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COMBINED REFERENCE STANDARD OF LOW ABSOLUTE PRESSURE AND RAREFIED GAS FLOW

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ABSTRACT

States of dynamic equilibrium of gas flows and absolute pressures in vacuum chambers are used in some methods of pressure unit realization in the range of low absolute pressure (high vacuum) to reduce (or divide) the initial relatively high pressure. Some of them are the continuous expansion (orifice flow) method and the static expansion method. For the basic methods of the flow unit realization ($Pa.m^3s^{-1}$), such as the method of constant pressure and the method of constant volume, the exact measurements of absolute pressure is necessary. These two physical quantities depend on each other in accordance with the flow definition and the methods of pressure unite realization. Some possibilities of employment of such pressure reference standards as piston gauge, manometer of McLeod, U-tube manometer to measure of rarefied gas pressure and flow are noted. Advantages of this "mutual cooperation" are shown. A development of a reference standard for realization of these two principal physical quantities of vacuum technique on the unique methodological and instrumental base is discussed. In present work a design of combined vacuum reference standard to be developed in accordance with the project PADRAVAC is proposed. This project was approved by the National Science Research Committee of Brazil (CNPq). A combination of static expansion method and classic two-orifice continuous expansion method is offered to be used for realization of low absolute pressure unit in the range ($10^3 – 10^{-5}$) Pa. The method of constant volume and the method of orifice flow (with long glass cylindrical capillaries) are proposed to be employed for realization of flow (throughput) unit in the range ($1\text{-}10^{-8}$) $Pa.m^3s^{-1}$ at the same apparatus. Interaction of mentioned methods under the realization of pressure and flow units is described. Calculations of range limits and expanded errors of every method have been carried out.

INTRODUCTION

The practice of reference standards development knows some facts of creation of the unique apparatus to realize the units of two physical quantities. For instance, there are time interval t and frequency f. These physical quantities depend each other in accordance with the following equation:

$$f = T^{-1} \qquad (1)$$

Due to the correlation between these physical quantities it was possible to create the combined standard for realization of the values of the time interval unit (second) and the frequency unit (Hertz) [1]. On the other hand, certain correlation between some physical quantities may exist according to their definitions. For example, to realize the constant electric current unit so-called "current balance" is used accordingly the following definition of ampere [2]:" The ampere is that constant current which, if maintained in two straight parallel conductors of infinite length, of negligible circular cross-section and placed 1 meter apart in vacuum, would produce between these conductors a force equal 2.10^{-7} Newton per meter of length." We could find many other examples of a similar tie between different physical quantities.

PRESSURE

In accordance with general definition, a pressure is force acting on a surface [3]. Modern reference standards realize this definition by means of different kinds of the piston manometers (pressure balances) [4] (Fig.1) and the U-tube manometers [5] (Fig.2).

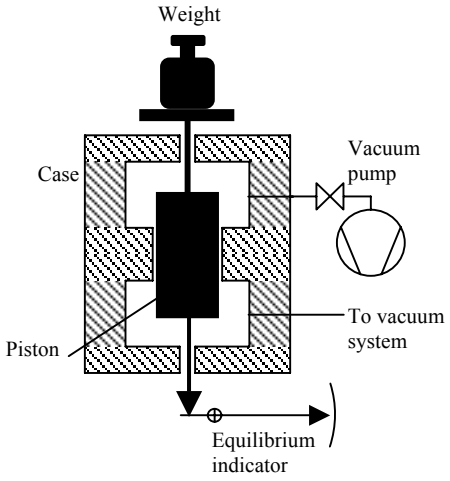

Figure 1. Piston manometer

The pressure p to be measured by the piston manometer, is given by:

$$p = \frac{mg}{S_e} - p_r \qquad (2)$$

where m is the mass of the weights; g is the gravity acceleration; S_e is the area of the piston cross-section; p_r – is the residual pressure at the upper side of the piston.

