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*भारतीय मानक मसौदा*

**निर्वात गेज के अंशशोधन की पद्धति — परीक्षण के तरीके  
भाग 1  $10^{-5}$  Pa से  $10^{-1}$  Pa की दाब सीमा में सतत प्रवाह से दाब में कटौती**

[आई एस 8276 (भाग 1) का पहला पुनरीक्षण]

*Draft Indian Standard*

**CALIBRATION OF VACUUM GAUGES — METHODS OF TEST  
PART 1 PRESSURE REDUCTION BY CONTINUOUS FLOW IN THE  
PRESSURE RANGE OF  $10^{-5}$  Pa to  $10^{-1}$  Pa**

[*First Revision of IS 8276 (Part 1)*]

ICS 23.160

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Chemical Engineering Plants and Related Equipment Sectional  
Committee, MED 17

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**FOREWORD**

*(Formal clause to be added later)*

This standard was originally issued in 1976. The present revision has been taken up with a view incorporating the modification found necessary as a result of experience gained in the use of this standard. Also, in this revision, the standard has been brought into the latest style and format of Indian Standards, and references to Indian Standards, wherever applicable have been updated.

International system (SI) units have been used in this standard. The relation of these units to other units is given below for guidance:

1 Pascal (Pa) = 1 newton/square metre ( $\text{N/m}^2$ )

1 torr = 1 mm Hg = 133.322 Pa

1 atm = 101.325 kPa

Throughput: 1 W = 1  $\text{N.m/s}$  = 7.5 torr l/s

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

## **1 SCOPE**

**1.1** This standard (Part 1) specifies a method for the calibration of vacuum gauges in the pressure range of  $10^{-1}$  to  $10^{-5}$  Pa whereby a known low pressure is established by the passage of a known flow of gas through a circular orifice of known conductance.

**1.1.1** The upper limit of pressure to which the method is applicable is set by the diameter of the orifice in relation to the mean free path of the gas molecules. The lower limit will depend on the type of gauge being calibrated, and on the amount of sorption or desorption of gas within the apparatus.

## **2 SYMBOLS**

The following symbols have been used in the standard:

$p^o$  = Absolute pressure of the gas in the throughput meter;

$p$  = Calculated pressure of the gas used for calibration in the calibration vessel;

$P_1$  = Value of  $p$  corrected to relate the calibration of the gauge to a specific temperature,  $T_o$ ;

$Q$  = Gas throughput, as measured at the temperature,  $T_Q$ ;

$S$  = Net volume rate of flow at the calibration vessel;

$L$  = Calculated conductance of the orifice;

$S_p$  = Measured volume rate of flow of the pumping system;

$T_o$  = Absolute temperature to which the calibration is to be referred;

$T_c$  = Absolute temperature of the calibration vessel, including the orifice, during the actual calibration;

$T_Q$  = Absolute temperature of the gas throughput meter during the actual measurement of the gas throughput in the calibration chamber;

$R$  = Gas constant;

$M$  = Molecular mass;

$K_1$  = Clausing factor allowing for the thickness of the orifice;

$K_2$  = Correction factor allowing for the diameter of the orifice relative to the mean free path of the molecules;

$A_L$  = Area of the circular orifice;

$r$  = Radius of the circular orifice;

$\bar{c}$  = Mean thermal velocity of the molecules ( $= \sqrt{\frac{8RT}{\pi M}}$ );

$l$  = Mean free path of the molecules; and

$\alpha$  = Correction factor for departure from the ideal gas law.

## **3 APPARATUS**

### **3.1 General Form**

The general form of the apparatus is shown schematically in Fig. 1. The gauges under calibration are connected to an expansion vessel (calibration chamber) into which gas is admitted through a flow measurement device and subsequently pumped away through an orifice.

