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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

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.

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.

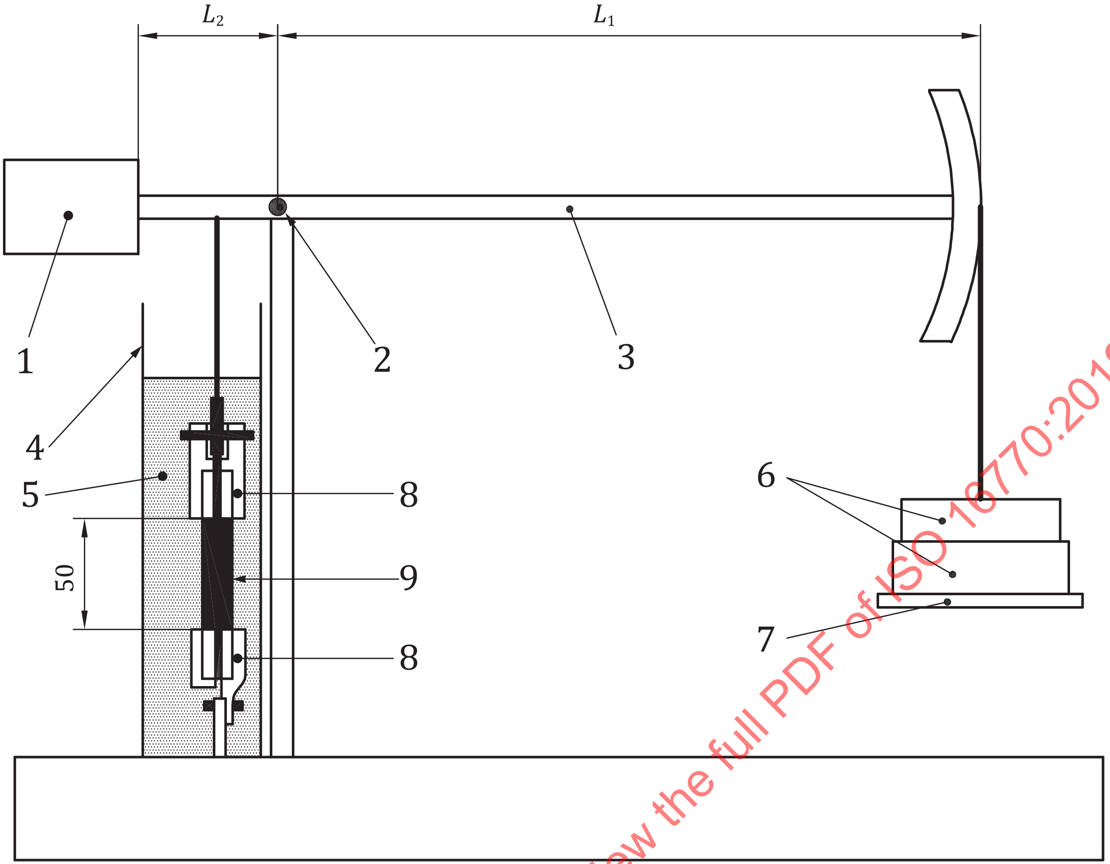
As the applied load is a critical parameter, the operation and calibration of the equipment shall be checked on a regular basis. Loading rate shall be kept as steady as possible to avoid shock-loading of the notched test specimen. Motor-driven loading system with speed control should be preferably used.

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

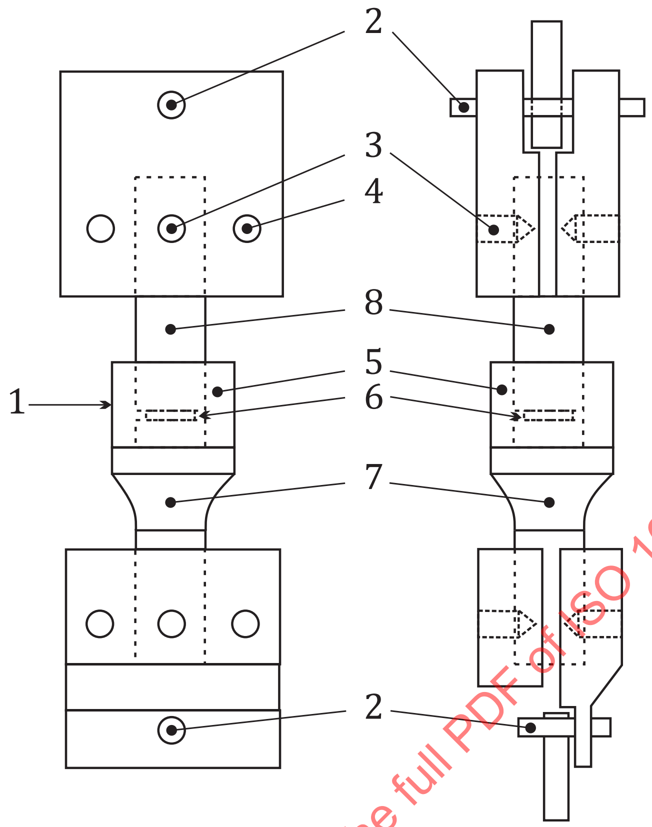


Key

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.

