INTERNATIONAL STANDARD

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Plastics — Film and sheeting — Determination of gas-transmission rate —

Part 1: Differential-pressure method

Plastiques — Film et feuille — Détermination du coefficient de transmission d'un gaz —

Partie 1: Méthode en pression différentielle

Reference number
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Contents

Page

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 3.

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 part of ISO 15105 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15105-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

ISO 15105 consists of the following parts, under the general title *Plastics — Film and sheeting — Determination of gas-transmission rate*:

— *Part 1: Differential-pressure method*

— *Part 2: Equal-pressure method*

Plastics — Film and sheeting — Determination of gas-transmission rate —

Part 1: Differential-pressure method

1 Scope

This part of ISO 15105 specifies a method for determining the gas transmission rate of any plastic material in the form of film, sheeting, laminate, co-extruded material or flexible plastic-coated material under a differential pressure.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 15105. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this part of ISO 15105 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 4593:1993, *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_2GTR).

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 equation (2) in clause 9.

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 Figure 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 permeation of gas through the specimen is indicated by an increase in pressure on the lower-pressure side.

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 Conditioning and test temperature

6.1 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.

6.2 Test temperature

Carry out the test in a room kept at 23 °C ± 2 °C, unless otherwise specified.

7 Apparatus and materials

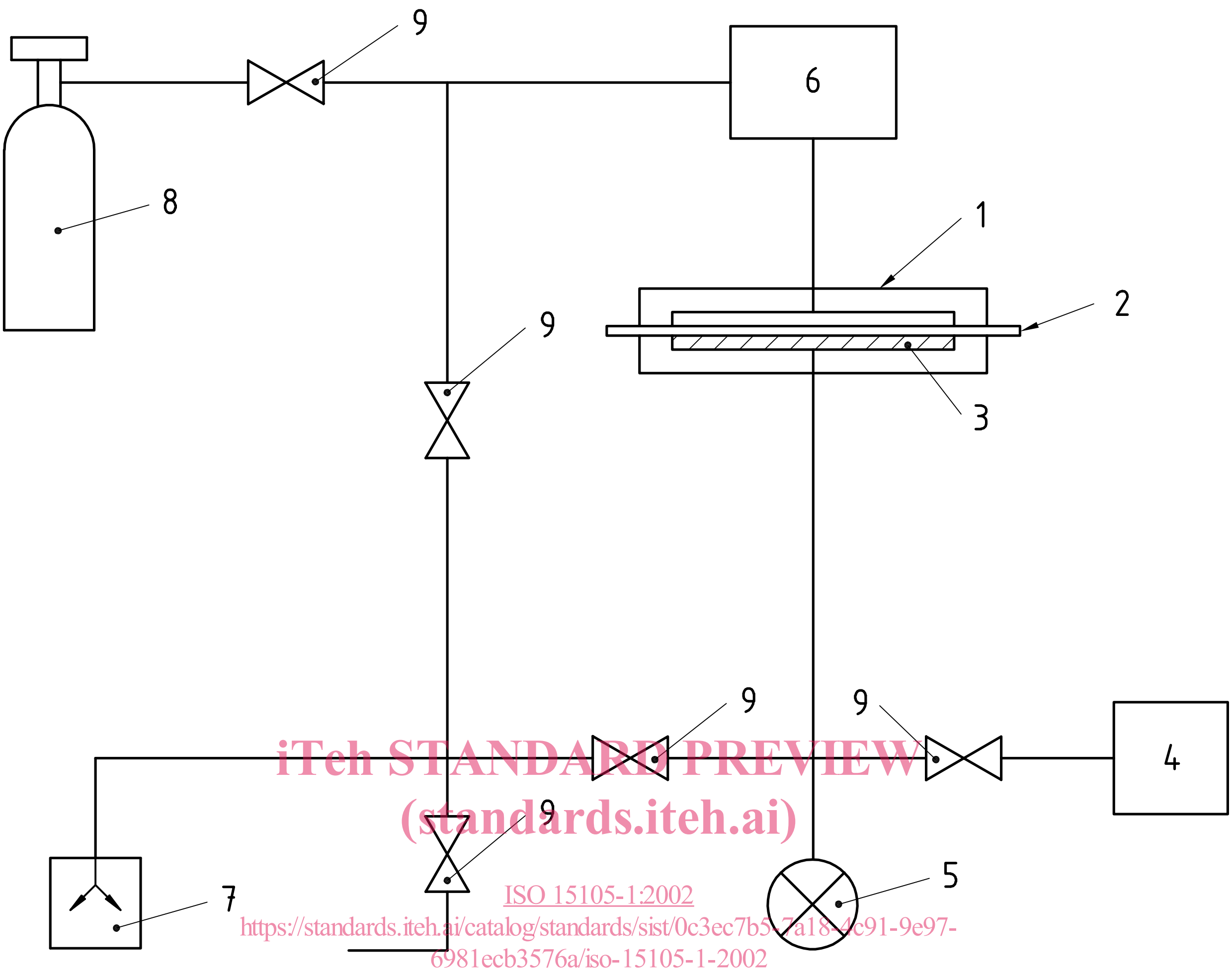
7.1 General

Figure 1 shows an example of an apparatus for determining gas transmission rate.

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.

7.2 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.



Key

1 Transmission cell
2 Specimen
3 Filter paper
4 Cell volume-control device
5 Pressure sensor
6 Gas feeder
7 Vacuum pump
8 Gas supply
9 Stop valve

Figure 1 — Example of gas transmission rate measurement apparatus

7.3 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.

7.4 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

7.5 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.

7.6 Gas

The gas used should preferably have a purity as specified by a relevant International Standard or other suitable standard. The use of gases of other purities shall be subject to agreement between the interested parties.

7.7 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.

8 Procedure

