International Standard 4611

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Determination of the effects of exposure to damp heat, water spray and salt mist

Plastiques — Détermination des effets d'une exposition à la chaleur humide, au brouillard d'eau et au brouillard salin

First edition — 1980-03-01

UDC 678.5/.8 : 678.019.32 : 620.193.2 **Ref. No. ISO 4611-1980 (E)**

Descriptors : plastics, tests, environmental tests, damp heat tests, salt spray tests, water resistance tests.

Price based on 7 pages

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 4611 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in October 1977.

It has been approved by the member bodies of the following countries :

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The member body of the following country expressed disapproval of the document on technical grounds :

Canada

Printed in Switzerland

Plastics — Determination of the effects of exposure to damp heat, water spray and salt mist

0 Introduction

0.1 Various test methods are available for the exposure of plastics to different aggressive agents acting in a combined and simultaneous fashion, such as natural weathering. Other test methods are available for the purpose of a separate evaluation of the action of individual aggressive agents. Among the latter there are, for example, tests for the resistance to specific chemicals and to radiations of a definite spectral range.

For some applications, it may be desirable to evaluate the behaviour of the materials in a hot damp atmosphere just below the saturation limit of water vapour, as well as in the presence of the liquid phase.

In these conditions not only water absorption or leaching of some ingredients of the compound may be observed but also degradation phenomena due to hydrolysis, exudation of plasticizers, etc.

It may also be desirable sometimes to evaluate the behaviour of materials in the presence of a highly corrosive electrolyte, such as a sodium chloride solution (salt mist), which is a principal aggressive agent present in marine climates and of particular importance in the case of nautical applications. It is well known that sodium chloride has no noticeable action on the polymers that are the basic components of plastics, and that salt solutions, owing to their higher osmotic pressure, are normally absorbed by plastics to a lesser degree than pure water, but it cannot be assumed *a priori* that they have no action on composite materials, containing fillers, reinforcing components or pigments, for instance.

Furthermore, the evaluation of the effect of salt mist can be very important for finished or semi-finished articles which, while basically consisting of plastic materials, do contain some metallic elements, such as moulded-in inserts, thin laminated foils, surface coatings applied by electro-plating or other procedures, or, lastly, metal cores sheathed with plastics by extrusion, dipping in pastes or fluidized-bed powders.

0.2 Methods and equipment for obtaining reproducible aggressive environments of the above types are well known and have been described by International Standards relevant to other materials and IEC (International Electrotechnical Commission) standards relevant to electrical and electronic components. The same pieces of equipment and procedures described in these standards can also be employed for plastics, with appropriate care and adjustments.

0.3 The present International Standard is intended to provide general guidance only, on the choice of suitable equipment and procedures for obtaining the exposure conditions described above and for the preparation of test specimens. It also only gives general guidance on the properties to be evaluated. Specific details are given in the various ISO and IEC publications.

For the expression of results, the present International Standard follows, as far as possible, the same criteria adopted in the existing test methods for the exposure to chemicals (see ISO 175) and to natural weathering (see ISO 4607).

0.4 These tests are intended to yield data about the effects of the described exposures on the materials; however, a direct correlation between the experimental results and the behaviour in service is not to be inferred.

1 Scope and field of application

1.1 This International Standard specifies the exposure conditions of plastics to

- damp heat,
- water spray,
- salt mist,

and the methods for the evaluation of the change of some significant characteristics after given exposure stages.

1.2 This International Standard is, in general, suitable for all plastics in the form of standard test specimens, and finished articles or parts thereof.

1.3 This International Standard considers separately methods for the determination of

- change of mass,
- change of dimensions and appearance,
- change of physical properties.

2 References

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

ISO 293, *Plastics — Compression moulding test specimens of thermoplastic materials.*[2]

ISO 294, *Plastics — Injection moulding test specimens of thermoplastic materials.*

ISO 295, *Plastics — Compression moulding test specimens of thermosetting materials.*

ISO 2557, *Plastics — Amorphous thermoplastic moulding materials — Preparation of test specimens with a defined level of shrinkage.*

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

ISO 3205, *Preferred test temperatures.*

ISO 3768, *Metallic coatings — Neutral salt spray test (NSS test).*

ISO 4607, *Plastics — Methods of exposure to natural weathering.*

IEC Publication 68, *Basic environmental testing procedures* :

68-2-3, *Test Ca : Damp heat, steady state.*

68-2-4, *Test D : Accelerated damp heat.*

68-2-11, *Test Ka : Salt mist.*

68-2-30, *Test Db : Damp heat, cyclic* (12 + 12-hour cycle).[3]

68-2-38, *Test Z/AD : Composite temperature/humidity cyclic test.*

3 Principle

Determination of one or more properties before and after given periods of exposure in the specified environmental conditions, and observation of any change in appearance. If required, the determination of one or more properties may be carried out after exposure and a subsequent drying treatment or a reconditioning treatment carried out with the aim of obtaining the same state of equilibrium with atmospheric humidity as that of the initial specimens.

