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Plastics — Film and sheeting — Determination of water vapour transmission rate —

Part 7: **Calcium corrosion method**

Plastiques — Film et feuille — Détermination du coefficient de transmission de vapeur d'eau —

Partie 7: Méthode de la corrosion du calcium

Reference number
ISO 15106-7:2015(E)

Contents

Page

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 61, *Plastics*, Subcommittee SC 11, *Products*.

ISO 15106 consists of the following parts, under the general title *Plastics — Film and sheeting — Determination of water vapour transmission rate*:

— *Part 1: Humidity detection sensor method*

— *Part 2: Infrared detection sensor method*

— *Part 3: Electrolytic detection sensor method*

— *Part 4: Gas-chromatographic detection sensor method*

— *Part 5: Pressure sensor method*

— *Part 6: Atmospheric pressure ionization mass spectrometer method*

— *Part 7: Calcium corrosion method*

Plastics — Film and sheeting — Determination of water vapour transmission rate —

Part 7: Calcium corrosion method

1 Scope

This part of ISO 15106 specifies a method for determining the water vapour transmission rate of plastic film, plastic sheeting, and multi-layer structures including plastics based on measurements of calcium degradation due to the reaction with water.

NOTE The method provides rapid measurement over a wide range of water vapour transmission rates.

2 Terms and definitions

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

2.1
water vapour transmission rate
amount of water vapour transmitted through unit area of test specimen per unit time under specified conditions

Note 1 to entry: Water vapour transmission rate is expressed in grams per square metre per 24 h [g/(m^2·24 h)].

2.2
seal material
adhesive materials between test specimen and glass slide for protecting the calcium film from the corrosive gas

2.3
encapsulant
encapsulation material for protecting the calcium film from the corrosive gas

3 Principle

The calcium test method is based on measuring the rate of reaction between calcium metal and water. A calcium film is deposited on a test specimen or a glass slide and sealed or encapsulated according to one of the three procedures described in Annex A, Annex B, and Annex C. The water vapour transmission rate is determined from the change of calcium film properties after exposure in an environmental chamber as described in Annex A, Annex B, and Annex C.

4 Test specimens

4.1 The test specimens shall be representative of the material, free from wrinkles, creases and pinholes, and have uniform thickness. Each test specimen shall have a larger area than the transmission area of the cell.

4.2 Three test specimens shall be tested unless otherwise specified or agreed between the interested parties.

NOTE For some products, testing more than three test specimens gives a more representative result.

5 Conditioning

Condition the test specimens in vacuum or inert atmosphere. The period of conditioning shall be as stated in the relevant specification for the material.

NOTE For some products, heating the test specimen is effective for rapid conditioning.

6 Apparatus, procedure, and calculation

6.1 Vacuum evaporator

The vacuum evaporator comprises a metal evaporation source, a substrate holder, a thickness monitor, and a shutter mechanism. The calcium metal shall be evaporated at less than 10^{-2} Pa. In addition, the substrate temperature shall be kept low enough to prevent heating damage of specimen. A deposition mask technique shall be used for the calcium film area control.

6.2 Glovebox

The water and oxygen content shall be less than 40 ppm in the glove box. The glove box shall have the mechanism for transporting a calcium film substrate from the vacuum chamber without exposure to atmosphere.

6.3 Environmental chamber

The temperature in the environmental chamber shall be kept within ±0,5 °C. In addition, the relative humidity shall be within ±3 %.

6.4 Of the several methods available for measuring the amount of water vapour permeating through a specimen, three are described in the annexes.

— Annex A: Optical measurement method

— Annex B: Electrical measurement method

— Annex C: Corrosion area evaluation method

7 Test conditions

The test conditions shall preferably be chosen from those given in Table 1.

Table 1 — Choice of test conditions

Test conditions	Temperature °C	Relative humidity %
1	25 ± 0,5	90 ± 3
2	40 ± 0,5	90 ± 3
3	60 ± 0,5	90 ± 3
4	85 ± 0,5	85 ± 3

Test conditions other than these shall be agreed upon by the interested parties.

