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**Rubber, vulcanized or  
thermoplastic — Determination of  
permeability to gases —**

**Part 1:  
Differential-pressure methods**

*Caoutchouc vulcanisé ou thermoplastique — Détermination de la  
perméabilité aux gaz —*

*Partie 1: Méthodes à pression différentielle*



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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: [Foreword — Supplementary information](#).

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 2782-1:2012), which has been technically revised. A new Clause on simplified pressure sensor method has been added (see [Clause 6](#)).

ISO 2782 consists of the following parts, under the general title *Rubber, vulcanized or thermoplastic — Determination of permeability to gases*:

- *Part 1: Differential-pressure methods*
- *Part 2: Equal-pressure method*

## Introduction

The measurement of the permeability of rubber to gases is important in the evaluation of compounds for products such as inner tubes, tubeless-tyre liners, hoses, balloons and other gas-containing products, as well as seals and diaphragms. The measurement is also of theoretical importance in the study of the characteristics of gas diffusion and gas solubility in relation to polymer structure.

This part of ISO 2782 proposes three different methods for the determination of the permeability to gases of vulcanized or thermoplastic rubber under a differential partial pressure, which are

- pressure sensor method which allows a complete characterization of a material,
- simplified pressure sensor method which is appropriate when only the gas permeability coefficient is needed (routine control, specification verification, design, etc.), and
- gas-chromatographic method.

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# Rubber, vulcanized or thermoplastic — Determination of permeability to gases —

## Part 1: Differential-pressure methods

**WARNING 1** — Persons using this part of ISO 2782 should be familiar with normal laboratory practice. This part of ISO 2782 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

**WARNING 2** — Certain procedures specified in this part of ISO 2782 might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

### 1 Scope

This part of ISO 2782 specifies three methods for the determination of the permeability to gases of vulcanized or thermoplastic rubber under a differential partial pressure.

The three methods specified are as follows:

- pressure sensor method (using vacuum): for determining the gas transmission rate, gas permeability coefficient, gas diffusion coefficient and gas solubility coefficient;
- simplified sensor method (using applied pressure): for determining the gas permeability coefficient only;
- gas-chromatographic method: for determining the gas transmission rate and gas permeability coefficient.

These methods apply to vulcanized and thermoplastic rubbers of hardness not less than 35 IRHD (international rubber hardness degrees) and to both single gases and mixtures of gases.

### 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

ISO 23529:2010, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

#### 3.1

##### **gas transmission rate**

number of moles of test gas passing through a test piece per unit area, per unit time, with unit partial-pressure difference between the two sides of the test piece

#### 3.2

##### **gas permeability coefficient**

number of moles of test gas passing through a test piece of unit thickness, per unit area, per unit time, with unit partial-pressure difference between the two sides of the test piece

#### 3.3

##### **gas diffusion coefficient**

quantity of test gas passing, by diffusion, through a test piece of unit thickness, per unit area, per unit time, where there is a unit gas concentration gradient across the test piece

#### 3.4

##### **gas solubility coefficient**

test gas concentration inside a test piece divided by the partial pressure of the test gas at the surface of the test piece

#### 3.5

##### **gas transmission curve**

<pressure sensor method> curve, plotted against time, of the pressure change on the low-pressure side of the test cell until gas transmission reaches a steady state

Note 1 to entry: A gas transmission curve is illustrated in [Figure 2](#).

### 4 Principle

The cavity of a test cell, maintained at a constant temperature, is divided by a test piece into a high-pressure and a low-pressure side. The high-pressure side of the cell is filled with the test gas. The quantity of gas that diffuses through the test piece to the low-pressure side is determined by a pressure sensor or by a gas chromatograph.

In the pressure sensor method, the gas transmission rate, gas permeability coefficient, gas diffusion coefficient and gas solubility coefficient are measured, producing an average value for a gas mixture. In the simplified pressure sensor method only the gas permeability coefficient is measured. In the gas-chromatographic method, measurements are possible on test gases containing water vapour, and it is also possible to analyse mixtures of gases to determine the components.

### 5 Pressure sensor method

#### 5.1 Apparatus

The apparatus consists of the test cell, pressure sensors, a test gas supply reservoir, a vacuum pump and associated tubing and valves. An example of a test apparatus is shown in [Figure 1](#).

