INTERNATIONAL STANDARD ISO 2528

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Sheet materials — Determination of water vapour transmission rate — Dish method

Produits en feuilles et en plaques — Détermination du coefficient de transmission de la vapeur d'eau — Méthode de la capsule

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2528 was drawn up by Technical Committee ISO/TC 6, *Paper, board and pulps,* and circulated to the Member Bodies in October 1971.

It has been approved by the Member Bodies of the following countries :

Austria	India	Spain
Belgium	Israel	Sweden
Bulgaria	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
Egypt, Arab Rep. of	Norway	Turkey
Finland	Poland	United Kingdom
France	Romania	U.S.A.
Hungary	South Africa, Rep. of	U.S.S.R.

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Australia
Germany
New Zealand

Printed in Switzerland

Sheet materials — Determination of water vapour transmission rate — Dish method

0 INTRODUCTION

The method described in this International Standard can in theory be applied to any sheet material. In practice its main use is for flat, usually thin, materials that can be processed to form a vapour-resistant barrier, as used in packaging, such as paper, board, plastics films or laminates of paper with films or metal foils, and for fabrics coated with rubber or plastics.

The water vapour pressure differential is the essential part of this test and in this instance it has not been possible to adopt the conditions recommended in ISO/R 554, *Standard atmospheres for conditioning and/or testing — Standard reference atmosphere — Specifications*. In addition, the limits of temperature and humidity control are more exacting than those required for normal testing.

1 SCOPE

This International Standard specifies a method for the determination of the water vapour transmission rate[1] of sheet materials, using dishes with a wax seal.

2 FIELD OF APPLICATION

This method is generally not recommended for use if the transmission rate is expected to be less than 1 g/m²d.

For some purposes it may be necessary to determine the transmission rate of creased material; a procedure for this is given in annex C.

The method cannot be applied to films that are damaged by hot wax or that shrink to an appreciable extent under the test conditions used.

The method should not normally be used for materials thicker than 3 mm.

3 DEFINITION

water vapour transmission rate (of a sheet material) : The mass of water vapour transmitted from one face to the other, under constant conditions of vapour pressure at the two faces, expressed per unit of surface area and during a given time. It is expressed in grams per square metre per 24 h (g/m²d) at the conditions of temperature and humidity defined at the two faces of the sheet. This transmission rate depends upon the thickness and permeability of the constituent material or materials and the conditions of temperature and humidity under which the test is carried out. (See annex A.)

4 PRINCIPLE

Dishes containing a dehydrating agent and closed by the sheet to be tested are placed in an enclosure with controlled temperature and relative humidity. (See annex A.)

These dishes are weighed at suitable intervals of time. The water vapour transmission rate is determined from the increase in mass when this increase has become proportional to the time interval.

5 SIGNIFICANCE OF THE TEST

This test is intended to give reliable values of water vapour transmission rate by means of simple apparatus.

The use of the results for any particular application must, however, be based upon experience.

Transmission rate is not a linear function of temperature nor, generally, of relative humidity difference. A determination carried out under certain conditions is not, therefore, necessarily comparable with one carried out under other conditions.

The conditions of test must, therefore, be chosen so as to be as close as possible to the conditions of use.

1) This characteristic is often erroneously termed "permeability".

6 APPARATUS AND EQUIPMENT

6.1 Dishes and accessories (Figure 1 shows examples of equipment which has proved satisfactory in use.)

All the following items must be in glass or in a metal as light as possible and resistant to corrosion under the test conditions. Aluminium of Grade Al 99,5 in accordance with ISO/R 209, protected by chemical or anodic oxidation, is suitable.

The assembly must be sufficiently rigid. Aluminium sheet 1 mm thick is satisfactory.

6.1.1 *Circular non-porous dishes* with a groove round the rim for sealing the test piece with wax. The groove must have a profile such that the test piece can be sealed over the opening of the dish and that no escape of water vapour can occur at or through the edges of the test piece.

The exact surface area of test piece exposed is defined by the diameter, *D*, of the template for the wax (see 6.1.2). The internal diameter of the ring portion of the dish on which the test piece rests shall be equal to or very slightly larger than diameter *D*. Moreover, the surface area of the bottom of the dish where it is filled with desiccant shall also be similar to that of the exposed surface, and there shall be no obstruction within the dish that might interfere with the flow of water vapour between the test piece and the desiccant. The internal depth of the dish below the plane of the test piece should be not less than 15 mm (deep dish) or 8 mm (shallow dish). Every dish shall be numbered.