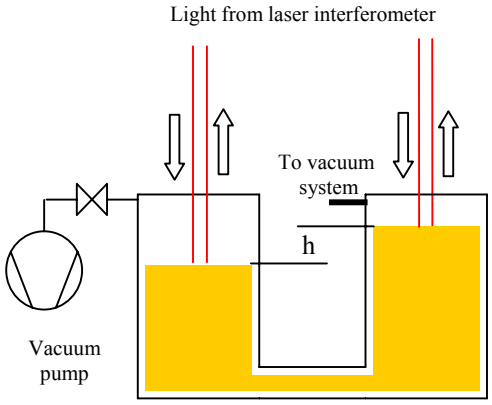

Figure 2. U-tube interferometric manometer.

The pressure to be measured by the U-tube manometers is given by:

$$p = \rho g h - p_r \qquad (3)$$

where ρ is the density of the liquid; h is the difference of the liquid column heights ; p_r – is the residual pressure over the oil surface in the right tube of the manometer. Considering the design of manometers, we can see that all these standards generate actually a differential pressure between two sides of their sensitive element (piston or liquid column) and it is necessary to keep very low pressure p_r at the one side of them. Because of low sensitivity of these instruments in the range of high vacuum, it was difficult to achieve an acceptable accuracy at the pressures lower 1 Pa. Some means of the pressure reducing are used to generate the pressure unit in the range of medium and high vacuum. There are well-known manometer McLeod [6] (Fig.3) and static expansion apparatus [7] (Fig.4), where isothermal compression and expanding of the gas are realized according to Boyle-Marriott's law:

$$p_1 V_1 = p_2 V_2 \qquad (4)$$

Compressing the gas in the volume V_2 at low pressure p_2, we can achieve much more high and more accurately measured pressure p_1 in the little volume V_1 by the McLeod manometer. Expanding gas from the little volume V_1 at high and precisely measured pressure p_1 into the large volume V_2, we can generate lower and accurate values of pressure p_2 by the static expansion apparatus.
The continuous expansion method [8] is used successfully in high- and ultrahigh vacuum. The method consists in reduction of relatively high primary pressure by means of gas flowing through two orifices situated one after the other when the second orifice conductance C_2 is much more than the first one C_1 (Fig.5). From the condition of gas flows equilibrium we can derive:

$$p_2 = p_1 \frac{C_1}{C_2} + p_r \qquad (5)$$

where p_r is the pressure determined by back flow of the gas from the vacuum pump inlet.

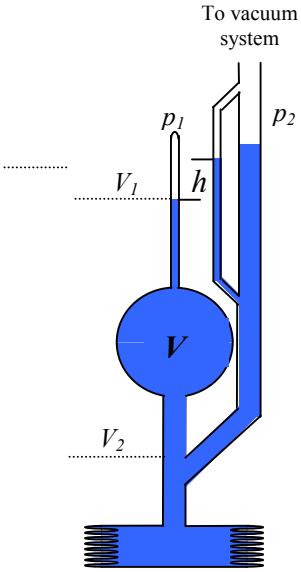

Figure 3. Manometer of McLeod

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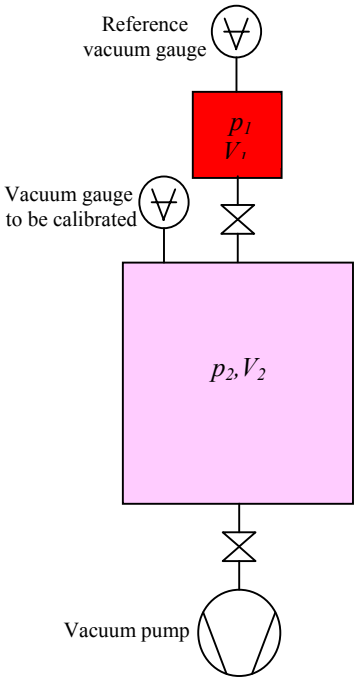

Figure 4. Static expansion method.

