INTERNATIONAL STANDARD

ISO 11542-2

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Plastics — Ultra-high-molecular-weight polyethylene (PE-UHMW) moulding and extrusion materials —

Part 2:
Preparation of test specimens and determination of properties

Plastics — Matériaux à base de polyéthylène à très haute masse moléculaire (PE-UHMW) pour moulage et extrusion —

Partie 2: Préparation des éprouvettes et détermination des propriétés

Reference number
ISO 11542-2:1998(E)

Foreword

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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.

International Standard ISO 11542-2 was prepared by Technical Committee ISO/TC 61, *Plastics,* Subcommittee SC 9, *Thermoplastic materials.*

ISO 11542 consists of the following parts, under the general title *Plastics — Ultra-high-molecular-weight polyethylene (PE-UHMW) moulding and extrusion materials*:

— *Part 1: Designation system and basis for specifications*

— *Part 2: Preparation of test specimens and determination of properties*

Annexes A and B form an integral part of this part of ISO 11542.

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Plastics — Ultra-high-molecular-weight polyethylene (PE-UHMW) moulding and extrusion materials —

Part 2:
Preparation of test specimens and determination of properties

1 Scope

This part of ISO 11542 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of PE-UHMW moulding and extrusion materials. Requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing are given here.

Procedures and conditions for the preparation of test specimens and procedures for measuring properties of the materials from which these specimens are made are given. Properties and test methods which are suitable and necessary to characterize PE-UHMW moulding and extrusion materials are listed.

The properties have been selected from the general test methods in ISO 10350-1. Other test methods in wide use for or of particular significance to these moulding and extrusion materials are also included in this part of ISO 11542, as are the designatory properties specified in part 1.

In order to obtain reproducible and comparable test results, it is necessary to use the methods of preparation and conditioning, the specimen dimensions and the test procedures specified herein. Values determined will not necessarily be identical to those obtained using specimens of different dimensions or prepared using different procedures.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 11542. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 11542 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 62:—[1)], *Plastics — Determination of water absorption.*

ISO 75-1:1993, *Plastics — Determination of temperature of deflection under load — Part 1: General test method.*

ISO 75-2:1993, *Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite.*

ISO 178:1993, *Plastics — Determination of flexural properties.*

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1) To be published. (Revision of ISO 62:1980)

ISO 179-1:— [2], *Plastics — Determination of Charpy impact properties — Part 1: Non-instrumented impact test.*

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO 527-1:1993, *Plastics — Determination of tensile properties — Part 1: General principles.*

ISO 527-2:1993, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics.*

ISO 527-4:1997, *Plastics — Determination of tensile properties — Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastics composites.*

ISO 899-1:1993, *Plastics — Determination of creep behaviour — Part 1: Tensile creep.*

ISO 1183:1987, *Plastics — Methods for determining the density and relative density of non-cellular plastics.*

ISO 1210/IEC 60695-11-10:— [3], *Determination of the burning behaviour of horizontal and vertical specimens in contact with a small-flame (50 W) ignition source.*

ISO 1628-3:1991, *Plastics — Determination of viscosity number and limiting viscosity number — Part 3: Polyethylenes and polypropylenes.*

ISO 2818:1994, *Plastics — Preparation of test specimens by machining.*

ISO 3146:— [4], *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods.*

ISO 3167:1993, *Plastics — Multipurpose test specimens.*

ISO 4589-1:1996, *Plastics — Determination of burning behaviour by oxygen index — Part 1: Guidance.*

ISO 4589-2:1996, *Plastics — Determination of burning behaviour by oxygen index — Part 2: Ambient-temperature test.*

ISO 8256:1990, *Plastics — Determination of tensile-impact strength.*

ISO 10350:1993, *Plastics — Acquisition and presentation of comparable single-point data.*

ISO 11542-1:1994, *Plastics — Ultra-high-molecular-weight polyethylene (PE-UHMW) moulding and extrusion materials — Part 1: Designation system and basis for specifications.*

IEC 60093:1980, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials.*

IEC 60112:1979, *Method for determining the comparative and the proof tracking indices of solid insulating materials under moist conditions.*

IEC 60243-1:1998, *Electrical strength of insulating materials — Test methods — Part 1: Tests at power frequencies.*

IEC 60250:1969, *Recommended methods for the determination of the permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelengths.*

IEC 60296:1982, *Specification for unused mineral insulating oils for transformers and switchgear.*

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[2] To be published. (Revision of ISO 179:1993)

[3] To be published. (Revision of ISO 1210:1992)

[4] To be published. (Revision of ISO 3146:1985)

3 Preparation of test specimens

It is essential that specimens are always prepared by the same procedure using the same conditions. The test specimens shall be prepared by compression moulding.