**5.1.1 Test cell**, consisting of a low-pressure side and a high-pressure side, such that, when a test piece is mounted in it, the gas transmission area is clearly defined. The high-pressure side has an inlet port to supply test gas, and a pressure sensor is connected to the low-pressure side to detect the change in pressure caused by the gas transmitted through the test piece. The surfaces of the two halves of the cell which make contact with the test piece shall be smooth and flat to prevent any leakage of gas. A seal such as an O-ring may be used between these areas and the test piece, in which case the gas transmission rate of the seal shall be considerably lower than that of the material being tested so that it does not affect the

result of the test. The material of the test cell shall be unreactive with regard to the test gas and shall not absorb the gas used. The diameter of the gas transmission area shall be within the range 10 mm to 150 mm, depending on the gas transmission rate expected.

The cell shall be equipped with a heating system capable of raising the temperature to 80 °C. The temperature accuracy shall be  $\pm 1$  °C for temperatures from 40 °C to 80 °C.

NOTE Examples of a heating system are an electric heating jacket and an oven designed to hold the test cell and test gas supply reservoir.

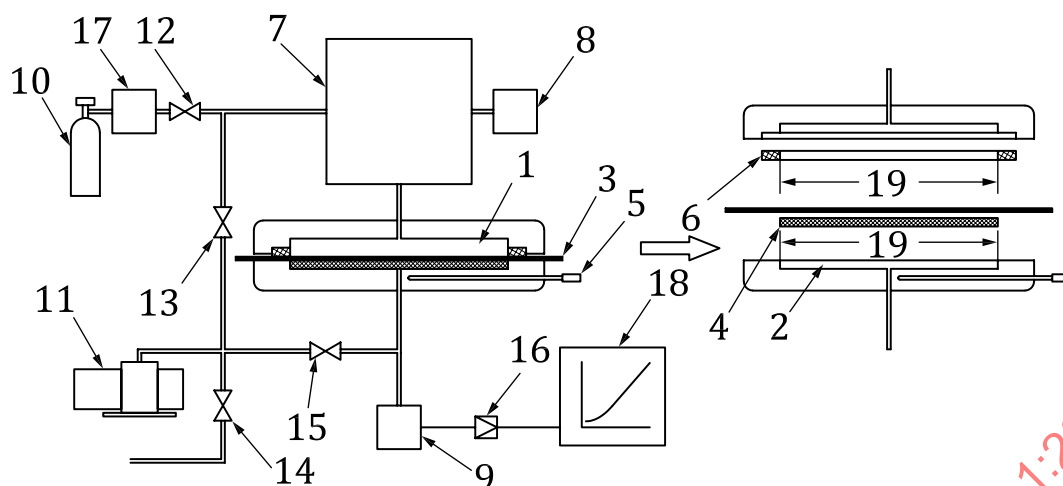
**5.1.2 Test piece support**, installed on the low-pressure side of the test cell in order to prevent deformation of the test piece due to the pressure difference between the high- and low-pressure sides. Any material, such as filter paper or wire mesh, that does not affect the result of the test may be used. When using filter paper, paper such as that used in chemical analysis is recommended, of thickness 0,1 mm to 0,3 mm, depending on the depth of the low-pressure side of the cell.

**5.1.3 Two pressure sensors**, the first, capable of reading to within 5 Pa or better, to measure the change in pressure on the low-pressure side of the test cell. A vacuum gauge with no mercury, an electronic diaphragm-type sensor or other suitable sensor shall be used as this pressure sensor. The second, capable of reading to within 1 % or better, is used to measure the pressure of the test gas supply reservoir.

**5.1.4 Test gas supply reservoir**, for supplying test gas at a constant pressure to the high-pressure side of the test cell. The volume of the reservoir shall be sufficient to ensure that the pressure drop on the high-pressure side, due to transmission of the test gas through the test piece to the low-pressure side during the test, does not exceed 1 % of the test pressure.

**5.1.5 Vacuum pump**, capable of evacuating the test cell to a pressure of 10 Pa or lower.

**5.1.6 Temperature sensor**, fitted in the test cell, for measuring the test temperature, and capable of reading to within 0,1 °C or better.