8 Test result

Calculate the test result as the arithmetic mean of the results obtained for each test specimen, rounding to two significant figures.

9 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.

10 Test report

The test report shall include the following information:

a) a reference to this part of ISO 15106, i.e. ISO 15106-7;

b) the test conditions;

c) the name of the apparatus used;

d) all details necessary for identification of the sample tested;

e) the method of preparation of the test specimens;

f) the side of the test specimen which faced the supply of water vapour, if necessary;

g) the transmission area of the test specimen;

h) the mean thickness of the test specimen;

i) the number of specimens tested;

j) details of specimen conditioning;

k) the test result;

l) the date of the test.

Annex A
(normative)

Optical measurement method

A.1 Apparatus

A.1.1 General

The apparatus includes a test cell and a spectrophotometer.

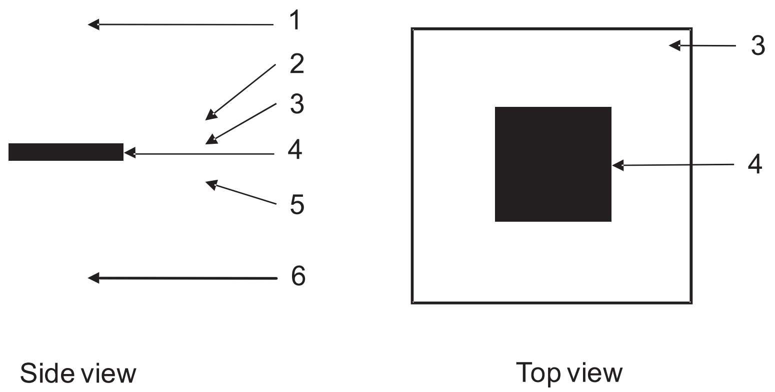
A.1.2 Test cell

The test cell structure is shown in Figure A.1. The test cell size shall typically be from 20 mm to 100 mm. The calcium film thickness shall typically be from 10 nm to 500 nm.

A.1.3 Spectrophotometer

The spectrophotometer shall measure the change of optical properties of the calcium film precisely.

The device has the performance that can detect an optical transmittance. The measurement shall be carried out under conditions of the constant temperature and humidity.



Key

1 light source
2 test specimen
3 seal material
4 calcium film
5 substrate
6 light detector

Figure A.1 — Example of calcium test cell structure

A.2 Procedure

A.2.1 Measure the water vapour transmission rate (WVTR) of each specimen as described in A.2.2 to A.2.5.

A.2.2 A measurement setup is shown in Figure A.1. The deposited calcium film is applied onto the substrate. The substrate is hermetically sealed using a seal material and the test specimen (or the glass slide) under inert atmosphere. Then, the test cell is transferred to an environmental chamber for accelerated ageing.

A.2.3 The optical transmission of the test cell is measured throughout the test period using a spectrophotometer.

A.2.4 The rate of calcium corrosion is calculated from change of optical transmission.

A.2.5 Repeat steps A.2.3 and A.2.4 and calculate the water vapour transmission rate (WVTR) each time. When the WVTR remains constant, indicating that permeation has reached a steady state, record this value.

A.3 Calculation

Calculate the water vapour transmission rate of each test specimen using Formula (A.1).

$$\mathrm{WVTR} = 2 \times \left(\frac{M_{\mathrm{H_2O}}}{M_{\mathrm{Ca}}} \right) \times d_{\mathrm{Ca}} \times \left(\frac{df(O_{\mathrm{p}})}{dt} \right) \times \left(\frac{S_{\mathrm{Ca}}}{S} \right) \tag{A.1}$$

where

WVTR	is the water vapour transmission rate of the test specimen, in [g/(m^2·24 h)];
$M_{\mathrm{H_2O}}$	is the molecular weight of water, in grams per mole;
M_{Ca}	is the molecular weight of calcium, in grams per mole;
d_{Ca}	is the density of calcium, in grams per cubic metre;
O_{p}	is the optical transmission;
$f(O_{\mathrm{p}})$	is the calibration function of calcium film thickness, in metres;
S_{Ca}	is the area of the calcium film, in square metres;
S	is the transmission area of the water vapour surrounded with the edge seal of the specimen, in square metres.

