International Standard 4600

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Plastics — Determination of environmental stress cracking (ESC) — Ball or pin impression method

Plastiques — Détermination de la fissuration sous contrainte dans un environnement donné (ESC) — Méthode par enfoncement de billes ou de goupilles

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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 4600 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in December 1978.

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

Austria	India	Poland
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The member bodies of the following countries expressed disapproval of the document on technical grounds :

Czechoslovakia
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Plastics — Determination of environmental stress cracking (ESC) — Ball or pin impression method

0 Introduction

Environmental stress cracking is exhibited by many materials, including plastics. When a plastic material is stressed or strained in air below its yield point, stress cracking can occur after a period of time which may be very long. These stresses may be internal or external, or a combination of both. Exposure to a chemical medium simultaneously with the same stress or strain may result in a dramatic shortening of the time to failure. This phenomenon is referred to as environmental stress cracking (ESC).

The cracks produced may penetrate completely through the thickness of the material, separating it into two or more pieces, or they may be arrested on reaching regions of lower stress or different material morphology.

The determination of ESC is complex because it is influenced by many parameters, including :

- test specimen dimensions;
- test specimen state (orientation, structure, internal stresses);
- stress and strain;
- temperature of test;
- duration of test;
- chemical medium;
- test method and failure criterion.

By keeping all but one parameter constant, the influence of the variable parameter on ESC can be assessed. The main objective of ESC measurements is to determine the effect of chemical media (environment) on plastics (test specimens and articles). The measurements may also be used to evaluate the influence of the moulding conditions upon the quality of an article when the failure mode corresponds to that obtained in actual service. It may not be possible, however, to establish any direct correlation between the results of short-duration ESC measurements on test specimens and the actual service behaviour of articles, because the behaviour of the latter may be more complex than that of test specimens.

1 Scope and field of application

This International Standard specifies methods for the determination of environmental stress cracking (ESC) of plastics by means of a constant strain test.

The test is applicable to finished products and to test specimens, prepared by moulding, and can be used for the assessment of both ESC of a plastic product or material exposed to different environments, and for the determination of ESC of different plastic materials exposed to a specific environment.

NOTE — A method for the determination of environmental stress cracking by means of a constant stress test is specified in ISO 6252.

2 References

ISO 178, *Plastics — Determination of flexural properties of rigid plastics.*

ISO 291, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 468, *Surface roughness — Parameters, their values and general rules for specifying surfaces.*[1]

ISO 527, *Plastics — Determination of tensile properties.*[2]

ISO 2557/1, *Plastics — Amorphous thermoplastic moulding materials — Preparation of test specimens with a defined level of shrinkage — Part 1 : Test specimens in the form of parallelepipedic bars (Injection moulding and compression moulding).*

ISO 6252, *Plastics — Determination of environmental stress cracking — Constant tensile stress method.*[3]

1) At present at the stage of draft. (Revision of ISO/R 468.)

2) At present at the stage of draft. (Revision of ISO/R 527.)

3) At present at the stage of draft.

3 Definitions

3.1 oversize (d_d) : The difference between the diameter of an impressed steel ball or pin (d_b) and the diameter of the hole (d_n) drilled into the test specimen.

$$d_d = d_b - d_n$$

3.2 deformation step : A determination made at a defined oversize.

3.3 deformation step zero : A determination made using test specimens that are drilled and reamed only, i.e. without impressing a ball or pin.

NOTE — Normally, the results of the flexural or tensile test on test specimens at deformation step zero are equivalent whether determined in air or in a chemical medium. If the value determined in the medium is higher, additional embrittlement should be suspected and, if it is lower, a softening of the material has occurred.

3.4 deformation series : A number of successive deformation steps beginning with deformation step zero.

NOTE — Normally, a deformation series consists of seven deformation steps of increasing severity.

3.5 failure limit : The oversize in a deformation series that produces failure.

Failure is defined :

a) in method A (for test specimens taken from products), by visible cracks, observed with the unaided eye;

b) in method B (for moulded or machined test specimens), by the following criteria (see figure 1) :

1) by a 5 % reduction in the maximum tensile force measured at deformation step zero (criterion B_1),

2) by a 5 % reduction in the maximum flexural force measured at deformation step zero (criterion B_2),

3) by a 20 % reduction in the tensile elongation at rupture measured at deformation step zero (criterion B_3).

If there is no rupture immediately after application of the maximum tensile force, the tensile elongation at 50 % of the preceding maximum tensile force (see figure 1) may be measured. Failure is then defined by a 20 % reduction in the value at deformation step zero (criterion B_4).