6.1.2 *Waxing templates,* to place the wax easily and to allow the test surface to be defined exactly. Their diameter, *D*, must be known to an accuracy better than 0,5 %.

The diameter should preferably be 79,8 ± 0,4 mm (an area of 50 cm²). If any other diameter or test area is used, this fact shall be mentioned in the test report. The diameter shall in no case be less than 56,4 ± 0,3 mm (an area of 25 cm²).

These templates may be either :

6.1.2.1 cross-braced ring templates which remain in place during the test. As many ring templates as dishes are required. The diameter, *D*, is the internal diameter of the ring; or

6.1.2.2 a cover template which must be taken off when the wax has been run in and cooled. It comprises a disk with a central handle, drilled with a small hole at a suitable point (see figure 1), and having the edge chamfered at an angle of approximately 45° in such a way that the smallest diameter is on the bottom. ~~The diameter, *D*,~~ is the diameter of this smaller circle.

Small guides can be fixed to the template to centre it automatically. A few templates are sufficient.

6.1.3 *Lids* (the rims of which are outside the dish) to close the dish assemblies sufficiently well to allow them to be brought out from the enclosure for weighing without loss of water vapour. They shall be numbered to correspond with each dish.

6.2 Enclosure, with controlled temperature and relative humidity in the conditions required for the test. (See annex A.)

The conditioned air shall circulate over the surface of the test pieces at a speed between 0,5 and 2,5 m/s (30 and 150 m/min).

The control shall be such that the specified conditions are re-established not more than 15 min after the door of the enclosure has been closed. The door shall be open for the shortest possible time; this is especially important with materials having high transmission rates.

6.3 Balance, for weighing the dishes to the nearest 0,5 mg.

6.4 Tongs or holders, for manipulating the dishes.

6.5 Desiccant : anhydrous calcium chloride in the form of granules passing a 4 mm sieve but retained on a 1,6 mm sieve, *or alternatively in the form of a friable flaked product* 1,5 to 2 mm in size.

NOTE — In particular circumstances it may be necessary to use another desiccant, for example silica gel, provided that the relative humidity above the desiccant does not exceed 1 %, and that the desiccant used is clearly stated in the test report.

6.6 Sealing material : a wax mixture (see annex B) which adheres strongly both to the dish and the test piece, and is not brittle at ordinary temperature, not hygroscopic and not susceptible to oxidation. A surface of 50 cm² of freshly melted wax when exposed for 24 h in condition B (see annex A), as previously indicated, shall not give a variation in mass of more than 1 mg.

6.7 Device for distributing the wax, such as a pipette with rapid rate of discharge and sufficient capacity (at least 25 ml) and a discharge tube of about 3 mm internal diameter.

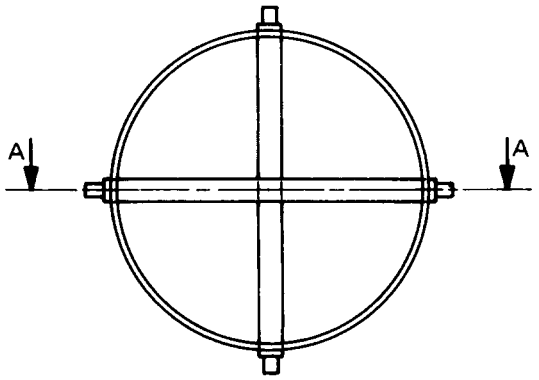
6.8 Cutting template. In order to save time, it is advisable to have a template for cutting the test pieces in the form of a ~~circular~~ disk with diameter equal to *D* + the width of the groove on the dish. This template may have a handle in the centre.

7 TEST PIECES

7.1 The test pieces shall be representative of the batch under test.

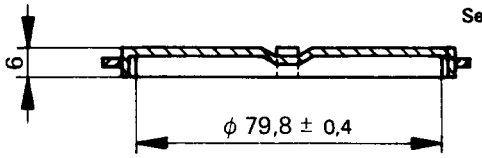
They shall be disks of diameter suitable for the dishes (diameter = *D* + the width of the groove; normally 90 mm) cut from the sheet using possibly the cutting template.