### Key

|    |                                                    |    |                                   |
|----|----------------------------------------------------|----|-----------------------------------|
| 1  | high-pressure side of test cell                    | 11 | vacuum pump                       |
| 2  | low-pressure side of test cell                     | 12 | valve 1                           |
| 3  | test piece                                         | 13 | valve 2                           |
| 4  | test piece support                                 | 14 | valve 3                           |
| 5  | temperature sensor                                 | 15 | valve 4                           |
| 6  | sealing ring                                       | 16 | signal amplifier                  |
| 7  | test gas supply reservoir                          | 17 | pressure-reducing valve           |
| 8  | pressure gauge for test gas supply reservoir       | 18 | data-processing unit              |
| 9  | pressure sensor for low-pressure side of test cell | 19 | diameter of gas transmission area |
| 10 | test gas cylinder                                  |    |                                   |

NOTE Diagram at right shows an exploded view of the test cell.

**Figure 1 — Example of apparatus for gas permeability measurement (pressure sensor method)**

## 5.2 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in [Annex A](#).

## 5.3 Test gas

Use a single gas, such as nitrogen, oxygen or hydrogen, or a mixture of gases, such as air, liquefied petroleum gas (in gaseous form) or coal gas. The purity of a single gas or the purity of each component in a gas mixture shall be 99,5 % by volume or higher, unless otherwise agreed between the interested parties, in which case a gas of lower purity may be used. The test gas shall not include any impurity that might affect the measurement.

When using a gas mixture, the purity of each component shall be verified in advance with a suitable instrument, such as a gas chromatograph.

When using a toxic and/or flammable gas, all necessary precautions should be taken in its use and in its recovery or disposal.

## 5.4 Test pieces

### 5.4.1 Shape and dimensions

The test pieces shall be of uniform shape and have a thickness of more than 0,1 mm but less than 2,2 mm, unless otherwise agreed between the interested parties. The test pieces shall be large enough to extend across the full width of the test cell and to be clamped between the flat edges of the two halves of the test cell (see [5.1.1](#)).

### 5.4.2 Preparation

The test pieces shall be cut out and prepared in accordance with ISO 23529. Any test piece containing foreign matter, air bubbles, scratches or holes shall be discarded.

### 5.4.3 Number of test pieces

Three or more test pieces shall be used except when testing for quality control purposes, in which case the number of test pieces may be reduced.

### 5.4.4 Measurement of thickness

Measure the thickness of each test piece at five or more points, including the centre of the gas transmission area, to the nearest 0,01 mm using method A specified in ISO 23529:2010, and take the arithmetic mean of the measurements. No single measurement on a test piece shall deviate by more than 10 % from the mean for that test piece, and the mean value for any one test piece shall not deviate by more than 10 % from the mean value for all the test pieces measured.

### 5.4.5 Time interval between forming and testing

The time interval between forming and testing shall be in accordance with ISO 23529.

Samples and test pieces shall be stored in accordance with ISO 23529 during the interval between forming and testing.

## 5.5 Conditioning

The minimum time between forming and the commencement of conditioning of the test pieces shall be 16 h.

Unless otherwise required in the material specification or agreed between the interested parties, the material shall be conditioned before testing for 16 h to 24 h at a standard laboratory temperature and humidity specified in ISO 23529. When using a test piece that can be easily affected by moisture, dry it for more than 48 h at the test temperature in a desiccator containing a suitable drying agent, such as anhydrous calcium chloride.

## 5.6 Test conditions

**5.6.1** The laboratory conditions shall be in accordance with ISO 23529.

**5.6.2** The test shall be conducted at the same temperature as was used to condition the test pieces (see [5.5](#)). The test temperature shall be measured by means of the temperature sensor fitted in the test cell.

## 5.7 Gas transmission area

The gas transmission area  $A$  shall be calculated from the internal diameter of the test cell or, if a sealing ring is used, from the internal diameter of the sealing ring.

## 5.8 Procedure

**5.8.1** Fit a suitable test piece support (4 in [Figure 1](#)) on the low-pressure side of the test cell.

**5.8.2** Apply vacuum grease lightly and uniformly to the flat edges of the two halves of the test cell which will make contact with the test piece and mount the test piece in the lower part of the cell, without any wrinkling or sagging.

**5.8.3** Place a sealing ring (if used) on the test piece, followed by the upper part of the cell. Clamp the two halves of the cell together with uniform pressure so that the test piece is completely sealed in place.

**5.8.4** When making measurements at a temperature other than a standard laboratory temperature, bring the test cell to the test temperature.

