INTERNATIONAL STANDARD

ISO 16770

First edition
2004-02-01

Plastics — Determination of environmental stress cracking (ESC) of polyethylene — Full-notch creep test (FNCT)

Plastiques — Détermination de la fissuration sous contrainte dans un environnement donné (ESC) du polyéthylène — Essai sur éprouvette entièrement entaillée (FNCT)

Reference number
ISO 16770:2004(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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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 16770 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

Plastics — Determination of environmental stress cracking (ESC) of polyethylene — Full-notch creep test (FNCT)

1 Scope

This International Standard specifies a method of determining the stress cracking resistance of polyethylene materials in any environment. The test is carried out on notched test specimens cut from compression-moulded sheet or finished products, as applicable. The test specimen is subjected to a static tensile load when immersed in an environment such as a surfactant solution held at a specified temperature, and the time to failure measured.

The method has been specifically developed for polyethylene materials but can be used to evaluate PE extrusions, such as pipe segments, PE fusion welds/fittings and blow-moulded PE containers to study the effect of aggressive environments, i.e. dangerous goods/chemicals. The method may also be adapted for other thermoplastic materials, e.g. polypropylene (PP). In this case, care must be taken in interpreting the results as the processing stresses/orientation in finished products may have an effect.

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
failure
complete separation of the two halves of the test specimen

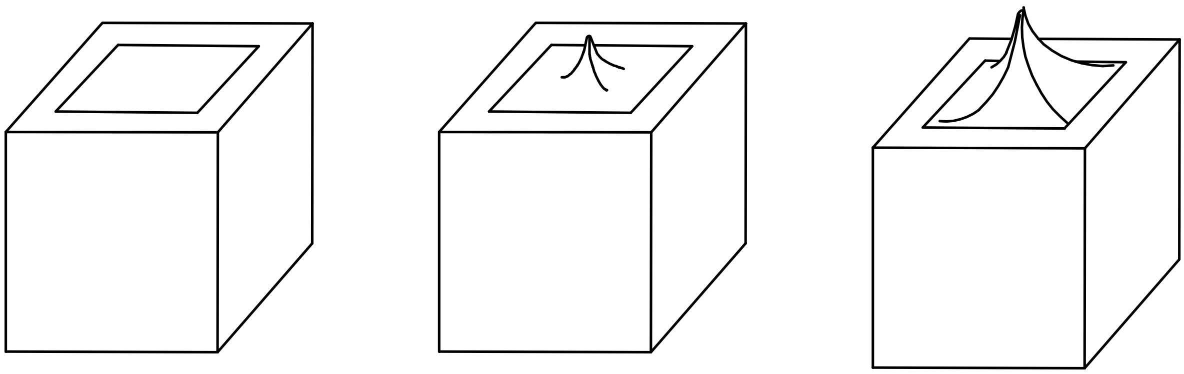
NOTE The description of the failure surface has been simplified in this International Standard (see 3.2 and 3.3). Further information is available in the literature (see the Bibliography).

3.2
brittle failure
failure in which the fracture surface exhibits no permanent material deformation to the naked eye, e.g. stretching, elongation or necking down [see Figure 1a)]

NOTE In tougher materials, an extended ligament may form in the centre [see Figure 1b)].

3.3
ductile failure
failure in which the fracture surface clearly exhibits permanent material deformation with stretching, elongation and necking down [see Figure 1c)]

3.4
ligament area
cross-sectional area remaining after notching



a) Brittle failure (no deformation)

b) Brittle failure (extended ligament may form at centre)

c) Ductile failure

Figure 1 — Failure surfaces

4 Principle

A test specimen, in the form of a square-section bar with coplanar notches in each face at the centre, is subjected to a static tensile load in a temperature-controlled environment, for example air, water, surfactant solution. 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 Apparatus

5.1 Loading device

A suitable device for applying the load is a lever-arm loading machine with an arm ratio between 4:1 and 10:1. A typical example of such a device is shown in Figure 2. The lever-arm ratio R is equal to L_1/L_2. When the lever-arm is fitted with the top specimen grip and the weight carrier, it shall be horizontal, i.e. balanced.

The specimen grips shall be designed to prevent slippage of the test specimen and to ensure that the load is transmitted axially through the test piece, e.g. via a low-friction coupling, to prevent bending and torsion of the test specimen during the test. A typical test specimen grip assembly is shown in Figure 3.

