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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)

Reference number
ISO 16770:2004(E)

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Foreword

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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

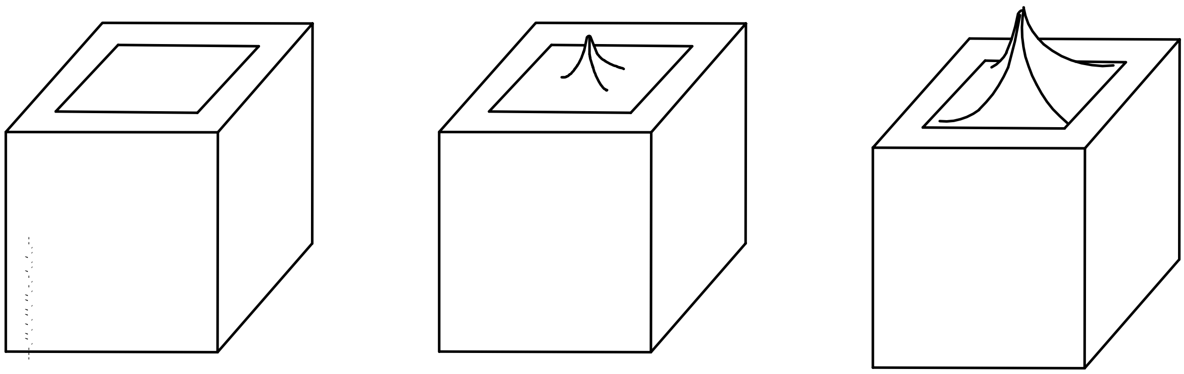
NOTE The description of the failure surface has been simplified in this International Standard (see 3.2 and 3.3). Further information is available in the literature (see the Bibliography).

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.

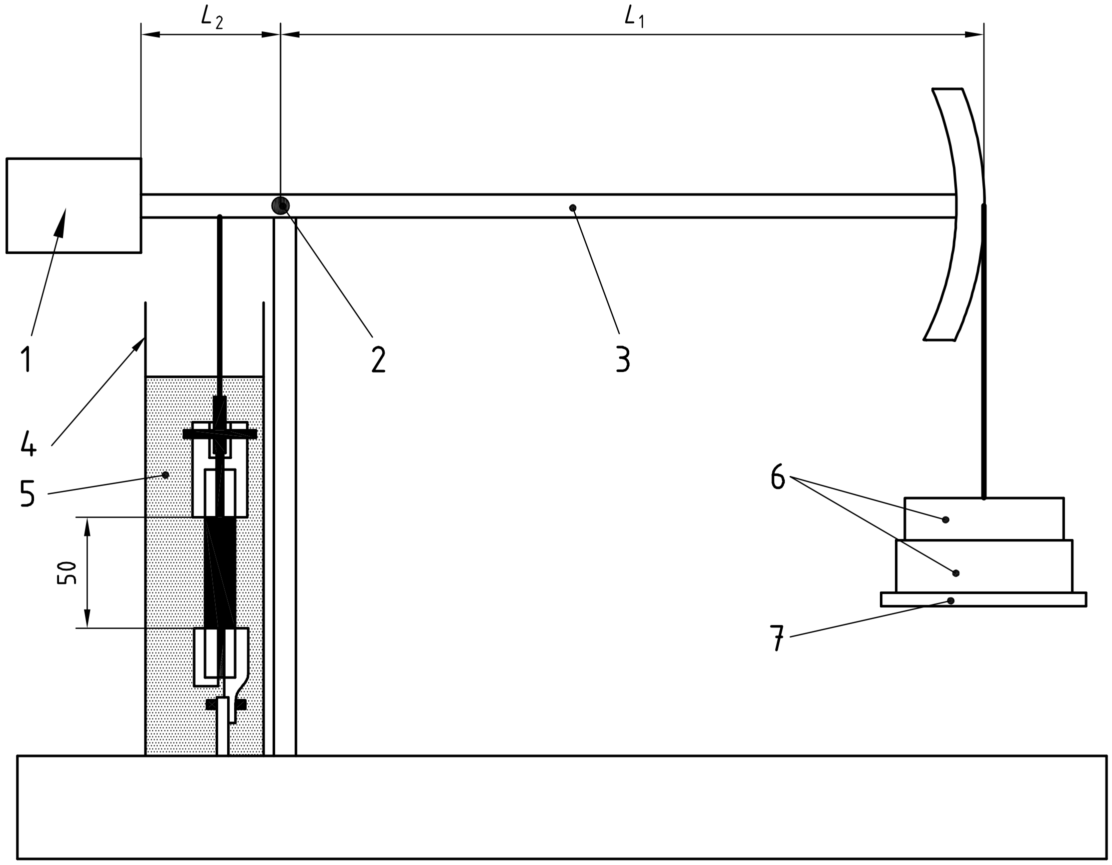
In addition to the above example, the tensile load may be applied directly using deadweights, pneumatically actuated loading or any other means of producing a constant load. The loading device shall be capable of applying the load to an accuracy of ± 1 %. The balanced loading apparatus as described in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