**5.8.5** Close valve 1 (12 in [Figure 1](#)), valve 2 (13) and valve 3 (14), and open valve 4 (15). Start the vacuum pump and then open valve 2 (13). Air will be evacuated first from the low-pressure side of the test cell, followed by the high-pressure side, so that the test piece fits snugly against the test piece support. Continue until evacuation is complete. Since it is necessary to remove all absorbed gas, allow sufficient evacuation time for a test piece of low gas transmission rate to be thoroughly degassed.

**NOTE** The evacuation time required will differ for different types of sample and different conditioning. A quantitative comparison between different types of sample can be made by fixing the exhaustion time. For samples with a low gas transmission rate, evacuating overnight is a common practice. Note that, with some types of sample, longer evacuation times might remove the more readily vaporized components from the test piece.

**5.8.6** Shut valve 2 (13) and valve 4 (15) to maintain the pressure on both the low-pressure side and the high-pressure side at 10 Pa or less. Stop the vacuum pump.

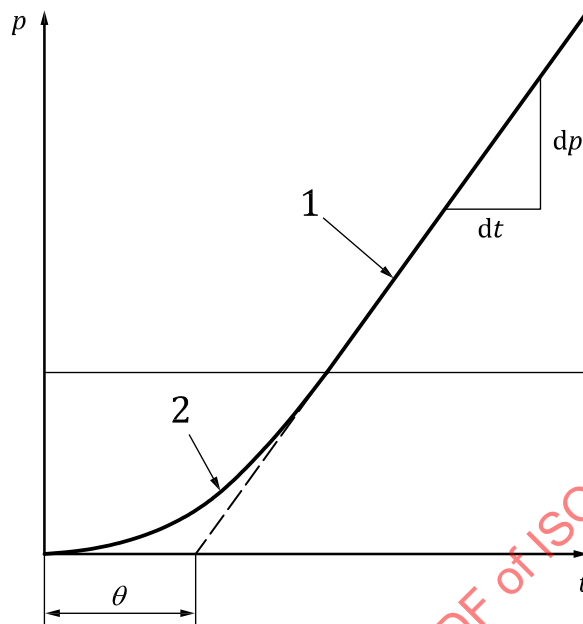
**5.8.7** If the pressure on the low-pressure side rises, repeat steps [5.8.2](#) to [5.8.6](#), as there is a possibility of gas leakage or gas being absorbed by the test piece.

**5.8.8** Introduce the test gas into the high-pressure side by opening valve 1 (12), shutting off the gas supply when the pressure (as measured by the pressure gauge, 8 in [Figure 1](#)) has reached the test pressure. Record the pressure on the high-pressure side  $p_h$  and the temperature  $T$  when the pressure on the low-pressure side starts increasing due to the permeation of the test gas from the high-pressure side to the low-pressure side.

**5.8.9** Draw the gas transmission curve by plotting the pressure on the low-pressure side against time. Continue taking measurements until a constant rate of gas transmission has been reached, as indicated by a straight line (see [Figure 2](#)). A gas transmission curve plotted by automatic recording may also be used.

**5.8.10** Determine the slope of the straight-line portion of the curve ( $dp/dt$ , see [Figure 2](#)). The slope may also be determined automatically by the recorder.

**5.8.11** When determining the gas diffusion coefficient, extrapolate the straight part of the gas transmission curve back to the x-axis and determine the delay time  $\theta$  (see [Figure 2](#)).



**Key**

- $t$  time (s)
- $p$  pressure (Pa)
- 1 steady-state region
- 2 non-steady-state region

**Figure 2 — Gas transmission curve**

## 5.9 Calculation and expression of results

### 5.9.1 Gas transmission rate

The gas transmission rate is determined from [Formula \(1\)](#):

$$\text{GTR} = \frac{V_c}{R \times T \times p_h \times A} \times \frac{dp}{dt} \quad (1)$$

where

GTR is the gas transmission rate [mol/(m<sup>2</sup>·s·Pa)];

$V_c$  is the volume of the low-pressure side of the test cell (m<sup>3</sup>);

$T$  is the test temperature (K);

$p_h$  is the pressure of the test gas on the high-pressure side of the test cell (Pa);

$A$  is the gas transmission area (m<sup>2</sup>);

$dp/dt$  is the pressure change on the low-pressure side of the test cell per unit time (Pa/s);

$R$  is the gas constant [8,31 m<sup>3</sup>·Pa/(K·mol)].