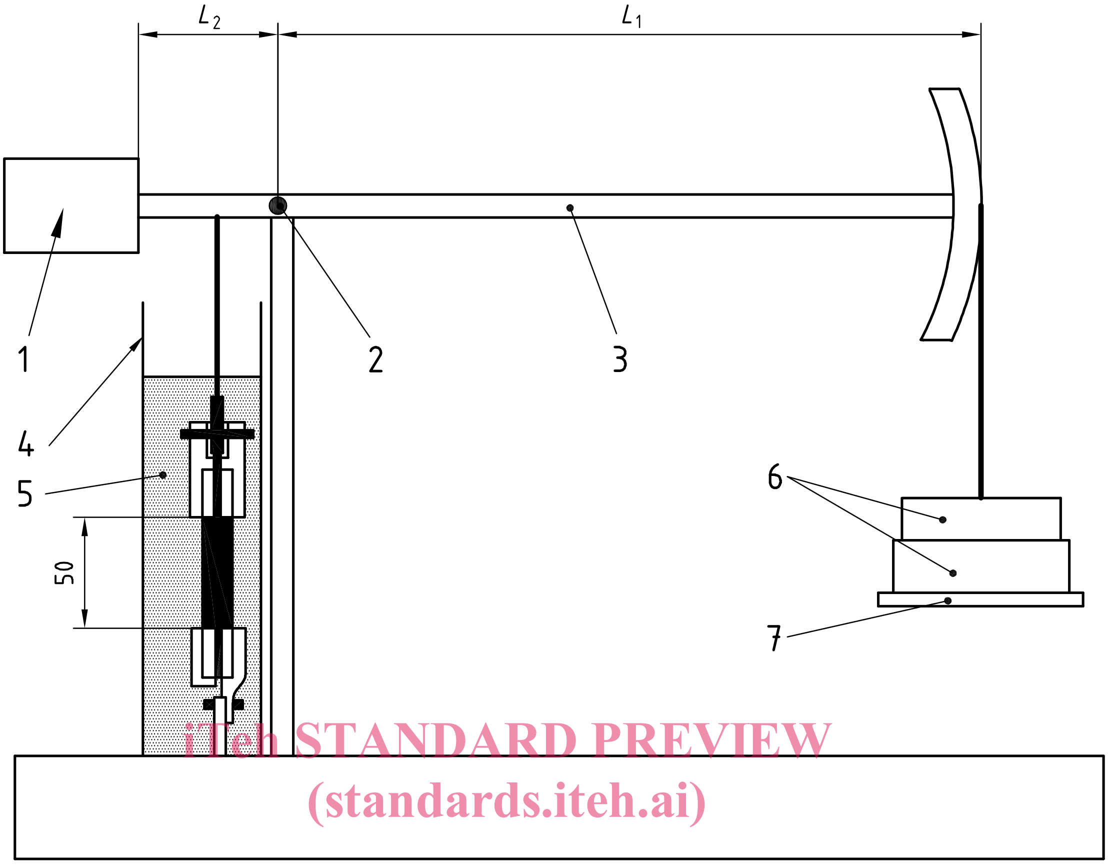
In addition to the above example, the tensile load may be applied directly using deadweights, pneumatically actuated loading or any other means of producing a constant load. The loading device shall be capable of applying the load to an accuracy of ± 1 %. The balanced loading apparatus as described in ISO 6252 has also been used satisfactorily.

The functioning and calibration of the equipment shall be checked on a regular basis because the applied load is a critical parameter. The calibration of a lever-arm machine can be checked by hanging a series of known weights on the specimen side of the lever-arm and counterbalancing these in turn with weights on the weight hanger. The ratio of the former to the latter provides a direct measure of the arm ratio and hence a check on the operation of the equipment.

In the case of multiple-specimen testing, care shall be taken to avoid undue disturbance of the remaining test specimens when one or more specimens fail.

NOTE Measurement of the extension of the test specimen or movement of the lever-arm can provide useful information. The rate of extension of the test specimen will increase when the initiation of the crack from the notch has occurred and will increase rapidly when failure is imminent.