5.5 Notching device, designed so that the notches in the specimen are coplanar and the plane of the 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.

Indentation using a razor blade of appropriate thickness is a preferred method for notching the tests specimens. Recommended razor blade thickness is 0,15 mm to 0,25 mm.

Alternatively, a cutting machine with a tool as broaching device can also be used to machine the notches, provided that the notch tip radius is less than 10 µm.

5.6 Optical device. A suitable optical device, such as a measuring microscope, comparator or similar device, is required to allow accurate measurement of the actual ligament area (area between the tips of the notches) after failure. The accuracy of the measurement shall be better than ±0,01 mm^2.

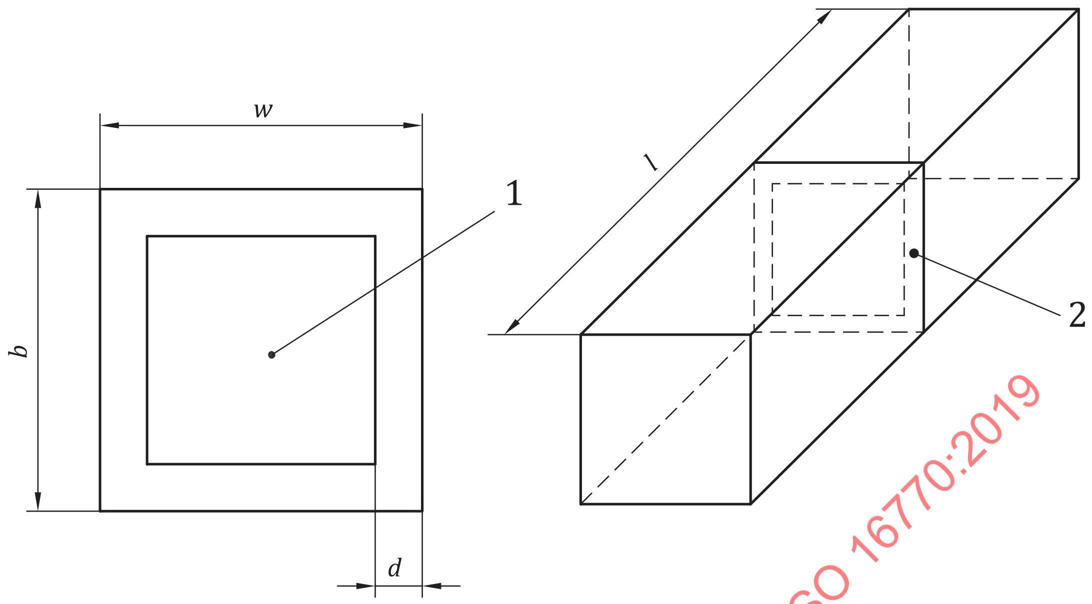
6 Test specimens

6.1 General

The method specifies preferred dimensions of the test specimens. Tests which are carried out on specimens of different dimensions or with different notches, or specimens which are prepared under different conditions, may give results which are not comparable. Other factors, such as conditioning of the test specimens, can also influence the test results. Consequently, all these factors shall be controlled and recorded.

6.2 Test specimens geometry

The test specimen geometry, showing notch and the ligament area is given in Figure 3.



Key

1 ligament area

2 notches

w width

b thickness

l length

d notch depth

Figure 3 — Test specimen showing the notch and the ligament area

The dimensions of the test specimens are given in Table 1. The appropriate test specimen type will be selected according to polymer end-use application.

Table 1 — Dimensions of the test specimens

Test specimen type	Length mm	Width mm	Thickness mm	Notch depth mm
A	100 ± 2	10,0 ± 0,1	10,0 ± 0,1	1,6
B	90 ± 2	6,0 ± 0,1	6,0 ± 0,1	1,0
C	100 ± 2	10,0 ± 0,1	4,0 ± 0,1	1,6/0,6
Type A test specimen is intended for pipe or sheet materials. Type B test specimen is intended for extrusion or blow moulding materials. Type C Test specimen is intended for injection moulding materials.				