Express the gas transmission rate as the arithmetic mean of the results obtained for all the test pieces.

### 5.9.2 Gas permeability coefficient

The gas permeability coefficient is determined from [Formula \(2\)](#):

$$Q = \text{GTR} \times d \quad (2)$$

where

$Q$  is the gas permeability coefficient [ $\text{mol} \cdot \text{m}/(\text{m}^2 \cdot \text{s} \cdot \text{Pa})$ ];

GTR is the gas transmission rate [ $\text{mol}/(\text{m}^2 \cdot \text{s} \cdot \text{Pa})$ ];

$d$  is the thickness of the test piece (m).

Express the gas permeability coefficient as the arithmetic mean of the results obtained for all the test pieces.

### 5.9.3 Gas diffusion coefficient

The gas diffusion coefficient is determined from [Formula \(3\)](#):

$$D = \frac{d^2}{6\theta} \quad (3)$$

where

$D$  is the gas diffusion coefficient ( $\text{m}^2/\text{s}$ );

$\theta$  is the delay time (s);

$d$  is the thickness of the test piece (m).

Express the gas diffusion coefficient as the arithmetic mean of the results obtained for all the test pieces.

### 5.9.4 Gas solubility coefficient

The gas solubility coefficient is determined from [Formula \(4\)](#):

$$S = \frac{Q}{D} \quad (4)$$

where

$S$  is the gas solubility coefficient [ $\text{mol}/(\text{m}^3 \cdot \text{Pa})$ ];

$Q$  is the gas permeability coefficient [ $\text{mol} \cdot \text{m}/(\text{m}^2 \cdot \text{s} \cdot \text{Pa})$ ];

$D$  is the gas diffusion coefficient ( $\text{m}^2/\text{s}$ ).

Express the gas solubility coefficient as the arithmetic mean of the results obtained for all the test pieces.

## 6 Simplified pressure sensor method

### 6.1 Apparatus

The apparatus consists of the test cell, pressure sensors, a test gas supply reservoir, and associated tubing and valves. An example of a test apparatus is shown in [Figure 3](#).

**6.1.1 Test cell**

See 5.1.1.

**6.1.2 Test piece support**

See 5.1.2.

**6.1.3 Two pressure sensors**

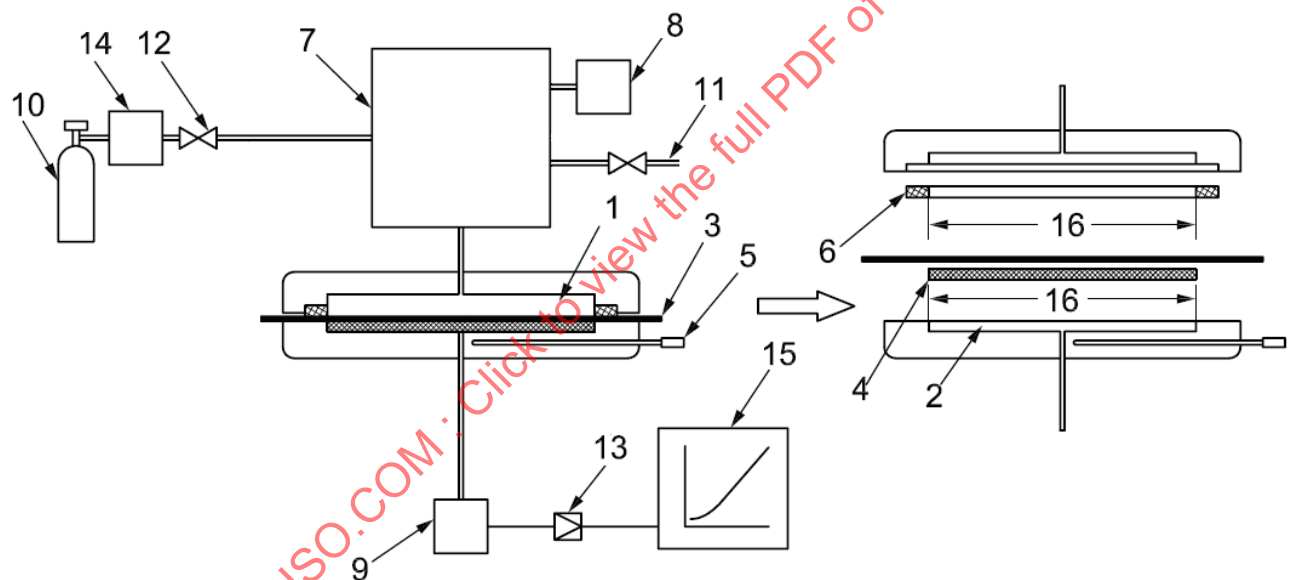
See 5.1.3.