3.1 Treatment of the material before moulding

Before processing, no pretreatment of the material sample is normally necessary.

3.2 Compression moulding

Compression-moulded sheets shall be prepared in accordance with ISO 293 using the conditions specified in table 1. The test specimens for the determination of the properties shall be machined from the compression-moulded sheets in accordance with ISO 2818 or stamped.

Table 1 — Compression-moulding conditions

Material	Moulding temperature °C	Average cooling rate °C/min	Demoulding temperature °C	Full pressure MPa	Full-pressure time min	Preheating pressure MPa	Preheating time min
All grades	210	15	⩽ 40	10	30	5	5 to 15

A type 1 (frame) mould may be used, but it is necessary to start cooling whilst simultaneously applying the full pressure. This avoids the melt being pressed out of the frame and avoids sink marks.

For thicker sheet (≈ 4 mm), a type 2 (positive) mould has been found to work satisfactorily. The preheating time depends on the type of mould and the type of energy input (steam, electricity). For frame moulds, 5 min is usually sufficient but for positive moulds, due to the bigger mass, a preheating time of 5 min to 15 min can be necessary, especially if electric heating is used.

4 Conditioning of test specimens

Test specimens shall be conditioned in accordance with ISO 291 for at least 40 h at 23 °C ± 2 °C and (50 ± 5) % relative humidity.

5 Determination of properties

In the determination of properties and the presentation of data, the standards, supplementary instructions and notes given in ISO 10350 shall be applied. All tests shall be carried out in the standard atmosphere of 23 °C ± 2 °C and (50 ± 5) % relative humidity unless specifically stated otherwise in the tables which follow.

Table 2 is compiled from ISO 10350, and the properties listed are those which are appropriate to PE-UHMW moulding and extrusion materials. These properties are those considered useful for comparisons of data generated for different thermoplastics.

Table 3 contains those properties, not found specifically in table 2, which are in wide use or of particular significance in the practical characterization of PE-UHMW moulding and extrusion materials.

Table 2 — General properties and test conditions (selected from ISO 10350)

Property	Unit	Standard	Specimen type (dimensions in mm)	Test conditions and supplementary instructions
Mechanical properties				
Tensile modulus	MPa	ISO 527-1, ISO 527-2 ISO 527-4	See ISO 3167	Test speed 1 mm/min
Yield stress	MPa			Test speed 50 mm/min
Yield strain	%			Test speed 50 mm/min
Nominal strain at break	%			Test speed 50 mm/min
Stress at 50 % strain	MPa			Test speed 50 mm/min
Stress at break	MPa			Test speed 5 mm/min. Only to be quoted if strain at break is < 10 %
Strain at break	%			
Tensile creep modulus	MPa	ISO 899-1	See ISO 3167	At 1 h / At 1 000 h: Strain $\leqslant 0{,}5$ %
Flexural modulus	MPa	ISO 178	$80 \times 10 \times 4$	Test speed 2 mm/min
Tensile notched impact strength	kJ/m^2	ISO 8256	$80 \times 10 \times 4$ double V-notch, $r = 1$	
Thermal properties				
Melting temperature	°C	ISO 3146	Powder	Method C (DSC or DTA). Use 10 °C/min.
Temperature of deflection under load	°C	ISO 75-1, ISO 75-2	$110 \times 10 \times 4$ edgewise or $80 \times 10 \times 4$ flatwise	0,45 MPa and 1,8 MPa
Coefficient of linear thermal expansion	°C^{-1}	TMA (see ISO 10350)	Prepared from ISO 3167	Parallel / Normal: Quote the secant value over the temperature range 23 °C to 55 °C
Flammability	mm/min	ISO 1210	$125 \times 13 \times 3$	Method A — linear burning rate of horizontal specimens
Ignitability	%	ISO 4589-1, ISO 4589-2	$80 \times 10 \times 4$	Procedure A — top surface ignition
Electrical properties				
Relative permittivity	—	IEC 250	$\geqslant 80 \times \geqslant 80 \times 1$	Frequency 100 Hz and 1 MHz (compensate for electrode edge effect)
Dissipation factor	—			
Volume resistivity	Ω·m	IEC 93	$\geqslant 80 \times \geqslant 80 \times 1$	Voltage 100 V
Surface resistivity	Ω			
Electric strength	kV/mm	IEC 243-1	$\geqslant 80 \times \geqslant 80 \times 1$ / $\geqslant 80 \times \geqslant 80 \times 3$	Use 25 mm/75 mm coaxial-cylinder electrode configuration. Immerse in IEC 296 transformer oil. Use short time (rapid rise) test
Comparative tracking index	—	IEC 112	$\geqslant 15 \times \geqslant 15 \times 4$	Use solution A
Other properties				
Water absorption	%	ISO 62	$50 \times 50 \times 3$ or $\varnothing\ 50 \times 3$ disc	24 h immersion in water at 23 °C
Density	kg/m^3	ISO 1183	$10 \times 10 \times 4$	Test specimen to be taken from moulded specimen

