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अनुभाग 6 फिल्म और शीटिंग — गैस-संचरण दर का निर्धारण

उपभाग 1 अंतर-दबाव पद्धतियाँ

(पहला पुनरीक्षण)

Plastics — Methods of Testing

Part 8 Permanence/Chemical Properties

Section 6 Film and Sheeting — Determination
of Gas-Transmission Rate

Subsec 1 Differential-Pressure Methods

(First Revision)

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This Indian Standard (Part 8/Sec 6/Subsec 1) (First Revision) which is identical to ISO 15105-1 : 2007 'Plastics — Film and sheeting — Determination of gas-transmission rate Part 1: Differential-pressure methods' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Methods of Sampling and Test for Plastics Sectional Committee and approval of the Petroleum, Coals and Related Products Division Council.

This Indian Standard was first published in 1997. This revision has been brought out to align the standard with the latest version of ISO 15105-1 : 2007 and ISO 15105-2 : 2003. As the ISO standard is available in two parts, the Committee decided to bifurcate the standards, as given below:

Subsec 1 Differential-pressure methods

Subsec 2 Equal-pressure method

The text of ISO standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'; and
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

The Committee has reviewed the provisions of the following International Standards referred in this adopted standard and has decided that it is acceptable for use in conjunction with this standard:

<i>International Standard</i>	<i>Title</i>
ISO 4593	Plastics — Film and sheeting — Determination of thickness by mechanical scanning

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

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PLASTICS — METHODS OF TESTING
PART 8 PERMANENCE/CHEMICAL PROPERTIES
SECTION 6 FILM AND SHEETING — DETERMINATION OF GAS-TRANSMISSION RATE
SUBSEC 1 DIFFERENTIAL-PRESSURE METHODS
(*First Revision*)

1 Scope

This part of ISO 15105 specifies two methods for determining the gas transmission rate of single-layer plastic film or sheet and multi-layer structures under a differential pressure. One method uses a pressure sensor, the other a gas chromatograph, to measure the amount of gas which permeates through a test specimen.

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 4593, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning*

3 Terms and definitions

For the purposes of this part of ISO 15105, the following terms and definitions apply.

3.1

gas transmission rate

GTR

volume of gas passing through a plastic material, per unit area and unit time, under unit partial-pressure difference between the two sides of the material

NOTE When the gas used is oxygen, the value obtained is the oxygen transmission rate (O_2 GTR).

3.2

gas permeability

coefficient of gas permeability

P

volume of gas passing through a plastic material of unit thickness, per unit area and unit time, under unit partial-pressure difference between the two sides of the material

NOTE 1 The theoretical value of P is given by $P = GTR \times d$ [see Equations (A.2) and (B.2)].

NOTE 2 Although P is a physical property of a polymeric material, differences in film preparation affecting polymer orientation and crystal structure will have an effect on the permeation properties.

4 Principle

A test specimen is mounted in a gas transmission cell (see Figures A.1 and B.1) so as to form a sealed barrier between two chambers. The lower-pressure chamber is evacuated, followed by evacuation of the higher-pressure chamber. A gas is introduced into the evacuated higher-pressure chamber and permeates into the lower-pressure chamber. The amount of gas which permeates through the specimen is determined by the increase in pressure on the lower-pressure side or by gas chromatography.

5 Test specimens

5.1 Test specimens shall be representative of the material under investigation, free from shrivelling, folds and pinholes, and of uniform thickness. They shall be larger than the gas transmission area of the measurement cell and be capable of being mounted airtight.

5.2 Use three specimens unless otherwise specified or agreed upon among the interested parties.

5.3 Mark the side of the material facing the permeating gas.

NOTE In principle, the test should replicate the actual conditions of use, with the gas passing from the inside to the outside of e.g. packaging material, or *vice versa*.

5.4 Measure the thickness of each specimen in accordance with ISO 4593, to the nearest 1 µm, at at least five points distributed over the entire test area, and record the minimum, maximum and average values.

6 Apparatus, procedure and calculation

Of the several methods available for measuring the amount of gas permeating through a specimen, two are described in the annexes:

- Annex A: pressure sensor method;
- Annex B: gas chromatography method.

7 Expression of results

Express the test result as the arithmetic mean of the results obtained for all the specimens, rounding to three significant figures.

8 Precision

The precision of these test methods is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added at the following revision.

