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ISO 7229

Second edition
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Rubber- or plastics-coated fabrics — Measurement of gas permeability

Supports textiles revêtus de caoutchouc ou de plastique — Mesure de la perméabilité aux gaz

Reference number
ISO 7229:2015(E)

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Foreword

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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).

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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 4, *Products (other than hoses)*.

This second edition cancels and replaces the first edition (ISO 7229:1997), which has been technically revised.

Introduction

The measurement of the permeability of rubber-or plastics-coated fabrics to gases is important in the evaluation of materials for products such as leisure boats, balloons or hoses, and other gas containers in addition to the materials for seals and diaphragms. The permeability of the material is crucial when a product is exposed to differential pressure conditioned environment in its service field.

Rubber- or plastics-coated fabrics — Measurement of gas permeability

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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 International Standard specifies two methods for measuring gas transmission through rubber- or plastics-coated fabrics, a property known as permeability.

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 2231, *Rubber- or plastics-coated fabrics — Standard atmospheres for conditioning and testing*

ISO 2286-3, *Rubber- or plastics-coated fabrics — Determination of roll characteristics — Part 3: Methods for determination of thickness*

3 Terms and definitions

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

3.1
gas transmission rate
volume 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
volume 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 transmission curve
in the 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 after starting the test

Note 1 to entry: See Figure 3.

4 Principle

A test piece is placed between two parts of a hermetically sealed measurement cell. Each part of the cell is vacuumed, then one part is filled with test gas to a certain pressure level. The quantity of gas that permeates through the test piece to the lower pressure side is measured and determined by a pressure sensor or by a gas chromatograph. In the gas chromatography method, measurement condition using equal pressure between two parts of the cell divided by the test piece is given for information in Annex A.

5 Test pieces

5.1 Shape and dimensions

The test piece shall be of uniform shape and have a thickness of more than 0,10 mm and less than 4,00 mm. When using test pieces other than this, the thickness shall be agreed between the interested parties. The test piece shall be large enough to cover the full area of the test cell.

5.2 Measurement of thickness

Measure the thickness of the test piece at five or more points including the centre part of the gas transmission area to the nearest 0,01 mm in accordance with ISO 2286-3 and take the arithmetic mean.