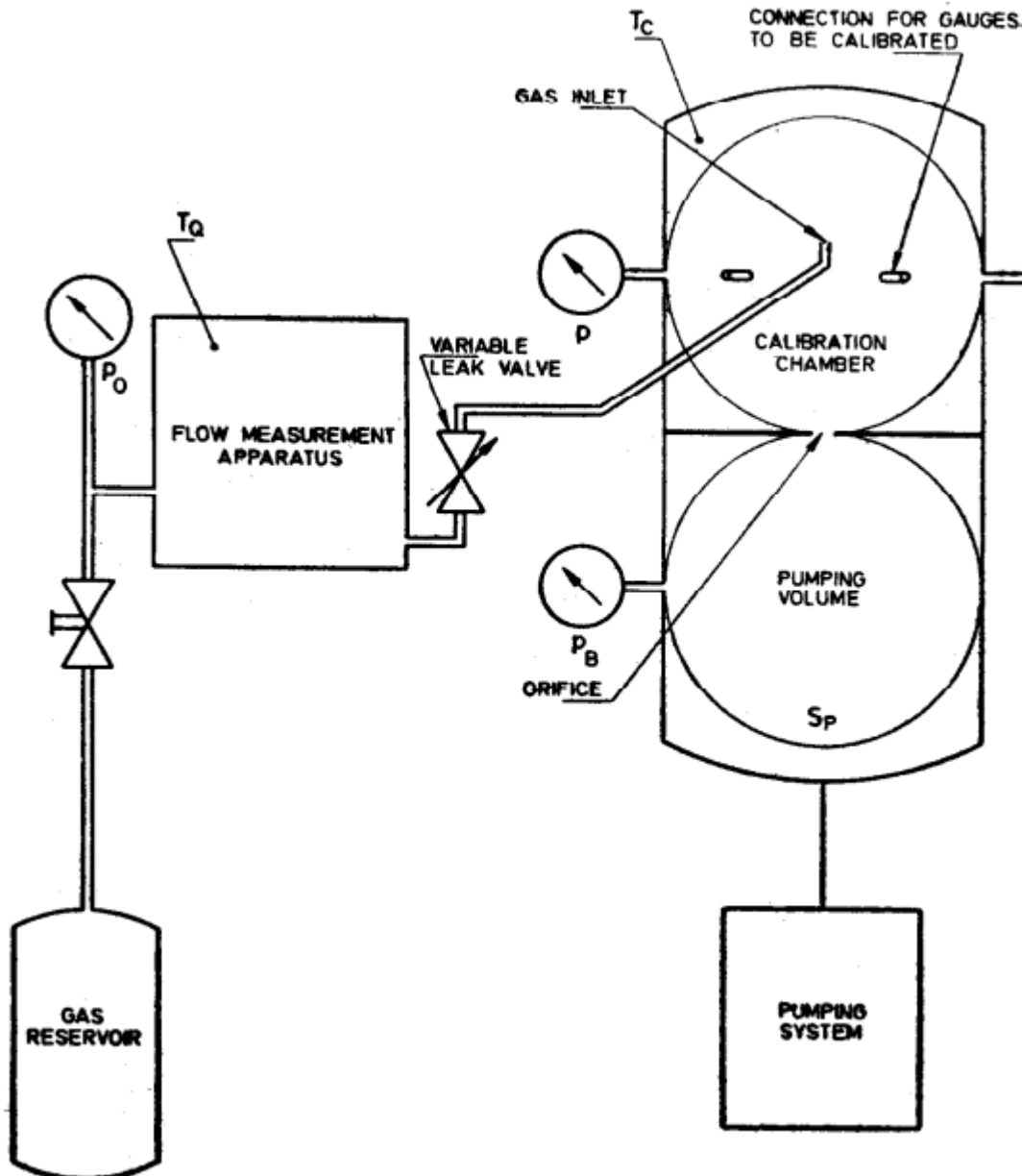


FIG. 1 SCHEMATIC DIAGRAM OF TYPICAL APPARATUS

### 3.2 Ratio of Cross-Sectional Area of Orifice to Surface Area of Calibration Vessel

The calibration chamber shall be in the form of a sphere or of a right cylinder. The orifice shall be circular, and its cross-sectional area shall be less than 1/1 000 of that of the inner surface of the inscribed sphere of the calibration chamber. The orifice shall be situated in the surface of the inscribed sphere. The volume between the orifice and the pumping system shall be comparable with that of the calibration chamber or even larger.

### 3.3 Arrangement of Gas Inlet

The gas shall be admitted in such a way that neither the orifice, nor the gauges to be calibrated, nor the gauge connections can be hit by the incoming gas molecules before they have hit the wall at least once.

### 3.4 Determination of Orifice Cross-Sectional Area and Conductance

The orifice cross-sectional area  $A_L$  shall be determined to an accuracy of  $\pm 0.1$  percent. The conductance  $L$  is calculated from the area by means of the equation:

$$L = A_L \frac{\bar{c}}{4} K_1 K_2 \dots\dots\dots(1)$$

The correction factors  $K_1$  and  $K_2$  vary with dimensions of orifice (*see* Annex A).

