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

Part 1: **General guidance**

Plastiques — Détermination de la fissuration sous contrainte dans un environnement donné (ESC) —

Partie 1: Lignes directrices générales



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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22088-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

ISO 22088 consists of the following parts, under the general title *Plastics* — *Determination of resistance to environmental stress cracking (ESC)*:

— Part 1: General guidance

— Part 2: Constant tensile load method (replacement of ISO 6252:1992)

— Part 3: Bent strip method (replacement of ISO 4599:1986)

— Part 4: Ball or pin impression method (replacement of ISO 4600:1992)

— Part 5: Constant tensile deformation method (new test method)

— Part 6: Slow strain rate method (new test method)

Introduction

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. Simultaneous exposure to a chemical environment and stress or strain may result in a dramatic shortening of the time to failure compared to that in an inert environment. The phenomenon is referred to as environmental stress cracking (ESC) and is exhibited by many materials, including plastics. The permissible long-term stress or strain may be reduced considerably by this phenomenon.

It is generally believed that ESC occurs via the following processes:

- 1) Formation of microvoids in specimens by microscopic stress concentrations after applying stress.
- 2) Formation and subsequent growth of macrovoids caused by the breakdown of intermolecular bonds in intervoids that is produced by the action of a chemical environment, and formation of crazes which are composed of interconnected voids and fibrils.
- 3) Growth of the crazes caused by the break-down of the fibrils due to the applied stress and contact with a chemical environment.
- 4) Finally, a crack starts at the tip of the craze, leading to brittle failure.

The cracks 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);
 specimen preparation;
 thermal history of specimen;
- stress and strain;
- temperature of test;
- duration of test;
- chemical environment;
- method of application of stress and strain;
- failure criterion.

By keeping all but one parameter constant, the relative influence of the variable parameter on ESC can be assessed. The main objective of ESC measurements is to determine the relative effect of chemical media exposure 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 is almost impossible, 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 is likely to be more complex than that of test specimens.

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

Part 1:

General guidance

1 Scope

- **1.1** This part of ISO 22088 provides information and general guidance relevant to the selection of the test method to be used to determine environmental stress cracking (ESC).
- **1.2** Part 2 describes a method in which a test specimen is subjected to a constant tensile load, while immersed in a stress cracking agent at a specified temperature. The time and/or stress at which the specimen breaks is recorded.
- **1.3** Part 3 describes a method in which strips of plastic are subjected to a fixed flexural strain and exposed to a stress cracking agent for a predetermined period.
- **1.4** Part 4 describes a method in which a hole of specified diameter is drilled in a specimen and an oversized steel ball or pin is inserted into the hole while the test specimen is brought into contact with a stress cracking agent.
- **1.5** Part 5 describes a method in which a constant tensile deformation is applied to a specimen which is immersed in a stress cracking agent at a temperature selected for testing.
- **1.6** Part 6 describes a method in which a slowly increasing strain is applied to a specimen immersed in a stress cracking agent.
- **1.7** These methods are applicable to thermoplastic materials only.
- **1.8** These are essentially ranking tests and are not intended to provide data to be used for design or performance prediction.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

- ISO 150, Raw, refined and boiled linseed oil for paints and varnishes Specifications and methods of test
- ISO 293, Plastics Compression moulding of test specimens of thermoplastic materials
- ISO 294-1, Plastics Injection moulding of test specimens of thermoplastic materials Part 1: General principles, and moulding of multipurpose and bar test specimens
- ISO 2818, Plastics Preparation of test specimens by machining

Terms and definitions

The following terms apply to all parts of ISO 22088 except as otherwise indicated:

3.1

test temperature

temperature at which the test specimens are in contact with the test medium while being stressed

3.2

test period

time during which the test specimens are in contact with the test medium while being stressed

3.3

test medium

liquid, gas, paste, solid or other chemical medium selected for contact with the test specimens during the test