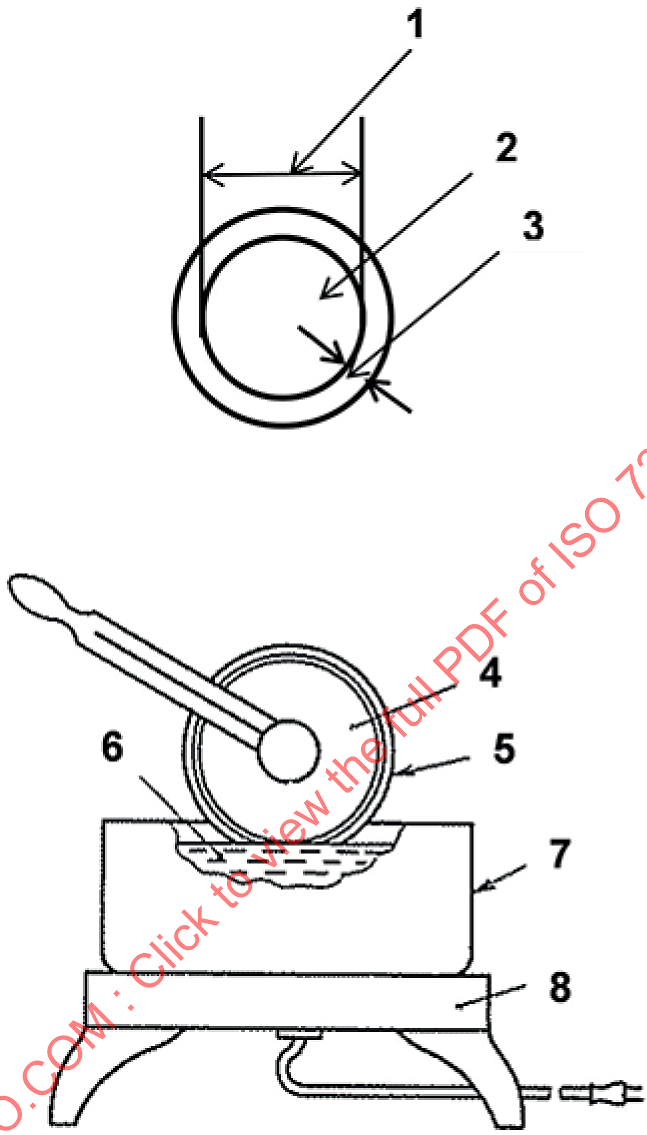
5.3 Number of test pieces

Three or more test pieces shall be used.

5.4 Sealing and masking

A substrate generally passes gas much more easily than coating materials, and even after the test piece is fixed in the cell, the test gas permeated through the coating material may leak out of the cell through the substrate. Cross section cut at the edge around the test piece shall be sealed or masked with wax or a solid type of adhesive which shall not cause any crack nor affect the gas permeability of the test piece during the test.

When a test piece of single-faced coated fabric is used, the surface of the fabric substrate outside the gas permeability measurement area shall be masked besides the edge as shown in Figure 1.



Key

1 10 mm – 150 mm (see 6.1.1)
2 gas transmission area *A* (see 5.7)
3 sealing and masking zone
4 plate to support for test piece
5 test piece
6 wax or adhesives
7 vessel
8 heater

Figure 1 — Example of the sealing and masking equipment

5.5 Conditioning

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

Unless otherwise required in the material specification, the material shall be conditioned before testing for 16 h to 24 h using method of conditioning "1" specified in ISO 2231. When using a test piece that can be easily affected by moisture, dry it for more than 48 h at test temperature in a desiccator containing a suitable drying agent such as anhydrous calcium chloride.

5.6 Test atmosphere

5.6.1 Laboratory conditions shall be (23 ± 2) °C (Atmosphere "D"), in accordance with ISO 2231.

5.6.2 When conducting test at a temperature different from the standard laboratory temperature, the temperature shall be agreed between the interested parties. The test temperature shall be recorded.

5.7 Gas transmission area

The gas transmission area A (m^2) shall be calculated from the internal diameter of the test cell. If a sealing ring is used, calculate the gas transmission area from its internal diameter.

6 Pressure sensor method

6.1 Apparatus

The testing apparatus consists of the test cell, pressure sensors, test gas supply reservoir, vacuum pump and associated pipes and valves. An example of the testing apparatus is shown in Figure 2.

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 a pressure sensor is connected to the low pressure side to detect the change of pressure caused by the transmitted gas. The mounting surfaces of the cell touching the sample shall be smooth and flat to prevent leakage of gas. The material of the test cell shall be inactive with regard to the test gas and shall not absorb the gas used. A seal such as an O-ring may be used to seal between the face of the test cell and the test piece, in which case, the transmission rate of the seal shall be considerably smaller than that of a test piece so that it does not affect the result of gas transmission test. The diameter of the gas transmission surface shall be within the range of 10 mm to 150 mm according to the range of gas transmission rates expected. The cell may be equipped with an electric or hot water heating jacket system capable of raising the temperature up to 80 °C. The temperature accuracy shall be controlled within ± 2 °C from 40 °C to 80 °C.