### 3.5 Ratio of Rim Thickness to Diameter of Orifice

The rim thickness of the orifice shall be less than 1/50 of its diameter. The influence of the rim thickness [*see* equation (1)] in 3.4 is taken into consideration by the clausuring factor  $K_1$ , the value of which may be taken from the table given in Annex A. The arrangement for locating the orifice is indicated in Fig. 2. Any device clamping the diaphragm, bearing the orifice, into the calibration chamber shall not protrude into the space occupied by the inscribed spheres above and below the orifice.

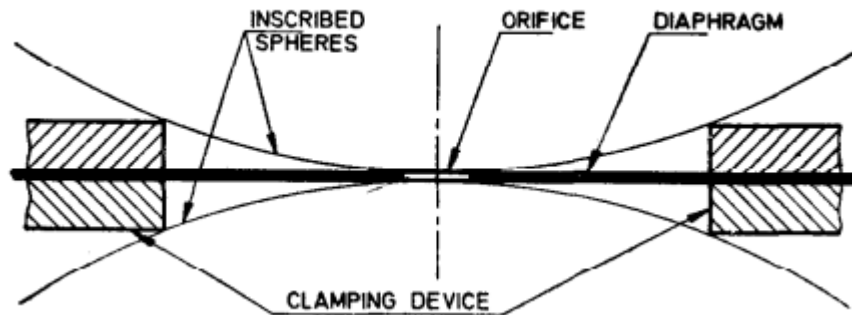


FIG. 2 ARRANGEMENT FOR LOCATION OF ORIFICE

### 3.6 Determination of Temperature of Calibration Vessel

Means shall be provided to measure the temperature  $T_c$  of the calibration chamber and orifice, and to keep this temperature constant during the period of the measurements.

### 3.7 Requirements for Pumping System

The pumping system shall provide for a volume rate of flow of gas through the orifice of at least 10 l/s for the gas used in the calibration. If a vapour pump is used, an adequate baffle or cold trap shall be provided to protect the gauges under calibration against disturbances due to the pump fluid.

### 3.8 Ratio of Volume Rate of Flow of Pumping System to Conductance of Orifice

The ratio of the measured volume rate of flow,  $S_p$  of the pumping system to the conductance  $L$  of the orifice shall exceed 50. If then  $S_p$  is measured with an accuracy of  $\pm 20$  percent, the resulting limit of uncertainty in  $S$  is  $\pm 0.4$  percent.

### 3.9 Arrangement of Gauges to be Calibrated

The gauges to be calibrated shall be arranged along the equator of the inscribed sphere of the calibration chamber, with the orifice situated at the pole of this sphere. For the connection of the gauge heads to be calibrated, the calibration vessel shall be provided with connecting pipes, the cross-sections of which correspond to the connecting cross-sections of the gauges, and the lengths of which do not exceed twice the diameter (*see 4.3.1*).

## **4 METHOD OF TEST**

### **4.1 Principle**

**4.1.1** The instrument to be calibrated is connected to a calibration chamber into which gas of accurately known throughput  $Q$  is admitted and pumped away through a gas sink of accurately known volume rate of flow  $S$ . Under the appropriate conditions the following equation determines the pressure  $p$  of the gas in the calibration vessel.

$$p = \frac{Q}{S} \dots\dots\dots (2)$$

**4.1.2** Assuming all parts of the calibration equipment to be at the same temperature. The required low pressure  $p$  is generated by the method of pressure reduction by continuous flow from a suitable supply of gas at an initial value of pressure sufficiently high to be accurately measurable (*see Annex B*).

### **4.2 Preliminary Requirements**

#### **4.2.1 Pre-treatment of Calibration Chamber**

In order to avoid errors due to other gas sources in the calibration chamber the rate of gas desorption of the apparatus shall be lowered by means of baking to such an extent that the residual gas pressure in the calibration chamber is less than 1/100 of the lowest pressure used during the calibration procedure. This residual gas pressure shall be taken into consideration in correcting the calibration results.

#### **4.2.2 Determination of Volume Rate of Flow**

The volume rate of flow  $S$  in the calibration chamber is determined by calculation from the conductance  $L$  (*see 3.4*) of the circular orifice and the measured effective volume rate of flow  $S_p$  of the pumping system, using the following formula.

$$S = \frac{L}{1+L/S_p} \dots\dots\dots (3)$$

#### **4.2.3 Determination of Effective Volume Rate of Flow of Pumping System**

The effective volume rate of flow  $S_p$  of the pumping system shall be determined for all conditions and pressure setting representative of the use of the apparatus. One of two methods described below may be used.