If other test specimens are used, these shall be made such that the ligament area is approximately 50 % of the total cross-sectional area of the specimen. This is to make sure that test specimen failure will occur under the prescribed conditions of brittle mode.

NOTE 1 The use of different test specimen geometries will give different test results with the same PE material.

NOTE 2 Comparisons between materials are valid only if the same test specimen geometry, preparation conditions and test conditions are used.

6.3 Test specimen preparation

6.3.1 General

The test specimens shall be prepared from compression moulded sheets, from injection moulded multipurpose test specimen type 1A according to ISO 20753, or from finished products (pipes, fittings, etc.).

6.3.2 Test specimens machined from compression moulded sheets

For compression moulding, the moulding conditions given in Table 2 shall be used. ISO 293[1], ISO 17855-2 and ISO 11542-2[2] give additional general guidelines for moulding and cooling conditions.

Table 2 — Conditions for compression moulding of the test specimens

Thickness mm	Moulding temperature °C	Average cooling rate °C/min	Preheating time min	Full pressure MPa	Full pressure time min
$6^{+0,5}_{-0,1}$	180	15 ± 2	20	5	10
$10^{+0,5}_{-0,1}$	200	3,5 ± 1,5	25	10	40

NOTE 1 Higher value of upper tolerance of the thickness enables adopting the sheet thickness to its nominal value.

NOTE 2 Preheating pressure is 0,5 MPa.

Use of a positive mould is necessary.

The demoulding temperature shall be ≤ 40 °C.

The control of moulding parameters, especially the cooling rate is critical during test specimen preparation. Inconsistent cooling rates can lead to significant deviations in test results due to the effect on crystallinity. It is critical that the moulding machine is capable of maintaining a constant cooling rate.

After compression moulding, annealing of the test specimens shall be carried out by conditioning the test specimens for 3 h in an oven at a temperature of 100 °C and then slowly cooled down to room temperature.

If the tested PE material is a powder, it may be necessary to calendar or compound the material prior to the compression moulding step. It is essential to make sure that the powder is heat-stabilized when this is done. The use of different moulding conditions will affect the test results.

The test specimens shall be machined to size from the moulded sheet or test specimen in accordance with ISO 2818 at least 24 h after moulding.

6.3.3 Test specimens machined from injection-moulded type 1A multipurpose test specimen

The test specimens shall be prepared by machining from multipurpose test specimen type 1A, prepared in accordance with ISO 294-1 and ISO 17855-2.

6.3.4 Test specimens machined from finished products

When testing finished products, the test specimens shall be cut from extruded or moulded products in accordance with ISO 2818.

6.4 Test specimen notching

For notching the test specimens, a suitable notching device (5.5) shall be used. The test specimens shall be notched at room temperature. Notching shall be performed at least 24 h after the test specimen preparation to allow for stress relaxation. Care shall be taken to avoid blunting the notch during this operation, e.g. to avoid use of excessive speed and/or force, as this will invalidate the test results.

When a razor blade is used, the speed of razor indentation shall not be higher than 0,5 mm/min. One razor blade shall be used for producing no more than 64 notches.

For any notching device used, the tolerance on the required notch depth shall be ±0,1 mm.

Notch integrity shall be checked microscopically after performing the test. Test specimens with non-conforming notches shall be discarded.

6.5 Conditioning of the test specimens

Unless otherwise specified, the notched test specimens shall be stored at (23 ± 2) °C, or at (27 ± 2) °C for tropical countries, and conditioned in the test environment at test temperature for a period between 16 h and 24 h after clamping in the loading apparatus, prior to loading. Conditioning the test specimens shall be started at least 24 h after notching the specimens.

Materials containing fillers or additives that are susceptible to moisture uptake shall be stored and conditioned at the appropriate temperature at (50 ± 10) % relative humidity.

7 Test environment

7.1 General

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

To maintain constant bath conditions, the activity of the detergent solution shall be monitored by suitable technique, for example by measuring the pH value or other relevant parameter depending on the detergent nature.

Specimens of a control material can be tested in the solution to verify that there is no change in activity.

The test results using different detergents are not comparable.