Table 3 — Additional properties and test conditions of particular utility to PE-UHMW moulding and extrusion materials

Property	Unit	Standard	Specimen type (dimensions in mm)	Test conditions and supplementary instructions
Mechanical properties				
Elongation stress	MPa	ISO 179-1	$120 \times 15 \times 10$ double V-notch $14° \pm 2°$	See annex A
Charpy notched impact strength	kJ/m^2			See annex B
Other properties				
Viscosity number	ml/g	ISO 1628-3	Powder	

Annex A
(normative)

Method for determining the elongational stress[5] of PE-UHMW moulding material

A.1 Scope

This annex specifies a method for the determination of the elongational stress as a characterization of the melt viscosity of PE-UHMW moulding powder.

NOTE The melt flow rate of this material cannot be determined by the method specified in ISO 1133 because of its extremely high molecular weight.

A.2 Definition

A.2.1 elongational stress, $F(150/10)$**:** The tensile stress (force divided by initial cross-sectional area) required to increase the measured length of a test specimen by 600 % at 150 °C over a 10 min period.

A.3 Apparatus (see figures A.1 and A.2)

Constant-temperature heating bath containing

— a mixer with motor (1)

— a heating coil (2)

— perforated plates (3), one fitted near the bottom of the bath, the other separating the mixer and the heating coil from the specimen

— a contact thermometer (4)

— a mercury-in-glass or equivalent thermometer (5), graduated in intervals of 0,5 °C, suitable for measuring temperatures within the range 150 °C $\pm$ 2 °C

— a stand (6) and clamps for supporting the specimen in its holder

— a specimen holder (7), in accordance with figure A.2, with arresting device (10)

— the test specimen (8)

— a set of weights (9), with hooks for suspension from the specimen holder such that the height of the weight, including its hook, is 41,5 mm in each case (for the masses of the weights, see table A.1)

— a heating-bath liquid (11)

— a stopwatch, accurate to 0,1 s

— measuring instruments, accurate to 0,02 mm, for measuring the width and thickness of the narrow parallel-sided section of the test specimen

