SLOVENSKI STANDARD
SIST ISO 4599:1996

01-junij-1996

Polimerni materiali - Ugotavljanje napetostne korozije (ESC) - Metoda z upognjenim trakom

Plastics -- Determination of resistance to environmental stress cracking (ESC) -- Bent strip method

Plastiques -- Détermination de la fissuration sous contrainte dans un environnement donné (ESC) -- Méthode de l'éprouvette courbée

Ta slovenski standard je istoveten z: **ISO 4599:1986**

ICS:

83.080.01	Polimerni materiali na splošno	Plastics in general

SIST ISO 4599:1996 **en**

International Standard 4599

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

Plastics — Determination of resistance to environmental stress cracking (ESC) — Bent strip method

Plastiques — Détermination de la fissuration sous contrainte dans un environnement donné (ESC) — Méthode de l'éprouvette courbée

First edition — 1986-04-15

UDC 678.5/.8 : 620.191.33 **Ref. No. ISO 4599-1986 (E)**

Descriptors : plastics, tests, environmental tests, cracking tests, determination, crazing resistance, stress factor.

Price based on 6 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4599 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Plastics — Determination of resistance to environmental stress cracking (ESC) — Bent strip method

0 Introduction

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 may occur after a period of time which may be very long. The 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. Cracking accelerated in this way is referred to as environmental stress cracking (ESC).

Other modes of failure than stress cracking may result in the shortening of the time to failure in this test, but such modes are included in the term "environmental stress cracking" as known in the trade.

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 resistance is a complex procedure because it is influenced by many parameters, including:

— test specimen dimensions;

— test specimen state (orientation, structure, internal stress);

— stress and strain;

— temperature of test;

— duration of test;

— test environment;

— failure criterion.

By keeping all but one parameter constant, the influence of the variable parameter on ESC resistance can be assessed. The main objective of ESC measurements is to determine the effect of chemical media 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-term ESC measurements on test specimens and actual long-term service behaviour.

1 Scope and field of application

This International Standard specifies a method for the determination of environmental stress cracking (ESC) resistance of plastics by means of a constant prestrain test. ESC will be indicated by the change of a suitably chosen indicative property of specimens that have been prestrained for a definite time in the environment. The method of test is suitable for determining the resistance of sheets and of flat test specimens to environmental stress cracking, especially the sensitivity of localized surface regions of specimens to ESC.

For the determination of the ESC sensitivity of finished articles or the bulk of a material subjected to a constant strain, see ISO 4600.

The bent strip method is suitable for the determination of ESC caused by gases and liquids as well as by solids containing migrating substances (e.g. polymeric adhesives and materials containing plasticizers) in contact with a specific polymer.

Preferably, this method is used to determine the ESC resistance of rigid plastics with only moderate stress relaxation in time.

For a constant strain test, refer to ISO 4600. For a constant stress test, refer to ISO 6252.

2 References

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

ISO 179, *Plastics — Determination of Charpy impact resistance of rigid plastics (Charpy impact flexural test).*

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

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

ISO 527, *Plastics — Determination of tensile properties.*

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 4600, *Plastics — Determination of environmental stress cracking (ESC) — Ball or pin impression method.*

ISO 6252, *Plastics — Determination of environmental stress cracking (ESC) — Constant tensile stress method.*

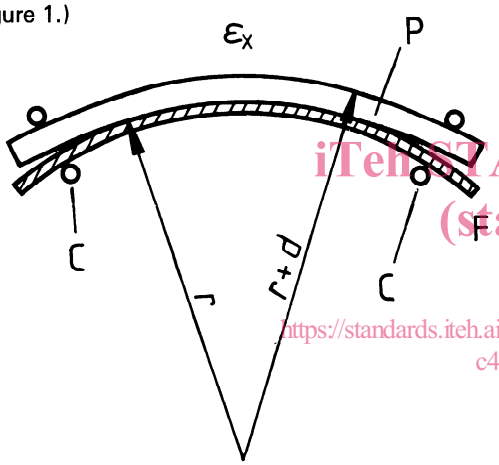
3 Definitions

For the purpose of this International Standard, the following definitions apply.

3.1 flexural strain, ε_x: the nominal value of the strain in the extended surface of a flat test specimen of thickness d, bent over the segment of a circle with radius r, calculated from the equation

$$\varepsilon_x = \frac{d}{2r + d} \times 100\ (\%)$$

(See figure 1.)



P : test specimen

d : thickness of the test specimen

F : former

r : radius of former

c : clamps

ε_x : nominal strain in the extended surface

Figure 1 — Test specimen P with defined strain in the outer surface

3.2 prestrain value: One of a series of strain levels applied to successive test specimens during exposure.

3.3 prestrain series: A number of prestrain values, including zero.

NOTES

1 Normally, the results of the mechanical test on test specimens with zero prestrain are equivalent whether determined in air or a medium. If the property measured after exposure to the medium at zero strain is different from that after exposure in air at zero strain, embrittlement or softening by the medium should be suspected.

