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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)

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastics*.

This second edition cancels and replaces the first edition (ISO 16770:2004), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the test conditions for bimodal PE materials with substantially increased crack-resistance have been extended on an alternative more aggressive detergent;

— the compression moulding conditions for 10 mm × 10 mm test specimen (Type A) have been modified;

— the specimen types have been updated, with injection moulded test specimens being included;

— precision data (repeatability) from interlaboratory testing have been included for Type A test specimen.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Since mid-2000, the full-notch creep test (FNCT) has been widely used as one of the tests characterizing the environmental stress cracking (ESC) of polyethylene (PE) materials used in demanding applications. Among current ESC tests, the FNCT is relatively simple, easy to perform and still a sensitive laboratory scale test method to characterize ESC resistance of PE materials. The advantage of FNCT is that it enables the evaluation of the whole PE product range by variation of test conditions (test specimen geometry, temperature, medium and load conditions).

The FNCT method was standardized in 2004 as a PE material property, with partial precision statement based on the repeatability for a 6 mm × 6 mm-cross-sectional test specimen (Type B). Since then, several interlaboratory tests have been performed, resulting in a repeatability statement for 10 mm × 10 mm-cross-sectional test specimen (Type A). Type A specimen is prepared by using modified compression moulding conditions, which give more consistent test results compared to the original moulding conditions.

In addition, due to recent development of new PE pipe materials, known as PE 100 RC, which exhibit substantially improved crack resistance, it became necessary to define test conditions for these materials. Testing of these materials using the conditions specified in the previous edition of this document (i.e. ISO 16770:2004) resulted in failure times of one year or longer. As a consequence, this document includes extended test conditions to cover PE materials with substantially increased crack resistance. This accelerated test procedure using extended test conditions allows failure times to be reduced substantially compared to conventional test conditions given in ISO 16770:2004. In addition, a good correlation between accelerated and conventional test methods was derived; see References [4], [7] and [9].

The FNCT is a material characterization and production monitoring test, which, by strictly maintaining the test conditions defined, enables relevant and reliable comparison among similar PE materials or group of PE materials.

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

1 Scope

This document specifies a method for determining the environmental stress cracking (ESC) resistance of polyethylene (PE) materials in a defined test environment. The test is carried out on notched test specimens machined from moulded sheets/specimens or from finished products. The test specimen is subjected to a static tensile load when immersed into an environment such as a surfactant solution held at a specified temperature. The time to failure is measured.

The method has been specifically developed for polyethylene materials but can be used to evaluate PE products, such as pipes, fusion welds/fittings and blow-moulded PE containers to study the effect of aggressive environments, i.e. dangerous goods and chemicals.

The method is suitable for use with test specimens moulded to chosen dimensions or machined from compression moulded sheets or injection moulded specimens, or from finished products, such as mouldings and pipes. When the test specimens are machined from extruded or moulded parts, the results can be affected not only by properties of the material, but also by stresses or orientation introduced during processing.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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*

ISO 7500-2, *Metallic materials — Verification of static uniaxial testing machines — Part 2: Tension creep testing machines — Verification of the applied force*

ISO 17855-2, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 20753, *Plastics — Test specimens*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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

3.2
brittle failure
failure (3.1), in which the fracture surface exhibits no permanent material deformation to the naked eye, e.g. stretching, elongation or necking down

Note 1 to entry: If the brittle area is less than 20 % of the total ligament, the failure is ranked as ductile.

Note 2 to entry: See Annex C for examples.

Note 3 to entry: The beginning of the transition to *ductile failure* (3.3) behaviour may be indicated by an extended ligament, which may form in the centre (see Annex C for examples).

3.3
ductile failure
failure (3.1), in which the fracture surface clearly exhibits permanent material deformation with stretching, elongation and necking down

Note 1 to entry: See Annex C for examples.

3.4
nominal ligament area
A_n
cross-sectional area of the test specimen remaining after notching

Note 1 to entry: It is expressed in square millimetres (mm^2).

