
**Plastics — Film and sheeting —
Determination of gas-transmission rate —
Part 2:
Equal-pressure method**

*Plastiques — Film et feuille — Détermination du coefficient de
transmission d'un gaz —
Partie 2: Méthode isobarique*

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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.

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 15105-2 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*

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

Part 2: Equal-pressure method

1 Scope

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

Specific examples, currently in use, of the method are described in the annexes.

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 4593:1993, *Plastics — Film and sheeting — Determination of thickness by mechanical scanning*
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3 Terms and definitions

For the purposes of this document, 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 (O₂GTR).

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 the equation

$$P = \text{GTR} \times d$$

where

P is expressed in mole metres per square metre second pascal [mol·m/(m²·s·Pa)];

GTR is expressed in moles per square metre second pascal [$\text{mol}/(\text{m}^2 \cdot \text{s} \cdot \text{Pa})$];

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

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. One chamber is slowly swept with a carrier gas. The second chamber is fed with the test gas. The total pressure is identical (atmospheric) in each chamber but, since the partial pressure of the test gas is higher in the second chamber, the test gas permeates through the barrier into the carrier gas in the first chamber.

The test gas which permeates through the specimen is carried by the carrier gas to a sensor the nature of which will depend on the material under test and the test gas used.

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 permeating 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 $1 \mu\text{m}$, 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

The specimens shall be

- either dried 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;
- or conditioned at the temperature and humidity specified for the test for a length of time appropriate to the nature of the material under test.

6.2 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.

7 Apparatus and materials

7.1 General

Figures A.1 and B.1 show examples of apparatus for determining gas-transmission rate.

The apparatus consists of a gas-transmission cell designed to allow a test gas to permeate through a specimen, a sensor to measure the amount of test gas which has permeated through the specimen, and gas-conditioning devices and flowmeters for the test gas and carrier gas.

7.2 Test gas

The test gas may be either a single gas, or a mixture of gases.

In the first case, the pressure of the gas shall be equal to atmospheric pressure.

In the second case, the pressure of the gas shall also be atmospheric and the partial pressure of each individual component will be determined by its concentration in the mixture.

7.3 Transmission cell

7.3.1 The transmission cell is divided into two chambers by a test piece, cut from the sheet of material under test, to allow permeation of the test gas (see 1 in Figures A.1 and B.1).

7.3.2 The test gas flows, under defined conditions, into chamber A and leaves this chamber through a suitably dimensioned outlet at ambient atmospheric pressure.

WARNING — Appropriate precautions should be taken with respect to the use, recovery, recycling and/or disposal of hazardous gases (toxic gases, flammable gases, etc.).

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7.3.3 A carrier gas flows through the other chamber (chamber B) of the cell, under defined conditions, such that the pressure difference between the two chambers is as low as possible to prevent distortion of the test piece.

7.3.4 The shape of the cell shall be such that the test specimen is swept on both sides by laminar gas flows.

In particular, the ratio of the effective permeation area to the volume of each chamber shall be such that the velocity of each gas is lower than a specified limit, depending on the shape of the cell.

7.3.5 The effective permeation area shall be adapted according to the range of values to be determined. It is generally between 1 cm² and 150 cm².

The effective permeation area of the test specimen may be reduced by means of a mask placed between the two chambers. In this case, the mask shall be glued on the test specimen and carefully checked for leaks, which would affect the measurements.

7.3.6 When the cell is fitted with gaskets, the material used for the gaskets shall have a gas-transmission rate which is negligible compared to that expected for the material under test.

7.4 Detection and measurement

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

- Annex A: coulometric sensor (for oxygen-transmission rate);
- Annex B: chromatographic sensor.

NOTE The method used will depend on:

- the permeability of the material to the test gas;
- the sensitivity required.

8 Diffusion conditions

Unless otherwise specified, the test gas and the carrier gas shall be conditioned under one of the sets of conditions of temperature and relative humidity given in Table 1.

Table 1 — Conditions for diffusion

Set of conditions	Temperature °C	Relative humidity %
1	23	0
2	23	50
3	23	60
4	23	75
5	23	85
6	10	85

NOTE 60 % is the relative humidity normally used for conditioning when using a test gas at 0 % RH and a carrier gas at 100 % RH.

9 Procedure

- 9.1 Remove a test specimen from the conditioning atmosphere.
- 9.2 Insert the specimen in the cell.
- 9.3 Inspect the specimen for visual defects such as creases made during insertion.
- 9.4 Connect the cell to the sensor.
- 9.5 Using the two valves immediately upstream of the cell, allow the carrier gas to flow through both chambers, A and B. The gas flow rate is generally set at between 5 ml/min and 100 ml/min.
- 9.6 Inspect the apparatus for leaks, then purge the apparatus completely, taking into account possible desorption from the specimen. Continue to purge the apparatus until a constant signal is received from the sensor.

9.7 Once a constant signal has been obtained, record this value as the zero value.

NOTE The time taken for the apparatus to stabilize may vary from less than one hour to several hours, depending on the material under test, its thickness and the test conditions.

9.8 Allow the test gas to flow into chamber A, under the specified conditions of flow, temperature and humidity. The gas flow rate is generally set at between 5 ml/min and 100 ml/min.

9.9 Proceed until a constant signal is obtained. Record this signal.

9.10 Repeat with the remaining specimens.

10 Expression of results

See Clause A.6 or B.7, depending on the method used.