FLOW

According to the definition, the gas flow in the units pV (or throughput) is expressed as [6]:

$$Q = \frac{\partial(pV)}{\partial t} = p\frac{\partial V}{\partial t} + V\frac{\partial p}{\partial t} = \\ = p\frac{\partial V}{\partial t}\Big|_{p=const} = V\frac{\partial p}{\partial t}\Big|_{V=const} \quad (6)$$

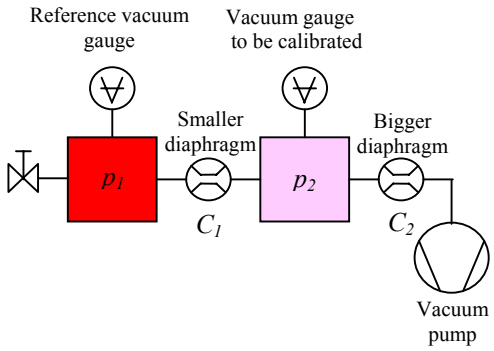

Figure 5. Continuous expansion method.

Thus, two methods of the flow unit realization are used in the reference standards of rarefied gas flow. There are constant pressure method [9] (Fig.6) and constant volume method [10] (Fig.7).

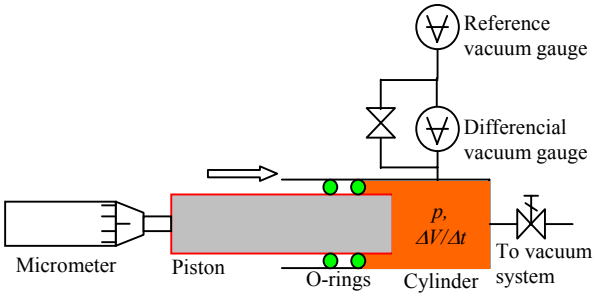

Figure 6. Constant pressure method.

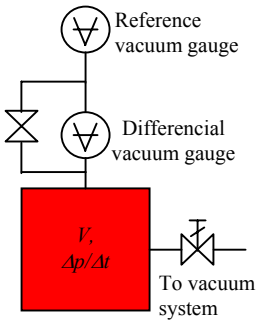

Figure 7. Constant volume method.

On the other hand, the gas flow passing through a certain element of the vacuum system, may be given by:

$$Q = C(p_1 - p_2) \quad (7)$$

where C is the conductance of the element, p_1 and p_2 are the pressures at the entrance and at the exit of the element, respectively. The value C is calculated according to the equations of rarefied gas dynamics [6]. Some elements with regular geometric form, such as a long cylinder tube and a thin circular orifice, were investigated in details and carefully for many years like theoretically as much experimentally. Accurate equations, describing their conductances within the error of ±(1-2)% were derived and are used in the rarefied gas flow measurements in general [6] and in the calibration practice in particular [11] (Fig.8).

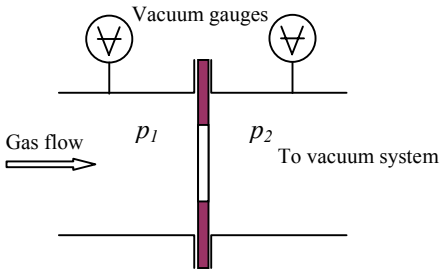

Figure 8. Conductance flowmeter.

The flowmeter with burette (Fig. 9) is the classic instrument for the rarefied gas flow measurements. Up to our time, the flowmeter with burette is still used in the vacuum practice [12]. The gas flow, measured by this flowmeter, one can determine from the following equation:

$$Q = \frac{p_a V}{t}\left(1 + \frac{\rho g h}{p_a}\left(\frac{V_0}{V} - 1\right)\right) \quad (8)$$

where p_a is the atmospheric pressure; V_0 is the internal volume of the burette; V and h are the volume and the height of the oil column; ρ is the oil density; g is the gravity acceleration. Here, we can see that both the pressure and the volume of gas are changed simultaneously during the measurement.