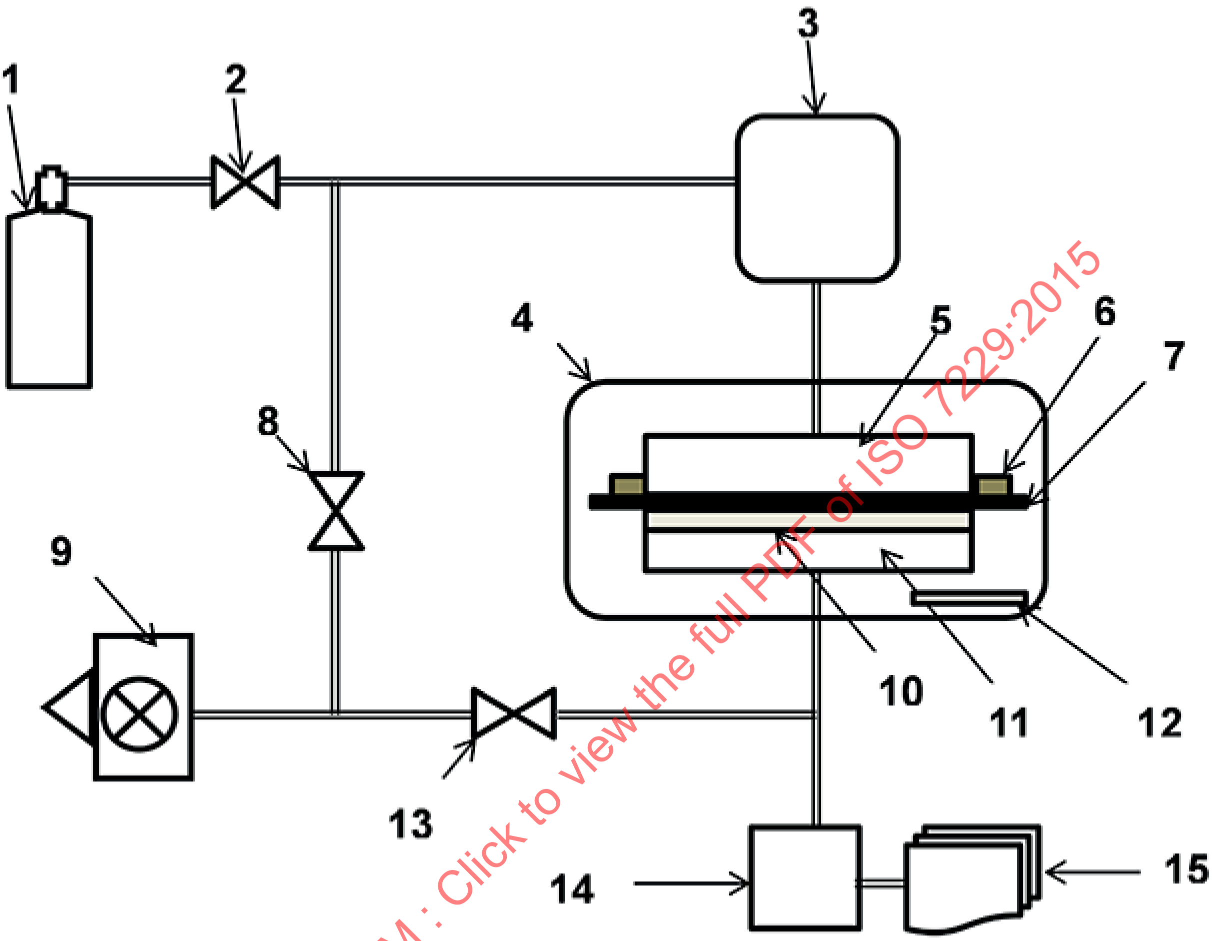
6.1.2 Test piece support, installed in the low pressure side in order to prevent the deformation of the test piece due to the pressure difference. Any material such as filter paper or wire mesh that does not affect the result of gas transmission test may be used. When using filter paper, paper such as that used in chemical analysis is recommended, of thickness 0,1 mm to 0,3 mm according to the depth of low pressure side of the cell.

6.1.3 Pressure sensors, a) to measure the change in pressure on the low pressure side of the cell, capable of reading at least to 5 Pa — vacuum gauge with no mercury, an electronic diaphragm-type sensor or other suitable sensor shall be used; b) to measure the pressure of the test gas supply tank, capable of reading at least to 100 Pa.

6.1.4 Test gas supply reservoir, with a pressure control system to supply test gas to the high pressure side at a constant pressure. The volume of the reservoir shall be sufficient to ensure that the pressure drop on the high pressure side due to the transmission of the test gas to the lower pressure side through a test piece is controlled within at least 1 % or less.