2 It is recommended that the test specimen for zero prestrain be clamped on to a flat former to prevent warping due to the effect of the medium.

3.4 failure prestrain, ε_F: The lowest prestrain in the prestrain series at which failure is observed.

3.5 indicative property, I: The property observed to determine failure using a criterion given in the table.

3.6 relative strain factor, M: The ratio of the value of failure prestrain determined in the test medium to that determined in the reference medium (usually air) for the same time of exposure.

3.7 prestrain period, t: The time during which the test specimens are in contact with the test environment while prestrained.

3.8 prestrain temperature, T: The temperature at which the test specimens are in contact with the test environment while subjected to a strain.

3.9 test environment: the chemical liquid, gas, paste, solid or other medium selected for contact with the test specimens during the prestrain period.

4 Principle

A test specimen suitable for the determination of the indicative property is clamped with one of its faces over a former of constant radius and brought into contact with the test environment. Due to the influence of the environment in the presence of strain, crazes may be generated which with time sometimes develop into visible cracks.

By using a series of formers with decreasing radii one obtains a series of test specimens with increasing strains in the outer surfaces.

After the agreed duration of contact with the test environment, the test specimens are visually observed, unclamped and assessed by mechanical or other test. The failure strain that corresponds to the failure criterion is obtained directly from the tabulated values or from a graph.

The criteria usually determined are:

— failure strain;

— relative strain factor.

NOTES

1 The maximum prestrain shall be less than the elongation at yield (ISO 527).

2 The failure strains for different indicative properties may be different.

5 Apparatus

5.1 Formers, made from chemical-resistant material either by machining or by bending metal sheet (for example, stainless steel sheet). For test specimens 2 to 4 mm thick, radii of curvature of 30 to 500 mm are adequate. The arc of the former shall have roughly the same length as the test specimen.

To increase the wetting of the test specimen by the test substance, the former may be perforated.

NOTE — The radius r of the segment of a circular arc of height h and chord length s is given by the equation

$$r = \frac{s^2}{8h} + \frac{h}{2}$$

5.2 Clamps, made from chemical-resistant material. Clamp the test specimen lightly; the contact between the test specimen and the former need not extend to the clamps, but shall extend over that length of test specimen which will be most highly stressed in the subsequent mechanical test. In no case shall the contact region (measured in the direction of the length of the test specimen) be less than 10 times the specimen thickness.

5.3 Vessels, Carefully cleaned glass containers with well fitting lids suitable for holding the mounted test specimens and the test environment. Other vessels are suitable when there is no interaction between their material and the test specimens or test environment. When the contact between the test specimens and the test media takes place under other conditions such as in a vapour or under a water spray, this fact shall be reported.

Table — Suggested indicative properties and failure criteria

(See note 1)

Designation	Indicative property	International Standard	Criterion of failure
A 1	State of surface (assessed by visual examination)	—	Cracks or crazes around the extended edges
A 2	State of surface (assessed by visual examination)	—	Cracks or crazes in the extended surface
A 3	State of surface (assessed by visual examination)	—	Any other observations; change in colour and appearance e.g.
B 1	Tensile stress at rupture	ISO 527	80 % of the value obtained on unprestrained unexposed test specimens
B 2	Flexural stress at maximum load	ISO 178	80 % of the value obtained on unprestrained unexposed test specimens
B 3	Percentage elongation at break	ISO 527	50 % of the value obtained on unprestrained unexposed test specimens
B 4	Charpy impact strength unnotched	ISO 179	50 % of the value obtained on unprestrained unexposed test specimens
B 5	Tensile impact strength	See note 2	50 % of the value obtained on unprestrained unexposed test specimens
B 6	Any other property agreed upon	—	To be agreed

NOTES

1 The state of the test specimens, the indicative property and the criterion of failure shall be selected with a view to the practical service conditions.

2 This question is under study.

5.4 Test environments, when applicable, these shall be of analytical quality. When technical-grade media are used, they shall be of agreed origin and quality and care shall be taken that only one manufacturing batch is used for all measurements in any one series.

NOTE — During long exposures and especially at elevated temperatures, the nature and composition of the test environment may change. This has to be taken into consideration. It may be necessary to agree on renewal after specified periods.

5.5 Micrometer, calibrated to determine the thickness of the test specimens to 0,01 mm.

5.6 Apparatus to determine the indicative property

(See 8.6.)

6 Test specimens

6.1 Form and dimensions

These shall comply with the requirements of the relevant material standard. When no material standard exists, the form and dimensions shall be in accordance with the test method standard.

NOTE — If the test specimens are machined from sheets or articles, their thickness shall be the thickness of the sheet or articles. This shall be reported, together with the original location in the sheet or article.

6.2 State

For amorphous thermoplastics this shall be determined according to ISO 2557 on five replicates. For partially crystalline thermoplastics and for thermosetting materials, the moulding conditions shall be reported.