9 Test report

The test report shall include the following information:

- a) a reference to this part of ISO 15105;
- b) the method of measurement used (pressure sensor or gas chromatography);
- c) all details necessary for identification of the test apparatus used (make, manufacturer, etc.), including, when a pressure sensor is used, the type of pressure sensor;

- d) all details necessary for identification of the sample tested;
- e) the method of preparation of the test specimens;
- f) the side of the specimen which faced the permeating gas;
- g) the pressure, composition and purity of the gas used;
- h) the average, minimum and maximum thickness of each specimen;
- i) the number of specimens tested;
- j) details of test specimen conditioning;
- k) the temperature and humidity of the laboratory;
- l) the test results;
- m) the date of the test.

Annex A (normative)

Method using a pressure sensor

A.1 Applicability

This method can be used to determine the gas transmission rate of any plastic material.

A.2 Apparatus and materials

A.2.1 General

Figure A.1 shows an example of an apparatus for determining gas transmission rate using a pressure sensor. The apparatus consists of a gas transmission cell designed to allow a gas to permeate through a specimen, a pressure sensor to detect the pressure change due to the permeation of the gas through the specimen, a gas feeder to supply the gas to the transmission cell, a cell volume-control device and a vacuum pump.

A.2.2 Transmission cell

The transmission cell shall consist of an upper (high-pressure) chamber and a lower (low-pressure) chamber, designed so that the gas transmission area is constant for any specimen mounted in the cell. The high-pressure chamber shall have an inlet for the gas and the low-pressure chamber shall be connected to a pressure sensor. The surfaces in contact with the specimen shall be smooth and flat so that leakage does not occur. The diameter of the gas transmission area shall be 10 mm to 150 mm.

A.2.3 Pressure sensor

The sensor shall be capable of determining the change in pressure on the low-pressure side with a minimum sensitivity of 5 Pa (0,038 mmHg). A vacuum gauge with no mercury, an electronic diaphragm-type sensor or another suitable type shall be used.

A.2.4 Gas feeder

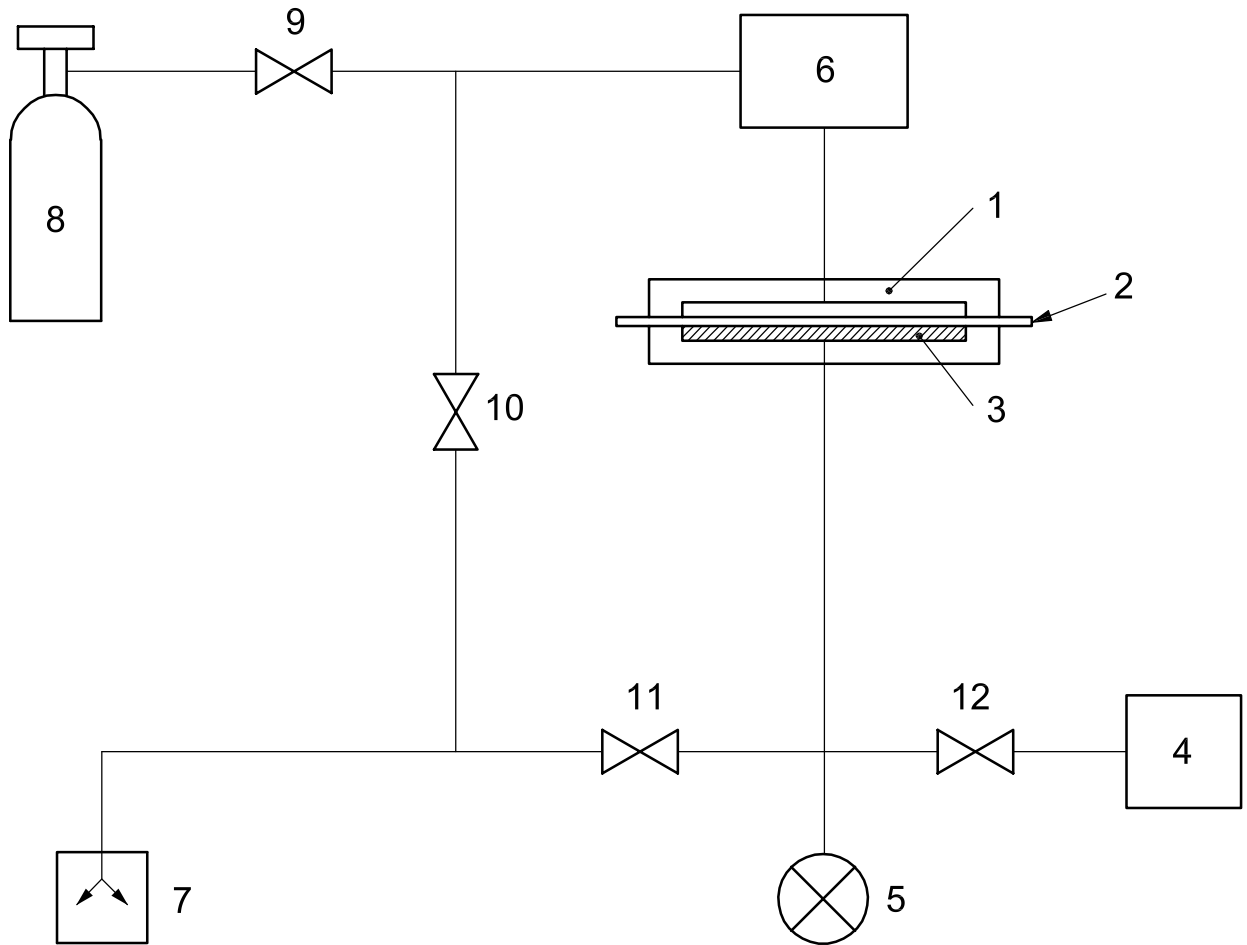
The gas feeder is basically a reservoir designed to store the gas. The gas is fed to the high-pressure side of the cell from the feeder. In order to determine the pressure in the reservoir, a manometer with a minimum sensitivity of 100 Pa (0,75 mmHg) is fitted. The reservoir shall have sufficient capacity such that permeation of the gas through the specimen does not cause any drop in pressure on the high-pressure side.