7.2 Recommended test environment

7.2.1 Test environment No. 1

Non-ionic neutral type nonylphenol ethoxylate detergents (CAS No. 9016-45-9)[1)]

The general formula of this type of detergent is shown in Figure 4:

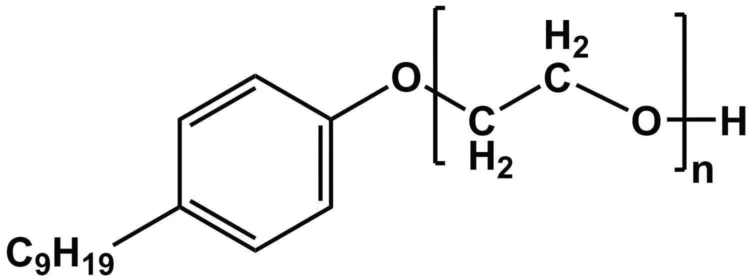


Figure 4 — Formula of the test environment No. 1

The value of *n* can be 10 or 11.

1) Arkopal® N100, N110 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

The test environment No. 1 is suitable for use in testing at elevated temperatures and is sufficiently aggressive to produce failures in reasonable times for standard PE materials defined in this document. A detergent with a value of n = 11 gives shorter failure times than one for which n = 10.

Using distilled water, a sufficient quantity of solution shall be prepared, of a concentration equivalent to 2 % mass fraction of detergent. A complete immersion of the test specimens shall be ensured. The concentration and designation of the environment shall be clearly specified in the test report, as the test result may depend on the surfactant used.

The solution shall be aged for 14 days at the test temperature before use to ensure consistent results. After a maximum of 2 000 h use, the solution shall be renewed due to acidification of the detergent especially at 80 °C and in contact with air[5]. It is recommended to avoid renewing the solution during the test, to limit the disturbance of the non-failed test specimens. For test specimens with a very long failure time, renewing the solution during the test is allowed, but disturbance shall be prevented as much as possible. If the solution is renewed during the test, it shall be stated in the test report. Aging is not required for the renewed solution while the test is running. New test specimens shall not be placed in the renewed solution before 14 days of aging at the test temperature.

Suggestion is to use a buffer to maintain the pH value of the solution at a level between 9 and 10. As an example, natrium carbonate can be used.

7.2.2 Test environment No. 2

Aqueous solution containing alkyl dimethyl amine oxide type of detergents, e.g. Lauramine oxide (CAS No. 85408-49-7)[2)]

The general formula of this type of detergent is shown in Figure 5:

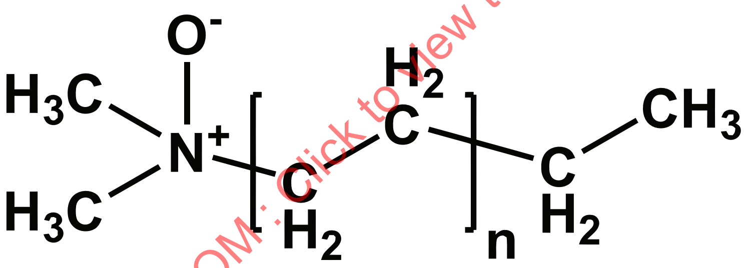


Figure 5 — Formula of the test environment No. 2

Test environment No. 2 is more stable at elevated temperature and is a more aggressive detergent and gives shorter failure times compared to the test environment No. 1[6]. For these reasons, such aqueous solution containing alkyl dimethyl amine oxide type of detergents is suitable for testing PE materials with substantially increased resistance to ESC, such as bimodal or multimodal PE pipe grades.

The test environment No. 2 has a non-ionic character when the pH value is higher than 7 and a cationic character with a pH value up to 3. The alkyl chain length has a range from n = 4 to 8 with n = 5 and 6 being predominant.

Using distilled water, a sufficient quantity of solution shall be prepared, of a concentration equivalent to 2 % mass fraction of detergent. A complete immersion of the test specimens shall be ensured.

The tests carried out by using freshly made up solutions of some detergents may give variable results, so the solution shall be aged for 4 days at the test temperature before using.

NOTE Changes in pH at the start of the test are not an indication of changes in aggressiveness of the detergent mixture.

2) Dehyton® PL is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

It is recommended to renew the detergent solution after each test, so that each test specimen is tested in a freshly made solution. If this is not possible or undesirable, it is recommended that the solution is renewed after 2 000 h to exclude possible variations even though aging is limited for test environment No. 2.

7.3 Other test environments

Other surface-active environments may be used, if specified in the relevant product or material standards or agreed upon between interested parties.

Generally, FNCT is suitable for comparative testing of PE materials with other chemicals, including distilled water. Therefore, tests addressing the ESC behaviour of PE-based container materials in contact with potentially aggressive environments, such as dangerous goods, can be performed.

