
**Notch tensile test to measure the
resistance to slow crack growth of
polyethylene materials for pipe and fitting
products (PENT)**

*Essai de traction d'une entaille pour mesurer la résistance à la
propagation lente de fissure des polyéthylènes pour tubes et raccords
(PENT)*



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Contents	Page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Apparatus	2
6 Test piece preparation	4
6.1 Test piece geometry	4
6.2 Test piece preparation	5
6.3 Test piece notching	5
6.4 Number of test pieces	6
7 Preconditioning	6
8 Procedure	6
8.1 Calculation of test mass	6
8.2 Loading the test piece	6
8.3 Checking temperature	7
8.4 Recording time to failure	7
8.5 Examination of failed test piece	7
9 Test report	7
Annex A (informative) Application of the test	8
Annex B (informative) Measurement of extension	9
Annex C (informative) Notch depth	10
Annex D (informative) Recommended minimum requirement	

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.

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ISO 16241 was prepared by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, Subcommittee SC 5, *General properties of pipes, fittings and valves of plastic materials and their accessories — Test methods and basic specifications*.

Introduction

This test method is based on ASTM F 1473 [1], which was originally developed at the University of Pennsylvania. It has been used for assessing and comparing the resistance to slow crack growth of a wide range of polyethylene pipe compounds used primarily for gas applications in the USA and Europe. The test had been evaluated by ten laboratories in the USA prior to acceptance as an ASTM standard. Further work was then carried out by an *ad-hoc* group of ISO/TC 138/SC4, *Plastics pipes and fittings for the supply of gaseous fuels*, to evaluate and refine the procedure, involving six laboratories in Europe and the USA.

Resistance to slow crack growth is an important consideration for polyethylene materials used for pipeline applications and tests to assess this property are incorporated in pressure pipe system standards. The test is also applicable to polyethylene materials used for other pipeline applications as well as to other thermoplastics materials used for other applications. Although seen primarily as a materials test, it has been applied to assess the performance of test pieces cut from pipe and fitting products and butt fusion welds.

Notch tensile test to measure the resistance to slow crack growth of polyethylene materials for pipe and fitting products (PENT)

1 Scope

This International Standard specifies a method for determining the resistance to slow crack growth of polyethylene (PE) materials for pipes and fitting products.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 293, *Plastics — Compression moulding of test specimens of thermoplastic materials*

ISO 1872-2, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

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

brittle failure

failure with a fracture surface which exhibits no visible permanent material deformation, e.g. stretching, elongation or necking down

3.2

ductile failure

failure with a fracture surface which clearly exhibits permanent material deformation with stretching, elongation and necking down

3.3

slow crack growth

slow extension of a crack with time which is of a brittle appearance

3.4

remaining ligament

remaining cross section after notching and crack growth

4 Principle

To assess the resistance to slow crack growth of a material, a test piece is cut from a compression-moulded sheet. The rectangular shaped test piece containing a centre notch in one face and side notches in the same plane is subjected to a constant static tensile load at a specified test temperature. The time to failure is recorded.

NOTE 1 The test may be carried out on test pieces machined from pipe and fitting products or butt welds (see Annex A).

The following characteristics are described in this method:

- the shape and dimensions of the test piece;
- the notch depth and the depth of the side grooves;
- the number of test pieces;
- the test temperature of the test pieces;
- the method of preparation of the test pieces.

The following parameters shall be specified by the referring standard (a standard making reference to this International Standard in its own provisions):

- the test piece thickness and notch depth if different from the preferred dimensions (see 6.1);
- the stress to be applied to the test piece for the duration of the test;
- the number of test pieces if differing from the number specified in 6.4;
- the test temperature if differing from the value specified in this method (see 5.2).

NOTE 2 The applied stress chosen is such as to preclude ductile behaviour by inducing a fracture surface which is flat, exhibiting no visible permanent material deformation (i.e. stretching, elongation or necking down). Such a surface as a result of the gradual extension of the crack with time is considered to be akin to slow crack growth. Typically, a load to give a stress level of 2,4 MPa at 80 °C will produce a mode of failure related to slow crack growth in polyethylene materials. Alternative stress levels may be used providing a similar mode of failure is produced.

