
**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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Published in Switzerland

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Pressure sensor method	2
5.1 Apparatus	2
5.2 Calibration	3
5.3 Test gas	3
5.4 Test pieces	4
5.5 Conditioning	4
5.6 Test conditions	4
5.7 Gas transmission area	4
5.8 Procedure	5
5.9 Calculation and expression of results	6
6 Gas-chromatographic method	7
6.1 Apparatus	7
6.2 Calibration	9
6.3 Test gas	9
6.4 Carrier gas	9
6.5 Test pieces	9
6.6 Conditioning	9
6.7 Test conditions	9
6.8 Gas transmission area	9
6.9 Calibration curve	9
6.10 Procedure	9
6.11 Calculation and expression of results	10
7 Test report	11
Annex A (normative) Calibration schedule	13

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 2782-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Together with Part 2, it cancels and replaces ISO 2782:2006, which has been technically revised.

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.

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

Part 1: Differential-pressure methods

WARNING — 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.

1 Scope

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

The two methods specified are as follows:

- a pressure sensor method: for determining the gas transmission rate, gas permeability coefficient, gas diffusion coefficient and gas solubility coefficient;
- a 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 referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18899:2004, *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 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 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 ± 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.

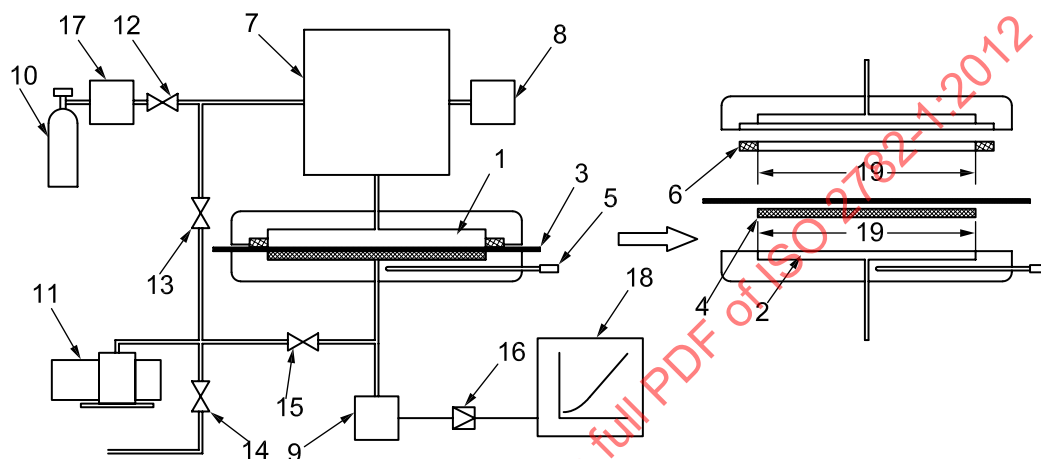
5.1.2 Test piece support, installed on the low-pressure side of the test cell in order to prevent deformation of the test piece due to the pressure difference between the high- and low-pressure sides. Any material, such as filter paper or wire mesh, that does not affect the result of the test may be 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 100 Pa 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	8	pressure gauge for test gas supply reservoir	15	valve 4
2	low-pressure side of test cell	9	pressure sensor for low-pressure side of test cell	16	signal amplifier
3	test piece	10	test gas cylinder	17	pressure-reducing valve
4	test piece support	11	vacuum pump	18	data-processing unit
5	temperature sensor	12	valve 1	19	diameter of gas transmission area
6	sealing ring	13	valve 2		
7	test gas supply reservoir	14	valve 3		

Figure 1 — Example of apparatus for gas permeability measurement (pressure sensor method)
(diagram at right shows an exploded view of the test cell)

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.

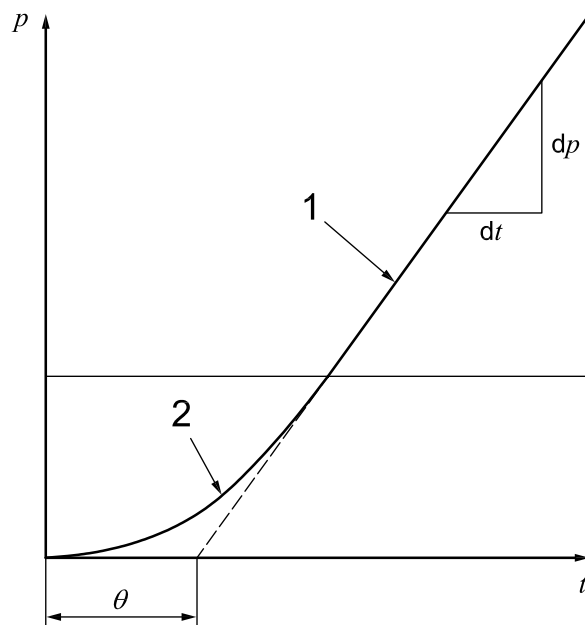
5.8.7 If the pressure on the low-pressure side rises, repeat steps 5.8.2 to 5.8.6, as there is a possibility of gas leakage or gas being absorbed by the test piece.