**4.2.3.1** Measurement of the pressure increase  $\Delta p$  in the calibration chamber, and of the pressure increase  $\Delta p_B$  in the volume between orifice and pumping system, when additional gas is introduced into the apparatus. The ratio  $L/S_p$  is then given by the following equation.

$$\frac{L}{S_p} = \frac{\Delta p_B}{\Delta p} \dots\dots\dots (4)$$

The pressure measurement necessitates two similar linear gauges of the same sensitivity, which, however, have not necessarily been calibrated, and which are connected to the calibration chamber and to the volume between orifice and pumping system respectively. In order that these pressure measurements should represent the true existing gas densities unaffected by distortion of the distribution function of the molecules in the volume between orifice and pumping system, this volume shall be about the same as that of the calibration chamber and of similar main dimensions (*see 3.2, Fig. 1 and Annex B*). The gauges shall be fitted on either side of the orifice on the equators of the two inscribed spheres, representing the volumes above and below the orifice, whose poles coincide at the orifice position.

NOTE — In the case of molecular pumps and diffusion pumps it is sufficient to use the effective volume rate of flow measured for each calibration gas in the required pressure range.

#### **4.2.3.2 Oatley procedure**

In this method readings of the pressure  $p$  in the calibration chamber are taken by a linear pressure gauge (not necessarily calibrated), at a constant throughput  $Q$  of gas, for a series of values of the conductance  $L$ . This procedure is repeated for several different values of  $Q$ . In order that the value of  $S_p$  may be measured with sufficient accuracy, the following conditions shall be met:

- a) The graphs of  $p$  when plotted against  $1/L$  for a constant value of  $Q$  shall not show a significant deviation from a linear relationship; and
- b) If  $S_p$  is constant, the straight lines obtained for various values of  $Q$  shall intersect at a single point on the axis of  $1/L$  given by the equation.

$$\frac{1}{L} + \frac{1}{S_p} = 0 \dots\dots\dots (5)$$

Any deviation from the above requirements gives an estimate of the error involved. The variation of  $1/L$  may be determined either by the use of a number of interchangeable orifices or by the use of a single orifice of variable conductance. The ratio of the largest to the smallest area of cross section of the orifice shall be at least 10 : 1. For all orifices used, or for all settings in the case of a variable orifice, the conditions of **3.5, 4.3.2** and Annex A shall be met. In the case of the Oatley method, however, an orifice of regular polygonal form may be used if desired.

### **4.3 Calibration Procedure**

#### **4.3.1 Connection of Gauge Heads to be Calibrated (see 3.9)**

The gauge heads to be calibrated shall be connected to the calibration chamber by means of connecting pipes, the cross-sections of which correspond to the connecting cross-sections of the gauges and the lengths of which do not exceed twice the diameter. The gauges shall be mounted along the equator of the inscribed sphere of the calibration chamber. The orientation of the gauges to be calibrated shall be well defined. Alternatively, it shall be ensured that their orientation does not significantly affect the calibration. Nude systems shall be connected in such a way that they are immersed as far as possible. If more than one nude gauge is attached to the calibration chamber, only one gauge shall be operated at any time, unless it is certain that a mutual influence is not possible.

#### **4.3.2 Limitation of Effects due to Pumping and Gas Desorption of the Gauges to be Calibrated**

In order to keep the errors resulting from the gauges to be calibrated acting as pumps or sources of gas, sufficiently small, the following conditions shall be met:

- a) The volume rate of flow due to pumping by the connected gauges, or their gas desorption rate expressed in the same units, shall not exceed 1/100 of the volume rate of flow  $S$  at the calibration chamber; and
- b) The volume rate of flow  $S$  at the calibration chamber shall be at least 10 l/s for the gases used in the calibration procedure, even if compliance with condition (a) shall allow a lower value.

**4.3.2.1** If the above conditions cannot be met, the results shall be corrected by means of the indications of a suitable auxiliary gauge. The conditions (a) and (b) above are especially important in the case of hot-cathode ionization gauges operating with gases, such as hydrogen, methane, oxygen and air.

**4.3.2.2** In certain cases it may be necessary to check for possible anomalous ion current caused by desorption of ions from the grid. For this purpose the ion current characteristic shall be recorded in order to ensure that the calibration relates to conditions where this characteristic is linear.

### **4.3.3** *Operation of Gauges to be Calibrated*

All the operating requirements specified by the manufacturer of the gauge and its associated parts shall be carefully followed. For gauges which have several indicated ranges the exact relations between them shall be the subject of a separate test.