3.4

ESC index

(general) ratio of the value of the failure-indicative property determined in the test medium to that determined in a reference medium (usually air), measured at the same test temperature after the same test period

3.4.1

ESC index

(ISO 22088-3) ratio of the value of the failure strain determined in the test medium to that determined in the reference medium (usually air) for the same time of exposure

3.4.2

ESC index

(ISO 22088-6) normalized value of the departure stress that gauges the ESC susceptibility of different materials to a particular environment

Principles of the test

- In Part 2, a test specimen is subjected to a constant tensile load while immersed in a stress cracking agent at a specified temperature. Three methods may be used. Method A determines the stress required to produce rupture at 100 h. Method B determines the time to rupture at a specified constant tensile stress. In method C, the time to rupture for a series of applied stresses is plotted to determine if the time to rupture meets a specified agreed-upon stress.
- **4.2** In Part 3, strips of plastic are subjected to a fixed flexural strain and exposed to a stress cracking agent for a predetermined period. Using a series of forms with decreasing radii, increasingly higher strains are produced in the outer surface. After a specified exposure to the stress cracking agent, specimens are removed, inspected and tested for the indicative property, such as tensile strength.
- In Part 4, a hole of specified diameter is drilled in the specimen and an oversized steel ball or pin is inserted into the hole while the test specimen is brought into contact with a stress cracking agent. After a specified period of exposure, specimens are inspected and/or tested for the indicative property. In some cases, a parallel test conducted in air is carried out for comparison purposes.
- In Part 5, a constant tensile deformation is applied to a specimen immersed in a stress cracking agent at a temperature selected for testing. The ESC of the test material is determined by comparing the amount of deviation of a defined critical stress determined in the stress cracking medium from that determined in air.
- In Part 6, a slowly increasing strain is applied to a specimen immersed in a stress cracking agent. Testing is conducted at relatively low strain rates to enhance the effect of the stress cracking medium on the specimen. Development of crazes causes the strain to be taken up by the crazes so that the stress is reduced compared to tests conducted in an inert environment.

5 Applicability of the test method

- **5.1** Environmental stress cracking tests are used as quality control tools and in research and development to evaluate stress crack resistance.
- **5.2** When selecting a test method, it is important to consider the types of stress and strain a material will experience in service. Care must be taken when using constant strain test methods, such as the bent strip method or the pin impression method, as the stress applied to the material will decay with time due to stress relaxation.

Annex A lists typical plastics that are characterized by each type of environmental stress cracking test.

5.3 Comparisons of materials must be based on identical test conditions for each material. Selection of the test conditions will depend on the material and the application.

6 Test specimen preparation

Environmental stress cracking of a specimen is influenced not only by the material, but also by the method of preparation. Materials can only be compared using specimens prepared in a similar manner and in the same state.

Specimens shall be prepared in accordance with the appropriate International Standard. If no procedure for specimen preparation is given, specimens shall be machined from sheet or from products by the methods specified in ISO 2818.

To obtain comparable results, the test specimens used shall have the same dimensions, state and age and shall have been prepared by the same method of preparation. When cut or machined (ISO 2818) from sheet or articles, they shall be cut from corresponding places and in corresponding directions. The machined surfaces and edges of the finished specimens shall be free of visible flaws, scratches and other imperfections.

Care shall be taken to handle only the ends of the test specimens. If the test specimens are not clean, they shall be cleaned, before mounting, with a liquid that has no effect on them. Since cleaning can influence the test results, the cleaning procedure, if used, shall be included in the test report.

Moulded test specimens often have a considerable amount of orientation. If the load is applied parallel to the direction of injection, the time to rupture may be significantly lower than in the transverse direction. If the specimens are anisotropic, it may be useful to carry out tests with the load applied in different directions relative to the direction of injection. If specimens are prepared by moulding, the procedures shall be in accordance with ISO 293 or ISO 294-1.

7 Conditioning and test conditions

7.1 Conditioning

Unless otherwise agreed between the interested parties, the test specimens shall be conditioned before testing for at least 24 h at (23 ± 2) °C and (50 ± 10) % relative humidity.

