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PLASTICS

DETERMINATION OF THE WATER VAPOUR TRANSMISSION RATE OF PLASTICS FILMS AND THIN SHEETS

DISH METHOD

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BRIEF HISTORY

The ISO Recommendation R 1195, *Plastics – Determination of the water vapour transmission rate of plastics films and thin sheets – Dish method,* was drawn up by Technical Committee ISO/TC 61, *Plastics,* the Secretariat of which is held by the American National Standards Institute (ANSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 820,which was circulated to all the ISO Member Bodies for enquiry in May 1965. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	France	Romania
Australia	Germany	Spain
Austria	Hungary	Sweden
Belgium	India	Turkey
Brazil	Israel	U.A.R.
Canada	Italy	United Kingdom
Chile	Japan	U.S.A.
Czechoslovakia	New Zealand	U.S.S.R.
Finland	Poland	

The following Member Body opposed the approval of the Draft :

Switzerland

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation | R 1195 | June 1970

PLASTICS

DETERMINATION OF THE WATER VAPOUR TRANSMISSION RATE* OF PLASTICS FILMS AND THIN SHEETS

DISH METHOD

1. SCOPE

This ISO Recommendation describes a method for the determination of the water vapour transmission rate of plastics films and thin sheets using dishes with wax seal.

2. DEFINITION

The water vapour transmission rate of a film or thin sheet is the mass of water vapour transmitted from one face to the other, under a constant differential vapour pressure, per unit of surface area and during a given time. It should be expressed in grammes per square metre per 24 hours at the conditions of temperature and humidity defined at the two faces of the film or sheet. This transmission rate depends, of course, upon the thickness of the film and upon the permeability of its constituent material.

3. PRINCIPLE

Dishes containing a dehydrating agent and closed by the film or sheet to be tested are placed in an enclosure with constant and regulated temperature and relative humidity.

These dishes are weighed at intervals of time previously fixed. The water vapour transmission rate is found from the increase in mass once this has become proportional to time.

4. SIGNIFICANCE OF THE TEST

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

The use of the results for any particular application should, however, be guided by experience.

Transmission rate is not a linear function of temperature nor, generally, of relative humidity. A determination carried out under certain conditions is not valid under other conditions.

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

5. APPARATUS AND MATERIALS

5.1 *Dishes and accessories.* 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 A5 protected by chemical or anodic oxidation is quite suitable.

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

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* This characteristic is often erroneously termed "permeability".

5.1.1 CIRCULAR DISHES, carrying a groove around the rim for sealing the test piece with wax.

Figures 1 and 2 show suitable designs. The grooves must have a profile such that the test pieces 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 clause 5.1.2). The interior diameter of the ring portion of the dish on which the test piece rests must be equal to or very slightly larger than this diameter *d*. Moreover, the surface area of the bottom of the dish where it is filled with desiccant must also be similar to that of the exposed surface, and there must not be any obstruction within the dish which could interfere with the flow of water vapour between the test piece and the desiccant. The depth, *h*, from the plane of the test piece must be about 15 mm.

It is advisable to number the dishes.

5.1.2 TEMPLATES FOR WAX. These are necessary in order to place the wax easily and allow the test surface to be defined exactly. Their diameter, *d*, must be known with an accuracy better than 0.5 %.

The diameter should be 79.8 ± 0.4 mm (area of 50 cm^2). However, it is possible to use apparatus of smaller diameter, but at least 56.4 mm (area of 25 cm^2).

These templates may comprise either

(*a*) *a cross-braced ring template* (see Fig. 1) which remains in place during the test. The number of templates must be the same as that of the dishes. The diameter, *d*, is the interior diameter of the ring;

or

(*b*) *a cover template* (see Fig. 2) 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, and having the edge chamfered at 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.

5.1.3 LIDS. These must close the dish assemblies sufficiently well to allow them to be brought out from the enclosure for weighing without loss of water vapour. They must be numbered to correspond with each dish.

5.2 *Enclosure*, with constant and regulated temperature and relative humidity in the conditions A or B.

Condition A : Temperature 25 ± 0.5 °C
Relative humidity 90 ± 2 %

Condition B : Temperature 38 ± 0.5 °C
Relative humidity 90 ± 2 %

The conditioned air must circulate over the surface of the test pieces at a speed of from 30 to 150 m/min (1.6 to 8.2 ft/s).

The regulating device must be such that the specified conditions are re-established not more than 15 minutes after the door of the enclosure has been closed. This necessitates that the door must only be kept open for the shortest possible time.