NOTES

1 It is sufficient to measure the elongation between the grips.

2 If the value for the tensile stress or flexural stress is required, refer the force to the smallest cross sectional area of the specimen at the location of the hole.

3.6 *relative stress cracking factor* : The ratio of the failure limits in the test environment to that in a reference environment, for example air, measured at the same test temperature after the same test duration.

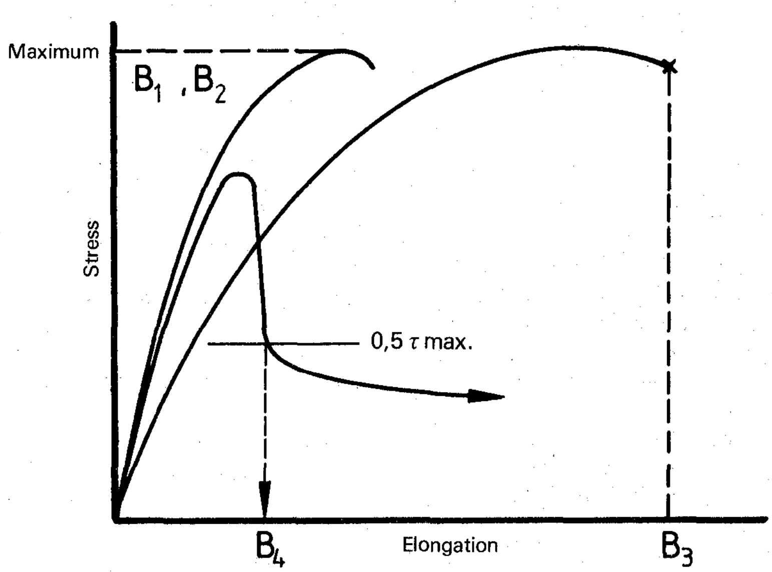

Figure 1 — Failure criterion for method B

4 Principle

A constant strain, produced by impressed balls or pins in a test specimen in a test environment, often generates microcracks which may, in time, develop to visible cracks. To shorten the time for the test, the failure may be accelerated by subsequent mechanical testing. If products cannot be assessed by mechanical tests, visual examination for cracks around the balls or pins may be undertaken.

A hole of specified diameter is drilled in the specimen, an oversize steel ball or pin is inserted into the hole and the test specimen is brought into contact with a chemical medium. This procedure is repeated using balls or pins of progressively greater diameter. After a specified time, the effect of the interaction may be determined by visual examination (method A) or by the determination of the tensile or flexural properties (method B). A parallel series of tests may be performed in which the test specimens are exposed to air and the comparative behaviour determined.

NOTE — Pins are preferred for a single series of test specimens or articles with thicknesses greater than 1 mm. The deformation of the test specimen is equivalent along the total length of the hole.

Balls are preferred for a number of series of test specimens or articles and for routine testing if the test specimens have a thickness of 2 to 4 mm. The deformation of the test specimen is greatest at the ball equator.

Due to the differences in deformation, the results of ball tests and pin tests may be different.

5 Apparatus

5.1 Drilling machine, operating at a suitable frequency of rotation, for example at 1 000 min^{-1}.

5.2 Drills, of diameter 2,8 mm.

5.3 Reamer, suitable for finishing a hole of diameter 3,00 ± 0,05 mm.

NOTE — A 3^{H7} reamer (3,004 to 3,008 mm) is suitable.

5.4 Plug gauges, or other suitable devices, for measuring the diameter of the reamed holes to within 0,005 mm.

5.5 Micrometer, for determining the diameter of the balls or pins with an accuracy of 0,001 mm.

5.6 Steel balls or pins.

NOTE — If steel is attacked in the test environment, other suitable hard materials, for example glass, may be used for the balls or pins.

5.6.1 Polished balls or pins, having tolerances of ± 0,001 mm on diameters up to 4 mm and ± 0,01 mm on diameters greater than 4 mm.

5.6.2 Pins, free of roughness or sharp edges, having a parallel part 10 to 50 mm long and a taper (1:5) at one end to reduce the entry diameter to 2,5 mm (see 8.3.2). The surface roughness of the pins shall be equal, preferably with $R_a < 0{,}02$ µm (see ISO 468).

NOTE — A longer parallel part of the pin will allow mounting several test specimens on the same pin.

The use of the range of diameters given in the table is recommended.

Table — Recommended range of diameters

Diameter mm	Increment mm
2,98 to 3,2	0,01
3,2 to 3,5	0,05
3,5 to 4,0	0,10
4,0 to 6,0	0,50

5.7 Jig, for drilling the holes.

5.8 Apparatus for pressing the balls or pins into the hole.

The spindle of the drilling machine or the tensile machine itself may be used.