To obtain comparable results, the test specimens used shall have the same dimensions, state, mode of preparation and age. When cut or machined (ISO 2818) from sheet or articles, they shall be cut from corresponding places and in corresponding directions. Cut edges shall have a clean finish.

6.3 Number

For each prestrain value, at least 3 test specimens are required and for zero prestrain 5, unless different numbers are prescribed in the material or testing standard.

7 Conditioning and testing atmospheres

7.1 Conditioning atmosphere

Unless otherwise agreed between the interested parties (e.g. for polyamides and ABS), the test specimens shall be conditioned for 48 h in the atmosphere 23 ± 2 °C and (50 ± 5) % R.H. (ISO 291) before exposure to the test and reference environments.

7.2 Test atmosphere

Unless otherwise agreed between the interested parties, the indicative property shall be determined in the atmosphere 23 ± 2 °C and (50 ± 5) % R.H. (ISO 291).

8 Procedure

8.1 Precautions

During all stages of testing, the test specimens shall be protected from all deleterious gases, vapours or liquids (other than the test environment), strong light and direct contact with the human skin.

8.2 Temperatures

During exposure of the prestained specimens to the test environment, the prestrain temperature shall normally be 23 ± 2 °C. Additionally, 40 ± 2 °C or 55 ± 2 °C or other specified temperatures may be used. During storage in the reference environment (normally air), the same temperature shall be used.

8.3 Mounting the test specimens

Clamp the cleaned test specimens to the formers, starting with zero strain and ending with the former having the smallest radius.

NOTE — Care should be taken to handle only the ends of the test specimens. If the test specimens are not clean, they should be cleaned before mounting with a liquid that has no effect on them.

8.4 Contact with test environment

Immediately after the specimens are mounted, place them in contact with the test environment.

Contact with liquids or gases is established by immersion. Pastes shall be carefully spread to a depth of 2 to 4 mm over the exposed surfaces of the test specimens. Solids, such as elastomers or plasticized sheet, shall be laid on the surface of the test specimen and sandwiched between this and a second test specimen. This ensures that the contact is established under a definite pressure (see figure 2).

8.5 Storage in contact with the test environment

8.5.1 Short-time test

Keep the mounted test specimens in contact with the test medium for 22 to 24 h.

8.5.2 Long-term test

Keep the mounted test specimens in contact with the test medium for an agreed period, or until a further increase of the period is no longer accompanied by a change of the indicative property concerned.

NOTES

1 A preliminary test may be carried out with only one test specimen for each prestrain value to locate the failure strain. Equal numbers of prestrain levels on either side of this failure strain should then be used.

2 For most practical purposes a strain period of 1 000 h is adequate.

3 If due to the influence of the test medium, the test specimen lifts off at the centre or warps, open the clamps and reclamp the test specimen so that its central part is in contact with the former. If this is not possible, carry out a new test.

8.6 Determination of the indicative property

Make the determination in accordance with the material standard or, if none exists, with the test method standard, except that any conditioning clauses shall be ignored and the following procedure substituted.

After the agreed prestrain time, observe the test specimen visually. When the test medium is a solid, discard it and the covering test specimen (see figure 2).

Demount the test specimen from the former and determine the indicative property, starting with test specimens with the lowest prestrain. Do not remove the residual test substance when this is a liquid that does not drain from the surface.

Determine the indicative property as soon as possible after demounting the specimen in the short-time test and within 22 to 24 h after demounting in the long-term test.

In the flexural or Charpy tests the surface of the test specimen that was not in contact with the former shall be laid on the supports.

NOTE — If there is no material standard, use the following test conditions:

— flexural test (ISO 178): flexural stress at maximum load;

— tensile test (ISO 527): tensile stress at rupture, v = 50 mm/min.

9 Expression of results

9.1 Calculation

Calculate the arithmetic mean value of the indicative property for each prestrain value.

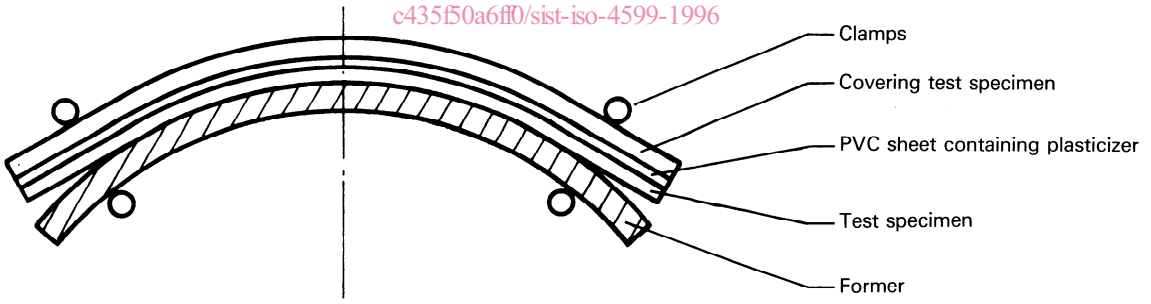


Figure 2 — Example of sandwiched test specimen