### **4.3.4** *Determination of Gas Throughput $Q$*

An effective volume rate of flow  $S$  at the calibration chamber shall be 10 l/s or more. The corresponding range of the throughput measurement is therefore between  $10^{-7}$  Pa.m<sup>3</sup>/s and a value above  $10^{-3}$  Pa.m<sup>3</sup>/s. This measurement shall have a typical uncertainty of  $\pm 0.5$  percent which shall be confirmed by periodic checks. The temperature  $T_Q$  of the gas during the measurement of throughput shall be known to within  $\pm 0.1$  percent, corresponding to  $\pm 0.3$  K at 300 K.

### **4.3.5** *Other Aspects of Calibration Technique*

#### **4.3.5.1** *Attainment of equilibrium*

In order that errors due to adsorption and desorption effects are made negligibly small, it is essential to wait for a sufficient time for all indications and readings to reach constancy within appropriate limits of uncertainty.

#### **4.3.5.2** *Temperature requirements*

The temperature of the gas throughput meter,  $T_Q$  and of the calibration chamber  $T_c$ , shall not deviate from the reference temperature  $T_0$  by more than  $\pm 10$  °C. The reference temperature shall lie in the range 20 °C to 25 °C but preferably at 23 °C. The temperature of the calibration chamber and orifice  $T_c$  and that of the throughput meter  $T_Q$  shall be kept constant during the period of measurements, this can be advantageously accomplished by means of a liquid closed-loop system. The values of  $T_c$  and  $T_Q$  shall be measured to within about  $\pm 0.1$  percent, corresponding to  $\pm 0.3$  K at 300 K.

## **4.4** *Choice of Calibration Gas*

### **4.4.1** *Purity*

For the calibration procedure pure gases should preferably be used. In each case the maximum content of any additional components present shall be known, so that their influence upon the calibration result can be evaluated. It shall be ensured that such additional components do not cause errors exceeding 0.1 percent.

#### 4.4.2 Drying

The gases shall be dried to a dewpoint which corresponds to a water vapour pressure of less than one part in  $10^5$  parts of gas by volume. This corresponds to a pressure  $p^\circ$  of 1 atm at a dewpoint of approximately  $-60^\circ\text{C}$ .

#### 4.4.3 Preferred Gases

For the calibration procedure, argon and nitrogen are preferred unless otherwise specified. In some technical applications, however, air is frequently used. As the molecular masses of the main components of air are very similar, it is permissible to take air as a gas of uniform composition as long as it is ascertained that all components are about equally well pumped by the pumping system. Ion pumps shall not be used in the latter case. An effective molecular mass for air shall be used which is given by:

$$M_{eff} = \left( \sum_i X_i \sqrt{M_i} \right)^2 = 28.9 \dots\dots\dots (6)$$

where  $X_i$  is the mole fraction of the component of molecular mass  $M_i$ . However, the calibration may correspond to a changed composition of air depending on the characteristics of the variable leak valve. If this valve exhibits molecular flow, the composition of the air in the calibration vessel is unchanged:

$X_i = 78.1$  percent,  $21.0$  percent and  $0.9$  percent for nitrogen, oxygen and argon respectively.

If the valve exhibits purely viscous flow, the composition of the air in the calibration vessel is:

$X_i = 76.8$  percent,  $22.1$  percent and  $1.1$  percent for nitrogen, oxygen and argon respectively.

4.4.3.1 Possible effects in calibration with gas mixtures are given in Annex D.

### 4.5 Interpretation of Measurements

#### 4.5.1 Effects of Non-uniformity of Temperature

In the ideal case where all parts of the equipment are at the same temperature, the pressure activating the gauge is given in terms of the throughput  $Q$  and the volume rate of flow of the orifice  $S$  by equation (2) (*see 4.1*). If, with the device for measuring the gas throughput  $Q$  at the absolute temperature  $T_Q$  and with the calibration chamber and therefore also the orifice at the absolute temperature  $T_c$  the calibration has to be related to a reference absolute temperature  $T_o$ , then instead of equation (2), the following equations apply:

a) For a gauge that responds to the pressure of the gas:

$$p_1 = \frac{Q}{S T_o} \frac{\sqrt{T_o T_c}}{T_Q} \dots\dots\dots (7)$$

b) For a gauge that responds to the density of the gas:



$$p_1 = \frac{Q}{S} \frac{T_0}{T_Q} \sqrt{\frac{T_0}{T_c}} \dots\dots\dots (8)$$

Equations (7) and (8) give the pressures  $p_1$  which should be used in determining the sensitivity of the gauge to be calibrated, if the calibration is to be related to the absolute temperature  $T_0$ .