The test report shall contain full details of the identity, concentration and producer of the chemical used, as well as the PE designation. Environments at higher temperatures, especially above 80 °C, can give different results due to absorption, chemical attack or crystalline changes in the polyethylene itself, which shall be taken into consideration when carrying out the test.

If surface-active environments other than those described in 7.2 are used, the ageing conditions shall be agreed between the interested parties.

8 Test procedure

8.1 Selection of detergent, reference tensile stress and test temperature

The test parameters as given by the relevant product standard shall be used. If none are given, the detergent, reference tensile stress and test temperature shall be selected from Table 3, depending on the PE material type to be tested, unless otherwise agreed upon between interested parties.

Table 3 — Test specimens and test conditions

Test specimen type	Test environment	Reference tensile stress MPa	Test temperature °C
A	No. 1	4,0	80
A	No. 2	5,0 or 4,0[a]	90
B	No. 1	9,0	50
C	No. 1	6,0	50

[a] If the tensile stress of 5,0 MPa results in ductile failure (see 8.6), then retesting using tensile stress of 4,0 MPa shall be performed.

NOTE 1 Alternative stress levels are allowed for obtaining reasonable testing times, if an unknown or less known PE material is being tested. Results of tests using alternative stress levels are not comparable with the test results using reference tensile stresses, as given in Table 3.

NOTE 2 Lower temperatures increase the failure times, higher temperatures decrease the failure times of the test specimens. However, when too high temperature is used, ductile failure, changes in crystallinity and an oxidative ageing can occur. The same applies when different environments are used.

NOTE 3 To ensure brittle failure, it can be necessary to investigate a broader range of stresses under certain conditions and to identify the transition from ductile behaviour and brittle behaviour, as outlined in 8.2 and Figure 6.

8.2 Number of test specimens to be tested

For one material, at least eight test specimens shall be tested, i.e. at least two test specimens at each of four nominal tensile stress levels, with two levels below and two above the selected reference tensile stress. This is to compensate for possible ligament variation introduced during the notching procedure.

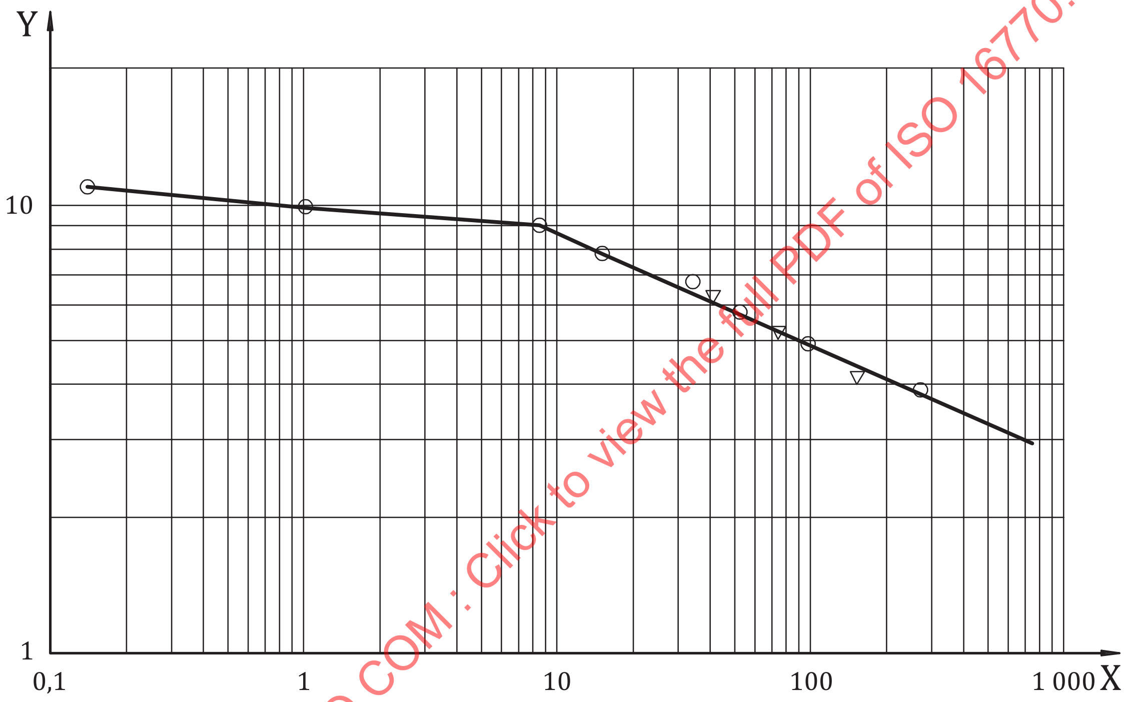
For example, having selected 4 MPa reference tensile stress, the recommended nominal tensile stress levels are 3,25 MPa, 3,75 MPa, 4,25 MPa and 4,75 MPa.

