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Plastics — Determination of water absorption

Plastiques — Détermination de l'absorption d'eau

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Foreword

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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.

International Standard ISO 62 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

This second edition cancels and replaces the first edition (ISO 62:1980), of which it constitutes a technical revision.

Annexes A and B of this International Standard are for information only.

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Introduction

Plastics exposed to water are subject to several different effects:

- a) dimensional changes (e.g. swelling) caused by absorption of water;
- b) extraction of water-soluble components; and
- c) changes in other properties.

However, exposure to humidity, immersion, and exposure to boiling water can result in distinctly different material responses. The equilibrium moisture content can be used to compare the amount of water absorbed by different types of plastics when they are exposed to moisture. Moisture content determined under non-equilibrium conditions can be used to compare different batches of the same material and to determine the diffusion constant of the material when determined under carefully controlled non-equilibrium exposure conditions to moisture and when using plastic specimens of defined dimensions.

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Plastics — Determination of water absorption

1 Scope

1.1 This International Standard describes a procedure for determining the moisture absorption properties in the “through-the-thickness” direction of flat or curved-form solid plastics. This International Standard also describes procedures for determining the amount of water absorbed by plastic specimens of defined dimensions, when immersed in water or when subjected to humid air under controlled conditions. The “through-the-thickness” moisture diffusion coefficient can be determined for single-phase material by assuming Fickian diffusion behaviour with constant moisture absorption properties through the thickness of the test specimen. This model is valid for homogeneous materials and for reinforced polymer-matrix composites tested below their glass transition temperature. However, some two-phase matrices such as hardened epoxies may require a multi-phase absorption model which is not covered by this International Standard.

1.2 Ideally the best comparison of the water absorption properties and/or diffusion coefficients of materials should be carried out only using the equilibrium moisture content of plastics exposed to identical conditions. The comparison of materials using properties at moisture equilibrium does not assume, and is therefore not limited to, single-phase Fickian diffusion behaviour.

1.3 Alternatively, water absorption of plastic specimens of defined dimensions exposed to immersion or humidity under controlled conditions but for an arbitrary time period can be used to compare different batches of the same material or for quality control tests of a given material. For this type of comparison, it is essential that all test specimens be of identical dimensions and, as nearly as possible, have the same physical attributes, e.g. surface smoothness, internal stresses, etc. However, moisture equilibrium is not reached under these conditions. Therefore, results from this type of test cannot be used to compare the water absorption properties of different types of plastics. For the more reliable results, simultaneous tests are recommended.

1.4 The results obtained using the methods described in this International Standard are applicable to most plastics but are not applicable to cellular plastics, granulates, or powders which can show additional absorption and capillary effects. Plastics exposed to moisture under controlled conditions for defined periods of time provide relative comparisons between them. The tests described for determination of diffusion coefficient may not be applicable to all plastics. Plastics that cannot retain their shape when immersed in boiling water should not be compared using method 2 in 6.3.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 175:—¹⁾, *Plastics — Determination of the effects of liquid chemicals, including water.*

ISO 294-3:1996, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates.*

ISO 2818:1994, *Plastics — Preparation of test specimens by machining.*

¹⁾ To be published. (Revision of ISO 175:1981)

3 Principle

Test specimens are immersed in distilled water at 23 °C or in boiling distilled water, or exposed to 50 % relative humidity, at given temperatures for prescribed times. The amount of water absorbed by the test specimen is determined by measuring its change in mass, i.e. the difference of its initial mass from that after exposure to water, and is expressed as a percentage of the initial mass. If required, the amount of water lost after drying the test specimens can also be determined.

In some applications, 70 % to 90 % relative humidity and temperatures of 70 °C to 90 °C may be necessary. Higher relative humidity and temperature conditions than those recommended in this International Standard may be used by agreement between interested parties. When relative humidity and temperature conditions other than those recommended are used, a complete description of these conditions (with appropriate tolerances) shall be included in the test report.