6.1.5 Vacuum pump, capable of evacuating the test cell to 10 Pa or lower.

6.1.6 Temperature sensor, installed in the test cell for measuring test temperature, and capable of reading at least to 0,1 °C.



Key

1 test gas tank
2 valve 1
3 test gas supply reservoir
4 test cell
5 high pressure side of test cell
6 sealing ring
7 test piece
8 valve 2
9 vacuum pump
10 test piece support
11 low pressure side of test cell
12 temperature sensor
13 valve 3
14 pressure sensor
15 data processor

Figure 2 — Example of apparatus for gas permeability measurement (pressure sensor method)

6.2 Test gas

Use a single gas or a mixture of gases such as air, nitrogen, oxygen, hydrogen, liquefied petroleum gas (in gaseous form) and coal gas. The purity of the single gas or the purity of each component used in the gas mixture shall be 99,5 % of the volume fraction or higher. When using a gas of purity less than this, it shall be agreed between the interested parties. The test gas shall not include any impurity that can affect the measurement.

WARNING — When using a toxic gas and/or inflammable gas, necessary measures shall be taken in its use and recovery.

6.3 Procedure

6.3.1 Install a suitable test piece support (10 in Figure 2) on the low pressure side of the test cell.

6.3.2 Lightly and uniformly apply vacuum grease to the surface of the test cell where the test piece touches and mount the test piece on this surface without any wrinkle or sag.

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

6.3.4 When making measurements at a different temperature from a standard laboratory temperature, bring the test cell to the test temperature.

6.3.5 Close valve 1 (2 in Figure 2) and valve 2 (8), and open valve 3 (13). Start the vacuum pump and then open valve 2 (8). Air will be evacuated first from the low pressure side, followed by the high pressure side so that the test piece stays close to the test piece support. Continue until evacuation is complete. Since it is necessary to thoroughly remove any absorbed gas, allow sufficient exhaustion time for a test piece of low gas transmission rate to be thoroughly degassed.

NOTE The exhaustion time required will differ for different types of samples and different conditioning. For those of low gas transmission rate, overnight is suggested. Long time exhaustion might remove sample's vaporized component depending on the type of sample.

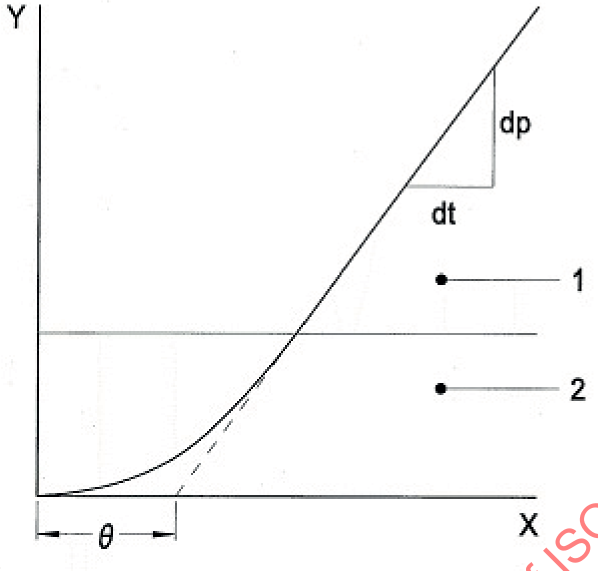
6.3.6 Close valve 2 (8) and valve 3 (13) to maintain the pressure on both the low pressure side and high pressure side at 10 Pa or less. Stop the vacuum pump.

6.3.7 If the pressure on the low pressure side rises, repeat steps 6.3.2 to 6.3.6 since there is a possibility of gas leakage or there is gas absorbed by the sample.

6.3.8 Introduce the test gas into the high pressure side by opening valve 1 (2), shutting off the gas supply when the pressure has reached the pressure level (P_u) set in the test gas supply reservoir. Keep the pressure of high pressure side consistent with the accuracy of 1 % or less. Record the pressure P_u and the temperature T when the pressure on the low pressure side starts increasing due to the permeation of the test gas.