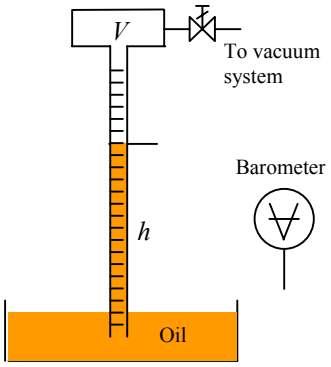

Figure 9. Flowmeter with burette.

INTERCHANCE

Observing the methods and the instruments mentioned above, we could note a certain likeness of their elements and operations. Really, the cylinder piston is used in the piston Manometer and in the constant pressure flowmeter. The liquid column is used in the U-tube manometer, in the McLeod manometer and in the flowmeter with burette. Generally, it would thus be desirable to note that for pressure measurements it is enough to determine the

position of the sensitive element (piston or liquid column). For rarefied gas flow measurements it is necessary to measure the velocity of the sensitive element movement or, at least, its start and final position over the certain time interval. Furthermore, the constant volume [10] and the constant pressure [9] flowmeters are used in the apparatus realizing the continuous expansion method for the vacuum gauge calibration, like the conductance flowmeters [11]. On the other hand, the apparatus for the vacuum gauge calibration, equipped by mass-spectrometer, are used for the diffusion reference leak calibration [13]. Here, a mass-spectrometer is applied as a comparator of the flows inputting from the conductance flowmeter and from the leak to be calibrated. The manometer of McLeod is used for the helium reference leak calibration by the cumulative method [14].

The combined reference apparatus intended for the calibration of the vacuum gauges and the rarefied gas flowmeters, have been developed by the author [15,16]. There, the methods of the direct comparison and the continuous expansion have been applied for the vacuum gauge calibration in the range of (10^{-5} – 10^{5}) Pa. The constant volume method, the method of the direct comparison and the comparison by the mass-spectrometer have been used for the flowmeters and for the reference helium leaks calibrations in the range of (10^{-8} –10) Pa.m^{3}s^{-1}. Diaphragm, thermocouple, Pirani, cold and hot cathode vacuum gauges; diffusion helium leaks, air orifice leaks, thermal mass flowmeters and flowmeters with burette were calibrated during the last years on this apparatus. The constant volume method was applied for periodic control of the first orifice conductance C_1 (capillary). The apparatus was employed at the Pump & Power Engineering Work "NASOSENERGOMASH" (Sumy, Ukraine) for calibration of the vacuum gauges, of the leaks and the flowmeters used at the testing of vacuum pumps and various vacuum equipments. The calibration of measuring instruments applied in different vacuum technologies and scientific research activities at various companies and institutes of Ukraine and Moldova has been carried out.

NEW PROJECT

Recently, the National Science Research Committee of Brazil approved the project PADRAVAC aimed to the development of the combined reference vacuum standard for generation of two principal physical quantities of vacuum. There are low absolute pressure and rarefied gas flow. The scheme of this standard is shown in Fig.10.

The initial pressure values are generated by the piston gauge in the range (10^{2} – 10^{5}) Pa. To realize the pressure unit, we propose to use the static expansion method in the range (10^{-2} – 10^{3}) Pa and the continuous expansion method in the range (10^{-5} –10^{-1}) Pa. The static expansion method is realized by the 3-chambers set (0,1;1,0 and 100 liters) with thermal stabilization. Expected uncertainties do not exceed ±2%. The uncertainty calculations for upper and lower range limits are given in the Appendix. The continuous flow method is realized by the classic two-orifice scheme. As a first orifice we chose a long glass cylindrical capillary. The conductance of the thin orifice C_o and the long capillary C_c at the free molecular gas flow regime are given by [6]:

$$C_o = \frac{\pi D^2}{4}\sqrt{\frac{RT}{2\pi M}} \tag{9}$$

$$C_c = \frac{\pi d^3}{3l}\sqrt{\frac{RT}{2\pi M}} \tag{10}$$

where D is the orifice diameter; d and l are the diameter and the length of the capillary; T and M are the temperature and the molar mass of the gas; R is the universal gas constant.

