuring pressures only below $10^{-1}$ N/m$^2$, the value of $C (h_0-h_{\text{in}}-\Delta_2)$ in the denominator of (1) can in this case be neglected (as compared with unity).

In Eq. (4) $H$ is the level of mercury in the wide tube; $\Delta_1' = \delta_{\text{in}} - \delta_T$; $\delta_T$ is the depression of mercury in the wide tube (normally a negligibly small quantity).

The remaining notations are the same as in expression (1).

Correction $\Delta_1'$ is determined experimentally during the evacuation of the manometer to sufficiently small pressures (below $10^{-5}-10^{-6}$ N/m$^2$).

In calibrating and testing various electrical discharge manometers by means of reference mercury compression manometers it becomes necessary to connect traps cooled with liquid nitrogen between the tested manometric transducers and the compression manometers in order to freeze out mercury vapors. The application of such traps contributes additional errors due to the pumping action of mercury vapors which move from the compression manometer to the cooling trap. It has been shown theoretically that the evacuation of the compression manometer by the mercury vapors can lead at room temperature to a reduction of the pressure in the compression manometer by 20-40% and, therefore, to a similar systematic calibration error.

In order to eliminate this error the VNIIM instruments [6] are provided with a long and thin connecting tube between the mercury container and the measuring bulb of the compression manometer. The pressure of the mercury vapor is reduced hundredfold in passing through the cooling tube and, therefore, their pumping action becomes insignificant. In order to eliminate the error due to the pumping action of mercury vapor it is advisable to make the mercury rise in the tube as quickly as possible (in 1-3 sec).

In order to raise the precision of the compression manometer's lower measuring limit, where the part played by random errors rises considerably, repeated readings were taken with a stepped raising of the mercury level in the compression manometer [3]. In this case the following measurement equation is used:

$$p = Cg_H \frac{1}{n} \sum_{i=1}^{n} (H_i - h_i - \Delta_i) (h_0 - h_i - \Delta_2).$$

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