**4.5.2 Corrections for Departures from the Ideal Gas Law**

In the case of real gases a correction may be necessary to account for departure from the ideal gas law over the range from the initial pressure  $p^0$  down to the very small calibration pressure  $p$ . If  $\alpha$  is the ratio of the  $pV$ -values at the expansion pressure  $p \rightarrow 0$  and the pressure  $p^0$  at the same temperature  $T_Q$ , then the true pressure in the calibration chamber is given by the following formula:

$$p_{\text{true}} = p \alpha, \text{ with } \alpha = \frac{pV(p^0 \rightarrow 0, T_Q)}{pV(p^0, T_Q)} \dots\dots\dots (9)$$

Here  $\alpha$  refers to the gas at temperature  $T_Q$ . If the initial starting pressure  $p^0$  is approximately 1 atm, then for the following gases the correction is less than  $\pm 0.1$  percent and may therefore be neglected:

Helium, neon, argon, hydrogen, oxygen, nitrogen, air free from carbon dioxide, and carbon monoxide.

**4.5.2.1** Correction factors for various gases are given in Annex E.

**4.5.3 Limits of Uncertainty**

Careful attention shall be paid to all factors influencing the accuracy of measurements and consideration of the corresponding limits of uncertainty (*see* Annex C).

**5 TEST REPORT**

As far as applicable to the various types of gauges, the test report shall contain the following information:

- a) The date of calibration;
- b) The manufacturer of the gauge, the model and serial number;
- c) The position and geometry of the connecting flange during calibration, and the orientation of the gauge;
- d) The geometry of important parts of the gauge and the material, as far as this is disclosed by the manufacturer, and if relevant to the purpose of the calibration;
- e) The temperature of the calibration chamber, of the gas throughput meter and of the ambient space;
- f) The reference temperature  $T_0$  for which the calibration is valid;
- g) The temperature and duration of baking, de-gassing and ageing;
- h) The previous history of the gauge as far as this is essential for the calibration;
- i) If the calibration of the gauge head is carried out separately from the supply unit, all currents, voltages and the other relevant quantities recorded during the calibration procedure;
- j) If the calibration of the gauge head is carried out together with the supply unit, all values set at the supply unit, the ranges used and the mains voltage;
- k) The calibration curve which shows the sensitivity related to the reference temperature as a function of pressure;
- l) The effective volume rate of flow at the calibration chamber during calibration;
- m) The composition of the gas used for calibration;

- n) The details of the calibration method used, including reference to any appropriate national or international standard;
- o) The limits of uncertainty of the calibration;
- p) Any noted tendency for the gauges to act as sources or sinks of gas; and
- q) The limits of uncertainty within which the calibration system is maintained in equilibrium during calibration.

**ANNEX A**

*(Clause 3.4, 3.5 and 4.2.3.2)*

**CORRECTIONS INVOLVED IN CALCULATION OF  
CONDUCTANCE OF ORIFICE**

**A-1 GENERAL**

**A-1.1** This annex gives further information on the values of the two correction factors  $K_1$  and  $K_2$  which may be involved in the calculation of the conductance of the orifice by means of the formula given in 3.4.

**A-2 CLAUSING FACTOR**

**A-2.1** The clausing factor allowing for thickness of orifice is given below:

<i>Sl No.</i>	<i>Thickness Radius</i>	<i>Clausing Factor, <math>K_1</math></i>
(1)	(2)	(3)
i)	0.001	0.999 5
ii)	0.002	0.999 0
iii)	0.003	0.998 5
iv)	0.004	0.998 0
v)	0.005	0.997 5
vi)	0.006	0.997 0
vii)	0.007	0.996 5
viii)	0.008	0.996 0
ix)	0.009	0.995 5
x)	0.010	0.995 0
xi)	0.011	0.994 5
xii)	0.012	0.994 0
xiii)	0.013	0.993 5
xiv)	0.014	0.993 0
xv)	0.015	0.992 6
xvi)	0.016	0.992 1
xvii)	0.017	0.991 6
xviii)	0.018	0.991 1
xix)	0.019	0.990 6
xx)	0.020	0.990 1

**A-3 UNCERTAINTY LIMITS**

**A-3.1 Relation between Orifice Dimensions and Uncertainty Limits of Calibration Pressure**

At the higher end of the pressure range used in the calibration, the mean-free path  $l$  of the molecules may no longer be very large compared with the radius  $r$  of the orifice. This resulting error may be corrected by the factor  $K_2$ , where

$$K_2 = 1 + \frac{r}{4l} \dots\dots\dots (10)$$

It is desirable that  $K_2$  should not exceed 1.03. If then one assumes an uncertainty in this correction of  $\pm 10$  percent, the maximum uncertainty resulting from the determination of the conductance will not exceed  $\pm 0.3$  percent. This implies that, in the case of argon at a pressure of  $10^{-1}$  Pa, the radius  $r$  of the orifice shall not exceed 5 mm.