4 General test conditions

4.1 Environmental conditions and equipment

4.1.1 Damp heat

The preferred test conditions are those described by the IEC Publications referred to in 4.1.1.1 and 4.1.1.2. Different temperature and/or humidity conditions may be specified, however, in the relevant product specifications or by agreement between the interested parties.

4.1.1.1 Steady state test

A suitable test method is specified in IEC Publications 68-2-3.

The following conditions are specified :

Temperature : 40 ± 2 °C

Relative humidity : $\left(93^{+2}_{-3}\ \%\right)$

4.1.1.2 Cyclic test

If a cyclic test is required, the conditions specified in IEC Publication 68-2-4 can be adopted. This publication specifies 24 h cycles, with temperature variations from :

25 ± 10 °C

to 55 ± 2 °C

Relative humidity is maintained between 80 and 100 % (95 % at the higher temperature).

IEC Publication 68-2-30 specifies 12 + 12 h cycles with temperature change from :

25 ± 3 °C

to a) 40 ± 2 °C

b) 55 ± 2 °C

Relative humidity is maintained at (93 ± 3) % at the higher temperature and not less than 95 % during the rest of the cycle.

NOTE — For composite temperature/humidity cycles, with the addition of a number of excursions to sub-zero temperatures, some guidance can be found in IEC Publication 68-2-38.

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[1] At present at the stage of draft. (Revision of ISO/R 175-1961 and ISO/R 462-1965.)

[2] At present at the stage of draft. (Revision of ISO 293-1974.)

[3] Under revision.

4.1.2 Water spray

The main difference between this exposure condition and that for damp heat/steady state (see 4.1.1) is the constant presence of the liquid phase, in the form of small water droplets.

Suitable equipment for obtaining these conditions is substantially identical to that for the salt mist exposure (see 4.1.3) and is described in the relevant specifications.

Distilled or de-ionized water, having a pH between 6 and 7, shall be used instead of the salt-solution.

The temperature in the test enclosure shall be 40 ± 2 °C.

4.1.3 Salt mist

Suitable equipment and procedure for the exposure to salt mist (or salt spray) are described in ISO 3768.

The general operating conditions are as follows :

Temperature inside the cabinet . . . . 35 ± 2 °C

Sodium chloride solution :

- concentration . . . . 50 ± 5 g/l
- pH . . . . 6,5 to 7,2

The solution is prepared by dissolving sodium chloride of recognized analytical grade in distilled or deionized water.

The purity requirements are specified in ISO 3768.

Quantity of "mist" collected over a period of 24 h on a horizontal collecting area of 8 000 mm^2 . . . . 1 to 2 ml/h

These conditions comply also with IEC Publication 68-2-11.

NOTE — In this test, the 35 °C temperature value has been retained, even though this value is not included among those recommended in ISO 3205, because it is specified in the ISO 3768 test referred to and by the majority of existing national standards.

4.2 Duration of tests

The test duration shall be as specified in the relevant standard or as agreed upon by the interested parties, with reference to the intended application.

It is recommended that the time periods be chosen from the following standard scale :

24, 48, 96, 144, 168 h

and, for long periods :

1, 2, 4, 8, 16, 26, 52, 78 weeks.

See 5.2, 6.2 and 7.2.

4.4 Conditioning

Unless otherwise agreed by the interested parties, the test specimens shall be conditioned before testing for at least 86 h at 23 ± 2 °C and (50 ± 5) % r.h.

NOTE — For certain materials, which are known to approach rapidly, or on the contrary very slowly, the state of equilibrium of temperature and, above all, of humidity, shorter or longer conditioning periods may be specified in the particular specifications concerning them (see the annex).