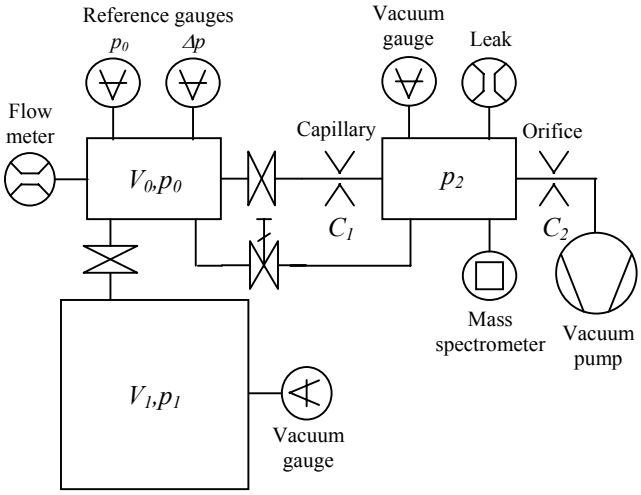

Figure 10. Scheme of combined reference standard.

The pressure p_2 in the continuous expansion method is expressed as [17]:

$$p_2 = p_1 \frac{C_c}{C_o} = p_1 \frac{4d^3}{D^2 l} \tag{11}$$

It would result that the value of the pressure p_2 depends only on the pressure p_1 and on the geometric dimensions of the orifice and the capillary. As such, this method may be considered as a fundamental one at the condition of strict free molecular flow in the orifice and in the capillary. To fulfill this requirement at the initial pressure p_1 up to 1 Pa, the first orifice is made as a lot of parallel thin glass capillaries with internal diameter lower 10^{-4} m. The second orifice was a disk with a lot of uniformly distributed on the disk plane small orifices. The initial pressures for the continuous expansion method are generated by the static expansion method. The absolute capacitance vacuum gauges CDG serve as the reference instruments for the measurement of initial pressure p_0. These gauges are calibrated *in situ* by the piston gauge. The general mode of the flow unit generation is the method of constant volume. Three chambers utilizing by the static expansion method are used by the constant volume flowmeter. Additionally, three differential CDG are connected to the chambers. These differential gauges are calibrated *in situ* just by the

static expansion method. If this flowmeter is used for the vacuum gauge calibration by the continuous expansion method, the differential gauges are connected to measure a decreasing pressure p_1. If this flowmeter is used for flowmeters calibration, the differential CDG are connected vise versa to measure the increasing pressure p_1. The measuring range of the flowmeter is (10^{-7}-10) Pa.m^{3}s^{-1}.
The conductance flowmeter consists of the long cylindrical capillaries, the CDG and the chambers realizing the static expansion method. The measured flow is calculated in accordance with Eq.7, taking into account the temperature and the gas specie property. The measuring range of this flowmeter is (10^{-6}-10^{-10}) Pa.m^{3}s^{-1}. The flowmeter is used for the helium leak calibration by the comparative method utilizing the quadrupole mass-spectrometer. The mass-spectrometer sensitivity and its stability are determined by the continuous expansion method. It would be noted that the measuring ranges of all methods are overlapped each other: for the piston gauge and the static expansion method in the range of (10^2-10^3) Pa; for the continuous expansion method and the static expansion one in the range of (10^{-2} – 10^{-1}) Pa; for the constant volume method and the conductance flowmeter in the range of (10^{-7}-10^{-6}) Pa.m^{3}s^{-1}. That it should be useful for estimation of intrinsic systematic uncertainties of every method mentioned above and for the periodic control of their repeatability. This is the principal advantage of the combined reference standard design, not counting a lower cost of the fabrication and the maintenance.

CONCLUSION

The dependence between such physical quantities as pressure and gas flow in accordance with their definitions is shown.
The likeness of sensitive elements of various manometers and flowmeters is mentioned.
Some examples of the vacuum reference standards application in the calibration both the vacuum gauges and the flowmeters are noted.
The design of the combined reference standard of low absolute pressure and rarefied gas flow is described**.**
The possibility and the usefulness of mutual control of the principal elements and the measuring methods utilized in the standard are detached.
A lower cost of the combined vacuum reference standard is pointed.