6.3.9 Draw the gas transmission curve by plotting the pressure on 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 3). A gas transmission curve plotted by automatic recording may also be used.

6.3.10 Determine the slope of the straight-line portion of the transmission curve (dp/dt, see Figure 3). The slope may also be determined automatically from the recorder.



Key

X time (h)

Y pressure (kPa)

1 steady-state region

2 non-steady-state region

Figure 3 — Gas transmission curve

6.4 Calculation and expression of results

6.4.1 Gas transmission rate

The gas transmission rate is determined using Formula (1):

$$GTR = \frac{V_C}{P_0 \times \left(T / T_0\right) \times P_u \times A} \times \frac{dp}{dt} \qquad (1)$$

where

GTR is the gas transmission rate (cm³/m²·24 h·Pa);

V_C is the volume of the low pressure side of test cell (cm³);

P_0 is the standard atmospheric pressure (Pa) (= 101 325)

T is the test temperature (K);

T_0 is the standard state temperature (K) (= 273,15);

P_u is the pressure of test gas in the high pressure side of test cell (Pa);

A is the gas transmission area (m²);

dp/dt is the pressure change of low pressure side cell per unit time (Pa/24 h).

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

6.4.2 Gas permeability coefficient

The gas permeability coefficient is determined using Formula (2):

$$Q = GTR \times d \quad (2)$$

where

Q is the gas permeability coefficient ($cm^3 \cdot m/m^2 \cdot 24\ h \cdot Pa$);

GTR is the gas transmission rate ($cm^3/m^2 \cdot 24\ h \cdot Pa$);

d is the thickness of test piece (m).

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

7 Gas chromatography method

7.1 Apparatus

The test apparatus consists of a test cell, a gas chromatograph, a test gas controller, a sampling loop, and a vacuum pump and associated pipes and valves. An example of the apparatus is shown in Figure 4.

7.1.1 Test cell, in accordance with 6.1.1.

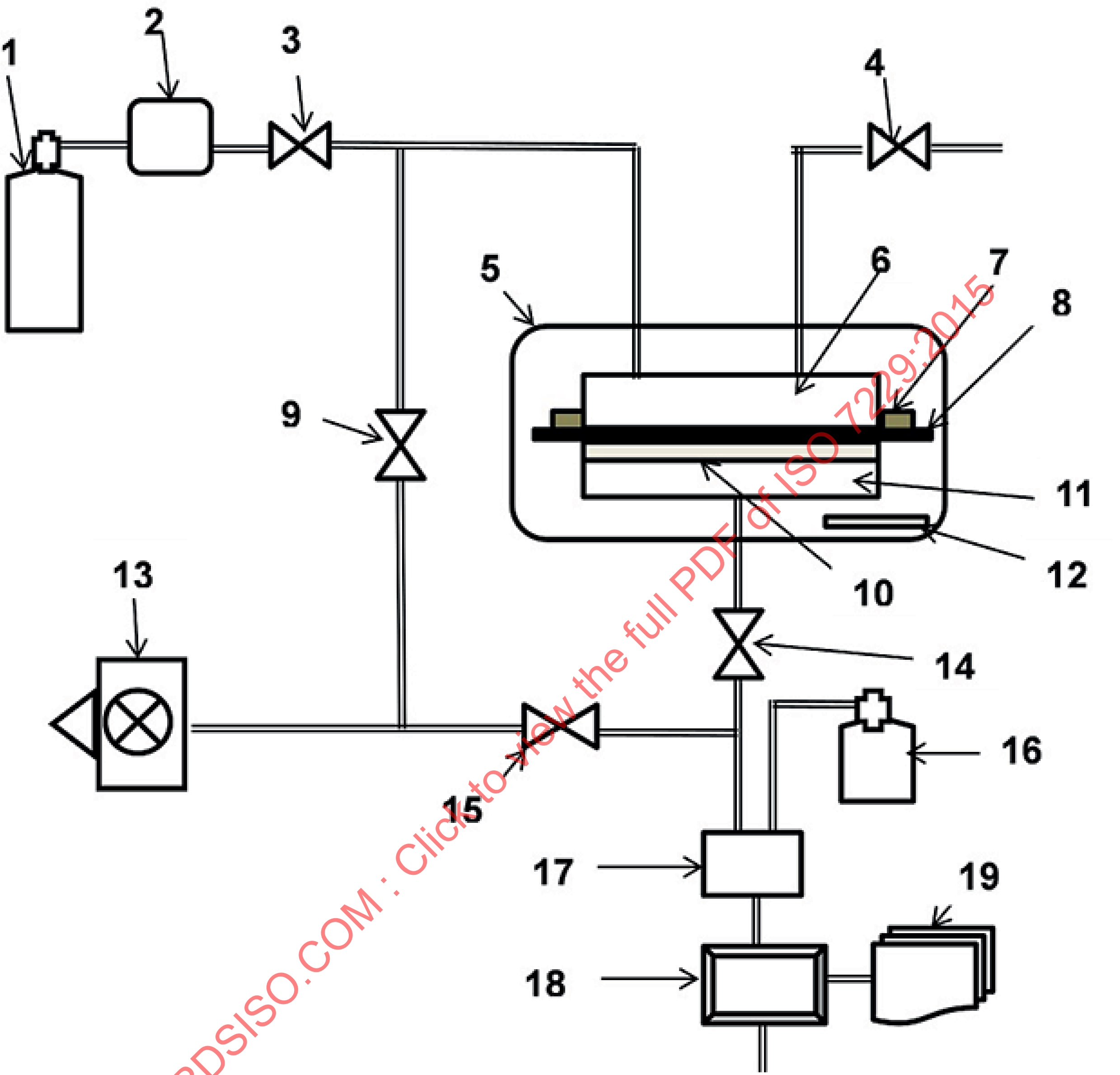
7.1.2 Test piece support, in accordance with 6.1.2.