For internal, routine testing four test specimens may be used, i.e. one test specimen at four nominal tensile stress levels, with two below and two above the selected reference tensile stress.

In case of dispute, at least eight test specimens shall be tested.

For all the nominal tensile stress levels, care shall be taken when selecting the upper nominal tensile stress levels, as these may cause occurrence of ductile failure. Test specimens showing ductile failure shall be discarded.

For a full material characterization, it may be useful to map out its behaviour over a broader range of stresses for a certain test environment at an appropriate test temperature. A typical example of this approach is given in Figure 6.



Key

X failure time (h)

Y actual tensile stress (MPa)

○ broached notch

▽ razored notch

Figure 6 — Typical stress/failure time plot

8.3 Calculation of the test load

The test load is calculated from Formula (1):

$$M = \frac{A_n \times \sigma_n}{9{,}81 \times R} \tag{1}$$

where

M	is the mass, in grams, of the weights used to apply the load;
A_n	is the nominal ligament area, in square millimetres;
σ_n	is the nominal tensile stress, in megapascals;
R	is the lever-arm ratio (equal to 1, if the load is applied directly);
9,81	is the conversion factor from mass, in kilograms, to applied load, in newtons.

8.4 Application of the load to the test specimen

The notched test specimen is carefully placed into the grips of lever-arm loading machine. Care shall be taken to avoid bending or twisting the specimen. The test specimen shall be positioned with half its length exposed between the grips and with the notch plane located at the centre. The specimen shall be immersed, held in the grips, into the test environment, ensuring that the notched area is in contact with the environment and is conditioned in accordance with 6.5. After conditioning, the calculated load to the lever-arm shall be applied gradually and carefully, avoiding shock loading of the specimen. At the same time, the timing device shall be started.

NOTE 1 Applying the load can be a major source for error.

NOTE 2 It has been found convenient to lower the weight carrier using a suitable jack or other means.

For alternative loading devices, the load shall be applied analogously.

NOTE 3 If the specimen has been machined from the finished product, it can contain internal stresses and be a little bent. Further guidance can be found in the particular product standard.

8.5 Removing individual specimens from a chamber

It is not recommended to remove any test specimen from the thermostatically controlled chamber (5.2) when other non-failed test specimens are in the same chamber. This is to prevent disturbance, and thus possible early failure, of the non-failed specimens. Specimens should be removed from the chamber only when all specimens in the same chamber have failed.

8.6 Measurement of the ligament area after failure

After allowing the specimen to cool to room temperature, the fracture surface of each tested specimen is examined to ensure that the fracture is of a brittle type. Use an optical microscope or SEM or laser microscope to calculate the brittle and ductile areas. If the brittle area is less than 20 % of the total ligament area, the failure is ranked as ductile and the specimen is discarded.

The actual ligament area (A_L) after failure is measured using a suitable optical device (5.6).

9 Expression of results

9.1 Calculation of the actual tensile stress

The actual tensile stress applied, $\sigma_{L,}$ is calculated using Formula (2):

$$\sigma_L = \frac{9{,}81 \times R \times M}{A_L} \tag{2}$$

where

σ_L is the actual tensile stress applied, in megapascals;

A_L is the measured ligament area, in square millimetres;

M is the mass, in kilograms, of the weights used to apply the load;

R is the lever-arm ratio.

9.2 Calculation of time to failure at the reference tensile stress

The time to failure of each test specimen t_f is plotted against the actual tensile stress applied σ_L.

It is necessary that at least 25 % of actual tensile stress levels are situated below the reference tensile stress or above it, respectively. If not, the set of experimental points shall be filled in to fulfil this condition; otherwise the test is not valid.

It is recommended that the time to failure at the reference tensile stress is calculated by plotting log t_f against log σ_L. A straight line of the form of log $t_f = A \log \sigma_{L.} + B$ is then fitted to the data to determine the values of A, B and the correlation coefficient r^2.