4 Apparatus

4.1 Balance, with an accuracy of $\pm 0,1$ mg (see clause 6, note 3).

4.2 Oven, with forced-air convection or vacuum, maintained at $(50,0 \pm 2,0)$ °C or at any other agreed temperature (see clause 6, note 2).

4.3 Containers, of distilled water, or water of equivalent purity, equipped with a means of heating and maintaining the temperature specified.

4.4 Desiccator, with a desiccant (e.g. P_2O_5).

4.5 Means of measuring dimensions of test specimens, to an accuracy of $\pm 0,1$ mm, if required.

5 Test specimens

5.1 General

For each material evaluated, test at least three test specimens. Test specimens of the required dimensions may be prepared by moulding or extrusion. The methods used for preparation of all test specimens shall be included in the test report.

NOTE The results of this method may be influenced by surface effects. For some materials, different results can be obtained between moulded specimens and specimens cut from a larger sheet.

Any contaminant on the test specimen surface which could influence water absorption shall be removed by a cleaning agent which does not attack the plastic, the degree of attack being determined in accordance with ISO 175:—, [see for example in 5.6, table 1, notation “none” (no change in appearance)]. Allow specimens to dry at 23 °C and 50 % relative humidity for at least 2 h after cleaning and before beginning the test. Wear clean gloves when handling test specimens to prevent contamination.

The cleaning agent shall have no effect on water absorption. When determining moisture equilibrium content according to 6.2 (method 1) and 6.5 (method 4) the effects of the cleaning agent can be ignored.

5.2 Square-shaped test specimens for homogeneous plastics

Unless otherwise specified or agreed by all interested parties, dimensions and tolerances of the square-shaped specimens should be the same as those given in ISO 294-3:1996, type D1. They can be prepared by moulding according to ISO 294-3:1996, using conditions given in standards applicable to the material being tested (or using conditions recommended by the material supplier). For some materials, such as polyamides, polycarbonates, and some reinforced plastics, use of a 1 mm thick specimen may not give meaningful results. Furthermore, some product specifications require use of thicker test specimens for the characterization of water absorption. In these cases, test specimens of $(2,05 \pm 0,05)$ mm thickness may be used. If specimens other than those 1 mm thick are

used, the specimen thickness used shall be indicated in the test report. There are no requirements for the radius of the edges and corners. However, they shall be smooth and clean to prevent material from the edges and corners from being lost during the test.

Some materials may exhibit mould shrinkage. If specimens of these materials are prepared using a mould with dimensions at the lower limits specified in ISO 294-3:1996, the final dimensions of the test specimens may be beyond the tolerances given in this International Standard and shall be noted in the test report.

5.3 Test specimens of reinforced plastics affected by anisotropic diffusion effects

For some reinforced plastic materials, such as carbon fibre reinforced epoxies, anisotropic diffusion effects caused by reinforcing material may produce erroneous results when small specimens are used. Where this is of concern, test specimens that meet one of the following requirements shall be used and the specific dimensions and methods of test specimen preparation shall be included in the test report.

- a) A nominally square-shaped plate or curved panel with dimensions that satisfies the relation:

$$w \leq 100 d$$

where

w is the nominal length of one side, in millimetres, and,

d is the nominal thickness, in millimetres.

- b) A 100 mm × 100 mm square-shaped plate with stainless steel or aluminium foil bonded to the edges so that moisture absorption through the edges is minimized. When this specimen is prepared, care shall be taken to weigh the specimen before and after bonding of the foil in order to obtain the mass increase due to the foil and the adhesive. Use only poorly absorbing adhesive which will not affect the results.

5.4 Tubular test specimens

Unless specifically required by other International Standards, tubular test specimens shall have the following dimensions.

- a) For tubes having an internal diameter of 76 mm or less, cut sections of tube that are (25 ± 1) mm in length from a longer tube of the material being evaluated. The cut shall be made perpendicular to the longitudinal axis of the tube. The cut can be made by machining, sawing, or shearing and shall produce smooth edges free from cracks.
- b) For tubes having an internal diameter larger than 76 mm, cut rectangular specimens (76 ± 1) mm in length (measured along the outside surface of the tube) by (25 ± 1) mm in width. The cut edges shall be smooth and free from cracks.