NOTE. – A more general ISO Recommendation for the determination of water vapour transmission rate, applying to all kinds of sheet materials, is being prepared. This general method also allows testing at 25 ± 0.5 °C and 75 % R.H.

5.3 *Balance*, for weighing the dishes to the nearest 0.1 mg.

5.4 *Tongs or holders*, for manipulating the dishes.

5.5 *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 crumbly flaked product 1.5 to 2 mm in size.

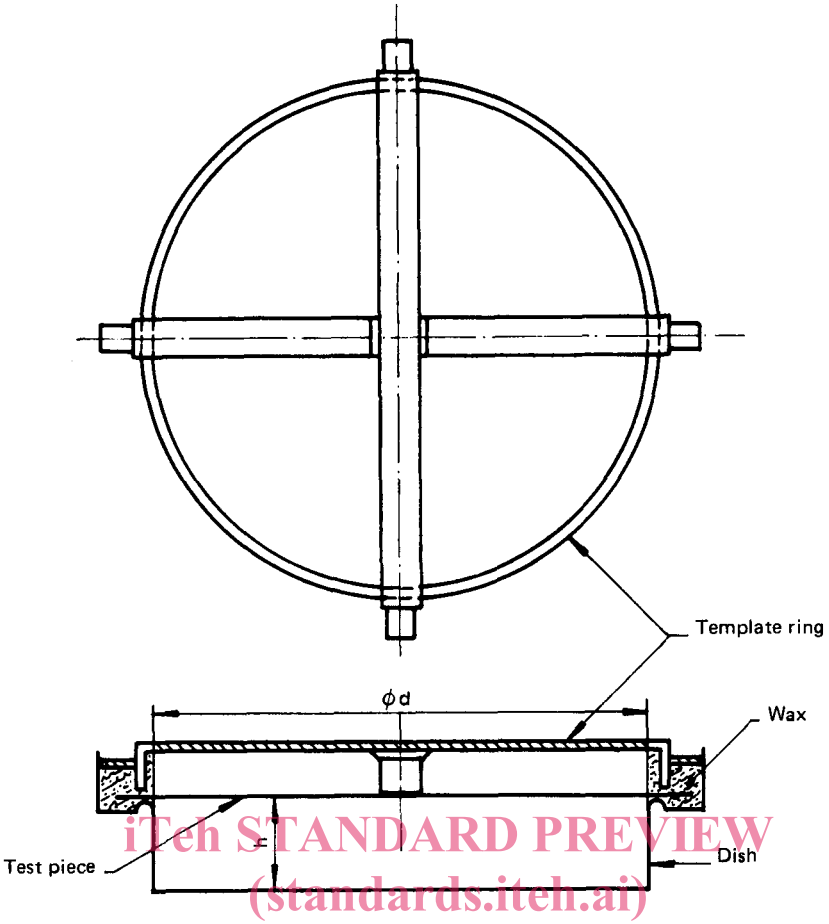

FIG. 1 – Cross-braced ring template and dish

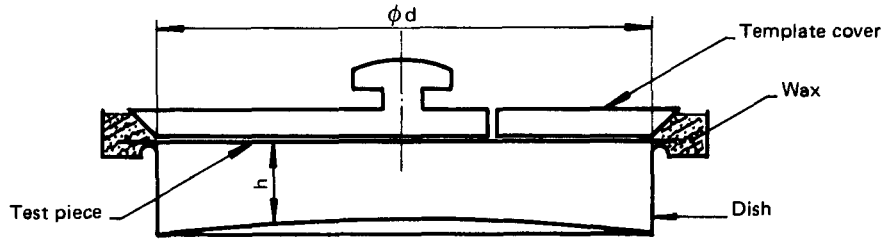

FIG. 2 – Cover template and dish

5.6 *Sealing material.* A wax mixture 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 cm^2 of freshly melted wax when exposed for 24 hours in condition B (see clause 5.2) must not give a variation in mass more than 1 mg (see clause 9.1).

5.7 *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.

5.8 *Template for cutting.* 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 + twice the half-width of the groove on the dish. This template may have a handle in the centre.

6. TEST PIECES

6.1 The test pieces must be representative of the batch under test.

They should be disks of diameter suitable for the dishes (diameter = d + width of the groove) cut from the film or sheet using the cutting template.

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

6.3 Prepare at least three test pieces for each batch and each surface to be tested, and allow one or two blank pieces if the material to be tested is hygroscopic or if a positive check is required (see clause 7.2.2).

6.4 If the film or 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 this residual solvent, details of treatment should be mentioned in the test report.