**6.1.4 Test gas supply reservoir**

See 5.1.4.

**6.1.5 Temperature sensor**

See 5.1.6.

**Key**

|   |                                              |    |                                                    |
|---|----------------------------------------------|----|----------------------------------------------------|
| 1 | high-pressure side of test cell              | 9  | pressure sensor for low-pressure side of test cell |
| 2 | low-pressure side of test cell               | 10 | test gas cylinder                                  |
| 3 | test piece                                   | 11 | valve 1                                            |
| 4 | test piece support                           | 12 | valve 2                                            |
| 5 | temperature sensor                           | 13 | signal amplifier                                   |
| 6 | sealing ring if necessary                    | 14 | pressure-reducing valve                            |
| 7 | test gas supply reservoir                    | 15 | data-processing unit                               |
| 8 | pressure gauge for test gas supply reservoir | 16 | diameter of gas transmission area                  |

NOTE Diagram at right shows an exploded view of the test cell.

**Figure 3 — Example of apparatus for gas permeability measurement (simplified pressure sensor method)**

## 6.2 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in [Annex A](#).

## 6.3 Test gas

See [5.3](#).

## 6.4 Test pieces

### 6.4.1 Shape and dimensions

See [5.4.1](#).

### 6.4.2 Preparation

See [5.4.2](#).

### 6.4.3 Number of test pieces

See [5.4.3](#).

### 6.4.4 Measurement of thickness

See [5.4.4](#).

### 6.4.5 Time interval between forming and testing

See [5.4.5](#).

## 6.5 Conditioning

See [5.5](#).

## 6.6 Test conditions

See [5.6](#).

## 6.7 Gas transmission area

See [5.7](#).

## 6.8 Procedure

**6.8.1** Fit a suitable test piece support (4 in [Figure 3](#)) on the low-pressure side of the test cell.

**6.8.2** See [5.8.2](#).

**6.8.3** See [5.8.3](#).

**6.8.4** When making measurements at a temperature other than a standard laboratory temperature, bring the test cell to the test temperature prior to clamping the two halves of the cell. After the test piece is mounted, condition it according to ISO 23529.

**6.8.5** Before beginning each test, make sure that the high pressure chamber is entirely filled with the test gas by purging it from air. Open valve 1 and valve 2 and let the gas flow for an appropriate time.

**6.8.6** Close valve 1 and maintain valve 2 open in order to introduce the test gas into the high-pressure side, shutting off the gas supply when the pressure (as measured by the pressure gauge, 8 in [Figure 3](#)) has reached the test pressure. Note down the pressure on the high-pressure side  $p_h$  and the temperature  $T$  when the pressure on the low-pressure side starts increasing due to the permeation of the test gas from the high-pressure side to the low-pressure side.

**6.8.7** See [5.8.9](#).

**6.8.8** See [5.8.10](#).

## 6.9 Calculation and expression of results

The gas permeability coefficient is determined from [Formula \(5\)](#):

$$Q = \frac{V_c \times d}{R \times T \times p_h \times A} \times \frac{dp}{dt} \quad (5)$$

The gas permeability coefficient referred to standard temperature and pressure is determined from [Formula \(6\)](#):

$$Q' = \frac{V_c \times d}{R \times T \times p_h \times A} \times \frac{dp}{dt} \times \frac{273}{101\,300} \quad (6)$$

where

- $Q$  is the gas permeability coefficient [ $\text{mol} \cdot \text{m} / (\text{m}^2 \cdot \text{s} \cdot \text{Pa})$ ];
- $Q'$  is the gas permeability coefficient referred to standard temperature and pressure [ $(\text{mol} \cdot \text{m} \cdot \text{K}) / (\text{m}^2 \cdot \text{s} \cdot \text{Pa}^2)$ ];
- $V_c$  is the volume of the low-pressure side of the test cell ( $\text{m}^3$ );
- $T$  is the test temperature (K);
- $p_h$  is the relative pressure of the test gas on the high-pressure side of the test cell (Pa);
- $A$  is the gas transmission area ( $\text{m}^2$ );
- $dp/dt$  is the pressure change on the low-pressure side of the test cell per unit time (Pa/s);
- $d$  is the thickness of the test piece (m);
- $R$  is the gas constant [ $8,31 \text{ m}^3 \cdot \text{Pa} / (\text{K} \cdot \text{mol})$ ].