5.5 Rod-shaped test specimens

Rod-shaped test specimens shall have the following dimensions.

- a) For rods 26 mm in diameter or less, cut a section of (25 ± 1) mm in length. The cut shall be made perpendicular to the longitudinal axis of the rod. The diameter of the test specimen shall be the diameter of the rod.
- b) For rods larger than 26 mm in diameter, cut a section of (13 ± 1) mm in length. The cut shall be made perpendicular to the longitudinal axis of the rod. The diameter of the test specimen shall be the diameter of the rod.

5.6 Specimens cut from finished products, extrusion compounds, sheets or laminates

Unless required by another International Standard, cut a piece from the product that:

- meets the requirements for the square-shaped test specimen, or

- has a length and width of (61 ± 1) mm and has the same shape (thickness and curves) of the material being tested.

The machining conditions used to prepare the test specimens shall be agreed by all interested parties. They also shall be in accordance with ISO 2818 and shall be included in the test report.

If the nominal thickness is greater than 1,1 mm and in the absence of special provisions in the relevant application, the thickness of the test specimen shall be reduced to 1,0 mm to 1,1 mm by machining on one surface only.

When the surface of a laminate is machined, the water absorption properties will be influenced to such an extent that the test results cannot be valid. Therefore, the specimen shall be tested at its original thickness and the dimensions shall be specified in the test report.

6 Test conditions and procedures

NOTE 1 Certain materials may require that the test specimens be weighed in a weighing vessel.

NOTE 2 Drying procedures other than those described in 6.2 to 6.5 may be used by agreement between the interested parties.

NOTE 3 When the water absorption of the material is greater than or equal to 1%, specimens having been weighed accurately to ± 1 mg and whose mass is constant to ± 1 mg are acceptable.

6.1 General conditions

6.1.1 Use at least 8 ml of distilled water per square centimetre of the total surface of the test specimen but no less than 300 ml per test specimen (see clause 5). This will prevent any extraction product from becoming excessively concentrated in the water during the test.

6.1.2 Place each set of three test specimens in a separate container (4.3) and completely immerse them in water.

When several samples of the same composition are to be tested, the test specimens may be placed together in the same container with at least 300 ml per test specimen. However, in this case, significant surface contact between test specimens or with the walls of the container is unacceptable and shall be prevented.

NOTE Grids of stainless steel may help to ensure a minimum distance between the test specimens.

For specimens having a density lower than that of water, immerse the specimens by placing them in a wire-grid stainless-steel basket connected to an anchor-weight by a stainless-steel wire. Avoid surface contact of the anchor-weight with the specimens.

6.1.3 Time intervals for water immersion are given in 6.2 and 6.3. However, longer immersion periods may be used by agreement between the interested parties. In such cases the following precautions shall be taken:

- for tests performed in water at 23 °C, agitate the water at least once daily, for example by swirling the contents of the beaker;
- for tests performed in boiling water, add boiling water as required in order to maintain the volume.

6.2 Method 1: Determination of water content absorbed after immersion in water at 23 °C

Dry all replicate test specimens in an oven (4.2) maintained at (50 ± 2) °C for (24 ± 1) h and allow them to cool to room temperature in the desiccator (4.4) before weighing them to the nearest 0,1 mg. Repeat this process until the mass of the specimens is constant (mass m_1) within $\pm 0,1$ mg. Then place the test specimens in a container (4.3) filled with distilled water, maintained at $23,0 \text{ °C} \pm 1,0 \text{ °C}$ or $\pm 2,0 \text{ °C}$ according to the relevant specification. In the absence of a specification, the tolerance shall be $\pm 1,0 \text{ °C}$.

After immersion for (24 ± 1) h, take the test specimens from the water and remove all surface water with a clean, dry cloth or with filter paper. Reweigh the test specimens to the nearest 0,1 mg within 1 min of removing them from the water (mass m_2).