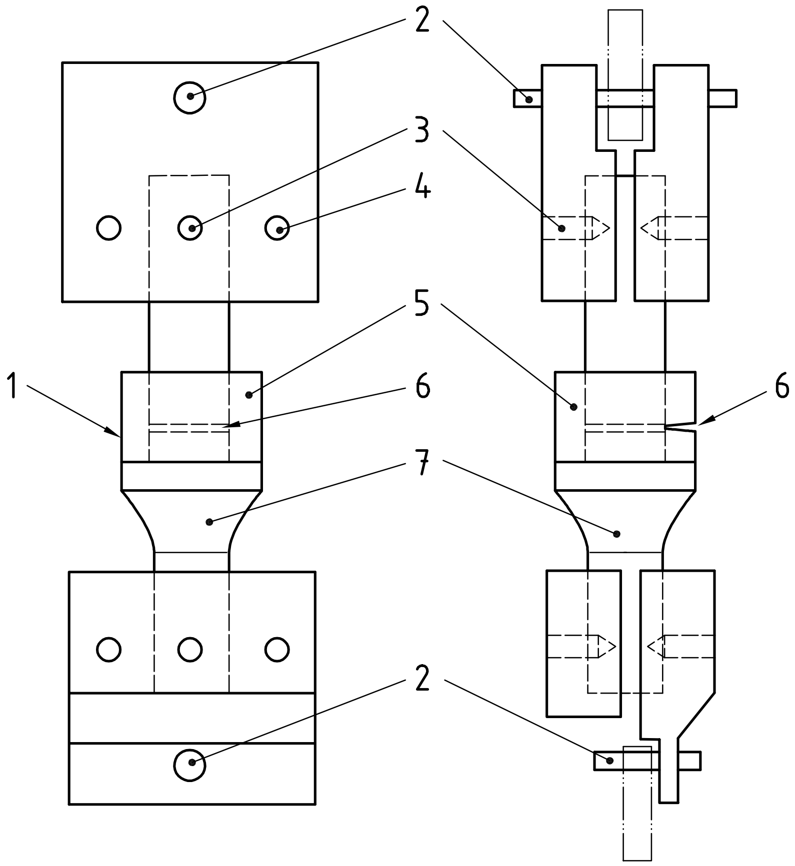
Dimensions in millimetres



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

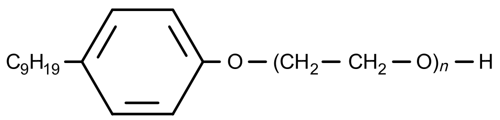
A microscope is required to allow accurate measurement of the actual ligament size (distance between the tips of the notches) after failure. It shall read to an accuracy of ± 100 µm.

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.