5 Apparatus

5.1 Test rig, consisting of the following.

5.1.1 Test machine, capable of applying, via grips (see 5.1.2) an axial load on the test piece controlled with an accuracy of within 0,5 % of the calculated or applied load, e.g. a lever-loading machine, with a lever arm ratio of around 5:1 to 10:1 be applied directly using dead weights or a constant load.

5.1.2 Grips, having a low friction universal action to prevent bending and to ensure that the load is transmitted axially through the test piece. The grips shall be designed to prevent slippage of the test piece (e.g. serrated grips).

NOTE Measurement of the extension of the test piece or movement of the lever arm provides useful information. The rate of extension of the test piece will increase when initiation of the crack has occurred and will increase rapidly when failure is imminent (see Annex B).

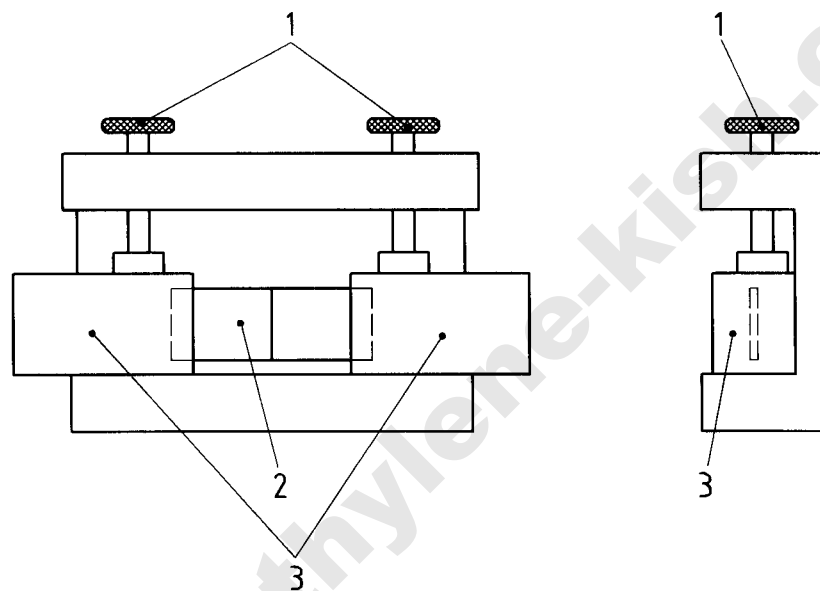
5.2 Heating chamber or oven, where the loading frame shall be located with air circulation by a fan or other means to maintain the test piece at $(80 \pm 1,0)$ °C or an alternative test temperature specified by the referring standard.

5.3 Temperature measuring device, i.e. calibrated thermometer or a thermocouple, capable of checking conformity to 5.2.

NOTE Temperature is a critical parameter and measurement made remote from the test piece may not be indicative of temperature of the test piece.

5.4 Clock or timing device, for recording the elapsed time, which shall automatically stop or record the point when the test piece is deemed to fail by either fracture or excessive displacement of the grips.

5.5 Alignment jig, as shown in Figure 1 or similar, to facilitate the alignment the grips and the test piece when it is being tightened in the grips.



Key

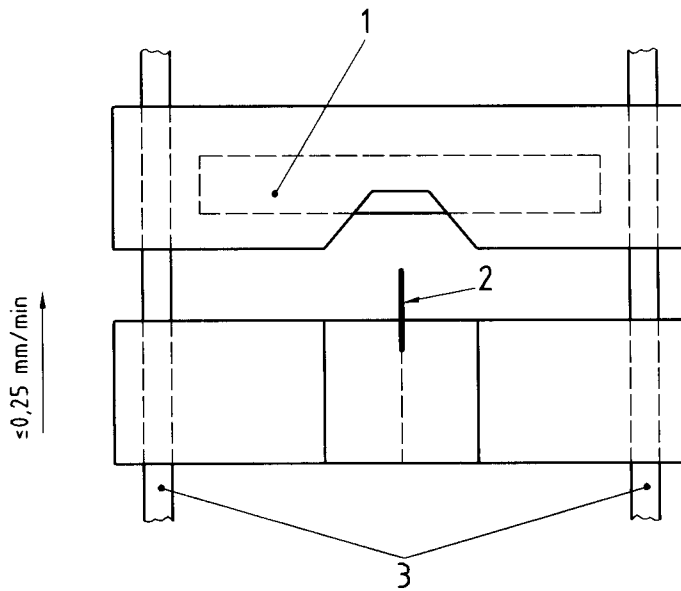
- 1 screw clamps
- 2 test piece
- 3 grips