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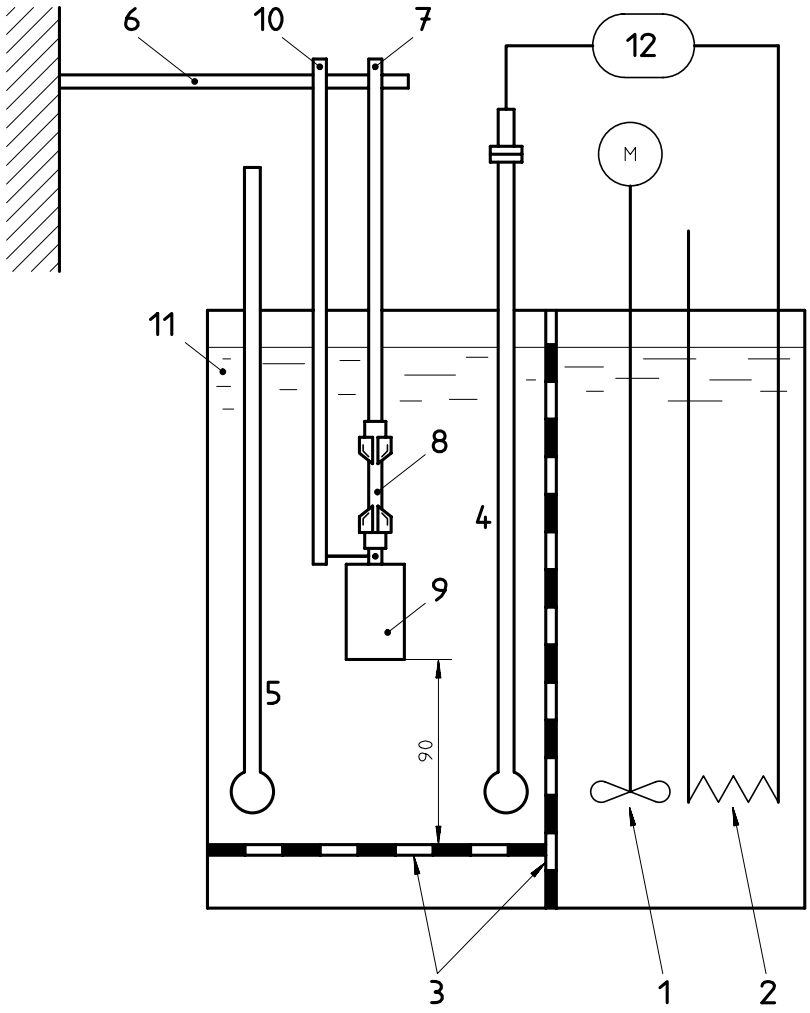
5) This property has been termed “flow value” in the past.

Table A.1 — Masses, in grams, of weights used to load specimen

100	120	150	180	200	250	300	350	400	500	600	700	800

Dimensions in millimetres

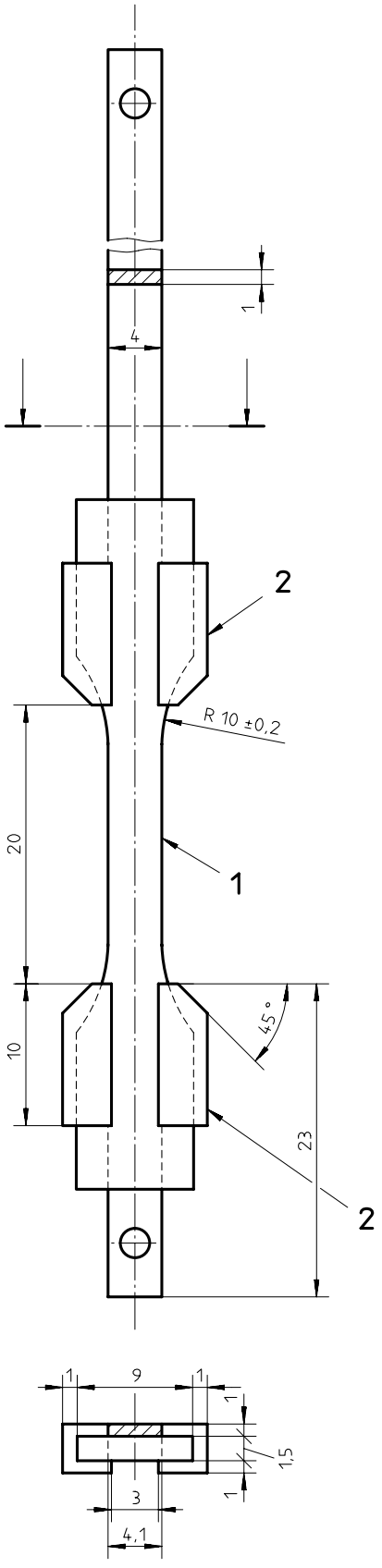


Key

1 Mixer with motor (M)
2 Heating coil
3 Perforated plates
4 Contact thermometer
5 Mercury-in-glass thermometer or equivalent
6 Stand
7 Specimen holder
8 Test specimen
9 Weight
10 Arresting device
11 Heating-bath liquid
12 Temperature-control unit