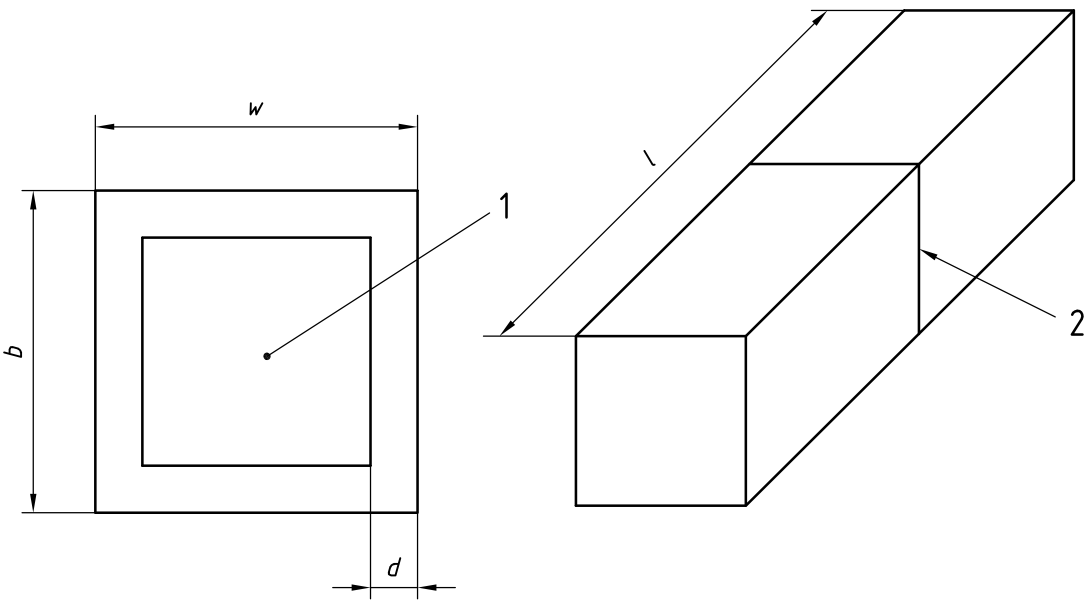
6.2 Other environments

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, especially above 80 °C, can give different results due to absorption, chemical attack or crystalline changes in the polyethylene itself, and this shall be taken into consideration when carrying out the test.

7 Preparation of test specimens

7.1 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.



Key

1 ligament area

2 notch

w width

b thickness

l length

d notch depth

Figure 4 — Test specimen showing notch and ligament area

7.2 Test specimen preparation

Except when testing finished products, prepare test specimens from compression-moulded sheet. ISO 1872-2:1993 (Subclause 3.3), ISO 11542-2:1998 (Table 1) and ISO 293 all give general guidelines for moulding and cooling conditions. However, thick sheets require the use of the conditions specified in Table 1 below. The use of different moulding conditions will affect the results. Machine the test specimens to size from the moulded sheet in accordance with ISO 2818 at least 24 h after moulding. Trim the specimen edges of any remaining swarf left from machining. When comparing finished products, cut the test specimens from extruded or moulded goods in accordance with ISO 2818. Always check the relevant product standard for any further details.

Table 1 — Conditions for compression moulding of test specimens

Thickness mm	Moulding temperature °C	Average cooling rate °C/min	Preheating time min	Full pressure MPa	Full-pressure time min
6	180	15 ± 2	20	5	10
10	180	2 ± 0,5	45	10	25
Demoulding temperature < 40 °C. Preheating pressure = contact pressure.					

If the material is a powder, it may be deemed 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.

7.3 Test specimen notching

Specimens shall be notched at room temperature. Due care shall be taken to avoid blunting the notch during this operation, e.g. avoid the use of excessive speed/force, as this will invalidate the results. If a razor blade is used, it shall be used for producing no more than a hundred notches. Whatever device is used for notching, the tolerance on the required notch depth is ± 0,1 mm.

NOTE Notch integrity may be checked microscopically.

7.4 Conditioning of test specimens

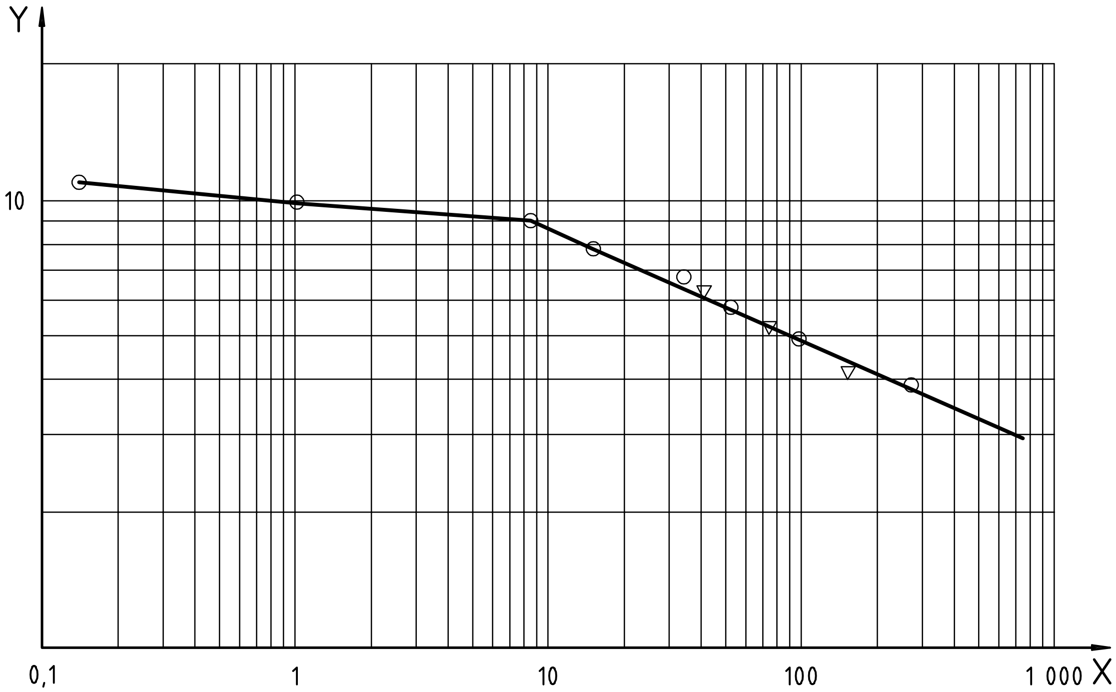
Normally, notched test specimens shall be stored at (23 ± 2) °C, although (27 ± 2) °C may be used in tropical countries. When they are required for use at other temperatures, they shall be conditioned in the environment at the test temperature for (10 ± 2) h after clamping in the loading apparatus and prior to loading.