Express the gas permeability coefficient as the arithmetic mean of the results obtained for all the test pieces.

## 7 Gas-chromatographic method

### 7.1 Apparatus

The apparatus consists of the test cell, a gas chromatograph, a test gas controller, a sampling loop, a vacuum pump and associated tubing and valves. An example of a test apparatus is shown in [Figure 4](#).

**7.1.1 Test cell**, in accordance with 5.1.1 except that the low pressure side is connected to a gas chromatograph through a sampling loop to detect the transmitted gas (see 7.1.3 and Figure 4).

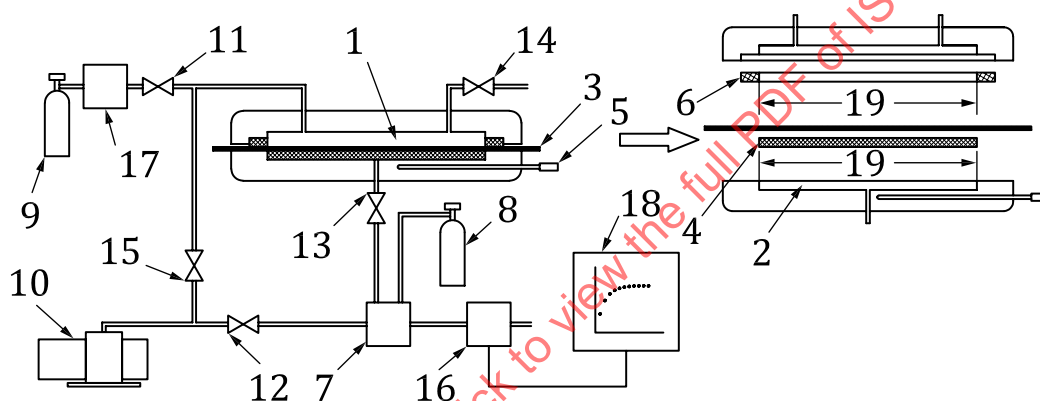
**7.1.2 Test piece support**, in accordance with 5.1.2.

**7.1.3 Gas chromatograph**, having a detector such as a thermal conductivity detector (TCD) or a hydrogen flame ionization detector (FID). The detector and column shall be suitable for the test gas used and of the required sensitivity. The gas chromatograph shall be capable of measuring the quantity of gas which permeates through the test piece with an accuracy of 5 Pa or better, expressed in terms of the pressure of the gas.

**7.1.4 Test gas controller**, capable of controlling the test gas flow rate and pressure, and of maintaining them constant. A flowmeter having an accuracy of  $\pm 3\%$  or better shall be used.

**7.1.5 Vacuum pump**, in accordance with 5.1.5.

**7.1.6 Temperature sensor**, in accordance with 5.1.6.



#### Key

|    |                                 |    |                                   |
|----|---------------------------------|----|-----------------------------------|
| 1  | high-pressure side of test cell | 11 | valve 1                           |
| 2  | low-pressure side of test cell  | 12 | valve 2                           |
| 3  | test piece                      | 13 | valve 3                           |
| 4  | test piece support              | 14 | valve 4                           |
| 5  | temperature sensor              | 15 | valve 5                           |
| 6  | sealing ring                    | 16 | gas chromatograph                 |
| 7  | sampling loop                   | 17 | test gas controller               |
| 8  | carrier gas cylinder            | 18 | data-processing unit              |
| 9  | test gas cylinder               | 19 | diameter of gas transmission area |
| 10 | vacuum pump                     |    |                                   |

NOTE Diagram at right shows an exploded view of the test cell.

**Figure 4 — Example of apparatus for gas permeability measurement (gas-chromatographic method)**

## 7.2 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in Annex A.

### 7.3 Test gas

See [5.3](#).