5.9 Vessels, for immersion of the specimens.

5.10 Clock.

5.11 Flexural or tensile testing machine (see ISO 178 and ISO 527), for the determination of flexural or tensile properties.

6 Test specimens

6.1 Form

In general, use the form of test specimen and method of preparation specified in the International Standard appropriate to the material or product concerned.

If the relevant International Standard contains no such specifications, the following form of test specimens shall be used.

6.1.1 Method A

Use the product or parts of it as the test specimen.

6.1.2 Method B

Use moulded or machined test specimens, according to the relevant International Standard.

Test specimens shall not be machined on the faces where the holes will be drilled. If test specimen dimensions are not specified for flexural testing, use a bar of dimensions 80 mm × 10 mm × 4 mm, as specified in ISO 178, and for tensile testing use the appropriate test specimen specified in ISO 527.

6.2 State

For tests which are intended to be comparable, the test specimens shall be in the same state. Equal state can be assumed when they have the same level of shrinkage. If finished articles are tested, the holes and pins shall be applied in the same area or an area agreed upon by the interested parties, especially if critical regions, such as weld lines, are examined.

The level of shrinkage of the test specimens, whether compression moulded, injection moulded or machined from sheet, shall be determined by the method specified in ISO 2557/1 on five test specimens before they are drilled and reamed.

NOTES

1 In the case of evaluation of moulding materials of crystalline polymers, such as polyethylene and polypropylene, the amount of crystallinity shall be standardized by annealing, as specified in the International Standard appropriate to the material concerned or as agreed between the interested parties.

2 The numerical value of the failure limit depends upon the method of determination and the distance between the edge of the hole and the side of the test specimen. The value decreases as this distance becomes smaller.

6.3 Number

The number of test specimens required depends upon the duration of the test, i.e. short (see 8.4.1) or long (see 8.4.2), and the method used. Three deformation steps shall lie on either side of the expected approximate failure limit.

6.3.1 Short duration test (up to 20 h in the test environment) (see 8.4.1).

6.3.1.1 Method A

Three complete deformation series shall be used for testing in the test environment. The required number of test specimens depends, therefore, upon the number of holes that can be drilled in the region of homogeneous state in one product.

6.3.1.2 Method B

Five test specimens shall be used for each deformation step.

6.3.2 Long duration test

The number of test specimens depends upon the test conditions (see 8.4.2).

7 Conditioning and test conditions

7.1 Conditioning

Unless otherwise agreed between the interested parties (for example for polyamides or polyolefins), the test specimens shall be conditioned before preparation and testing for at least 24 h at 23 ± 2 °C and 50 ± 5 % relative humidity (see ISO 291).

7.2 Test conditions

7.2.1 Unless otherwise agreed between the interested parties (for example for polyethylene), the temperature during insertion of the ball or pin shall be 23 ± 2 °C.

7.2.2 Unless otherwise specified, the temperature during immersion shall be 23 ± 2 °C and the test specimens shall be stored in air at 23 ± 2 °C and 50 ± 5 % relative humidity.

7.2.3 Tensile or flexural testing shall be performed at 23 ± 2 °C and 50 ± 5 % relative humidity, or 23 ± 2 °C if the relative humidity is not critical.

7.3 Chemical medium

The chemical medium used for the test shall be that specified in the relevant International Standard. If there is no such specification, use either the chemical medium with which the material will be in contact in the expected application or a product agreed upon between the interested parties.

8 Procedure

Test specimens, balls and pins shall be clean and free of grease, fat, perspiration or other substances that could affect the test result.

NOTE — Exposure of test specimens to intensive artificial light or sunlight could also affect the result.

8.1 Drilling the test specimens

8.1.1 Method A

Drill holes of diameter 2,8 mm in each conditioned test specimen and ream them to 3 mm. The holes shall be perpendicular to the surface of the test specimens, at least 15 mm apart and 15 mm from the edges of the test specimen. Use a coolant during this operation (for example compressed air, water or other media known to have no effect on the material under test).

NOTE — Specimen preparation is difficult and critical and care shall be exercised (see 6.2).

8.1.2 Method B

Drill a hole of diameter 2,8 mm in each conditioned test specimen and ream them to 3 mm. Drill the hole perpendicularly to the surface of the test specimen, so that it passes through the intersection of the axes of symmetry to within 0,2 mm longitudinally and 0,02 mm transversely. Drill the set of test specimens for each deformation series consecutively with the minimum time delay, using a coolant (see 8.1.1).