**ANNEX B**  
*(Clause 4.1.2 and 4.2.3.1)*

**PREREQUISITES FOR THE APPLICABILITY OF EQUATION (2)**

**B-1 PREREQUISITES**

**B-1.1** Equation (2) holds strictly only in the case of an undistorted distribution function of the gas molecules in the calibration chamber. A distortion of the distribution may arrive from either the gas inlet or the gas outlet regions, and this may restrict the applicability of equation (2) in three ways:

- a) The gas pressure in the calibration vessel may no longer be well defined in the sense of statistical mechanics;
- b) The gas density may no longer be uniform; and
- c) The calculation of the conductance of the orifice from its geometry may require a gas-kinetic correction.

**B-1.1.1** For these reasons the chamber, gas inlet and orifice shall be so dimensioned and arranged that the distortion becomes sufficiently small. In a spherical calibration chamber the distortion caused by gas inlet and gas sink may easily be evaluated. In the case of molecules characterized by an undistorted velocity distribution function an equal number of molecules impinges on all unit surface elements of the inner surface of the hollow sphere. Also, the molecules emitted by any surface element are distributed evenly over the inner surface, provided Lambert's law (cosine law) applies. It follows, as an adequate approximation in the case of molecular flow that the proportionate disturbance caused by a circular hole, such as the outlet orifice does not exceed the ratio of the cross-sectional area of the orifice to the inner surface area of the sphere. If the vessel is not a hollow sphere, the greatest inscribed sphere shall be used as a basis for the dimensioning of the system. The geometry of the gas inlet shall take account of the fact that the directions of the entering molecules usually do not comply with the cosine law. This is guaranteed, to a great extent, after the first impact on a wall.

**B-1.1.2** Similarly, the volume between the orifice and the pumping system shall be as large as the calibration chamber to ensure an undisturbed velocity distribution function in the volume which is responsible for back diffusion of the calibration gas through the orifice.

**ANNEX C**  
*(Clause 4.5.3)*

**DISCUSSION OF ERRORS**

**C-1 GENERAL EXPRESSION FOR THE ERROR**

According to equations (7) and (8) the pressure  $p_1$  is given by:

$$p_1 = \frac{Q \sqrt{T_0 T_c}}{s T_0 T_Q} \text{ or } p_1 = \frac{Q T_0}{s T_0 T_Q} \sqrt{\frac{T_0}{T_c}}$$

Using equation (7) and taking into consideration the equations (3) and (1) it follows that:

$$p_1 = \frac{Q}{A_L \frac{c}{4} K_1 K_2 \left( \frac{1}{1+L/S_p} \right) T_Q} \sqrt{T_0 T_c} \dots\dots\dots (11)$$

and with a corresponding expression when equation (8) is used. From this the relative error is given by:

$$\frac{\delta p_1}{p_1} = \frac{\delta Q}{Q} + \frac{\delta A_L}{A_L} + \frac{\delta K_2}{K_2} + \frac{\delta \beta}{\beta} + \frac{1}{2} \frac{\delta T_c}{T_c} + \frac{\delta T_Q}{T_Q} \dots\dots\dots (12)$$

In this equation,  $\beta = \frac{1}{1+L/S_p}$

Because of this smallness the relative error of the clausius correction has not been taken into consideration. A corresponding expression may be obtained for the error in the case of the Oatley procedure, for determining the effective volume rate of flow of the pumping system (see 4.23).

**C-2 MAGNITUDE OF TOTAL ERROR**

The total relative error (see equation 12) is numerically made up in the following way:

- a)  $\frac{\delta Q}{Q}$  shall be of the order of  $\pm 0.5$  percent and depends on the type of throughput meter used. The relative error of the throughput measurement shall be determined under the prevailing conditions for each gas and pressure setting;
- b)  $\frac{\delta A_L}{A_L} = 0.1$  percent (see 3.4);
- c)  $\frac{\delta K_2}{K_2} = 0.3$  percent (see Annex A);
- d)  $\frac{\delta \beta}{\beta} = 0.4$  percent (see 3.8 and 4.2.3);
- e)  $\frac{\delta T_c}{T_c} = 0.1$  percent (see 3.6); and
- f)  $\frac{\delta T_Q}{T_Q} = 0.1$  percent (see 4.3.4).