### 7.4 Carrier gas

Use a suitable carrier gas of purity preferably greater than 99,99 % by volume.

### 7.5 Test pieces

#### 7.5.1 Shape and dimensions

See [5.4.1](#).

#### 7.5.2 Preparation

See [5.4.2](#).

#### 7.5.3 Number of test pieces

See [5.4.3](#).

#### 7.5.4 Measurement of thickness

See [5.4.4](#).

#### 7.5.5 Time interval between forming and testing

See [5.4.5](#).

### 7.6 Conditioning

See [5.5](#).

### 7.7 Test conditions

See [5.6](#).

### 7.8 Gas transmission area

See [5.7](#).

### 7.9 Calibration curve

Inject a known quantity of the test gas into the gas chromatograph using a syringe or a gas sampler. Determine the area of the peak in the chromatogram corresponding to the gas of interest. Repeat the above measurements with at least three different levels of concentration and prepare a calibration curve from the data obtained.

For a test gas mixture, a calibration curve for each component of the mixture shall be produced.

### 7.10 Procedure

**7.10.1** Fit a suitable test piece support (4 in [Figure 1](#)) on the low-pressure side of the test cell.

**7.10.2** Apply vacuum grease lightly and uniformly to the flat edges of the two halves of the test cell which will make contact with the test piece and mount the test piece in the lower part of the cell, without any wrinkling or sagging.

**7.10.3** Place a sealing ring (if used) on the test piece, followed by the upper part of the cell. Clamp the two halves of the cell together with uniform pressure so that the test piece is completely sealed in place.

**7.10.4** When making measurements at a temperature other than a standard laboratory temperature, bring the test cell to the test temperature.

**7.10.5** Close valve 1 (11 in [Figure 3](#)), valve 4 (14) and valve 5 (15), and open valve 2 (12). Start the vacuum pump and then open valve 5 (15). Air will be evacuated first from the low-pressure side of the test cell, followed by the high-pressure side, so that the test piece fits snugly against the test piece support. Continue until evacuation is complete. Since it is necessary to remove all absorbed gas, allow sufficient evacuation time for a test piece of low gas transmission rate to be thoroughly degassed.

NOTE The evacuation time required will differ for different types of sample and different conditioning. See also the note to [5.8.5](#).

**7.10.6** When all the air has been evacuated, stop evacuating the high-pressure side of the test cell by closing valve 5 (15). Introduce the test gas into the high-pressure side through the test gas controller (17) by opening valve 1 (11), and maintain the high-pressure side at a constant pressure. The test gas will start permeating from the high-pressure side to the low-pressure side and will be evacuated by the vacuum pump. Record the pressure on the high-pressure side  $p_h$  and the temperature  $T$ .

**7.10.7** Close valve 2 (12) to collect gas which has permeated through the test piece in the sampling loop (7). After a predetermined time  $t$  (see the note), close valve 3 (13) and sweep the gas from the sampling loop (7) into the chromatographic column (16) with carrier gas (8). Measure the area of the peak in the chromatogram corresponding to the gas of interest. Determine, from the calibration curve prepared in accordance with [6.9](#), the amount of gas  $V_s$ , in cubic metres, that collected in the sampling loop during time  $t$ .

NOTE The time  $t$  will depend on the permeability to gas of the sample. A suitable time can be determined by carrying out one or more trial runs.

**7.10.8** Repeat the procedure from [7.10.5](#) to [7.10.7](#) until a steady-state is reached. The steady-state is assumed to have been reached when the measurements of the amount of gas which permeates through the test piece in time  $t$  are substantially constant. An apparatus that automatically plots a gas transmission curve from the peak areas in chromatograms at each of a number of predetermined intervals may be used to do this.

**7.10.9** Either before or after the test, carry out a blank run to determine the (small) amount of gas present, under the steady-state conditions, in the sampling loop at the beginning of time  $t$ . Do this by simultaneously closing valve 2 (12) and valve 3 (13), thus trapping the gas which is flowing through the loop under the steady-state conditions, and subsequently determining the amount of gas  $V_b$  trapped.

## 7.11 Calculation and expression of results

### 7.11.1 Gas transmission rate

The gas transmission rate is determined from [Formula \(7\)](#):

$$\text{GTR} = \frac{T_0 \times (V_s - V_b) \times k}{0,022\,7 \times T \times A \times t \times p_h} \quad (7)$$

where