3.5
measured ligament area
A_L
actual cross-sectional area of the test specimen remaining after notching determined after testing

Note 1 to entry: It is expressed in square millimetres (mm^2).

3.6
nominal tensile stress

σ_n

normal force per unit area of the *nominal ligament area* (3.4) of the test specimen

Note 1 to entry: It is expressed in megapascals (MPa).

3.7
actual tensile stress
σ_L
normal force per unit area of the *measured ligament area* (3.5) of the test specimen

Note 1 to entry: It is expressed in megapascals (MPa).

3.8
reference tensile stress
$\sigma_{L,ref}$
selected normal force per unit area of the *measured ligament area* (3.5) of the test specimen used for determination of comparable time to *failure* (3.1)

Note 1 to entry: It is expressed in megapascals (MPa).

3.9
time to failure at the reference tensile stress
$t_{f,ref}$
time to failure corresponding to the *reference tensile stress* (3.8), calculated by interpolating in the measured dependence of time to failure vs. *actual tensile stress* (3.7) for individual tested specimens

Note 1 to entry: It is expressed in hours (h).

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 load in a temperature-controlled environment containing a surface-active detergent solution. The geometry of the test 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.

NOTE 1 Distilled water is also a suitable environment for carrying out this test.

NOTE 2 For testing of PE-based container materials, other environments can also be used, such as dangerous goods and chemicals or suitable liquid test media.

5 Apparatus

5.1 Loading device, constructed and maintained in accordance with ISO 7500-2. The class of the apparatus shall be stated and reported.

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 device is shown in Figure 1. 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.

Other suitable loading mechanisms are permitted.

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 specimen, for example via a low-friction coupling, to prevent bending and torsion of the test specimen during the test. An example of test specimen grip assembly is shown in Figure 2.

In addition to the above example, the tensile load may be applied directly using deadweight 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 22088-2[3] can also be used.

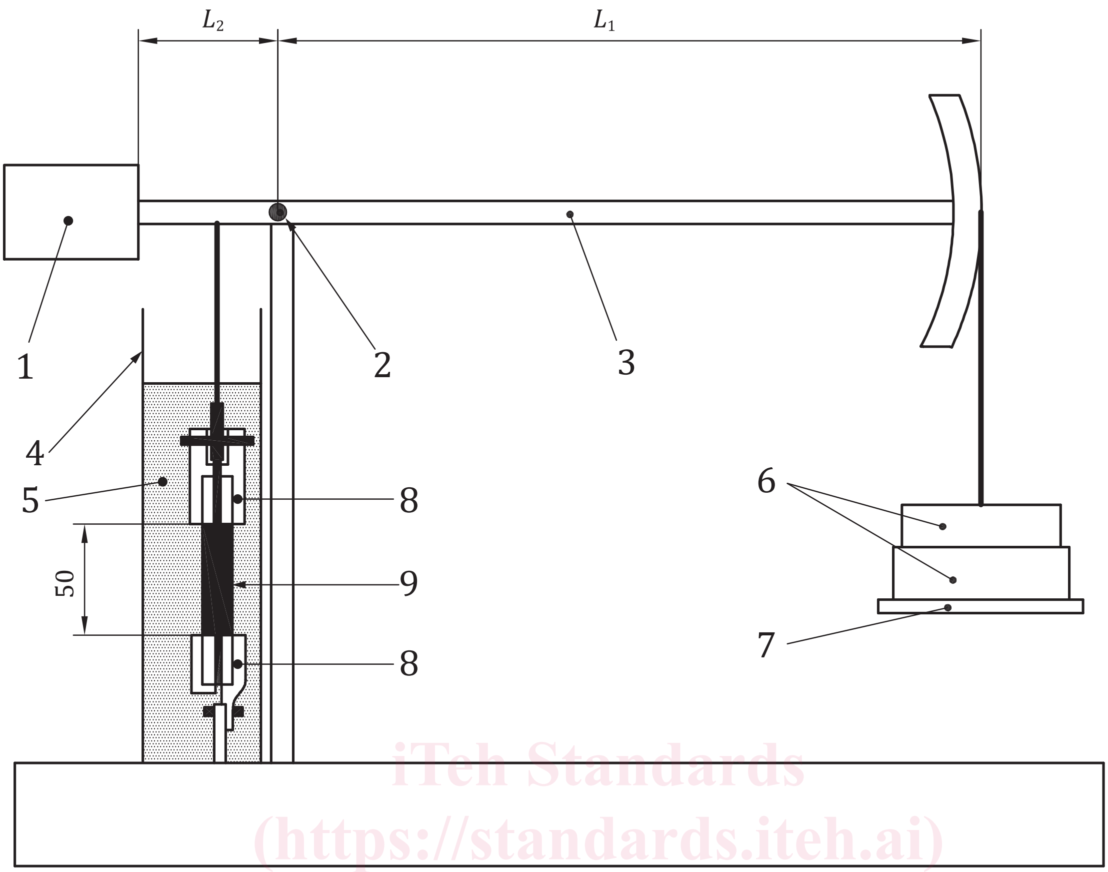
As the applied load is a critical parameter, the operation and calibration of the equipment shall be checked on a regular basis. Loading rate shall be kept as steady as possible to avoid shock-loading of the notched test specimen. Motor-driven loading system with speed control should be preferably used.