11 Precision

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

12 Test report iTeh STANDARD PREVIEW

See Clause A.7 or B.8, depending on the method used. (standards.iteh.ai)

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Annex A (normative)

Method for the determination of the oxygen-transmission rate using a coulometric sensor

A.1 General

This method is used to determine the amount of oxygen permeating through the material, using a coulometric sensor which measures the amount of oxygen which has permeated through the material and been carried out of the cell by the carrier gas.

The sensor generates an electric current proportional to the amount of oxygen flowing through the sensor per unit time.

A.2 Apparatus

Figure A.1 shows a typical apparatus.

Valve 12 bypasses the sensor (9), thus avoiding the cell (1) being swamped with air when the test specimen (2) is being placed in the cell. The valves (7) immediately upstream of the transmission cell allow either carrier gas or test gas to be passed into chamber A of the cell. The catalyst bed (11) is intended to eliminate any traces of oxygen which might be present in the carrier gas.

NOTE Any other suitable means of removing oxygen from the carrier gas is acceptable.

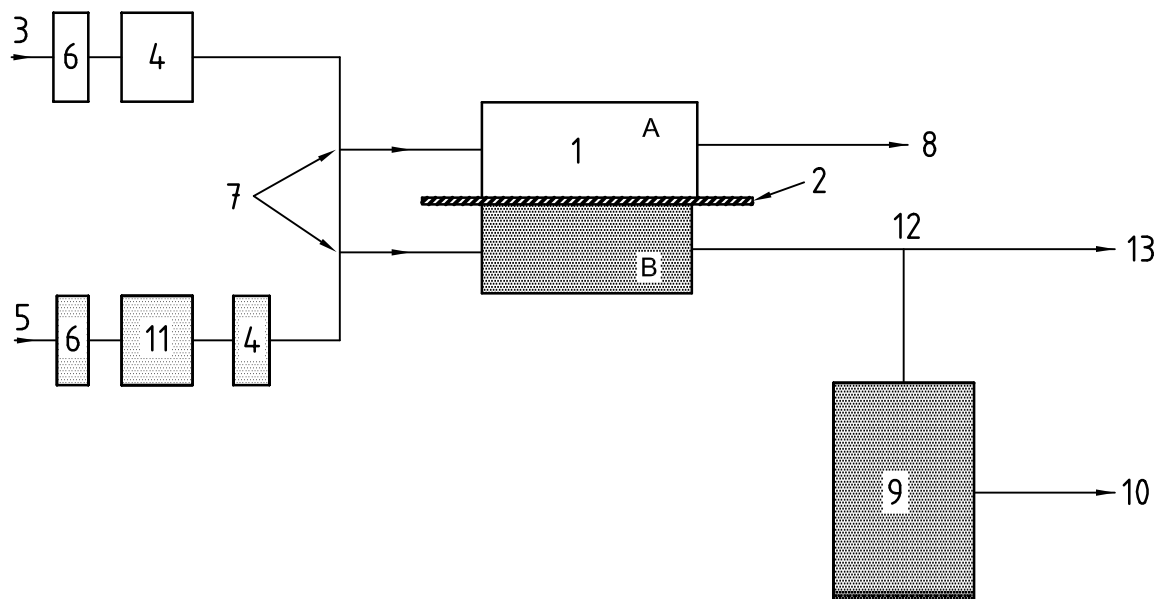
A.3 Carrier gas and test gas

A.3.1 The carrier gas shall be a mixture of dry nitrogen with 0,5 % to 3 % by volume of hydrogen. The volume fraction of oxygen in the carrier gas shall not exceed 100 µl/l.

A.3.2 The test gas shall be dry oxygen containing at least 99,5 % by volume of O₂.

NOTE For materials with a high permeability to oxygen, a mixture of nitrogen and oxygen, such as air (21 % oxygen), may be used. It is also possible to decrease the effective permeation area of the specimen by means of a mask as indicated in 7.3.5.

A.3.3 Gas-conditioning devices (see 4 in Figure A.1) shall be placed upstream of the cell in order to achieve one of the sets of conditions given in Table 1. Devices for monitoring the humidity of the gases may be fitted in the carrier and/or test gas circuits.



Key

- | | | | |
|---|---|----|---|
| 1 | transmission cell with chambers A and B | 8 | oxygen outlet (or carrier gas outlet when purging) |
| 2 | specimen | 9 | coulometric sensor for measurement of O ₂ in carrier gas |
| 3 | oxygen inlet | 10 | carrier gas outlet |
| 4 | gas-conditioning device | 11 | catalyst bed, to eliminate traces of O ₂ in carrier gas |
| 5 | carrier gas inlet | 12 | 3-way valve |
| 6 | flowmeter | 13 | carrier gas outlet when purging the chambers |
| 7 | 3-way valves | | |

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Figure A.1 — Typical example of apparatus for determination of oxygen-transmission rate using a coulometric sensor

A.4 Calibration

It is recommended that the sensor be checked periodically against known reference materials.

NOTE The oxygen sensor used in this method is a coulometric device that yields a linear output as predicted by Faraday's law. In principle, the sensor produces four electrons for each molecule of oxygen passing into it. Considering that the sensor is known to have a basic efficiency of 95 % to 98 %, it may be considered an intrinsic standard that does not require calibration, and this method can thus be used as a reference method. However, a sensor may be damaged or may become depleted to the extent that efficiency and response are impaired.