8.1 Spread a filter paper having the same size as the gas transmission area in 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.

8.2 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.

8.3 Place a rubber sealing ring on the specimen, followed by the upper part of the cell, pressing down with uniform pressure so that the specimen is completely sealed in place.

8.4 Start the vacuum pump. Air will be evacuated first from the low-pressure side, followed by the high-pressure side.

8.5 When all air has been evacuated, stop the vacuum pump, shutting off the relevant valve to maintain the vacuum. Care is necessary here, because the time taken to completely exhaust the cell will depend on the permeability of the specimen.

8.6 If the pressure on the low-pressure side rises, repeat 8.3 to 8.5 to ensure that no air is leaking into the cell and to complete any degassing.

8.7 Introduce the gas into the high-pressure side, shutting off the gas supply when a pressure of about one atmosphere has been reached. Record the pressure p_{u} on the high-pressure side as indicated by the manometer associated with the gas feeder. An increase in pressure on the low-pressure side will confirm transmission of the gas.

8.8 Plot a curve of the pressure on the low-pressure side versus time, continuing until equilibrium has been reached as indicated by a straight line.

8.9 Determine the slope of the straight-line portion of the transmission curve ($\mathrm{d}p/\mathrm{d}t$, see clause 9). An automatically recorded transmission curve may also be used.

9 Calculation

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

a) **Gas transmission rate**

$$\text{GTR} = \frac{V_c}{R \times T \times p_u \times A} \times \frac{\mathrm{d}p}{\mathrm{d}t} \qquad (1)$$

where

GTR is the gas transmission rate, expressed in moles per square metre second pascal [mol/(m^2·s·Pa)];

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

T is the test temperature, expressed in kelvins;

p_u is the pressure of the gas on the high-pressure side, expressed in pascals;

A is the transmission area, expressed in square metres;

$\mathrm{d}p/\mathrm{d}t$ is the change in pressure per unit time on the low-pressure side, expressed in pascals per second;

R is the gas constant ($= 8{,}31 \times 10^3$), expressed in litre pascals per kelvin mole [(l·Pa)/(K·mol)].

b) **Gas permeability, or coefficient of gas permeability**

$$P = \text{GTR} \times d \qquad (2)$$

where

P is the gas permeability, or coefficient of gas permeability, expressed in mole metres per square metre second pascal [mol·m/(m^2·s·Pa)];

GTR is the gas transmission rate, expressed in moles per square metre second pascal [mol/(m^2·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 [cm^3/(m^2·24 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 [cm^3·mm/(m^2·24 h·atm)].

10 Test results

Calculate the arithmetic mean of the results obtained for all the specimens, rounding to three significant figures.

11 Precision

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