7. PROCEDURE

7.1 Preparation of dishes

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

Always begin by carefully cleaning and drying the dishes and also the ring templates or the cover template.

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. (See clauses 7.1.1 and 7.1.2.) *The work should be done rapidly* to keep the absorption of water by the desiccant to the minimum.

7.1.1 *Use of wax and a cover template.* Fill each dish with calcium chloride up to 3 to 4 mm below the final position of the test piece and level by tapping. Put the test piece on centrally 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 with a small gas flame.

Remove the cover template and examine the assembly to make sure that the joint is satisfactory. To ensure the cover template coming away easily, it is advisable previously to smear some vaseline around the edge. Cover the assembly with a numbered lid.

7.1.2 *Use of wax and a ring template.* Melt the wax on a water bath at 100 °C, then fill the dispensing device. Run the molten wax into the circular groove round the dish until a slight meniscus is produced on the inner edge.

Fill the 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 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 with a small gas flame. Remove the weight and leave the ring in place.

7.2 Test

7.2.1 *General method*

(*a*) Weigh all the dishes with their lids, to the nearest 0.1 mg.

(*b*) Place the dishes in the enclosure controlled to conditions A or B after having removed the lids, which should be left outside.

(*c*) Carry out successive weighings of the dishes at suitable intervals of time.

THE WEIGHINGS should be carried out as follows :

Remove the dishes from the controlled enclosure using the tongs or holder. Cover them with their respective lids and leave them to reach ambient temperature. Weigh the assemblies to the nearest 0.1 mg, then return them to the enclosure after having 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.

It is also possible to work without the lids, but in this case it is advisable to use blank assemblies (see clause 7.2.2), and transport and cooling of the dishes must be done in a closed vessel with calcium chloride desiccant.

THE INTERVAL between weighings should be 24, 48 or 96 hours. The choice depends on the transmission rate of the film being tested; the gain in mass between two successive weighings should be at least 5 mg.

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

(*d*) Continue the weighings until the increase in mass per unit time becomes constant to within 5 % for two successive weighings (see clause 9.2).

(*e*) The test must be finished before the efficiency of the desiccant is reduced appreciably. (In practice, 20 g of calcium chloride may be permitted to absorb up to 2 g of water.)

7.2.2 *Use of blank assemblies.* If the sample is of low transmission rate and considerable thickness, and is appreciably hygroscopic, it is advisable to test two or more *blank assemblies* in addition to each ten normal test assemblies, prepared in the same manner but without desiccant. All the measured masses are then corrected for each time interval by the mean change of mass of the blank assemblies which are put through the same treatment.

8. EXPRESSION OF RESULTS

Calculate the water vapour transmission rate, in grammes per square metre per 24 hours (see clause 9.3), for each assembly, from the formula

$$\frac{240\,m}{t\,S}$$

where

S is the area, in square centimetres, of the tested surface of the test piece;

t is the total duration, in hours, of the last two exposure periods;

m is the increase in mass, in milligrammes, of the assembly during the time t.

For several assemblies corresponding to a single sample of test material, and to a single face, calculate the arithmetic mean and ensure that the maximum variation of the individual results from the arithmetic mean does not exceed 10 % of the arithmetic mean.

9. NOTES

9.1 The following wax mixtures are considered suitable :

(*a*) 60 % of micro-crystalline wax and 40 % of refined crystalline paraffin wax.

(*b*) 90 % of micro-crystalline wax and 10 % of plasticizer.

(*c*) 80 % of paraffin wax with melting point of 50 to 52 °C and 20 % of viscous consistency polyisobutylene (relatively low degree of polymerisation).

(*d*) Mixture of waxes with melting points 60 to 75 °C. Oil content 1.5 to 3 %.

If the wax contains traces of water, it is advisable to heat it to 105 to 110 °C, with stirring, to eliminate the water.

9.2 The total increase in mass can also be represented graphically as a function of time, and the test can end when three or four points lie on a straight line showing a constant rate of passage of water vapour.

9.3 If the weighings are plotted as a function of time, the transmission rate can be calculated from the straight line giving the best fit with the last three or four points.

10. TEST REPORT

The test report should include :

(*a*) full identification of the material tested, in particular its thickness and identification of the outside face during the tests;

(*b*) the controlled atmosphere used, A or B;

(*c*) the individual results obtained;

(*d*) the arithmetic mean, if the greatest of all the differences between the individual results and this mean does not exceed 10 % of the mean value;

(*e*) any details of procedure which are optional, such as treatments eliminating residual solvent (see clause 6.4), or not included in this ISO Recommendation, together with any other information which may have a bearing on the results.

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