NOTE — To centre the hole when drilling, the type of fixture shown in figure 2 is recommended.

8.2 Measurement of hole diameter

(Methods A and B)

Store the drilled and reamed test specimens for 24 ± 2 h in the atmosphere specified in 7.2.2.

Measure the diameter of five holes selected at random to within 0,005 mm. Check that the range of values is less than 0,01 mm and then calculate the arithmetic mean. This mean value shall be taken as the hole diameter for the series.

8.3 Insertion of balls or pins (Methods A and B)

8.3.1 Balls

Insert one ball into each hole using a ball impression apparatus or other suitable means, for example the spindle feed of the drilling machine (see 5.8). Ensure that the position of the balls is symmetrical to the thickness of the test specimens.

8.3.2 Pins

Wet the pin with the chemical medium to be tested, insert the tapered end into the hole in the test specimen and press it in until the parallel part of the pin is in contact with the wall of the hole along its entire length.

8.4 Immersion in the chemical medium

(Methods A and B)

Store the prepared test specimens for 60 ± 5 min in the atmosphere specified in 7.2.2 prior to immersion in the test medium.

8.4.1 Short-duration test

8.4.1.1 Liquid medium

Immerse the test specimens in the liquid medium contained in a vessel (5.9) for 20 h at the specified temperature (see 7.2.2). Remove the specimens, wipe off the medium using blotting-paper and allow them to stand for 3 h in the atmosphere specified in 7.2.2 before determining stress cracking.

8.4.1.2 Viscous medium

If the medium is viscous (for example paste or grease), cover the area of the hole on both surfaces of the test specimen with the medium. Store at the specified temperature for 20 h, wipe off the medium using blotting-paper and allow the specimens to stand for 3 h in the atmosphere specified in 7.2.2 before determining stress cracking.

8.4.2 Long-duration test

Proceed as described in 8.4.1 and subject the test specimens to the medium for a specified or agreed period. If no period of contact is specified or agreed, it has been found convenient to carry out the test using periods of 1, 2, 4, 8, 16, etc. days to determine the influence of time on the failure limit.

NOTE — Before determining the failure limit (see 3.5), wipe off the medium using blotting-paper and allow the test specimens to stand for 3 h in the atmosphere specified in 7.2.2. The period of 1 h storage in the atmosphere specified in 7.2.2 prior to contact with the medium and the period of 3 h storage after contact with the medium are included in the time of test.

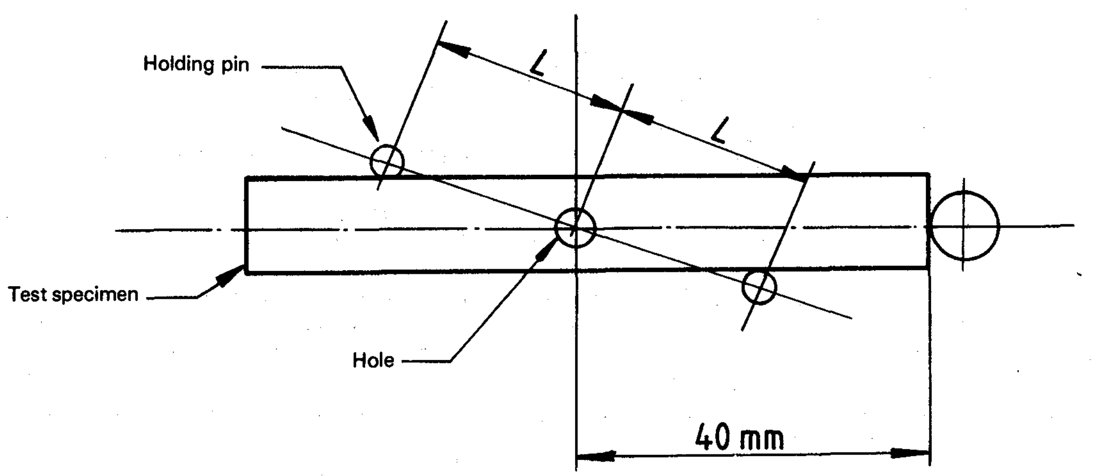

Figure 2 — Fixture for drilling holes in test specimens

8.5 Exposure in air (Methods A and B)

If a simultaneous test in air is to be performed, store the test specimens in the atmosphere specified in 7.2.2 for 24 h for the short-duration test or for the test period(s) used for the long-duration test.

8.6 Determination of stress cracking

8.6.1 Method A

Determine by visual observation, or by means of a lens of magnification X 5, the failure limit for products exposed to air and immersed in the chemical medium.