A.2.5 Cell volume-control device

In order to extend the transmission rate measurement range, the volume of the low-pressure chamber may be adjusted by a cell volume-control device such as an additional reservoir or an adapter.

A.2.6 Gas

The gas used should preferably have a purity greater than 99,5 %. The use of gases of other purities shall be subject to agreement between the interested parties.



Key

- | | |
|------------------------------|---------------|
| 1 transmission cell | 7 vacuum pump |
| 2 specimen | 8 gas supply |
| 3 filter paper | 9 valve 1 |
| 4 cell volume-control device | 10 valve 2 |
| 5 pressure sensor | 11 valve 3 |
| 6 gas feeder | 12 valve 4 |

Figure A.1 — Example of gas transmission rate measurement apparatus using a pressure sensor

A.2.7 Vacuum pump

A vacuum pump capable of producing a vacuum better than 10 Pa (0,075 mmHg) in the low-pressure chamber shall be used.

A.3 Conditioning and test temperature

A.3.1 Conditioning

Dry the specimens for not less than 48 h at the same temperature as that at which the test is to be carried out, using calcium chloride or another suitable drying agent in a desiccator. Drying will not normally be required for non-hygroscopic materials.

A.3.2 Test temperature

Carry out the test in a room kept at $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, unless otherwise specified. Other test conditions, for example those specified in ISO 291, can be chosen subject to agreement between the interested parties.

A.4 Procedure

A.4.1 Spread a filter paper (3 in Figure A.1) having the same size as the gas transmission area over the bottom of the low-pressure chamber.

NOTE The filter paper is used to support the specimen film. A filter paper of the type generally used for chemical analysis, of thickness about 0,2 mm to 0,3 mm, is recommended for this purpose.

A.4.2 Coat the flat edges of the two halves of the transmission cell thinly and uniformly with vacuum grease, and mount the specimen over the lower chamber so that no creasing or slackness occurs.

A.4.3 Place a rubber sealing ring on the specimen, followed by the upper part of the cell. Clamp the two halves of the cell together with uniform pressure so that the specimen is completely sealed in place.

A.4.4 Close valve 1 (9 in Figure A.1) and valve 2 (10), and open valve 3 (11). Start the vacuum pump and then open valve 2 (10). Air will be evacuated first from the low-pressure chamber, followed by the high-pressure chamber. Continue until evacuation is complete. Care is necessary here because the time taken to completely exhaust the cell will depend on the permeability of the specimen.