7.1.3 Gas chromatograph, having a detector such as thermal conductivity detector (TCD) or hydrogen flame ion detector (FID). The detector and the column shall be suitable for the test gas used and of the required sensitivity. The gas chromatograph shall be able to measure the quantity of permeated gas with an accuracy of at least 5 Pa, expressed in terms of the pressure of the gas.

7.1.4 Test gas controller, capable of controlling the test gas flow and pressure, and of maintaining them constant. A flowmeter having at least ± 3 % accuracy shall be used.

7.1.5 Vacuum pump, in accordance with 6.1.5.

7.1.6 Temperature sensor, in accordance with 6.1.6.



Key

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

Figure 4 — Example of apparatus for gas permeability measurement (gas chromatography method)

7.2 Test gas

See 6.2.

7.3 Carrier gas

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

7.4 Calibration curve

Inject a known quantity of 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 mixed test gases, it is necessary to produce a calibration curve for each component of the mixture.

7.5 Procedure

7.5.1 Install a suitable test piece support on the low pressure side of the test cell.

7.5.2 Lightly and uniformly apply some vacuum grease to the surface of the test cell which will be in contact with the test piece, and mount the test piece on this surface without any wrinkle or sag.

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

7.5.4 When measuring at a different temperature from a standard laboratory temperature, bring the test cell to the test temperature.

7.5.5 Close valve 1 (3 in Figure 4), valve 2 (4) and valve 3 (9), and open valve 4 (14) and valve 5 (15). Start the vacuum pump. Then open valve 3 (9). Air will be evacuated first from the low pressure side cell, followed by the high pressure side cell so that the test piece stays close to the test piece support. Continue until evacuation is complete. Since it is necessary to thoroughly remove the absorbed gas, allow sufficient exhaustion time for test pieces of low gas transmission rate.

NOTE The exhaustion time will differ for different types of samples and different conditioning. For those of low gas transmission rate, overnight is suggested. Long time exhaustion might remove sample's vaporized component depending on the type of sample.

7.5.6 When all the air has been evacuated, stop evacuating the high pressure side of test cell by closing valve 3 (9). Introduce the test gas into the high pressure side through the test gas controller (2) by opening valve 1 (3) and maintain the high pressure side at a constant pressure (P_u). The test gas will start permeating from high pressure side to low pressure side and will be evacuated by the vacuum pump. Record the pressure P_u and the temperature T.