8.6.2 Method B

Determine the failure limit by the selected method (see 3.5).

In case of dispute, the ball or pin shall be removed before the tensile or flexural tests. The pin shall always be removed before the flexural test.

9 Expression of results

9.1 Method A

Record the oversize at which any cracks are visible and the time for the appearance of cracking either in the first of a group of specimens or at the 50 % value.

NOTE — For routine testing, it may simply be recorded whether or not a crack is visible after exposure to the chosen conditions, i.e. oversize, chemical medium, time.

9.2 Method B — Graphical evaluation

Plot the arithmetical mean values of the tensile or flexural values determined at each deformation step on graph paper against the oversize of the corresponding ball or pin as abscissa. Draw a smooth curve through the points. (See figure 3 for examples.)

Calculate the failure limit as described in 3.5.

If the tensile force at deformation step zero is 2 000 N, the 95 % value is 2 000 N × 95/100 = 1 900 N. Draw a line through this value, parallel to the abscissa. At the intersection of this line with the curve draw a line perpendicular to the abscissa. The oversize value at the intersection is the failure limit, in millimetres. Record the value to three significant figures.

Proceed similarly when the tensile elongation or flexural force is the failure criterion.

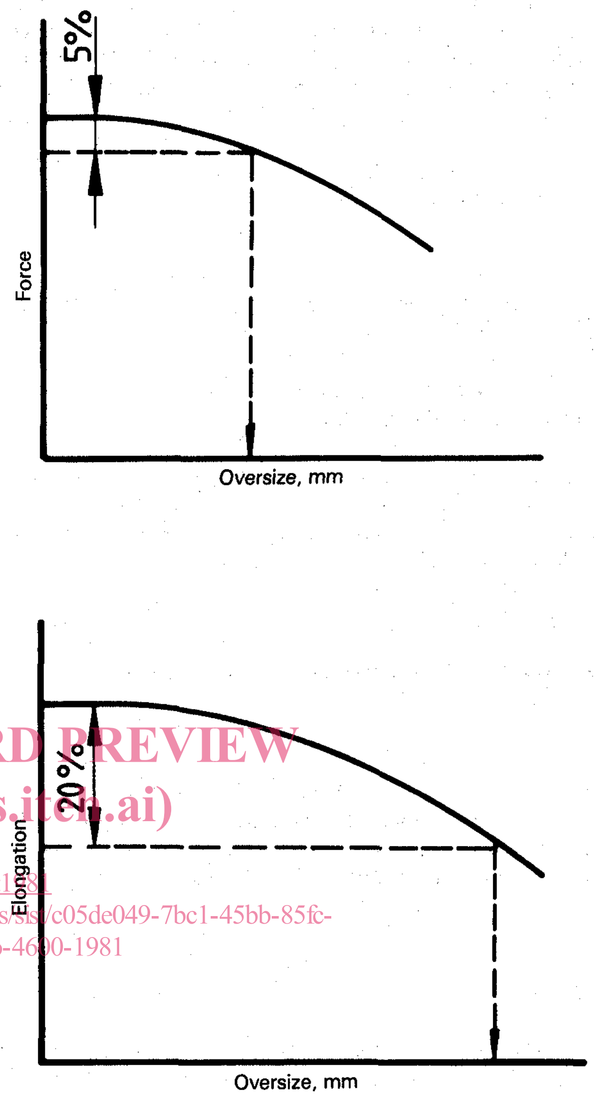

Figure 3 — Examples of graphical evaluations of results

10 Test report

The test report shall include the following information :

a) a reference to this International Standard, indicating the method used (A or B);

b) complete identification of the material tested (type, source, manufacturer, code number, form or principal dimensions, previous history);

c) type and dimensions of test specimens;

d) state of specimens;

e) procedure for conditioning and/or annealing (if applicable);

f) method of cleaning specimens (if applicable);

g) deformation series dimensions [diameter of hole (8.2), diameter of balls or pins (5.6)];

h) chemical medium;

j) duration of test;

k) other observations (change in colour, appearance, mass);

m) any deviation from the procedure specified in this International Standard.

In addition, the following shall be included according to the method used :

a) for method A only

1) the oversize at which the first crack or cracking at the 50 % level is observed;

2) for routine testing :

i) the test conditions, i.e. oversize, medium and time,

ii) whether cracks are visible or not;

b) for method B only

1) the mechanical test used;

2) the failure limit, for example : ESC, method B_2 (23 °C, 2 days) = 3,35 mm;

3) a graphical presentation of the results as required in 9.2;

4) the relative stress cracking factor, if requested, together with the reference environment used.