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 θ (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 Equation (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 [$\text{mol}/(\text{m}^2 \cdot \text{s} \cdot \text{Pa})$];
 V_c is the volume of the low-pressure side of the test cell (m^3);
 T is the test temperature (K);
 p_h is the pressure of the test gas on the high-pressure side of the test cell;
 A is the gas transmission area (m^2);
 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 [$8,31 \text{ m}^3 \cdot \text{Pa}/(\text{K} \cdot \text{mol})$].

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 Equation (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 Equation (3):

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

where

D is the gas diffusion coefficient (m^2/s);

θ 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 Equation (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 (m^2/s).

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

6 Gas-chromatographic method

6.1 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 3.

6.1.1 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 the low-pressure side is connected to the gas chromatograph through the sampling loop to detect

the transmitted gas. 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 test piece, 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 ± 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 gas supply reservoir.

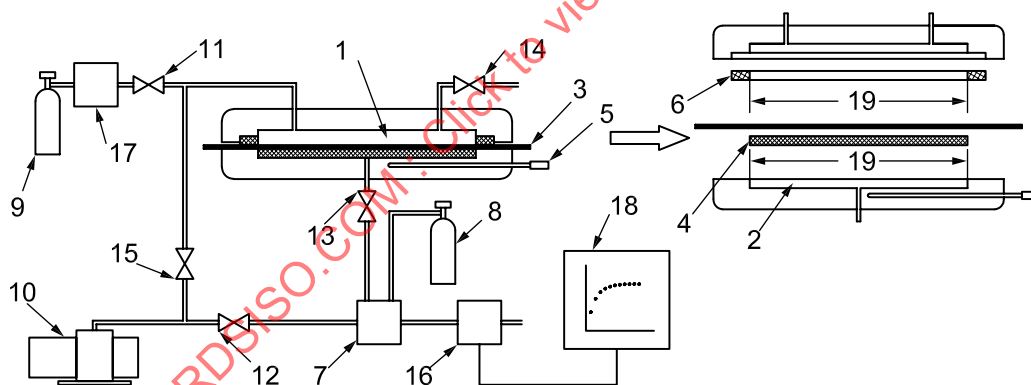
6.1.2 Test piece support, in accordance with 5.1.2.

6.1.3 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.

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

6.1.5 Vacuum pump, in accordance with 5.1.5.

6.1.6 Temperature sensor, in accordance with 5.1.6.



Key

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

Figure 3 — Example of apparatus for gas permeability measurement (gas-chromatographic method)
(diagram at right shows an exploded view of the test cell)

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 Carrier gas

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

6.5 Test pieces

6.5.1 Shape and dimensions

See 5.4.1.

6.5.2 Preparation

See 5.4.2.

6.5.3 Number of test pieces

See 5.4.3.

6.5.4 Measurement of thickness

See 5.4.4.

6.5.5 Time interval between forming and testing

See 5.4.5.

6.6 Conditioning

See 5.5.

6.7 Test conditions

See 5.6.

6.8 Gas transmission area

See 5.7.

6.9 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.

6.10 Procedure

6.10.1 Fit a suitable test piece support (4 in Figure 1) on the low-pressure side of the test cell.

6.10.2 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.

6.10.3 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.

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

6.10.5 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.

6.10.6 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 .

6.10.7 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.

6.10.8 Repeat the procedure from 6.10.5 to 6.10.7 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.

6.10.9 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.

6.11 Calculation and expression of results

6.11.1 Gas transmission rate

The gas transmission rate is determined from Equation (5):

$$\text{GTR} = \frac{T_0 \times (V_s - V_b) \times k}{0,022\,7 \times T \times A \times t \times p_h} \quad (5)$$

where

GTR is the gas transmission rate [mol/(m²·s·Pa)];

T	is the test temperature (K);
T_0	is the standard-state temperature, in K (= 273,15);
t	is the time during which test gas was collected in the sampling loop (s);
V_s	is the amount of test gas collected in the sampling loop (see 6.10.7) (m ³);
V_b	is the blank reading (see 6.10.9) (m ³);
p_h	is the pressure on the high-pressure side of the test cell (Pa);
A	is the gas transmission area (m ²);
k	is a conversion factor for converting the sampling-loop volume to the total volume of the low-pressure chamber (the value of k depends on the particular type of apparatus and is given by the manufacturer);
0,022 7	is the volume, in m ³ , of 1 mol of gas at a pressure of 0,1 MPa [the volume of 1 mol of gas at 1 atm and 0 °C is 0,022 4 m ³ (22,4 l), but it is 0,022 7 m ³ (22,7 l) at 0,1 MPa (1 bar)].

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

6.11.2 Gas permeability coefficient

The gas permeability coefficient is determined from Equation (6):

$$Q = \text{GTR} \times d \quad (6)$$

where

Q	is the gas permeability coefficient [mol·m/(m ² ·s·Pa)];
GTR	is the gas transmission rate [mol/(m ² ·s·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.

7 Test report

The test report shall include the following information:

- a) a full description of the sample and its origin;
- b) a full reference to the test method used, i.e. the number of this part of ISO 2782 and the method used (e.g. ISO 2782-1, pressure sensor method);
- c) test details:
 - 1) the type of test piece used,
 - 2) the method of preparation of the test pieces from the sample, for example moulded or cut,
 - 3) the laboratory temperature,
 - 4) the time and temperature of conditioning of the test pieces prior to testing,
 - 5) the number of test pieces tested,