Figure 1 — Typical arrangement for an alignment jig

5.6 Notching apparatus, for notching the test piece (see Figure 2) so that the main notch and any side notches are coplanar and the plane of the notching is perpendicular to the tensile axis of the test piece. A blade of approximate thickness of 0,15 mm to 0,25 mm shall be used for preparing the notch. The notch-machining equipment shall have a blade penetration speed of not more than 0,25 mm/min and be capable of producing a specified notch depth to within $\pm 0,1$ mm.

NOTE The indicated notch depth can be checked by notching and measurement of a dummy test piece.

5.7 Microscope, suitable for measuring the notch depth and examining the fracture surface.



Key

- 1 test piece
- 2 razor blade
- 3 guides

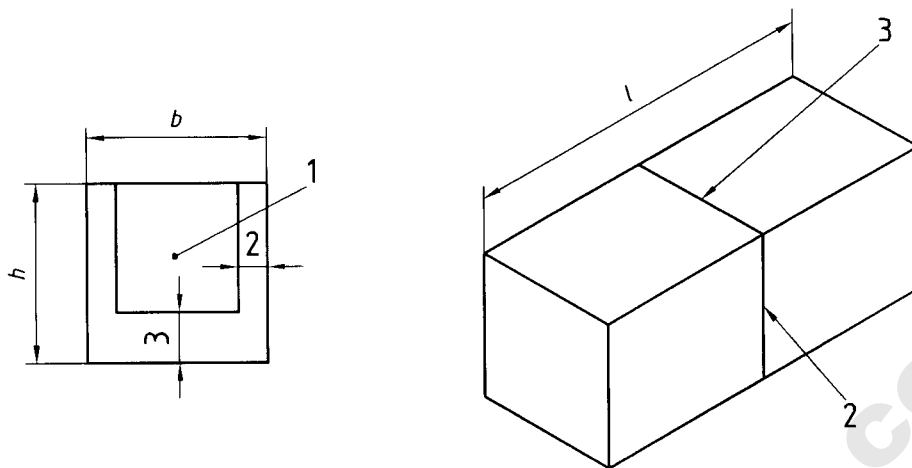
Figure 2 — Typical arrangement for apparatus for notching test pieces

6 Test piece preparation

6.1 Test piece geometry

The test piece geometry is shown in Figure 3, with a preferred nominal width, b , not less than 25 mm and not greater than the width of the grips used, a thickness, h , of a nominal 10 mm, a principal notch depth (3) of 3,5 mm and with side notch depths (2) of 1 mm. Alternative thickness samples with appropriate notch depths are given in Table C.1. The depth of the principal notch for test pieces of thickness other than those given in the table may be obtained by linear interpolation. The length of the test piece, l , shall be sufficient to allow at least 10 mm between the end of each grip and the notch, see 8.2.

NOTE Significant variation from the preferred width could influence the result.



Key

- l* length
- b* width
- h* thickness (sheet thickness)
- 1 ligament area
- 2 side notch
- 3 principal notch

Figure 3 — Test piece geometry

6.2 Test piece preparation

Test pieces shall be prepared from compression-moulded sheets made from the material to be tested. Alternatively, test pieces can be prepared from pipe and fitting products or butt welds (see Annex A).

For the compression moulding of the sheets from polyethylene materials, general instructions are given in ISO 1872-2 and ISO 293, but the following specific procedure shall be applied.

Heat the material in granular form in the range of 140 °C to 160 °C and then apply and remove the pressure three times. Increase the temperature in the range of 170 °C to 190 °C for between 10 min and 15 min without pressure. Then apply and remove the pressure three times in order to eliminate voids. Turn off the heat and apply a nominal pressure (force per unit area) of 1,7 MPa. Allow cooling at a rate such that either the time to cool between 130 °C and 90 °C is greater than 80 min or so that the time to cool from the moulding temperature to about room temperature is greater than 5 h. During cooling the pressure shall be maintained.