8 Test procedure

8.1 Choice of stress and temperature

For a known material or category of material, select from Annex A a stress and temperature that will cause brittle failure of the test specimens. Test at least four specimens, at nominal stresses above and below the selected value; this is to compensate for variation in the ligament area introduced during the notching operation. For example, having selected a stress of 9 MPa, a series of nominal stress values, such as 8,25 MPa, 8,75 MPa, 9,25 MPa and 9,75 MPa, could be used to give a spread above and below 9 MPa.

If an unknown polyethylene material is being tested, then it is useful to map out its behaviour over a broad range of stresses at an appropriate temperature. A typical example of this approach is shown in Figure 5.



Key

X failure time (h)
Y actual stress (MPa)
O broached notch
∇ razored notch

Figure 5 — Typical stress/failure time plot

8.2 Calculation of test load

Calculate the test load from the equation:

$$M = \frac{A_\text{n} \times \sigma}{9{,}81R}$$

where

- M is the mass, in kilograms, of the weights used to apply the load;
- A_n is the nominal ligament area, in square millimetres;
- σ is the required 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.

8.3 Application of the load to the test specimen

Place a notched test specimen in the grips of the lever-arm loading machine (see Figures 2 and 3), taking care to avoid bending or twisting the specimen. Position the specimen with half its length exposed between the grips and with the notch plane located at the centre. Immerse the specimen, held in the grips, in the environment, ensuring that the notched area is in contact with the environment, and condition it in accordance with 7.4. After conditioning, gradually apply the calculated load to the lever-arm, avoiding shock loading of the specimen. At the same time, start the timing device.

If the specimen has been cut from a finished product, it may contain internal stresses and be a little bent. Refer to the particular product standard for further guidance.

NOTE 1 It has been found convenient to lower the weight carrier using a suitable jack or other means.

NOTE 2 Lower temperatures will increase the time to failure of the test specimen. Higher temperatures will decrease the time to failure, but if too high a temperature is used changes in crystallinity and possibly also oxidative ageing may occur. The same will apply when different environments are used.

8.4 Calculation of results

Examine the fracture surface of each test specimen to ensure that the fracture is of the brittle type (see Figure 1). Measure the dimensions of the ligament using a travelling microscope and calculate the ligament area. Calculate the actual stress applied, σ_L, from the equation:

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

where

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;

σ_L is the actual tensile stress applied, in megapascals.

Plot the time to failure t_f against the actual stress applied σ_L and fit a power-law curve of the form