Figure A.1 — Schematic diagram of apparatus for determining elongational stress

Dimensions in millimetres



Key

1 Specimen

2 Clamp

Figure A.2 — Specimen holder

Dimensions in millimetres

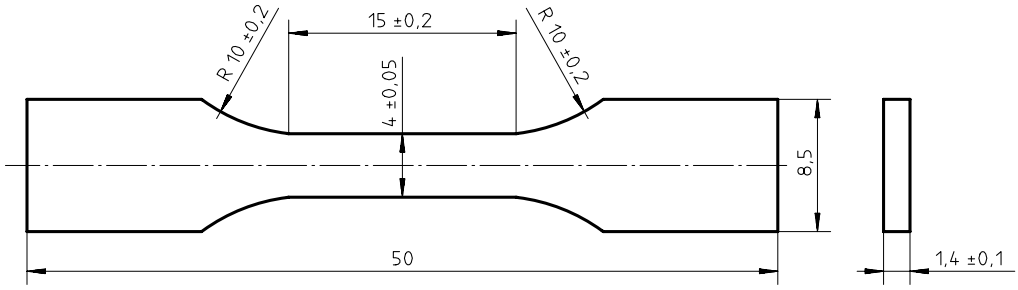


Figure A.3 — Test specimen

A.4 Compression moulding of sheet

Use the conditions specified in table 1 of this part of ISO 11542. Mix stabilizer, at a concentration capable of reducing crosslinking, homogeneously into the moulding powder. If the PE-UHMW resin or the stabilizer is in granular or pellet form, grind or pulverize it so that a homogeneous mixture is obtained.

A.5 Procedure

A.5.1 **Test specimens** (see figure A.3)

Punch six specimens out of the same sheet. Each one is for use with a different weight.

A.5.1.1 Measurement of cross-section

Measure the width and thickness of the narrow parallel-sided section of each of the six test specimens to the nearest 0,02 mm. Record the measurements.

A.5.2 Determination

A.5.2.1 Fill the heating bath with a suitable liquid (e.g. silicone oil) and raise the temperature to 150 °C ± 2 °C.

A.5.2.2 Clamp one of the test specimens in the holder as shown in figure A.2, hook a weight to the holder and suspend the specimen and weight in the bath liquid as shown in figure A.1 with the holder arrested by the arresting device so that the specimen in not loaded by the weight. Ensure that the base of the weight is 90 mm ± 1 mm above the bottom perforated plate.

A.5.2.3 Five minutes after the specimen has entered the bath liquid, free the holder from the arresting device and simultaneously start the stopwatch.

A.5.2.4 At the moment that the descending weight touches the perforated plate, stop the watch and record the time (i.e. the time needed to reach 600 % elongation of the narrow parallel-sided section of the test specimen).

A.5.2.5 Repeat the operations described in A.5.2.2 to A.5.2.4 for each of the five remaining specimens, using a different weight with each.

The choice of the six different weights used to load the test specimens from the thirteen weights listed in table A.1 depends upon the molecular weight of the PE-UHMW sample. Select the weights so that times between 1 min and 20 min are obtained.

NOTE Elongation of the test specimens does not take place at constant speed.

A.6 Calculation of results

For each of the six separate determinations, the tensile stress σ, expressed in MPa, is given by the equation

$$\sigma = \frac{m \times 9{,}81}{b \times s} \times \left(1 - \frac{\rho_m}{\rho_w}\right)$$

where

m is the mass, in g, of the weight used;

b is the initial width, in mm, of the narrow parallel-sided section of the test specimen;

s is the initial thickness, in mm, of the narrow parallel-sided section of the test specimen;

ρ_m is the density of the heating-bath liquid at 150 °C;

ρ_w is the density of the weight at 150 °C;

9,81 is the acceleration due to gravity, in m/s^2.

NOTE As is usual in practice, the mass of the lower test specimen holder has been neglected but the attached weight has been corrected for buoyancy.

Using a log/log scale, plot the tensile stress for the six specimens against the corresponding times for 600 % elongation recorded in A.5.2.4 and A.5.2.5. Draw a straight line through the six points and, from this graph, read off the tensile stress corresponding to a period of 10 min (see figure A.4). This value represents the elongational stress F(150/10) in MPa.