4.5 Treatment after exposure

The exposed specimens shall be tested, either

a) directly after the exposure, or

b) after the exposure and subsequent drying or reconditioning

The first procedure shall be used when it is required to know the state of the material while still containing the amount of water it had absorbed at the end of the exposure. The second procedure shall be used when it is required to determine the changes in properties of the material as a result of the exposure only. In the case of reconditioning, the specimens shall be brought back, as far as possible, to the same state as the initial state before exposure with regard to equilibrium with atmospheric humidity (see 4.4).

4.5.1 Test after exposure only

After rinsing with distilled or deionized water, if necessary, and wiping dry, the exposed specimens shall be brought to 23 ± 2 °C in a closed container; usually 4 h are sufficient for this purpose.

4.5.2 Test after exposure and drying or reconditioning

After rinsing and wiping dry, the specimens shall be dried or reconditioned to equilibrium with the same atmospheric conditions as before the exposure (see 4.4), taking due account of the procedures described in A.3.1 or A.3.2 of the annex. Unless otherwise specified in the relevant product standard or agreed upon between the interested parties, the specimens shall be dried in an oven at 50 ± 2 °C for 24 h and cooled to 23 ± 2 °C in a desiccator.

5 Change of mass

5.1 General

5.1.1 In this type of test, the changes of mass are, at least partially, due to water absorption, and they are, therefore, particularly influenced by the conditioning and recovery conditions of the test specimens.

It is important, therefore, that the precise conditions of test be specified in the relevant product specifications.

Normally the specimens are weighed immediately after exposure, rinsing and wiping dry, or are tested as described in 4.5.1.

If the determination of mass after drying or reconditioning is required, the specimens shall be dried or reconditioned as specified in 4.5.2.

NOTE — This test is not applicable to cellular materials.

5.1.2 The changes of mass are generally proportional to the surface area of the test specimens, but are influenced by their thickness.

IMPORTANT — It is emphasized that the comparison of different plastics by means of this test is valid only if the test specimens used are of the same form, the same dimensions and in as nearly as possible the same state (of surface, internal stresses, etc.).

5.2 Test specimens

The test specimens may be obtained directly by moulding or by machining. In the latter case, the cut surfaces shall be smooth and shall not show any trace of charring that may be due to the method of preparation.

5.2.1 Moulding and extrusion compounds

The test specimens shall be in the form of a square of 50 ± 1 mm side and 3 ± 0,2 mm thick. Rectangular specimens having the same surface area (for example 100 mm × 25 mm, i.e. 2 500 mm^2) may be used.

The specimens may be cut from a sheet of the same thickness, prepared by compression or injection moulding or by extrusion under the conditions given in the relevant specification of the material (or under the conditions prescribed by the supplier of the material).

Moulding materials may be, alternatively, directly moulded in a mould of the prescribed dimensions.

The state of internal stress, if necessary, shall be determined according to ISO 2557.

NOTE — The general principles for preparing moulded and machined test specimens are the subject of the following International Standards : ISO 293, ISO 294, ISO 295, ISO 2557, ISO 2818.

5.2.2 Sheet

The specimens shall be 50 ± 1 mm square or rectangular, having the same surface area, and shall be cut from the sheet under test.

The thickness of the test specimen shall be the same as that of the sheet under test if the nominal thickness of the sheet is equal to or less than 25 mm.

If the nominal thickness is greater than 25 mm and in the absence of special provisions in the relevant specification, the thickness of the test specimen shall be reduced to 25 mm by machining on one surface only.

The machined surface shall not be directly exposed to water or salt spray.

A complete description of the machining, if any, shall be included in the test report.

5.2.3 Semi-finished and finished products (other than sheets)

The specimens shall be as similar in shape and size as possible to the specimens described in 5.2.1, and be prepared according to the product specification or as agreed upon between the interested parties.

A complete description of the machining, if any, shall be included in the test report.

5.2.4 Number of test specimens

At least three specimens shall be tested.

5.3 Conditioning

See 4.4.

5.4 Procedure

5.4.1 Determine the mass (m_1) of each specimen to the nearest 0,001 g.

5.4.2 Expose the specimens to the test environment chosen from those listed in clause 4.

5.4.3 Rinse, if necessary (for example in the case of exposure to salt mist), and wipe the specimens dry.

5.4.4 Immediatly determine the mass (m_2) of each specimen to the nearest 0,001 g.

5.4.5 Dry or recondition the specimens, if required, according to 4.5.2 and determine the mass (m_3) of each specimen to the nearest 0,001 g.