The total relative error therefore is:

$$\frac{\delta p_1}{p_1} \leq 1 \text{ percent} + \frac{\delta Q}{Q} \dots\dots\dots (13)$$

The relative error  $\frac{\delta Q}{Q}$  shall be determined afresh for each measurement.

### **C-3 DIMENSIONING OF THE APPARATUS**

Dimensions for the various components of the apparatus are not given. However, the dimensions, particularly of the gas throughput meter, shall be such that the expression  $\delta Q/Q$  becomes a minimum. The most suitable dimensions depend essentially upon the properties of the components and their stability in time, as well as upon a number of other requirements such as the pressure range of the calibration, the time required for one calibration.

## **ANNEX D**

*(Clause 4.4.3.1)*

### **ASPECTS OF CALIBRATION USING GAS MIXTURES**

#### **D-1 GENERAL**

Complications may arise from the use of gas mixtures due to the fact that molecular flow through the orifice causes the components to become separated if the molecular masses are different. Similarly, separation may occur during the flow from the gas throughput meter, through the variable leak valve, into the calibration vessel. Such errors caused by the variable leak valve can be eliminated by using an uncalibrated fixed leak which exhibits either:

- a) Purely viscous flow; or
- b) Purely molecular flow, in the pressure range used.

It should also be noted that the effective volume rates of flow of the pumps and the mean-free-paths of the molecules flow are different for the various components.

**D-2 USE OF GAS MIXTURES WITH A VISCOUS FLOW LEAK**

**D-2.1** When using a fixed leak exhibiting purely viscous flow for the expansion, instead of the equations (7) and (8) the following expressions shall be used:

- a) For gauges responding to pressure:

$$p_1 = \frac{Q\sqrt{T_0T_c}}{T_Q} \sum_i \frac{X_i}{S_iT_0} \dots\dots\dots (14)$$

- b) For gauges responding to density:

$$p_1 = \frac{QT_0}{T_Q} \sqrt{\frac{T_0}{T_c}} \sum_i \frac{X_i}{S_iT_0} \dots\dots\dots (15)$$

where

$X_i$  = The mole fraction of the gas component  $i$  with  $\sum_i X_i = 1$ ; and  
 $S_i$  = The corresponding rate of flow.

The calibration now no longer refers to the original gas mixture having the molecular fractions  $X_1$ , but to a mixture in the expansion volume with the new molecular fractions.

$$X_{ii} = \frac{X_i\sqrt{M_i}}{\sum_i X_i\sqrt{M_i}}$$

**D-3 USE OF GAS MIXTURES WITH A MOLECULAR FLOW LEAK**

When using a fixed leak exhibiting purely molecular flow for the expansion, the equations (7) and (8) remain valid if the effective molecular mass:

$$M_{eff} = \left( \sum_i X_i\sqrt{M_i} \right)^2$$

is substituted for  $S$  in the expression, the calibration referring to the original gas mixture.





**ANNEX E**  
(Clause 4.5.2.1)

**TABLE OF CORRECTIONS ARISING FROM DEPARTURES FROM THE IDEAL GAS LAW**

**E-1 CORRECTION FACTOR**

The following table lists the correction factor  $\alpha$  given by the expression:

$$\alpha = \frac{pV(p \rightarrow 0, 25^\circ\text{C})}{pV(1 \text{ atm}, 25^\circ\text{C})}$$

corresponding to the gas temperature 25 °C, by which the pressure  $p$  estimated from equations (7) and (8) shall be multiplied to obtain the true pressure,

$$p_{\text{true}} = p \alpha$$

He	0.999 5
H <sub>2</sub> , D <sub>2</sub>	0.999 5
NH <sub>3</sub>	1.012 0
CH <sub>4</sub>	1.001 9
C <sub>3</sub> H <sub>6</sub>	1.014 4
Ne	0.999 6
O <sub>2</sub>	1.000 6
N <sub>2</sub> O	1.005 0
C <sub>2</sub> H <sub>6</sub>	1.007 8
Ar	1.000 7
SF <sub>6</sub>	1.011 7
CO	1.000 4
C <sub>2</sub> H <sub>4</sub>	1.005 4
Kr	1.002 2
N <sub>2</sub>	1.000 2
CO <sub>2</sub>	1.005 5
C <sub>2</sub> H <sub>2</sub>	1.006 9
Xe	1.005 5
Air free from CO <sub>2</sub>	1.000 4
CF <sub>4</sub>	1.003 8
C <sub>3</sub> H <sub>8</sub>	1.015 4

The figures given in this table refer to the case where the gas is expanded at room temperature starting from about 1 atm.