The six points plotted should lie in a straight line. An undue amount of scatter ($R^2 \geqslant 0{,}95$) indicates that partial crosslinking has occurred in the test specimens. In such a case, prepare further specimens using an increased amount of stabilizer (see clause A.4), and repeat the whole procedure.

NOTE The slope of the line can be given as an additional characterization parameter to compare different PE-UHMW products with the same elongational stress.

A.7 Precision

The precision of this method is not known because inter-laboratory data are not available. However a coefficient of variation of about ± 5 % could be expected.

A.8 Test report

The test report shall include the following information:

a) all details necessary for identification of the PE-UHMW moulding powder tested;

b) the elongational stress F(150/10), in MPa;

c) details of any departures from the standard method specified herein, plus the reasons why;

d) the date of the test.

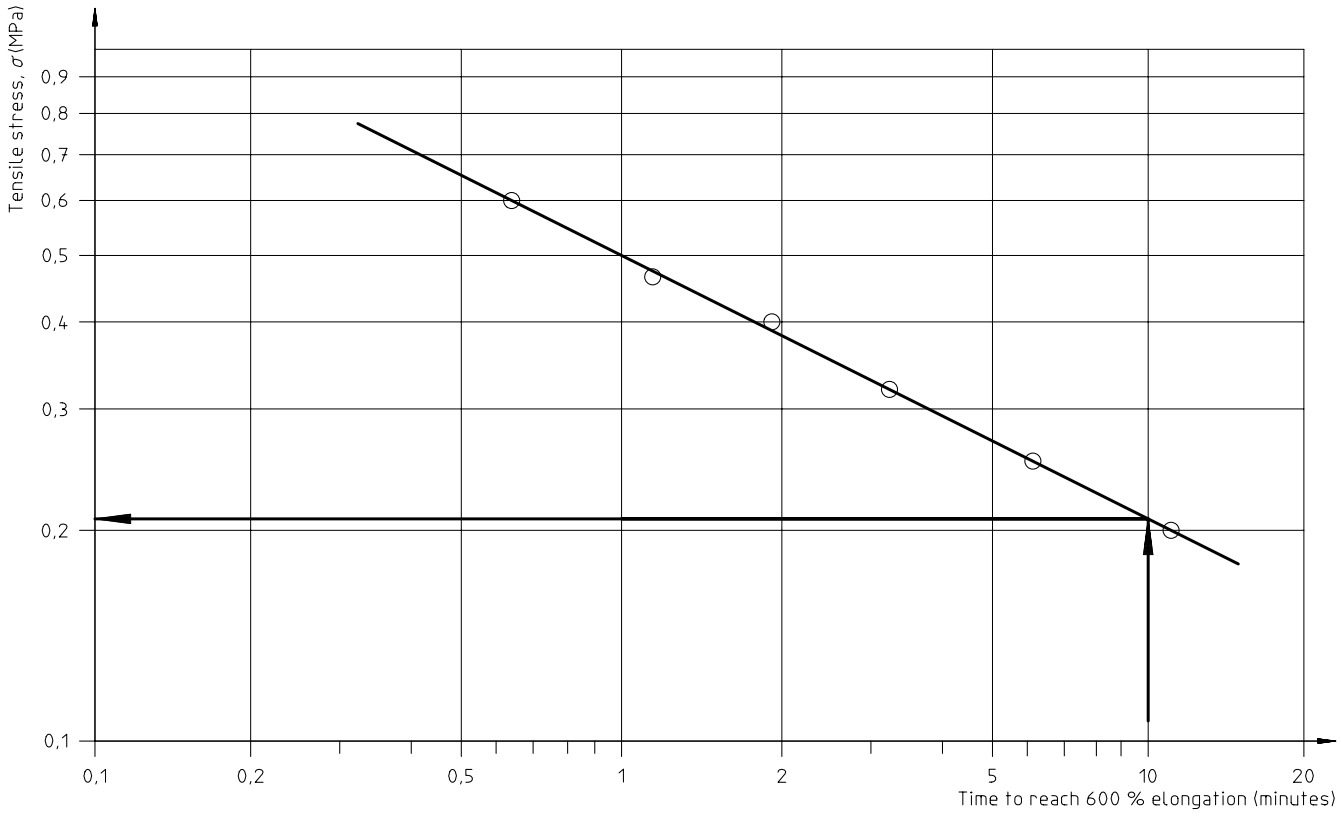


Figure A.4 — Typical curve for determining the elongational stress

Annex B
(normative)