After allowing at least one day of storage of the sheet following moulding, the test pieces shall be machined from the sheet to the required geometry (see 6.1) in accordance with ISO 2818.

NOTE 1 Higher moulding temperatures of ≤ 200 °C are advised for lower MFR materials, i.e. those of MFR $\leq 0,5$ (5 kg load, Condition T, in accordance with ISO 1133).

NOTE 2 The cooling conditions specified are crucial and any deviation from the specified conditions could influence the result of the test. The moulding and thermal processing cycle specified above might not be representative of the manufacturing process to which the material will be subjected.

6.3 Test piece notching

The principal notch shall be machined by pressing a sharp blade, e.g. a razor blade with a blade tip radius of less than 10 μm , into the specimen at a speed of not more than 0,25 mm/min; the tolerance on the required

notch depth is $\pm 0,1$ mm. Each blade shall be used for one day only and for notching not more than six test pieces. The side notches (see Figure 3) shall be coplanar to the principle notch. Specimens shall be notched at room temperature. If stored prior to testing, the test piece shall be kept at (23 ± 2) °C according to ISO 291, prior to conditioning. The depth of the notch shall be checked and recorded by examining the side of the test piece with a microscope prior to testing.

NOTE 1 For the preparation of the side notches, the notching rate is of no importance.

NOTE 2 The accuracy of the notching equipment to give the required notch depth can be checked by notching a dummy test piece.

6.4 Number of test pieces

Unless otherwise specified in the referring standard, a minimum of three test pieces shall be prepared and used.

7 Preconditioning

Unless otherwise specified in the referring standard, the preferred test piece (thickness 10 mm, see 6.1) shall be preconditioned prior to loading (see 8.2) at (80 ± 1) °C, or at an alternative test temperature specified by the referring standard, for $6 \text{ h} \pm 30 \text{ min}$, whilst set in the clamps and located in the test rig.

If applicable, test pieces of different geometry will require different conditioning periods dependent on thickness. The conditioning periods related to thickness detailed in ISO 1167 should be applied.

8 Procedure

8.1 Calculation of test mass

Calculate the test mass, m , using the following equation:

$$m = \sigma \times b \times h / 9,807$$

where

σ is the stress, in megapascals (MPa);

b is the measured test piece width, in millimetres (mm);

h is the measured test piece thickness, in millimetres (mm).

If a lever-loaded machine is used, the value of the mass m shall be divided by the lever arm ratio, if applicable.

NOTE The variables b and h are based on the un-notched cross section in the plane of the notch.

8.2 Loading the test piece

Using an alignment jig (see 5.5), centre the test piece in the grips so that the axis of the test piece is aligned with the grips. The ends of the grips shall be at least 10 mm from the notch. Tighten the grips without causing any bending or twisting of the test piece.

Transfer the combination of the grips and test piece from the alignment jig to the test rig. Ensure that the required preconditioning time at the test temperature is observed (see Clause 7). Apply m , to within $\pm 0,5$ %, gradually and within a period of between 5 s to 10 s, without causing any shock loading of the test piece and then activate the timing device.

In a multi-station test rig, care should be taken not to disturb other test stations when loading test specimens or during the period of testing.

Contamination of the test piece with fluids such as detergents should be avoided.

8.3 Checking temperature

Place a thermocouple or thermometer close enough to the notched part of the test piece to enable checking and recording of the temperature at regular intervals to ensure conformity to 5.2.

8.4 Recording time to failure

When the test piece fails, record the time to failure. Failure is indicated by division of the test piece into two separate pieces or when elongation and stretching occurs in the remaining ligament, (see Annex B).

8.5 Examination of failed test piece

After allowing to cool to room temperature, examine the failed test piece with a microscope in order to measure and record the depth of the main notch to within $\pm 0,1$ mm, if possible.

Record the failure type:

- a) completely separated at the notch of the two halves of test piece (brittle failure);
- b) crack growth and deformation in the remaining ligament;
- c) extensive material deformation with no evidence of crack growth [(e.g. extension, elongation, stretching), (ductile failure)].