NOTE The low-pressure chamber is evacuated first to ensure that the specimen fits close to the filter paper.

A.4.5 When all the air has been evacuated, stop the vacuum pump, shutting valve 2 (10) and valve 3 (11) to maintain the vacuum.

A.4.6 If the pressure on the low-pressure side rises, repeat A.4.3 to A.4.5 to ensure that no air is leaking into the cell and to complete any degassing.

A.4.7 Introduce the gas into the high-pressure chamber by opening valve 1 (9), shutting off the gas supply when a pressure of about one atmosphere has been reached. Record the pressure p_h in the high-pressure chamber as indicated by the manometer associated with the gas feeder. An increase in pressure in the low-pressure chamber will confirm transmission of the gas.

A.4.8 Plot a curve of the pressure in the low-pressure chamber versus time, continuing until equilibrium has been reached as indicated by a straight line.

A.4.9 Determine the slope of the straight-line portion of the transmission curve (dp/dt , see Clause A.5). An automatically recorded transmission curve may also be used.

A.5 Calculation

Calculate the gas transmission rate and the gas permeability, or coefficient of gas permeability, from Equations (A.1) and (A.2).

a) Gas transmission rate

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

where

GTR is the gas transmission rate, expressed in moles per square metre second pascal [mol/(m²·s·Pa)];

V_c is the volume of the low-pressure chamber, expressed in litres;

T is the test temperature, expressed in kelvins;

p_h is the pressure of the gas in the high-pressure chamber, expressed in pascals;

A is the transmission area of the specimen, expressed in square metres;

dp/dt is the change in pressure per unit time in the low-pressure chamber, expressed in pascals per second;

R is the gas constant (= 8,31 × 10³), expressed in litre pascals per kelvin mole [(l·Pa)/(K·mol)].

b) Gas permeability, or coefficient of gas permeability

$$P = \text{GTR} \times d \quad (\text{A.2})$$

where

P is the gas permeability, or coefficient of gas permeability, expressed in mole metres per square metre second pascal [mol·m/(m²·s·Pa)];

GTR is the gas transmission rate, expressed in moles per square metre second pascal [mol/(m²·s·Pa)];

d is the average thickness of the specimen, expressed in metres.

NOTE 1 GTR is generally expressed in cubic centimetres per square metre 24 h atmosphere [cm³/(m²·24 h·atm)], the volume of the gas being converted to standard conditions under a pressure difference of one atmosphere.

NOTE 2 P is generally expressed in cubic centimetre millimetres per square metre 24 h atmosphere [cm³·mm/(m²·24 h·atm)].

Annex B (normative)

Method using a gas chromatograph

B.1 Applicability

This method is used to determine the gas transmission rate using a gas chromatograph which is equipped with columns suited to the nature of the gas or mixture of gases concerned. The method is especially suitable for the determination of the gas transmission rate of each component of a gas mixture.

B.2 Apparatus and materials

B.2.1 General

Figure B.1 shows an example of apparatus for determining the gas transmission rate by gas chromatography. The apparatus includes a gas transmission cell, a sampling loop for holding gas which has permeated through the test specimen, switching valves, a gas chromatograph, a test gas controller and a vacuum pump.

B.2.2 Transmission cell

The transmission cell shall consist of a high-pressure chamber (upper side of the cell in Figure B.1) and a low-pressure chamber (lower side of cell). The high-pressure chamber shall have an inlet for a test gas and the low-pressure chamber shall be connected to a gas chromatograph via a sampling loop. The surfaces in contact with the specimen shall be smooth and flat so that leakage does not occur. The diameter of the gas transmission area shall be from 10 mm to 150 mm.

B.2.3 Gas chromatograph

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.

B.2.4 Test gas controller

The test gas controller shall be capable of adjustment to maintain the high-pressure chamber at a specified pressure.