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 case of multiple specimens testing, care shall be taken to ensure that when one or more specimens fail, the remaining test specimens remain unaffected.

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 increases when the initiation of the crack from the notch has occurred and increases rapidly when failure is imminent.

Dimensions in millimetres

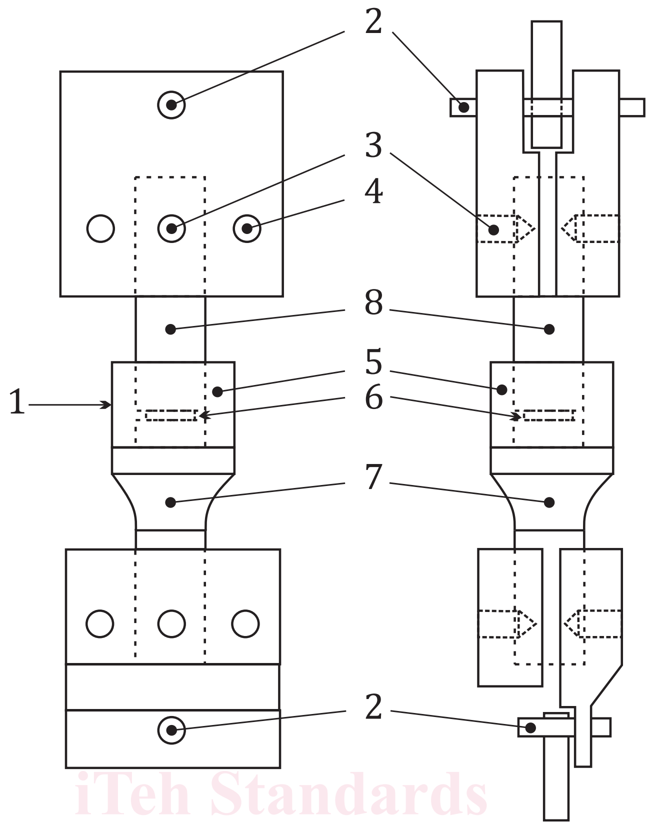


Key

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

NOTE The distance between the grips is 50 mm corresponding with the specimen length of 100 mm.

Figure 1 — Loading device



Key

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

Figure 2 — Specimen grip assembly

5.2 Thermostatically controlled chamber, designed to contain the test environment and ensure immersion of at least the notched area of the test 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 K of the specified test temperature throughout the duration of the test.

The homogeneity and uniform dispersal of the environment shall be ensured. If the cloud point of the environment solution is lower than the test temperature, phase separation occurs and so moderate laminar flow or adequate stirring equipment is required. 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 ±0,2 K is suitable.

5.4 Timing device, with an accuracy of the timing device shall be ±1 min. The timing device shall automatically indicate or record the point when the test specimen fails by excessive displacement of the grips.