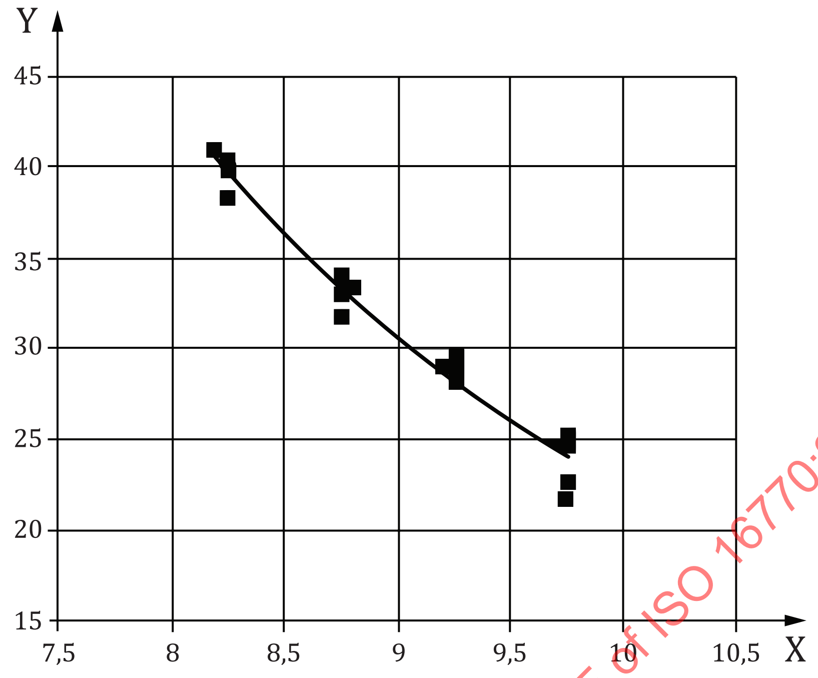
The time to failure at the reference tensile stress is then given by the antilogarithm of log ($t_{f,ref}$). See Formulae (3) and (4):

$$\log(t_{f,ref}) = A \log\left(\sigma_{L,ref}\right) + B \tag{3}$$

$$t_{f,ref} = \log^{-1}\left[\log(t_{f,ref})\right] \tag{4}$$

Alternatively, a power-law curve may be used (see Figure 7). In this case a power-law curve is fitted to the data to determine the values of the constants C, n and correlation coefficient r^2. The time to failure at the reference tensile stress $\sigma_{L,ref}$ is then given by Formula (5):

$$t_{f,ref} = C \times \left(\sigma_{L,ref}\right)^n \tag{5}$$



Key

X applied stress (MPa)

Y time to failure (h)

Figure 7 — Example of a typical stress/failure time plot (PE-grade at 50 C, test environment No.1)

The used evaluation method (log-log or power-law) shall be recorded in the test report.

10 Precision

A precision statement is given in Annex A.

Major sources of error are the following.

a) During test specimen preparation, the moulding conditions are not strictly respected.

b) If the load is applied too quickly, blunting of the notch may occur, invalidating the results.

c) The notch is too blunt after notching, and/or various parts of the notch are not coplanar.

d) The tolerances on the temperature of the test environment are not respected.

e) The environment has aged or is not homogeneous.

f) Specimens are removed from the temperature-controlled chamber while other specimens in the same chamber have not failed yet.

11 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 16770:2019;

b) all details necessary for complete identification of the test material, such as manufacturer, production data, etc.;

c) all details necessary for identification of the test specimens, such as test specimens preparation method and conditions;

d) the test specimen dimensions;

e) the method of notching used, i.e. indentation of a razor blade or broaching;

f) all details necessary for complete identification of the test environment used;

g) the temperature and concentration of the test environment;

h) the actual tensile stress, based on the measured ligament area, applied to the test specimens;

i) the time to failure (or duration of test if failure did not occur);

j) the evaluation method (log-log or power-law);

k) any deviations from the method specified in this document, such as moulding conditions;

l) the date and time of the start and end of the test.

An example of a recommended test report is given in Annex B.

Annex A
(informative)

Precision statement

A.1 General

A.1.1 Reproducibility for Type A test specimen (10 mm × 10 mm cross-section)

A.1.1.1 Interlaboratory tests were performed in 2008 with 10-mm thick test specimen, machined from compression moulded sheets (see Table 1, type A). The aim of the tests was to verify modified compression moulding conditions used. These conditions gave more homogeneous test results, compared to results obtained using moulding conditions from the previous edition of this document (i.e. ISO 16770:2004). The results have subsequently been confirmed by smaller-scale interlaboratory testing performed in 2013.

A.1.1.2 The 2008 interlaboratory tests included one PE-HD extrusion material and 13 laboratories from eight European countries. All the test specimens were compression moulded and notched by one laboratory, which also distributed the detergent for dilution to all participants. The tests strictly followed the ISO 16770:2004 requirements. The moulding conditions as given in Table 2 for specimen type A were used. Three test specimens were tested per each nominal tensile stress level (3,25 MPa, 3,75 MPa, 4,25 MPa and 4,75 MPa), i.e. a total of 12 test specimens tested by each participant.