7.2 Test temperature

The test temperature shall normally be (23 ± 2) °C. If required, other temperatures may be used, preferably selected from the following:

$$(40 \pm 2)$$
 °C, (55 ± 2) °C, (70 ± 2) °C, (85 ± 2) °C, (100 ± 2) °C,

or as agreed upon by the interested parties.

7.3 **Test medium**

The test medium used for the test shall be that specified in the relevant International Standard for the material tested. If nothing is specified, use either the agent which will be in contact with the material in the expected application or a reference product agreed upon between the interested parties.

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

NOTE Examples of reference products are:

- 95 % (by volume) ethanol pharmaceutical quality;
- a 1 % (by mass) solution of nonylphenoxy-poly(ethylene-oxy)ethanol ¹⁾ in distilled water; b)
- refined linseed oil (see ISO 150).

¹⁾ This detergent is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

Annex A

(informative)

Examples of test methods for various plastics

The following list gives examples of the types of material that can be examined using these test methods. [1] This is not a complete list and if a material is not listed it does not mean that it is inappropriate to use the test method.

ISO 22088

constant tensile load method	(Part 2)	PE	ABS	ABS+PC	ASA
bent strip method	(Part 3)	PMMA	PC	ABS	ABS+PC
ball or pin impression method	(Part 4)				
constant tensile deformation method	(Part 5)	PVC	PA		
slow strain rate test method [2]	(Part 6)	PC	ABS		

Other standards

bent strip method with notch (Bell method) (ASTM D 1693) [3]	PE	PFA
full notch creep test (FNCT) (ISO 16770) [4]	PE	
notch tensile test method (PENT) (ASTM F 1473) [5]	PE	

Other test methods

17 Composition 10 The Committee of Committee	1/4 ellipse bent method	PC .	ABS	POM	PBT	PPS	PS	PΡ	PVC	PΑ	PMMA	ABS+PC
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1/2 parabola bent method ASA ABS

cantilever beam method PC

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- [1] Report on an investigation concerning ESC test methods in Japan in December, 2001, Materials Technical Committee No. 4.5.1 (2002), The Japan Plastics Industry Federation
- [2] Polymer Testing, 19 (2000), pp. 117-129
- [3] ASTM D 1693, Standard Test Method for Environmental Stress-Cracking of Ethylene Plastics
- [4] ISO 16770, Plastics — Determination of environmental stress cracking (ESC) of polyethylene — Fullnotch creep test (FNCT)

A test specimen, in the form of square section bar with coplanar notches at the centre of each face, is subjected to a static tensile load in a temperature-controlled environment. The geometry of the specimen is such that plane strain conditions are obtained and brittle failure occurs under appropriate tensile load and temperature conditions. The time for this brittle failure to occur after loading is recorded.

[5] ASTM F 1473, Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resins

This test method determines the resistance of polyethylene materials to slow crack growth under conditions specified within. The test is generally performed at 80 °C and at 2,4 MPa, but may also be done at temperatures below 80 °C and with other stresses low enough to preclude ductile failure and thereby eventually induce brittle failure. Generally, polyethylene will ultimately fail in a brittle manner by slow crack growth at 80 °C if the stress is below 2,4 MPa.

- ISO 22088-2, Plastics Determination of resistance to environmental stress cracking (ESC) [6] Part 2: Constant tensile load method
- ISO 22088-3, Plastics Determination of resistance to environmental stress cracking (ESC) [7] Part 3: Bent strip method
- [8] ISO 22088-4, Plastics — Determination of resistance to environmental stress cracking (ESC) — Part 4: Ball or pin impression method
- [9] ISO 22088-5, Plastics — Determination of resistance to environmental stress cracking (ESC) — Part 5: Constant tensile deformation method
- ISO 22088-6, Plastics Determination of resistance to environmental stress cracking (ESC) [10] Part 6: Slow strain rate method
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