NOTE The calibration function of the calcium film thickness is determined from the relationship between optical transmission and calcium film thickness.

Annex B
(normative)

Electrical measurement method

B.1 Apparatus

B.1.1 General

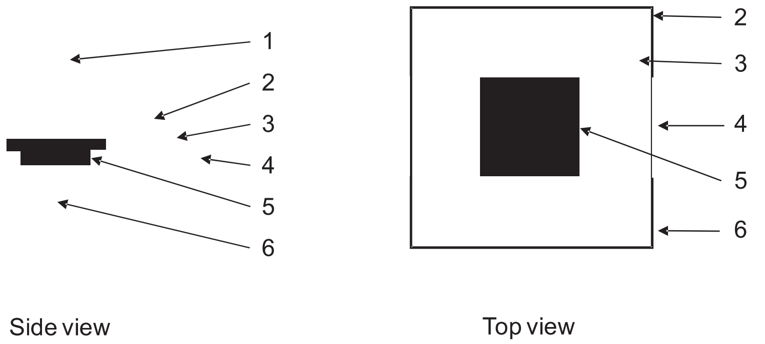
The apparatus includes a test cell and ohmmeter.

B.1.2 Test cell

The test cell structure is shown in Figure B.1. The test cell size shall typically be from 20 mm to 100 mm. The calcium film thickness shall typically be from 10 nm to 500 nm.

B.1.3 Ohmmeter

The ohmmeter has to be able to measure the change of electrical resistance of the calcium film precisely.



Key

1 ohmmeter
2 test specimen
3 seal material
4 electrode
5 calcium film
6 substrate

Figure B.1 — Example schematic representation of calcium test setup

B.2 Procedure

B.2.1 Measure the water vapour transmission rate (WVTR) of each specimen as described in B.2.2 to B.2.6.

B.2.2 A measurement setup is shown in Figure B.1. The electrodes for the resistivity measurement are deposited onto the substrate. After a deposited calcium film has been applied, it is hermetically sealed under inert atmosphere using a seal material and a test specimen.

B.2.3 The test cell is transferred to an environmental chamber for accelerated ageing under a specific condition. The ohmmeter is connected to the electrode.

B.2.4 The resistivity is measured throughout the test period using an ohmmeter.

B.2.5 The rate of calcium corrosion is calculated from a change in resistivity of the calcium film.

B.2.6 Repeat steps B.2.4 or B.2.5 and calculate the water vapour transmission rate (WVTR) each time. When WVTR remains constant, indicating that permeation has reached steady state, record this value.

B.3 Calculation

Calculate the water vapour transmission rate of each test specimen using Formula (B.1).

$$\mathrm{WVTR} = -2 \times \left(\frac{M_{\mathrm{H_2O}}}{M_{\mathrm{Ca}}} \right) \times d_{\mathrm{Ca}} \times \rho \times \left(\frac{d(1/R)}{dt} \right) \times \left(\frac{a}{b} \right) \times \left(\frac{S_{\mathrm{Ca}}}{S} \right) \quad \text{(B.1)}$$

where

WVTR	is the water vapour transmission rate of the test specimen, in [g/(m^2·24 h)];
$M_{\mathrm{H_2O}}$	is the molecular weight of water, in grams per mole;
M_{Ca}	is the molecular weight of calcium, in grams per mole;
d_{Ca}	is the density of calcium, in grams per cubic metre;
ρ	is the volume resistivity, in [Ω·m];
$d(1/R)/dt$	is the rate of change of the conductance, in [1/(Ω·24 h)];
a	is the length of the calcium film, in metres;
b	is the width of the calcium film, in metres;
S_{Ca}	is the area of the calcium film between electrodes, in square metres;
S	is the water vapour transmission area surrounded with the edge seal of the specimen, in square metres.