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APPENDIX. Uncertainties calculation.

Static expansion method.
The uncertainty budget for the maximum (10^3 Pa) and for the minimum (10^{-2} Pa) measuring range limits is shown in Table 1:

Table 1. The uncertainty budget for the maximum (10^3 Pa) and for the minimum (10^{-2} Pa) measuring range limits

Source	Value,%		Notice
	Max	Min	
Initial pressure	0,58	0,08	See [18]
Initial pressure	0,2	0,07	Calibration *in situ*
Volume ratio	0,1	0,05	1:1000/1:100 [19]
Out gassing	0,2	<0,01	Estimation
Temperature	0,2	0,2	Passive thermostat
Temperature	0,05	0,05	Active thermostat
Total	0,9	0,35	Maximum values
Total	0,6	0,17	Minimum values

The estimation of the outgassing is very approximate because the chambers material, technology of their fabrication, kind of the gas and preliminary bakeout of the chambers influence notably on the outgassing flow value.

Continuous expansion method.
The uncertainty budget for the maximum (10^{-1} Pa) and for the minimum (10^{-5} Pa) measuring range limits is shown in Table 2:

Table 2. The uncertainty budget for the maximum (10^{-1} Pa) and for the minimum (10^{-5} Pa) measuring range limits

Source	Value, %		Notice
	Max	Min	
Initial pressure	0,5	0,2	
Conductance ratio	0,7	0,7	
Unstable flow	0,7	0,3	Estimation
Intermediate flow	<0,01	0,3	Estimation
Back stream effect	0,1	0,1	Estimation
Total	2,0	1,6	

The expansion conductance ratio uncertainty δR_c was calculated from Eq. (11) in accordance with real sizes of the capillary and the orifice:

$$\delta R_c = \sqrt{9(\delta d)^2 + (\delta l)^2 + 4(\delta D)^2} \qquad (1A)$$

The intermediate gas flow in the orifice becomes significant at the upper measuring limit. There, some elements of this gas flow regime are appeared under the Knudsen number Kn<10. Corresponding uncertainty is given by:

$$\delta C_{\text{int}} = \frac{C_t - C_{ti}}{C_{ti}} + \frac{C_o + C_{oi}}{C_{oi}} \qquad (2A)$$

where C_{ti} and C_{oi} are the conductances of the capillary and the orifice calculated for the intermediate gas flow regime according to [20].The back streaming uncertainty is caused by the pressure difference between the calibration chamber and the high vacuum pump inlet. It depends on the pumping speed and on the ultimate pressure of the pump. It may be obtained from:

$$\delta p_b = \sqrt{(p_3 / p_2)^2 (\delta p_3)^2} \qquad (3A)$$

where p_3 is the pump inlet pressure; δp_3 is the uncertainty of the pump inlet pressure measurement.

Constant volume method.
The uncertainty budget for the maximum (10 $Pa.m^3s^{-1}$) and for the minimum (10^{-7} $Pa.m^3s^{-1}$) measuring range limits is shown in Table 3:

Table 3. The uncertainty budget for the maximum (10 $Pa.m^3s^{-1}$) and for the minimum (10^{-7} $Pa.m^3s^{-1}$) measuring range limits

Source	Value, %		Notice
	Max.	Min.	
Initial dif. pressure	0,58	0,08	See [18]
Initial dif. pressure	0,2	0,07	Calibration *in situ*
Unstable flow	0,7	0,3	
Volume	0,1	0,01	1 and 100 L
Out gassing	0,2	<0,01	Estimation for N_2
Temperature	0,3	0,5	Passive thermostat
Temperature	0,05	0,1	Active thermostat
Time interval	<0,01	0,05	
Total	1,9	1,0	Maximum values
Total	1,3	0,6	Minimum values

According to Eq. (6), the generation of the gas flow (or throughput) unit by the constant volume method is carried out under the condition, where the pressure is changed exponentially. (It is the gas outflow through the leak valve from the chamber with constant volume). Hence, the value of the flow isn't constant. This is the principal disadvantage of the method. To decrease this instability it would necessary to linearize this process in time and to keep the following condition:

$$\Delta p << p_1 \qquad (4A)$$

Accordingly [10] this uncertainty does not exceed 0,63% for the time intervals up to 2000s.