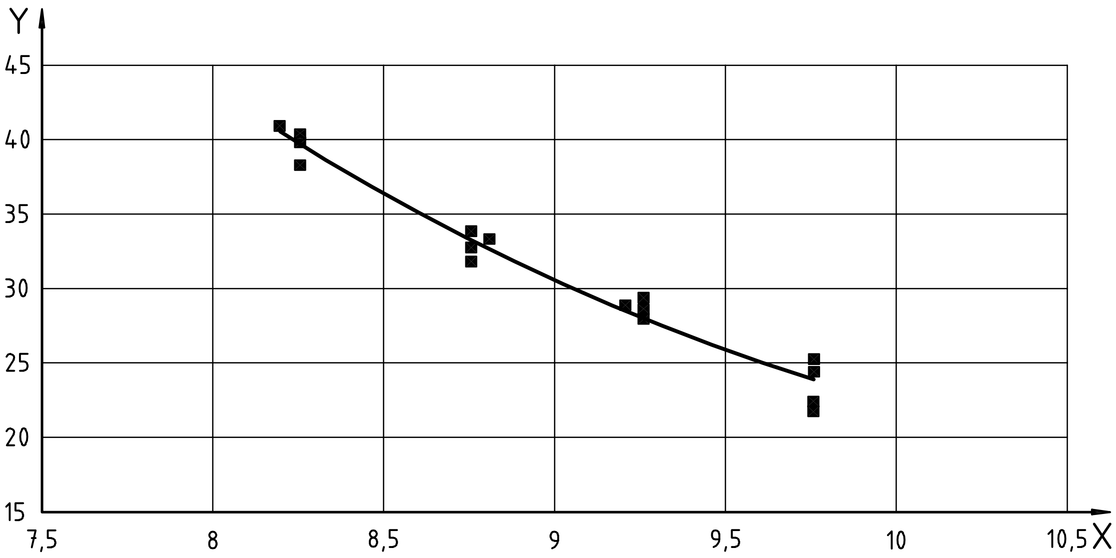
$$t_f = C \times (\sigma_L)^n$$

to the data to determine the values of the constants C and n. The time to failure at the reference stress σ_{ref} (see Annex A) is then given by $C \times (\sigma_L)^n$.

Alternatively, plot log t_f against logσ_L fit a straight line of the form log $t_f = A$ log$\sigma_L + B$ to the data to determine the values of A and B. The time to failure at the reference stress is then given by the antilogarithm of log t_f, where log $t_f = A$ log$\sigma_L + B$.

9 Repeatability and reproducibility

The precision of this test method is not known because sufficient interlaboratory data are not yet available. Interlaboratory data are being obtained, and an indication of the repeatability is given in Figure 6. A full precision statement will be added at the next revision, when the work is complete. Figure 6 illustrates the repeatability for five specimens tested at four different stresses. For a reference stress of 9MPa, the failure time is given as 30,5 h with 95 % confidence limits of ± 0,5 h. The standard deviation from the regression line is 1 h.



Key

X applied stress (MPa)

Y failure time (h)

Figure 6 — Data for moulding-grade PE at 50 °C in 2 % Arkopal solution

Major sources of error are listed below:

a) if the load is applied too quickly, blunting of the notch may occur, rendering the results invalid;

b) the notch is too blunt after notching;

c) the various parts of the notch are not coplanar;

d) the tolerances on the temperature of the environment are not respected;

e) the environment has aged or has not been stirred.

10 Test report

The test report shall contain the following:

a) a reference to this International Standard;

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

c) all details necessary for identification of the test specimens, e.g. cut from compression-moulded sheet, from pipe, from mouldings, etc.;

d) the specimen dimensions, as given in Annex A;

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

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

g) the temperature and concentration of the environment;

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

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

j) any deviations from the method specified in this International Standard, e.g. in the moulding conditions;

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

Annex A
(informative)

Specimen dimensions and environment

A.1 Surface-active agent

Any suitable product that meets the requirements of 6.1, e.g. Arkopal N110 (n = 11), may be used.

A.2 Test specimen dimensions and test conditions

Specimens A and B (see Table A.1), have been found suitable for very high stress crack resistant polyethylenes used, for instance, for pressure pipes and fittings. Completely brittle failures do not always occur at 95 °C, and caution must be exercised in the interpretation of the results. Table A.2 gives some indication of the expected range of failure times. Experience has shown that specimens with a cross-section below 6 mm × 6 mm are too small to achieve plane-strain conditions and thus obtain brittle failure with lower-ESC materials.

Table A.1 — Test specimens and test conditions

Test specimen	Specimen dimensions (length × width × thickness) mm (tolerance: ± 0,2 mm)	Notch depth mm	Reference stress MPa	Temperature °C
A	100 × 10 × 10	1,60	4,5	95
B	100 × 10 × 10	1,60	4,00 or 6,00	80
C	90 × 6 × 6	1,00	9,00	50
D	90 × 6 × 6	1,00	12,00	23
E	100 × 10 × 4	1,60	3,5	80
F	100 × 10 × 4	1,60	9,0	50
G	100 × 10 × 4	1,60	12,0	23

Table A.2 — Expected range of failure times

Application	Test specimen	Failure-time range h
Pipe	A or B	10 to 1 000
Moulding	C or F	5 to 100
Moulding	D, E or G	5 to 50

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