NOTE Failure type c) is indicative of carrying out the test at too high a stress for the material concerned.

9 Test report

The test report shall include the following information:

- a) reference to this International Standard and to the referring standard;
- b) complete identification of the material tested, including type, source, manufacturer's designation, test piece (manufacturer, method of manufacture, production date);
- c) stress based on the cross-sectional area of the un-notched test piece;
- d) depth of the main notch and of the side grooves recorded prior to the test, and afterwards if measurement is possible;
- e) calculated load and cross-sectional dimensions of test piece;
- f) test temperature and any recorded variation;
- g) time to failure and type of failure (separation or extension) for each test piece or the duration of the test if failure has not occurred;
- h) any factors which may have affected the results, such as any incidents or any operating details not specified in this International Standard;
- i) date and time of start and end of test.

Annex A (informative)

Application of the test

This method could be used to determine the resistance to slow crack growth of other thermoplastics providing suitable test parameters are established.

It may be used to measure the resistance to slow crack growth of test pieces taken from pipe and fitting products or butt fusion welds. Specimens should be taken from products in such a way that the machined notch reflects the direction in which crack growth could occur in service. However, it is noted that processing thermal history and geometry could influence the result and therefore the test result on such test pieces might not be comparable to the result obtained on test pieces taken from compression-moulded sheet using the same material.

To obtain test pieces from pipe, specimens should be cut oversize from the pipe to allow residual stresses from manufacture to relax for a period of time of at least 12 h before finally machining to the required dimensions. The main notch should be machined in the surface closest to the bore of the pipe.

NOTE It is important that the notch is in the heat-affected zone in the interface between the two pipes in test pieces taken from butt welds.

Annex B (informative)

Measurement of extension

Measurement of extension provides a useful indication of imminent failure. This is easily achieved by fitting a dial gauge or a displacement transducer to monitor extension by increasing displacement of a lever arm or a deadweight load. The extension with time can be plotted and a steep change in slope of the curve provides an indication of imminent failure.

Annex C
(informative)

Notch depth

Table C.1 gives notch depth as a function of test piece thickness. A 10 mm thickness is preferable but alternative thickness test pieces with the appropriate notch depth may be selected from Table C.1 (see 6.1). The preferred width of the test piece is 25 mm, see 6.1. Thinner test pieces may promote ductile behaviour in some materials.

Table C.1 — Notch depth as function of test piece thickness

Dimensions in millimeters

Thickness	Notch depth	Thickness	Notch depth
4,00	1,90	13,00	4,20
5,00	2,30	14,00	4,40
6,00	2,50	15,00	4,50
7,00	2,80	16,00	4,70
8,00	3,10	17,00	4,90
9,00	3,30	18,00	5,00
10,00	3,50	19,00	5,10
11,00	3,70	20,00	5,20
12,00	3,90		

Annex D (informative)

Recommended minimum requirements for polyethylene

The following information is offered for guidance as a recommended minimum requirement for polyethylene (PE) materials intended for pressure pipe applications.

Table D.1 — Recommended test parameters and requirements for PE 80 and PE 100

Material class	Nominal stress MPa	Temperature °C	Requirement h
PE 80	2,4	80	≥ 100
PE 100	2,8	80	≥ 100

NOTE The PE 80 material used to determine this requirement was the same as that used to determine the recommended requirements in ISO 13479 and is equivalent to a 165 h requirement in that test.

Annex E
(informative)

Precision

Interlaboratory trials (round robins) conducted with ten laboratories in the USA and a further six laboratories in Europe gave a standard deviation of the test results within laboratories which was less than 15 %. The standard deviation of the average values from the different laboratories is less than 17 %. With a confidence level of 95 %, it is concluded that the precision of tests carried out within laboratories are not significantly different.

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Bibliography

- [1] ASTM F 1473, *Standard test method for notch tensile test to measure the resistance to slow crack growth of polyethylene pipes and resins*
- [2] ISO 1167, *Thermoplastics pipes for the conveyance of fluids — Resistance to internal pressure — Test method*
- [3] ISO 1133, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*
- [4] ISO 13479, *Polyolefin pipes for the conveyance of fluids — Determination of resistance to crack propagation — Test method for slow crack growth on notched pipes (notch test)*

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