Annex C
(normative)

Corrosion area evaluation method

C.1 Apparatus

C.1.1 General

The apparatus includes a test cell and a microscope.

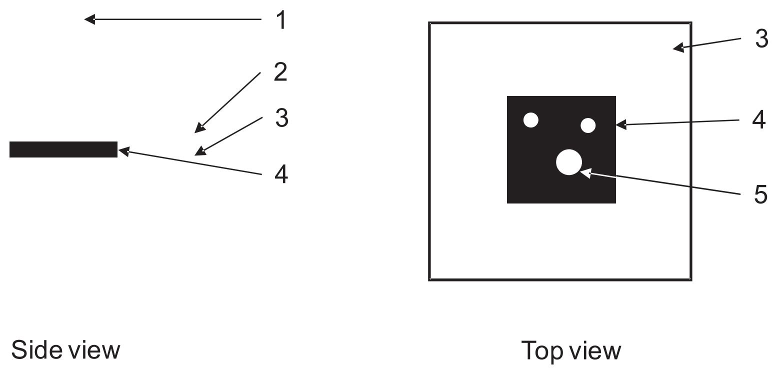
C.1.2 Test cell

The test cell structure is shown in Figure C.1. The test cell size shall typically be from 20 mm to 100 mm. The calcium film thickness shall typically be from 10 nm to 500 nm.

mark

C.1.3 Microscope

The microscope shall detect the colour change of the calcium film due to the reaction with water. The device has the performance that can evaluate a corrosion area from the colour change area. The measurement shall be carried out under conditions of constant temperature and humidity. Microscope strength shall typically be over 5×.



Key

1 microscope

2 test specimen

3 encapsulant

4 calcium film

5 corrosion area

Figure C.1 — Example of a calcium test cell structure

C.2 Procedure

C.2.1 Measure the water vapour transmission rate (WVTR) of each specimen as described in C.2.2 to C.2.5.

C.2.2 A measurement setup is shown in Figure C.1. The deposited calcium film is applied onto the test specimen. The side of the specimen with the Ca film is smeared with seal material under inert atmosphere. Then, the test cell is transferred to an environmental chamber for accelerated ageing.

C.2.3 The corrosion area of the test cell is evaluated throughout the test period using a microscope.

C.2.4 The rate of calcium corrosion is calculated from the change of the corrosion area.

C.2.5 Repeat steps C.2.3 and C.2.4 and calculate the water vapour transmission rate (WVTR) each time. When the WVTR remains constant, indicating that permeation has reached steady state, record this value.

To prevent inaccurate values of measurement, the calcium film thickness shall be monitored by observing the colour of the calcium film.

C.3 Calculation

Calculate the water vapour transmission rate of each test specimen using Formula (C.1).

$$\mathrm{WVTR} = 2 \times \ell \times \left(\frac{M_{\mathrm{H_2O}}}{M_{\mathrm{Ca}}} \right) \times d_{\mathrm{Ca}} \times \left(\frac{dA}{dt} \right) \times \left(\frac{1}{S_0} \right) \tag{C.1}$$

where

WVTR	is the water vapour transmission rate of the test specimen, expressed in grams per square metre per 24 h [g/(m^2·24 h)];
$M_{\mathrm{H_2O}}$	is the molecular weight of water, in grams per mole;
M_{Ca}	is the molecular weight of calcium, in grams per mole;
ℓ	is the thickness of calcium film, in metres;
d_{Ca}	is the density of calcium, in grams per cubic metre;
dA/dt	is the rate of change of the corrosion area, in square metres per day;
S_0	is the deposition area of the calcium film, in square metres.

Bibliography

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

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