B.2.5 Test gas

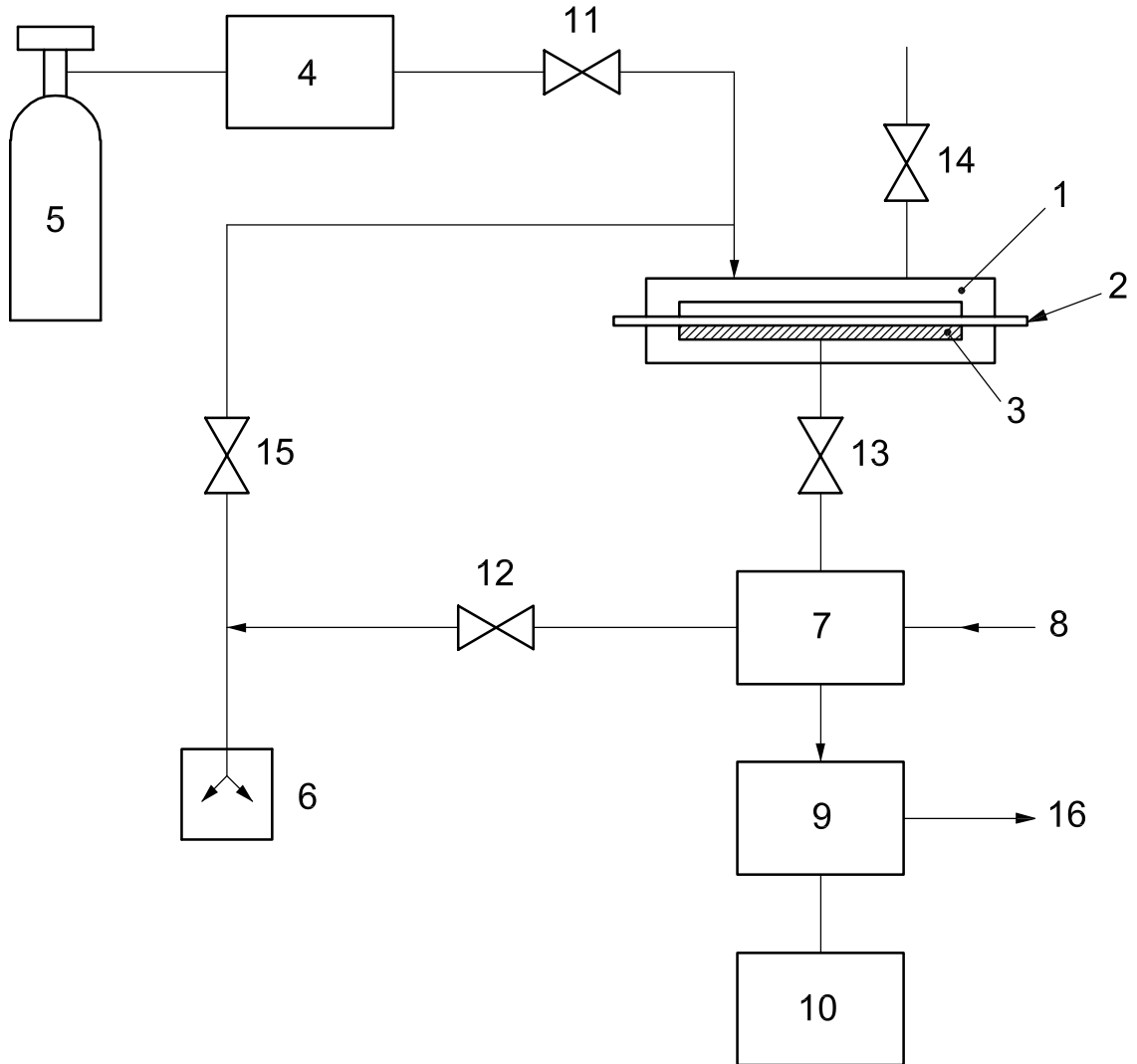
When a single gas is used, the gas shall have a purity of greater than 99,5 % by volume. When a mixture of gases is used, the composition of the mixture shall be determined for the calculation of the GTR either before or after the test.

B.2.6 Carrier gas

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

B.2.7 Vacuum pump

A vacuum pump capable of producing a vacuum better than 10 Pa (0,075 mmHg) in the low-pressure chamber shall be used.



Key

- | | |
|-----------------------|---------------------|
| 1 transmission cell | 9 gas chromatograph |
| 2 specimen | 10 data processor |
| 3 filter paper | 11 valve 1 |
| 4 test gas controller | 12 valve 2 |
| 5 test gas supply | 13 valve 3 |
| 6 vacuum pump | 14 valve 4 |
| 7 sampling loop | 15 valve 5 |
| 8 carrier gas in | 16 carrier gas out |

Figure B.1 — Example of gas transmission rate measurement apparatus using a gas chromatograph

B.3 Calibration graph

Inject a known quantity of sample 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 at different levels of concentration and prepare a calibration graph from the data obtained.