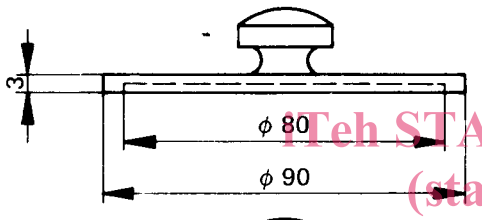
Dimensions in millimetres



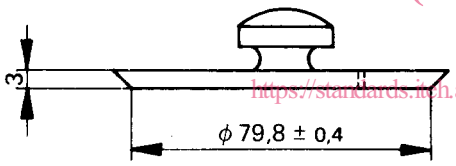
Ring template (6.1.2.1)

Section A-A

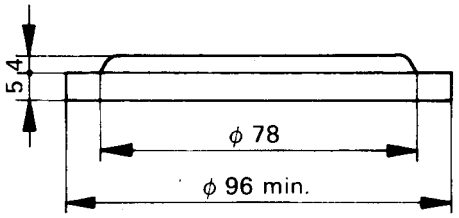




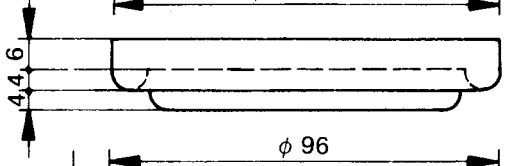
Cutting template (6.8)



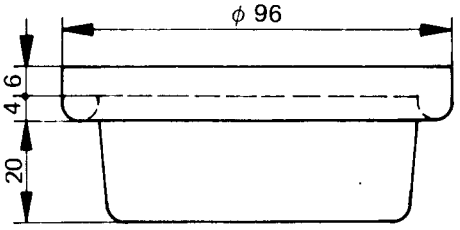
Cover template (6.1.2.2)



Lid (6.1.3) with rim to fit outside dish



Shallow dish (6.1.1) for materials with normal transmission rate



Deep dish (6.1.1) for materials with transmission rate greater than 100 g/m²d. (Tapered to nest)

Dimensions are shown for test areas of 50 cm². Values for dishes and lids show inside dimensions, except the overall diameter of the dishes, which is an outside dimension.

Only the dimension 79,8 ± 0,4 mm shall be strictly respected; the other dimensions are approximate.

FIGURE 1 – **Examples of test dishes and templates**

7.2 If the two surfaces of the sheet are not identical, the face which is exposed to the humid atmosphere shall be indicated in the test report. If measurements are to be made on both surfaces, two sets of test pieces are required.

7.3 Prepare at least three test pieces for each batch and each surface to be tested, and allow at least two blank test pieces if the material to be tested is hygroscopic or if a greater safety is required (see 8.2.2).

7.4 If the sheet has been prepared by a process involving solvents, the results may be affected by the residual solvent in the test pieces.

If the test pieces are treated to eliminate residual solvent, details of the treatment shall be mentioned in the test report.

7.5 It is recommended that the specimen be conditioned to equilibrium before placing in the dish, especially if the water vapour transmission rate is known to be high.

8 PROCEDURE

8.1 Preparation of dishes

The method of preparation is slightly different according to whether a cover or ring template is used.

Always begin by carefully cleaning and drying the dishes and the templates.

Introduce the desiccant, then put on the test piece and the template and make a vapour-tight seal between the test piece and the dish. The details for the different types of equipment are given below in 8.1.1 and 8.1.2. The work must be done rapidly in order to keep the absorption of water by the desiccant to a minimum.

8.1.1 *Use of wax and a cover template*

Fill each dish with desiccant up to 3 to 4 mm below the final position of the test piece and level by tapping. Place the test piece centrally in position, followed by the cover template. Melt the wax on a water bath at 100 °C and fill the dispensing device. Run the molten wax into the annular cavity until it reaches the level of the upper surface of the cover template and, after cooling, complete the joint by removing air bubbles and hair cracks with a small gas flame. A warm spatula may be run over the wax to assist in this process, so that shrinkage cracks that may have developed during cooling will be closed.

Remove the cover template and examine the assembly to make sure that the joint is satisfactory. To ensure that the cover template comes away easily, it is advisable previously to smear a thin film of petroleum jelly aound the edge and to wipe away any excess which could contaminate the test piece. Cover the assembly with a lid numbered to correspond with the number of the dish.

8.1.2 *Use of wax and a ring template*

Fill each dish with desiccant up to a level of 3 to 4 mm below the final position of the test piece and level by tapping. Melt the wax on a water bath at 100 °C and fill the dispensing device. Run the molten wax into the circular groove round the dish until a slight meniscus is produced above the inner edge of the groove.

Place the test piece centrally in position, followed by the ring template, and load it with a 1 kg weight.