The water content at saturation is measured by re-immersing the test specimens and reweighing them at given time intervals. A typical immersion time scale is 24 h, 48 h, 96 h, 192 h, etc. At each of these intervals (± 1 h), remove the test specimens from the water, remove all surface water and reweigh each test specimen to the nearest 0,1 mg within 1 min of removing them from the water (e.g. $m_{2/24\text{ h}}$).

6.3 Method 2: Determination of water content absorbed after immersion in boiling water

Dry all replicate test specimens in an oven (4.2) maintained at $(50,0 \pm 2,0)$ °C for (24 ± 1) h and allow them to cool to room temperature in the desiccator (4.4) before weighing them to the nearest 0,1 mg. Repeat this process until the mass of the specimens is constant (mass m_1) within $\pm 0,1$ mg.

Place the test specimens in a container (4.3) of boiling distilled water, so that they are supported on edge and are entirely immersed. After (30 ± 2) min, remove the test specimens from the boiling water and cool them in distilled water maintained at room temperature. After cooling the test specimens for (15 ± 1) min, remove them from the water, one at a time. Remove all surface water with a dry cloth, then immediately weigh them to the nearest 0,1 mg (mass m_2). If the thickness of the test specimens is less than approximately 1,5 mm, a small, but measurable amount of water desorption may occur during the weighing procedure. In this case, it is preferable to weigh the specimens in a weighing bottle.

The water content at saturation is measured by re-immersing the test specimen and reweighing it after each (30 ± 2) min interval. After each of these intervals, the test specimens shall be removed from the water, cooled in distilled water, dried and weighed as described above.

Cracks may be formed by repeated successions of immersion and drying. The number of cycles at which the cracks are observed shall be noted in the test report.

6.4 Method 3: Determination of water-soluble matter lost during immersion

When materials are known or suspected to contain appreciable amounts of water-soluble ingredients, it is necessary to correct for the water-soluble matter lost during the immersion test. For this purpose, after immersion according to 6.2 or 6.3, recondition the test specimens to constant mass (mass m_3) as used in the original drying period of 6.2 and 6.3. If this reconditioned mass m_3 is less than the conditioned mass m_1 , the difference shall be considered as the water-soluble matter lost during the immersion test. For such materials, the water absorption value shall be taken as the sum of the increase in mass following immersion and the mass of the water-soluble matter.

6.5 Method 4: Determination of water content absorbed after exposure to 50 % relative humidity

Dry all replicate test specimens in an oven (4.2) maintained at $(50,0 \pm 2,0)$ °C for (24 ± 1) h and allow them to cool to room temperature in a desiccator (4.4) before weighing them to the nearest 0,1 mg. Repeat this process until the mass of the specimens is constant (mass m_1) within $\pm 0,1$ mg.

Then place the test specimens in a container or room containing air with a relative humidity of (50 ± 5) %, maintained at $23,0$ °C $\pm 1,0$ °C or $\pm 2,0$ °C according to the relevant specification. In the absence of a specification, the tolerance shall be $\pm 1,0$ °C. After conditioning the test specimens for (24 ± 1) h, reweigh them to the nearest 0,1 mg (mass m_2) within 1 min after the removal from the container or the room containing air with a relative humidity of (50 ± 5) %. The moisture equilibrium content is measured by repeating exposure of the test specimens to 50 % relative humidity, following the same weighing procedure and time intervals as given in method 1 (6.2).

7 Expression of results

7.1 Percentage by mass of water absorbed

For each test specimen, calculate the percentage change in mass, c , relative to the initial mass by using the appropriate formula:

$$c = \frac{m_2 - m_1}{m_1} \times 100$$

or

$$c = \frac{m_2 - m_3}{m_1} \times 100$$

where

m_1 is the mass of the test specimen, in milligrams (mg), after initial drying and before immersion;

m_2 is the mass of the test specimen, in milligrams (mg), after immersion;

m_3 is the mass of the test specimen, in milligrams (mg), after immersion and final drying.

Express the result as the arithmetic mean of the three values obtained at the same exposure time.