B.4 Conditioning and test temperature

B.4.1 Conditioning

The specimens shall be conditioned at the same temperature and humidity at which the test is to be carried out, for a length of time appropriate to the nature of the material under test.

NOTE Placing specimens in a cell having an automatic conditioning facility ensures that these conditions are met.

B.4.2 Test temperature

Carry out the test at $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, 0 % RH or at $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, 50 % RH, unless otherwise specified. Other test conditions, for example those specified in ISO 291, can be chosen subject to agreement between the interested parties.

B.5 Procedure

B.5.1 Spread a filter paper (3 in Figure B.1) having the same size as the gas transmission area over the bottom of the low-pressure chamber.

NOTE The filter paper is used to support the specimen film. A filter paper of the type generally used for chemical analysis, of thickness about 0,2 mm to 0,3 mm, is recommended for this purpose.

B.5.2 Apply a thin, uniform coating of vacuum grease on the flat edges of the two halves of the transmission cell and mount a specimen over the lower chamber so that no creasing or slackness occurs.

B.5.3 Place a rubber sealing ring on the specimen, followed by the upper part of cell. Clamp the two halves of the cell together with uniform pressure so that the specimen is completely sealed in place.

B.5.4 Close valve 1 (11 in Figure B.1), valve 4 (14) and valve 5 (15), and open valve 2 (12) and valve 3 (13). Start the vacuum pump. Then open valve 5 (15). Air will be evacuated first from the low-pressure chamber, followed by the high-pressure chamber. Continue until evacuation is complete. Care is necessary here because the time taken to completely exhaust the cell will depend on the permeability of the specimen.

NOTE The low-pressure chamber is evacuated first to ensure that the specimen fits close to the filter paper.

B.5.5 When all the air has been evacuated, stop evacuation from the high-pressure chamber by closing valve 5 (15) and introduce the test gas into the high-pressure chamber through the gas controller by opening valve 1 (11) sufficiently to keep the high-pressure chamber at a constant pressure. Test gas will permeate from the high-pressure chamber to the low-pressure chamber through the specimen and be evacuated by the vacuum pump.

B.5.6 By closing valve 2 (12), collect permeated gas in the sampling loop (7). After a predetermined time t , close valve 3 (13) and sweep the gas in the sampling loop (7) into the chromatographic column (9) with carrier gas (8). Using the integrator of the chromatograph, determine the area of the peak in the chromatogram corresponding to the gas of interest. Determine, from the calibration graph prepared in Clause B.3, the amount of gas V_s , in litres, which collected in the sampling loop during time t .

B.5.7 Repeat B.5.6 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 specimen in time t are substantially constant.

B.5.8 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 shutting 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.

B.6 Calculation

Calculate the gas transmission rate and the gas permeability, or coefficient of gas permeability, from Equations (B.1) and (B.2).

a) Gas transmission rate

$$\text{GTR} = \frac{273 \times (V_s - V_b) \times k}{22,4 \times T \times A \times t \times p_h} \quad (\text{B.1})$$

where

GTR is the gas transmission rate, expressed in moles per square metre second pascal [mol/(m²·s·Pa)];

T is the test temperature, expressed in kelvins;

t is the time during which test gas was collected in the sampling loop, expressed in seconds;

V_s is the amount of test gas collected in the sampling loop, expressed in litres;

V_b is the blank reading (see B.5.8), expressed in litres;

p_h is the pressure in the high-pressure chamber, expressed in pascals;

A is the transmission area of the specimen, expressed in square metres;

k is a conversion factor for converting the sampling loop volume to the total volume of the low-pressure chamber.

NOTE The value of k depends on the particular type of apparatus and is given by the manufacturer.

b) Gas permeability, or coefficient of gas permeability

$$P = \text{GTR} \times d \quad (\text{B.2})$$

where

P is the gas permeability, or coefficient of gas permeability, expressed in mole metres per square metre second pascal [mol·m/(m²·s·Pa)];

GTR is the gas transmission rate, expressed in moles per square metre second pascal [mol/(m²·s·Pa)];

d is the average thickness of the specimen, expressed in metres.

Bibliography

- [1] ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

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