7.5.7 Close valve 5 (15), to collect permeated gas in the sampling loop (17). After a predetermined time t, close valve 4 (14) and sweep the gas from the sampling loop (17) into the chromatographic column (18) with the carrier gas (16). Measure the area of the peak in the chromatogram corresponding to the gas of interest. Determine, from the calibration curve prepared in 7.4, the amount of gas volume D_V, which is collected in the sampling loop during time t.

NOTE A suitable predetermined time t differs depending on the gas permeability of the sample.

7.5.8 Repeat the procedure from 7.5.5 to 7.5.7 until a steady-state is reached. The steady-state is assumed to have been reached when the measurement of the amount of gas which permeates the test piece in time t is substantially constant. A gas transmission curve plotted by automatic recording may also be used.

7.5.9 Either before or after the test, carry out a blank test to determine the blank quantity of transmitted test gas D_b, Do this by simultaneously closing valve 1 (3) and valve 3 (9), thus trapping the gas which is flowing through the loop under the steady-state conditions and subsequently determining the amount of gas D_b trapped in time t.

7.6 Calculation and expression of results

7.6.1 Gas transmission rate

Gas transmission rate is determined from the following Formula (3):

$$GTR = \frac{\left(D_v - D_b\right) \times k}{(T / T_0) \times A \times (t / 1\ 440) \times Pu} \quad (3)$$

where

GTR	is the gas transmission rate ($cm^3/m^2 \cdot 24$ h·Pa);
T	is the test temperature (K);
T_0	is the standard state temperature (K) (= 273,15);
t	is the time during which test gas is collected in the sampling loop (min);
D_b	is the blank quantity of transmitted test gas (cm^3);
D_v	is the measured quantity of transmitted test gas (cm^3);
P_u	is the pressure of test gas in the high pressure side of test cell (Pa);
A	is the gas transmission area (m^2);
k	is the device constant for determining the total volume of the low pressure side.

NOTE The value k is the ratio of the total volume of the low pressure side including pipes and sample loop to the volume of the sample loop which is actually used for trapping the transmitted test gas. The value depends on the particular type of apparatus and is given by the manufacturer.

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

7.6.2 Gas permeability coefficient

The gas permeability coefficient is determined using Formula (4):

$$Q = GTR \times d \quad (4)$$

where

Q	is the gas permeability coefficient ($cm^3 \cdot m/m^2 \cdot 24$ h·Pa);
GTR	is the gas transmission rate ($cm^3/m^2 \cdot 24$ h·Pa);
d	is the thickness of test piece (m).

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

8 Test report

The test report shall include the following information:

a) test piece details:

   1) a full description of the test piece and its origin,

   2) the method of pretreatment of the test piece (for example, sealing, masking, or both),

   3) the type of test pieces used;

b) test method:

   1) the number of this International Standard, i.e. ISO 7229;

   2) the test method used (sensor or gas chromatography);

c) test details:

   1) the laboratory temperature,

   2) the time and temperature of conditioning prior to test,

   3) details of any procedures not specified in this International Standard;

d) test results:

   1) the number of test pieces used,

   2) the individual test results,

   3) the mean results;

e) test gas:

   1) the gas used for measuring permeability,

   2) if gas mixture is used, its composition and density;

f) date(s) of test.

Annex A
(informative)

Gas chromatography method with equal pressure condition

A.1 General

A test cell, maintained at atmospheric pressure, is divided into supply side and transmission side by the test piece (see Figure A1). The test gas is supplied to the supply side and a carrier gas is passed through the transmission side. The pressure on both sides of the cell is equal and constant. The quantity of test gas passing through the sample is measured by gas chromatograph. In this method, measurements using a test gas containing water vapour and component analysis (if a mixed test gas is used) are also possible.

A.2 Apparatus

The test apparatus is same as given in 7.1. An example of the test apparatus assembled is shown in Figure A1.