5.5 Expression of results

5.5.1 The change of mass per unit area, in grams per square metre, is given by the formula

$$\frac{m_2 - m_1}{S} \quad \text{or} \quad \frac{m_3 - m_1}{S} \quad \text{respectively}$$

where

m_1, m_2 and m_3 are as defined in 5.4.1, 5.4.4 and 5.4.5;

S is the initial total surface area (including the edges of the specimens) in square metres.

5.5.2 The change of mass, as a percentage, is given by the formula

$$\frac{m_2 - m_1}{m_1} \times 100 \quad \text{or} \quad \frac{m_3 - m_1}{m_1} \times 100 \quad \text{respectively.}$$

The change is positive for increase of mass and negative for reduction of mass.

5.5.3 Calculate the mean value of the test results for all the specimens from a given sample.

6 Change of dimensions and appearance

6.1 General

Change of dimensions may result from either volume changes due to water absorption or leaching of some components, or to relaxation of internal moulded-in stresses, or both.

It is important, therefore, that the precise conditions of test be specified in the relevant product specifications.

For anisotropic materials, such as calendered or extruded sheets or extruded rods, the changes in linear dimensions in the machine direction (lengthwise) and in the transverse direction (crosswise) may be different; it is therefore necessary to determine the changes in both directions.

NOTE — In order to differentiate between the effect of the relaxation of moulded-in stresses and that of the action of water, one may, by agreement between the interested parties, also carry out the test on a set of annealed specimens.

6.2 Test specimens

Prepare the test specimens as described in 5.2. For anisotropic materials, the sides shall be parallel to the machine and transverse directions, respectively (see 6.1).

NOTE — The dimensional measurements may normally be carried out on the same specimens used for the determination of the changes in mass, immediately after weighing them.

6.3 Conditioning

See 4.4.

6.4 Procedure

6.4.1 Measure the thickness of each specimen at four marked points to an accuracy of 0,01 mm using a dial micrometer, and calculate the mean value ($\bar{d}_1$).

Measure individually the four sides of the square or the rectangle to an accuracy of 0,1 mm and calculate the mean values of the dimensions in the two perpendicular directions respectively (length $\bar{l}_1$ and width $\bar{b}_1$). For different specimens, for example from semi-finished or finished products, measure the most meaningful dimensions.

6.4.2 Expose the specimens to the test environment chosen from those listed in clause 4.

6.4.3 Rinse, if necessary (for example in the case of exposure to salt mist), and wipe the specimens dry.

6.4.4 Remeasure the specimens in the same manner as before exposure, i.e. determine the dimensions after exposure ($\bar{l}_2$, $\bar{b}_2$ and $\bar{d}_2$).

NOTE — If the specimens have undergone severe warpage, the linear measurements should be carried out with a tape measure.

6.4.5 Note any visible change in appearance.

6.4.6 Dry or recondition the specimens, if required, as described in 4.5.2 and determine the dimensions after drying or reconditioning ($\bar{l}_3$, $\bar{b}_3$, $\bar{d}_3$).

6.5 Expression of results

6.5.1 The results may be expressed in either of two ways :

a) as the percentage change in dimension, based on the initial dimension using the formulae

$$\frac{\bar{l}_2 - \bar{l}_1}{\bar{l}_1} \times 100, \quad \frac{\bar{b}_2 - \bar{b}_1}{\bar{b}_1} \times 100, \quad \frac{\bar{d}_2 - \bar{d}_1}{\bar{d}_1} \times 100$$

or (after exposure and drying or reconditioning)

$$\frac{\bar{l}_3 - \bar{l}_1}{\bar{l}_1} \times 100, \quad \frac{\bar{b}_3 - \bar{b}_1}{\bar{b}_1} \times 100, \quad \frac{\bar{d}_3 - \bar{d}_1}{\bar{d}_1} \times 100$$

The change is positive for increase and negative for decrease in the dimension.

b) as the percentage of the final dimension relative to the initial dimension, using the formulae

$$\frac{\bar{l}_2}{\bar{l}_1} \times 100, \quad \frac{\bar{b}_2}{\bar{b}_1} \times 100, \quad \frac{\bar{d}_2}{\bar{d}_1} \times 100$$

or

$$\frac{\bar{l}_3}{\bar{l}_1} \times 100, \quad \frac{\bar{b}_3}{\bar{b}_1} \times 100, \quad \frac{\bar{d}_3}{\bar{d}_1} \times 100 \text{ respectively.}$$

In this case, 100 % means no change, values lower than 100 % a reduction and values higher than 100 % an increase in the dimensions.