Method for determining the “double notch” Charpy impact strength of PE-UHMW moulding material

B.1 Scope

This annex specifies a method of determining the Charpy notched impact strength of PE-UHMW moulding material using double notches. Other conventional standard methods, e.g. ISO 179-1 (determination of Charpy impact with different notch types) and/or ISO 8256 (determination of notched tensile-impact strength), are not suitable because a brittle break cannot be achieved for all UHMW polyethylenes. Complete brittle breakage of the test specimens is obtained by this new method with a lower standard deviation compared to other standard tests, including Izod (ISO 180).

NOTE This procedure is a modification of ISO 179-1. The use of specimens with sharp double notches can make significant differentiation possible between UHMW polyethylenes.

B.2 Definition

B.2.1 Charpy impact strength of double-notched specimens: The impact energy absorbed in breaking a notched specimen, referred to the original cross-sectional area of the specimen, at double notches (see figure B.1). It is expressed in kilojoules per square metre.

B.3 Principle

The test specimen, supported as a horizontal beam, is broken by a single swing of a pendulum, with the line of impact midway between the supports, directly into the notch, with a flatwise direction of blow.

B.4 Apparatus

See ISO 179-1 except that the span between the supports shall be 70 mm.

Use a pendulum, having an energy of 50 J, in accordance with ISO 179-1 and a device for notching the test specimen (see figure B.1).

B.5 Test specimens

Prepare compression-moulding sheets using the conditions specified in table 1 of this part of ISO 11542. From the sheets, machine test specimens, measuring 120 mm $\times$ 15 mm $\times$ 10 mm, in accordance with ISO 2818.

For the preparation of the notches on both sides of the test specimens, use a suitable notching device with a thick (0,23 mm $\pm$ 0,03 mm) single-edged razor blade with 14° $\pm$ 2° included angle at the cutting edge. Make notches 3 mm deep in the middle of the specimens. An example of a suitable device is shown in figure B.1. The notching speed shall be less than 500 mm/min and a new blade shall be used after notching 40 specimens. A set consisting of four specimens is sufficient for testing. Condition the test specimens as describe in ISO 179-1.

NOTES

1 In the notching device shown in figure B.1, the test specimen is placed on the lower block in position A, making sure that it is positioned so that the notch will be cut at the centre. A 3-mm-deep notch is cut by pressing the upper block down on the specimen. The specimen is then turned over and placed in position B, and the procedure repeated. Slight differences in specimen thickness are compensated for by this particular device which ensures that the distance between the two notch roots is always 4 mm.

2 The use of different notching speeds up to 500 mm/min has been shown to have no significant effect on the results obtained.

B.6 Procedure

The procedure is carried out in accordance with ISO 179-1, with the only difference that the pendulum hits flatwise against the notch cut first (the marked side of the test specimen). Due to the design of the notching device, it is not necessary to measure the thickness between the notch roots for each test specimen (always 4 mm), but only the width, which shall be measured and recorded to the nearest 0,02 mm.

B.7 Calculation of results

Calculate the impact strength a_{cN} of each specimen, expressed in kilojoules per square metre, using the equation

$$a_{cN} = \frac{W}{bh_N} \times 10^3$$

where

W is the energy, in joules, absorbed by the test specimen breaking, corrected for frictional-energy loss;

b is the exact width, in millimetres, of the test specimen;

h_N is the thickness, in millimetres, between the roots of the notches in the test specimen (always 4 mm).

Calculate the mean impact strength for the set of specimens tested.

B.8 Precision

Interlaboratory testing by PE-UHMW producers has shown that the standard deviation of the mean is about 8 %.