Run more wax into the annular space so formed and, after cooling, complete the joint by removing any air bubbles and hair cracks with a small gas flame. A warm spatula may be run over the wax to assist in this process, so that shrinkage cracks that may have developed during cooling will be closed. Remove the weight and leave the ring in place.

8.2 Determination

8.2.1 *General method*

8.2.1.1 Weigh all the dishes with their lids to the nearest 0,5 mg.

8.2.1.2 Place them upright in the enclosure controlled to the conditions of the test (see annex A) after having removed the lids, which are to be left outside.

8.2.1.3 Carry out successive weighings of the dishes with their lids at suitable intervals of time.

The weighings are to be carried out as follows :

Remove the dishes from the controlled enclosure using the tongs or holders. Cover them with their respective lids and leave them to reach ambient temperature. Weigh the assemblies to the nearest 0,1 mg, then return to the enclosure after having again taken off the lids.

Take care to work rapidly, taking the dishes in small groups always containing the same number, so that the whole weighing operation always lasts about the same time (not exceeding 30 min).

It is also possible to work without the lids, but in this case it is advisable to use blank assemblies (see 8.2.2), and transport and cooling of the dishes must be done in a closed vessel with calcium chloride desiccant.

The interval between weighings should preferably be 24, 48 or 96 h, but shorter time intervals (for example 3, 4 or 8 h) may be necessary for materials with a high transmission rate. The choice depends on the transmission rate of the sheet being tested; the gain in mass between two successive weighings should be at least 5 mg. This choice is to be made at the beginning of the test.

If the first weighing shows a gain in mass too large or too small, the subsequent time interval for weighing may be modified.

8.2.1.4 Continue the weighings until the increase in mass per unit time of exposure to the selected atmosphere becomes constant to within 5 % of two successive weighings (see clause 9).

8.2.1.5 The test must be completed before the efficiency of the desiccant is reduced appreciably. (In practice, the total increase in mass should not exceed 1,2 g for shallow dishes and 3,2 g for deep ones.)

8.2.2 *Use of blank assemblies*

If the sample is of low transmission rate and considerable thickness, for example rubber, plastics or polyethylene-coated board, or is appreciably hygroscopic, it is advisable to test two or more blank assemblies, prepared in the same manner but without desiccant, in addition to each ten normal test assemblies. All the measured masses are then corrected at each time interval by the mean change of mass of the blank assemblies which undergo the same treatment.

8.2.3 *Creased sheet*

A method for use when the transmission rate of a creased sheet is needed is given in annex C.

9 EXPRESSION OF RESULTS

9.1 Express the test results by one of the following two methods :

9.1.1 For each dish, represent the total increase in mass graphically as a function of time of exposure, the test being completed when three or four points lie on a straight line (see 8.2.1.4), showing a constant rate of passage of water vapour.

Using this straight line, the water vapour transmission rate for each dish is then calculated, in grams per square metre per 24 h, from the formula

$$\frac{240 \times m_1}{S}$$

where

m_1 is the increase in mass, in milligrams per hour, determined from the graph;

S is the area, in square centimetres (normally 50 cm^2), of the tested surface of the test piece.

9.1.2 If weighings are made at identical time intervals, it is possible to calculate the transmission rate for each dish directly from the results, without preparing a graph, by using the above formula but taking $\frac{m_2}{t}$ as the value for m_1,

t being the total duration, in hours, of the last two exposure periods (see 8.2.1.4);

m_2 being the increase in mass, in milligrams, of the assembly during the time t.

9.2 For several assemblies corresponding to a single sample of test material, and to a single face, calculate the arithmetic mean of the results obtained by either 9.1.1 or 9.1.2.

9.3 Report the mean water vapour transmission rate for values

over 100 g/m^2d : to the nearest 10 g/m^2d;

from 10 g/m^2d to 100 g/m^2d : to the nearest whole number;

less than 10 g/m^2d : to the first decimal place.

10 TEST REPORT

The test report shall include the following particulars :

a) reference to this International Standard;

b) full identification of the material tested, in particular grammage, thickness (if required) and identification of the outside face during tests;

c) depth of dish;

d) test conditions (see annex A);

e) the individual results obtained;

f) the arithmetic mean if the largest difference between individual results and the arithmetic mean does not exceed 10 % of this mean;

g) any details of procedure that are optional, such as creasing, treatments eliminating residual solvent, or not included in this International Standard, together with any other information that may have a bearing on the results.