A.1.1.3 The 2013 interlaboratory tests verified the 2008 test results. These tests included the identical PE-HD material and identical test conditions. The tests were conducted by five laboratories from five European countries. An improved moulding machine was used for compression moulding the test sheets, enabling control of the moulding parameters, namely cooling rate. Due to insufficient testing points, a full statistics analysis was not performed; however, a lower standard deviation of the average values compared to 2008 results was achieved.

A.1.1.4 Table A.1 is based on interlaboratory tests performed in 2008. Due to the nature of the FNCT method, a relevant statistical evaluation (95 %-repeatability limit r) could be derived for this test, only considering the following assumption: failure times for individual stress levels from regression analysis were taken as arithmetic mean values with corresponding confidence intervals. Based on these principles, the basic statistics for FNCT were determined and repeatability identified.

A.1.1.5 Table A.2 is based on verification tests performed in 2013 using identical material and test conditions.

A.1.2 Reproducibility for type B test specimen (6 mm × 6 mm cross-section)

A.1.2.1 A partial precision statement was included in ISO 16770:2004, based on interlaboratory tests conducted in 2000 on test specimens machined from compression moulded sheets (see Table 1, type B). Moulding conditions were used as given in Table 1. In 2005, the 2000 interlaboratory tests results were verified by a smaller-scale testing programme conducted by four European laboratories. A similar PE-HD material was used. The test conditions were in both cases identical - three test specimens were tested per each nominal tensile stress level of 8,25 MPa, 8,75 MPa, 9,25 MPa and 9,75 MPa, i.e. a total of 12 test specimens tested by each participant. In both interlaboratory testing programmes, unnotched test specimens were distributed by one laboratory to the participants for notching and testing, the same as the detergent for dilution.

A.1.2.2 From the 2000 interlaboratory tests: Repeatability for type B test specimens at four different stresses was identified. For a reference tensile stress of 9 MPa, the failure time was 30,9 h with 95 % confidence limits of ±0,5 h. The standard deviation from the regression line was 1 h.

A.1.2.3 From the 2005 interlaboratory tests: The repeatability for Type B test specimens at four different stress levels was identified for similar PE-HD material. For a reference tensile stress of 9 MPa, the failure time was 24, 2 h. The confidence limits and standard deviation from the regression line were confirmed. Therefore, the compression moulding conditions as defined in ISO 16770:2004 have been found satisfactory and are maintained in this document.

A.2 Statistical evaluation

CAUTION — Due to a limited number of laboratories, materials and the specifics of the FNCT, the following explanation of *r* (see A.2.1) is only intended to present a meaningful way of considering the approximate precision (repeatability) of this test method. The data in Tables A.1 and A.2 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the interlaboratory tests and may not be representative of other conditions, materials or laboratories.

A.2.1 Concept of *r*

Repeatability: two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the *r* value for that material. *r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment in the same laboratory.

The judgements in Tables A.1 and A.2 will have an approximately 95 % (0,95) probability of being correct.

A.2.2 Statistical properties

In Tables A.1 to A.3, the statistical properties used are:

s_r within laboratory standard deviation

r 95 % repeatability limit = 2,8 s_r

n number of laboratories

A.2.3 Statistical parameters for Type A test specimen

Table A.1 — 2008 Precision data for FNCT failure times $t_{f,ref}$ for σ_f = 4 MPa

Values in hours

Material	Evaluation	n	Failure time *h*	s_r *h*	*r*
PE-HD1	Power-law function	13	86,4	9,6	26,8
	Log-log scale	13	84,6	6,5	18,3

Table A.2 — 2013 Confirmation of 2008 data for FNCT failure times $t_{f,ref}$ for σ_f = 4 MPa

Values in hours

Material	Evaluation	n	Failure time *h*	s_r *h*	*r*
PE-HD1	Power-law function	5	81,9	2,13	6,0
	Log-log scale	5	81,9	2,07	5,8

A.2.4 Statistical parameters for Type B test specimen

Table A.3 — Precision data for FNCT failure times $t_{f,ref}$ for σ_f = 9 MPa

Material	Evaluation	n	Failure time *h*	s_r *h*	$s_{r, rel}$ %
PE-HD2	Log-log scale	8	30,9	5,3	17,1
PE-HD3	Log-log scale	4	24,2	3,4	14,0