Conductance flowmeter.
The uncertainty budget for the maximum (10^{-7} $Pa.m^3s^{-1}$) and the minimum (10^{-10} $Pa.m^3s^{-1}$) measuring range limits is shown in Table 4.
This uncertainty budget is calculated with accordance to Eq. (7) and Eq. (10).

$$\delta Q_{con} = \sqrt{\begin{matrix} 9(\delta d)^2 + (\delta l)^2 + 0{,}5[(\delta T)^2 + (\delta M)^2] \\ + (\delta p_1)^2 + (p_2 / p_1)^2 (\delta p_2)^2 \end{matrix}} \qquad (5A)$$

Table 4. The uncertainty budget for the maximum (10^{-7} $Pa.m^3s^{-1}$) and the minimum (10^{-10} $Pa.m^3s^{-1}$) measuring range limits

Source	Value, %		Notice
	Max	Min	
Initial dif. pressure	0,6	0,2	
Capillary dimensions	0,6	0,6	
Viscosity and molar mass of gas	0,1	0,1	Standard reference data
Intermediate flow	<0,01	0,3*	* Pressure 1 Pa
Temperature	0,05	0,05	
Total	1,4	1,3	

It would necessary to note that all these calculations are preliminary estimations and the uncertainties values will be determined more accurately in the experimental research.

REFEREFCES

1. ESSEN, L.; PARRY, J.V.L., Nature **176**, 280 (1955).
2. CONMETRO resolution; 1988:12:28.
3. The International System of Units (SI). NIST Special Publication 330; 2001:7.
4. RENDLE, C.G.; ROSENBERG H., Metrologia **36**, 613 (1999).
5. UEKI, M.; OOIWA, A., Metrologia **30**, 579 (1993).
6. LAFFERTY, J.M., Foundation of Vacuum Science and Technology: Wiley Interscience, 1998.
7. JITSCHIN, W.; MIGWI, J.K.; GROSSE, G., Vacuum **40**, 293 (1990).
8. BERGOGLIO, M.; CALCATELLI, A.; MARZOLA, L.; RUMIANO, G., Vacuum **38**, 887 (1988).
9. JOUSTEN, K.; MESSER, G.; WANDREY, D., Vacuum **44**, 135 (1993).
10. LEVINE, P.D.; SWEDA, J.R.; J. Vac. Sci. Technol. A **14**, 1297 (1996).
11. JITSCHIN, W.; WEBER, U.; HARTMANN, H.K., Vacuum **46**, 821 (1995).
12. SHARMA, J.K.N.; SHARMA, D.R., J. Vac. Sci. Technol. **14**, 829 (1977).
13. ABBOTT, P.J.; TISON, S.A., J. Vac. Sci. Technol. A **14**, 1242 (1996).
14. KUZMIN, V.V., Measurement techniques (Izmeritel'naya tehnika) 11, 41 (1988). [in Russian].
15. KUZMIN, V.V.; ODNORALOV, V.N., Metrologia 10, 45 (1985). [in Russian].
16. ODNORALOV, V.N., News of Kiev Polytechnic Institute. Instrumentation **19**, 10 (1989). [in Russian].
17. REDHEAD, P.A.; HOBSON, J.P.; KORNELSEN, E.V., The physical basics of ultrahigh vacuum. New York: AIP: 1993. p. 260.
18. SULLIVAN, J.J., J. Vac. Sci. Technol. A **3**, 1721 (1985).
19. JITSCHIN, W., Metrologia **39**, 249 (2002).
20. SHARIPOV, F.; SELEZNEV, V., J. Phys. Chem. Ref. Data **3**, 657 (1998).