ANNEX A

TEST CONDITIONS

Although other conditions may be required for special purposes, certain standard temperatures and relative humidities have been established for testing paper and plastics. These are :

Condition A : Temperature 25 ± 0,5 °C
Relative humidity 90 ± 2 %

Condition B : Temperature 38 ± 0,5 °C
Relative humidity 90 ± 2 %

Condition C : Temperature 25 ± 0,5 °C
Relative humidity 75 ± 2 %

ANNEX B

SEALING WAXES

Suitable wax compositions are :

B.1 60 % microcrystalline wax and 40 % refined crystalline paraffin wax.

B.2 90 % microcrystalline wax and 10 % plasticizer[1].

B.3 80 % paraffin wax with a melting point of 50 to 52 °C and 20 % viscous consistency polyisobutylene (relatively low degree of polymerization).

B.4 Mixture of waxes with melting points 60 to 75 °C and oil content 1,5 to 3 %.

If the wax contains traces of water, they can be eliminated by heating to 105 to 110 °C and stirring.

The oil content of the microcrystalline wax should be below 3 %, and that of the refined paraffin wax below 1 %.

1) Mobil Oil Company Limited microcrystalline wax Mobilwax 2305 with Mobil-Kote 338 as plasticizer has been found suitable.

ANNEX C

CREASING

If the water vapour transmission rate of creased material is required, the creasing should be carried out using one of the methods given in this annex.

C.1 DEFINITIONS

C.1.1 transmission rate of a creased sheet : The rate of transmission measured on a test piece cut after having been creased in a standardized manner and after the sheet has again been straightened.

C.1.2 transmission rate of the creases : The difference between the transmission rate of the creased sheet and the transmission rate of the uncreased sheet, both given in g/m^2d; it is expressed in grams per 100 linear metres per 24 h (g/100 md).

C.2 PRINCIPLE

A test piece is cut and creased so that finally there is a double series of creases in accordion fashion forming a pattern of squares, i.e. a series of parallel creases and a series of perpendicular creases having the same spacing.

The spacing of the pattern of squares is such that in the final test piece the value of the total length of the creases, in centimetres, contained in the area S (normally 50 cm^2) is the same number as the area, in square centimetres.

The test piece is cut and put into the dish in such a manner that the centre of the circular dish is at the centre of gravity of one of the squares formed by the creases.

C.3 APPARATUS

C.3.1 Creasing table, in the form of a flat rectangular plate, the width of which is slightly larger than the larger dimension of the test piece.

C.3.2 Cutting template, of square shape having the dimensions of the test piece before creasing. This template may have notches making it possible to mark the position of the creases (see figure 2).

C.3.3 Pressing plate : a rigid rectangular flat plate, the length of which is about 175 mm and the width either 15 mm (Procedure A) or 30 mm (Procedure B). This plate is suitably loaded for the creases to be pressed under a load of 9,8 N per 10 mm of crease.

The creasing may also be done by a suitable press.

C.3.4 Ruling plate (or wooden rule), size approximately 200 mm × 30 mm, with smooth straight edges.

C.4 PREPARATION OF TEST PIECES FOR CREASING

The number of test pieces to be prepared for creasing is the same as that provided for in 7.3 of this International Standard.

Using a template, (C.3.2), cut the test pieces in a square shape, the side of which has a dimension greater than that of the test piece that will finally be fixed into the dish.

The precise dimension of the test piece for creasing will depend on the manner in which the creasing is carried out (see C.6).

If a sheet with a particular direction (for example, machine direction) is used, the cutting shall be carried out in such a manner that this direction is parallel to one of the sides of the test piece (except if it has been specified that the cutting is to be made diagonally, in which case there shall be an angle of 45° between the particular direction and the sides of the square).

If a template with notches is used, mark each crease (with a notch or pencil line, for example) on the periphery of the test piece for creasing.

C.5 CONDITIONING OF THE TEST PIECES BEFORE CREASING

Condition the test pieces in the conditions usual for the material, i.e. in accordance with the requirements of one of the following documents :

– ISO/R 187, *Method for the conditioning of paper and board test samples;*

– ISO/R 471, *Standard atmospheres for the conditioning and testing or rubber test pieces;*

– ISO/R 291, *Plastics – Standard atmospheres for conditioning and testing;*

– ISO 2231, *Fabric coated with rubber or plastics – Standard atmospheres for conditionning and testing.*

In the absence of any particular recommendation, choose one of the above documents.

C.6 CREASING

C.6.1 Principle

The creasing consists of preparing a double series of creases in accordion fashion (adjacent creases being the opposite sides of the sheet), in such a way as to form the square pattern indicated in clause C.2.