Report any apparent deformation such as warpage, curling, delamination, or visible signs of surface degradation such as :

- change in colour and/or gloss, presence of crazing, cracks;
- blisters, etc.;

- exudation of plasticizers, tackiness;
- blooming of solid ingredients;
- corrosion of metal components, if any;

and assign, if possible, a qualification such as slight, moderate, severe, etc.

7 Change of physical properties

7.1 General

Any physical property may be determined; the most significant are usually the mechanical, optical and electrical properties.

It is important that the precise conditions of test be specified in the relevant product specifications.

7.2 Test specimens

The size, shape and number of test specimens shall conform to those specified in the International Standard for the determination of the relevant property.

If the test is a destructive one, the number of test specimens has to be doubled.

NOTE — Exposed specimens may be in the form of a sheet from which the specimens for the particular test are subsequently cut. Since the results obtained on specimens prepared before the exposure may be different from those obtained on specimens cut after the exposure, owing to the action of the exposure on the cut edges, the procedure of preparation of the specimens shall be precisely stated in the test report.

7.3 Conditioning

See 4.4.

7.4 Procedure

7.4.1 Determine the value (P_1) of each of the intended properties on a set of test specimens.

7.4.2 Expose the specimens to the test environment chosen from those listed in clause 4.

7.4.3 Rinse and wipe dry

7.4.4 Determine the value (P_2) of each of the properties after exposure.

7.4.5 If required, expose a third set of specimens, rinse, wipe and dry or recondition according to 4.5.2; determine the value (P_3) of each of the properties after exposure and drying or reconditioning to the same moisture equilibrium state as that of the specimens used to determine P_1.

7.5 Expression of results

The results may be expressed in either of two ways :

a) as the percentage change of property, using the formula

$$\frac{P_2 - P_1}{P_1} \times 100 \quad \text{or} \quad \frac{P_3 - P_1}{P_1} \times 100 \text{ respectively}$$

b) as the percentage of the final property, relative to the initial one, using the formula

$$\frac{P_2}{P_1} \times 100 \quad \text{or} \quad \frac{P_3}{P_1} \times 100 \text{ respectively.}$$

8 Test report

The test report shall include the following particulars :

a) reference to this International Standard;

b) complete identification of the material or product;

c) type of exposure;

d) treatment of the specimens before and after the exposure;

e) properties examined;

f) details of the preparation of the specimens, with particular mention of any machining;

g) type of specimens;

h) values of the properties before and after the test;

j) corresponding changes calculated according to 5.5, 6.5 and 7.5 as appropriate including the signs;

k) if available, curves of properties as a function of time of exposure;

m) any observation of changes in appearance of the exposed specimens.

Annex

Absorption of moisture by a test specimen of a plastic material in equilibrium with its conditioning atmosphere

A.1 The amount and rate of absorption of moisture by a test specimen conditioned in a humid atmosphere varies significantly according to the nature of the plastics tested.

A.2 The normal conditions for conditioning established in this International Standard (see 4.4) are very generally satisfactory, except for the following :

A.2.1 Materials known to reach equilibrium with their conditioning atmosphere only after a very long period of time (for example, certain polyamides).

A.2.2 New materials or those of unknown structure, for which no *a priori* forecast can be made either of their capacity for absorbing moisture or of the time required for reaching equilibrium.

A.3 In the last two cases one of two procedures can be followed :

A.3.1 Drying the material at elevated temperature. This procedure has the disadvantage that certain properties, mechanical in particular, in the dry state differ from those obtained after conditioning at 23 ± 2 °C and (50 ± 5) % r.h.

A.3.2 Conditioning the test specimen at 23 ± 2 °C and (50 ± 5) % r.h. until equilibrium is achieved. In this case, a suitable criterion could be one of the following :

a) Mass constant within 0,1 % for two determinations separated by an interval of d^2 weeks (d being the thickness of the test specimen in millimetres).

b) For certain polymers, it is sufficient to plot the graph of mass against time, with intervals much less than d^2 weeks, equilibrium being considered achieved for practical purposes when the gradient of the curve, expressed as a percentage, is equal to 0,1 %.