B.9 Test report

The test report shall include the following information:

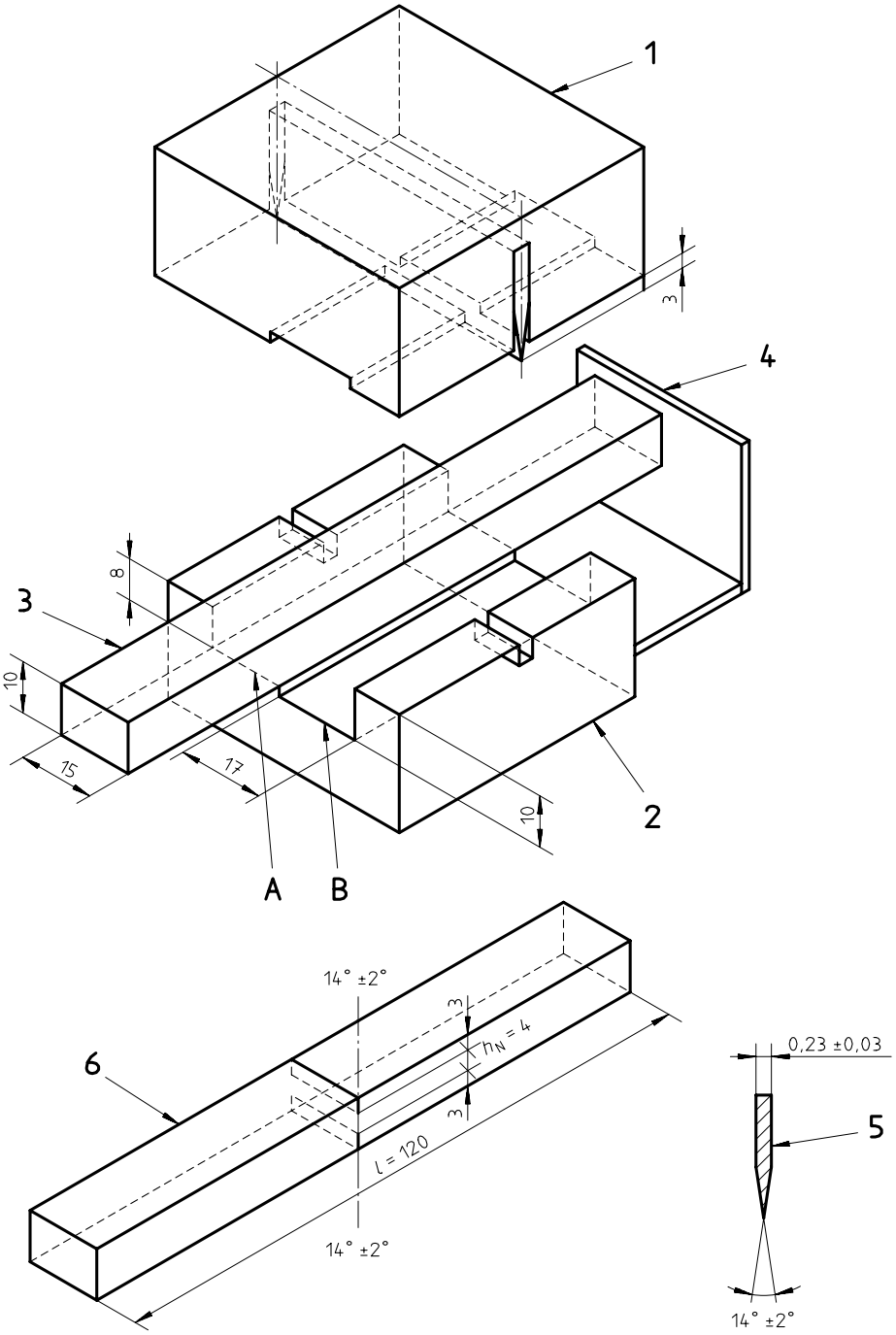
a) all details necessary for complete identification of the material tested, including the manufacturer's designation and the grade and form of the material;

b) the orientation of the test specimen in relation to the sheet from which it was cut;

c) the mean double-notch Charpy impact strength, in kilojoules per square metre, for the set of specimens tested;

d) the date of the test.

Dimensions in millimetres



Key
1 Upper block
2 Lower block
3 Test specimen
4 Back stop
5 Razor blade
6 Notched specimen

Figure B.1 — Notching device and notched test specimen

ICS 83.080.20

Descriptors: plastics, thermoplastic resins, moulding materials, extrusion materials, polyethylene, tests, determination, properties, test specimens, specimen preparation.

Price based on 14 pages