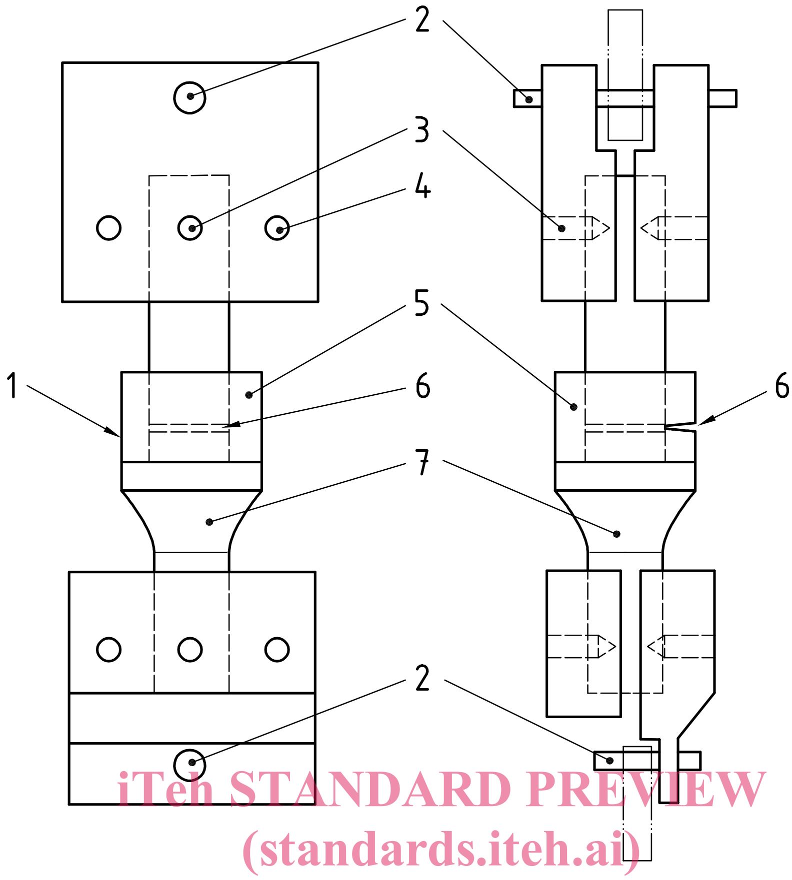
Dimensions in millimetres



Key

1 counterweight
2 low-friction roller on hinge
3 balance lever-arm
4 example of environmental chamber
5 environment
6 weights
7 weight carrier

Figure 2 — Loading device



Key

1 small environmental chamber
2 coupling pin
3 grub screw to prevent slipping
4 clamp bolt
5 glass tube
6 notch
7 heat-shrink tube

Figure 3 — Specimen grip assembly

5.2 Thermostatically controlled chamber

This chamber shall be designed to contain the environment and ensure immersion of at least the notched area of the specimen(s). The chamber shall be constructed of material(s) which do not affect the environment and which are not affected by it. The temperature of the environment shall be controlled to maintain the test specimens within ± 1,0 °C of the specified test temperature. Where the environment is aggressive, the chamber can be very small as shown in Figure 3.

If the cloud point of the environment solution is lower than the test temperature, phase separation will occur and so moderate laminar flow is required in the environment to ensure uniform dispersal. It shall also be ensured that the results achieved at each location in the immersion bath are the same.

5.3 Temperature-measuring device

A calibrated thermometer, thermocouple or thermistor with an accuracy of ± 0,1 °C is suitable.

5.4 Timing device

This shall automatically indicate or record the point when the test specimen fails by excessive displacement of the grips. The accuracy of the timing equipment shall be ± 1 min.

5.5 Notching apparatus

This machine shall be designed so that the notches are coplanar and the plane of notching is perpendicular to the tensile axis of the test specimen. The machine shall have a device to ensure that the notches are placed in the centre of the test specimen. The notch tip radius shall be less than 10 µm. Razor blades are preferred. However, a cutting machine with a tool like a broaching device is acceptable as an alternative provided the notch tip radius is less than 10 µm.

NOTE A device, appropriately dimensioned, such as illustrated in ISO 11542-2:1998, Figure B.1, would be satisfactory.

5.6 Microscope

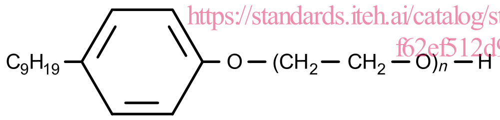
A microscope is required to allow accurate measurement of the actual ligament size (distance between the tips of the notches) after failure. It shall read to an accuracy of ± 100 µm.

6 Preparation of test specimens

6.1 Test specimen geometry

Typical test specimen geometries are given in Annex A. If other specimens are used, these shall be made such that the ligament area is approximately 50 % of the total cross-sectional area of the specimen (see Figure 4). This is to make sure that specimen failure will occur under the prescribed conditions. A "dog-bone" shaped specimen is easier to clamp, but a parallel-sided section of at least 15 mm on either side of the notch is required. The use of different test specimen geometries will give different results with the same polyethylene. Comparisons between materials are only valid if the same specimen geometry and specimen preparation technique are used.

A neutral-type nonylphenoxy-(ethyleneoxy)-ethanol detergent of the general formula shown below is required:



The value of n can be 10 or 11. Such detergents are suitable for use in testing at elevated temperatures and are sufficiently aggressive to produce failure in a reasonable timescale. A detergent with a value of n of 11 gives shorter failure times than one for which n = 10.

Using deionized water, prepare a sufficient quantity of a solution, of a concentration equivalent to 2 % by mass of the detergent, to ensure complete immersion of the test specimens. Other surface-active agents may be used if specified in the relevant product standard or by agreement between the interested parties. If, for example, Igepal CO630 is used, its concentration and designation shall be clearly specified in the test report because the result may depend on the surfactant used.

NOTE The effect of a detergent on polyethylene varies with the density of the material. For lower-density PE, this can be more severe than if water or air alone is used.

Tests carried out using freshly made-up solutions of some detergents can give variable results, so the solution shall be "aged" for 14 days at the test temperature to ensure that the alcohol groups are converted to acid groups. This is said to improve the reproducibility of the results. The solution may continue to age, and it is suggested that a check be made after 2 500 h of use. Specimens of a control material can be tested in the solution to verify that there is no difference in activity.

6.2 Other environments

The FNCT is suitable for comparative testing of polyethylene test specimens with other chemicals, including distilled water. The test report shall contain full details of the identity, concentration and producer of the chemical used, as well as the designation of the polyethylene. Environments at higher temperatures,