NOTE In certain instances, it may be required to express the content of absorbed water as a percentage by mass with respect to the test specimen after drying, using the following formula:

$$c = \frac{m_2 - m_3}{m_3} \times 100$$

7.2 Determination of the water content at saturation and the diffusion coefficient using Fick's laws

At temperatures well below the glass transition temperature of the wet polymer, water absorption of most polymers (methods 1, 3 and 4) correlates well with Fick's laws (see annex A), and a diffusion coefficient independent of time and concentration^[1] can be calculated. In this case, the water content at saturation c_s as well as the diffusion coefficient D , expressed in millimetres squared per second (mm^2/s), can be determined by fitting the experimental data to Fick's law for sheets^[2,3] without waiting for constant mass. The water content at saturation is expressed as c_s when the test specimens are immersed in water according to methods 1, 2 or 3 and $c_s(50\%)$ when the test specimens are exposed to air at 50 % relative humidity (method 4). Graphical methods may be used to verify the Fickian diffusion behaviour of the test specimens, substituting the calculated D value, e.g. by a log plot which is then fitted to the theoretical data or by using a commercially available software package. In order to verify that the water absorption of a polymer follows Fickian diffusion behaviour, the polymer has to be in equilibrium with the water.

Figure A.1 in annex A shows a fairly exact solution of Fick's laws for sheets. A slope of 0,5 has been found for

$$c \leq 0,51 c_s$$

or

$$c/c_s \leq 0,51$$

or

$$\frac{D\pi^2 t}{d^2} \leq 0,50$$

where

t is the time of immersion of the test specimen in water or humid air, in seconds;

d is the test specimen thickness, in millimetres.

In the instance where

$$D\pi^2 t/d^2 \geq 5$$

use the value

$$c = c_s$$

Additional values are given in table 1.

Table 1 — Theoretical dimensionless values from Fick's law for sheets

$D\pi^2t/d^2$	c/c_s
0,01	0,07
0,10	0,22
0,5	0,51
0,7	0,60
1,0	0,70
1,5	0,82
2,0	0,89
3,0	0,96
4,0	0,99
5,0	1,00

EXAMPLE

After fitting the experimental data to the theoretical graph, take the experimental concentration $c_{70\%}$ from the calculation $c/c_s = 0,7$, and calculate:

$$c_s = \frac{c_{70\%}}{0,7}$$

where c_s and $c_{70\%}$ are expressed as milligrams per gram or as a percentage by mass.

The experimental time t_{70} at $c_{70\%}$ enables the diffusion coefficient D , expressed in millimetres squared per second (mm^2/s), to be calculated from

$$\frac{D\pi^2 t_{70}}{d^2} = 1$$

or

$$D = \frac{d^2}{\pi^2 t_{70}}$$

If t_{70} is given in seconds, π^2 is approximated as 10 and the thickness for a flat test specimen is 1 mm, then:

$$D = \frac{1}{10t_{70}}$$

NOTE Typical values of D for plastics at 23 °C are $10^{-6} \text{ mm}^2/\text{s}$, which gives a t_{70} of 10^5 s (or 1 day) with the 1 mm test specimen. With this thickness the immersion time necessary to calculate c_s and D will not normally exceed one week.

8 Precision

The precision of this test method is not known because inter-laboratory data are not available. Inter-laboratory data are being obtained and a precision statement will be added at the following revision.

9 Test report

The test report shall include the following:

- a) a reference to this International Standard;
- b) all details necessary for complete identification of the material or product tested;
- c) the type of test specimen used, the method of preparation, stating whether the specimens have been cut or not, their dimensions, their initial mass, and, if applicable, their initial surface area and their surface condition (for example whether they are machined or not);
- d) the method (1, 2, 3 or 4) used and the immersion period(s);
- e) the water absorption calculated by one or more of the ways of expressing results given in clause 7, reporting the mean and the standard deviation of the results (if the calculations described in 7.1 and 7.2 give a negative value for water absorption, this fact shall be stated clearly in the test report);
- f) the water absorption at saturation, c_s or $c_s(50\%)$ at 23 °C calculated according to 7.2;
- g) the diffusion coefficient at 23 °C calculated according to 7.2;
- h) any incidents likely to have affected the results;
- i) the date(s) of testing.

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