# INTERNATIONAL STANDARD

ISO 21304-2

First edition 2021-01

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

Part 2:

Preparation of test specimens and determination of properties

Plastiques — 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





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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 <a href="www.iso.org/directives">www.iso.org/directives</a>).

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 <a href="https://www.iso.org/patents">www.iso.org/patents</a>).

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 <a href="https://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

This document was prepared by Technical Committee ISO/TC 61, Plastics, Subcommittee SC 9, Thermoplastic materials, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, Plastics, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This first edition of ISO 21304-2 cancels and replaces ISO 11542-2:1998, which has been technically revised. It also incorporates the Technical Corrigendum ISO 11542-2:1998/Cor 1:2007.

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

- updated the normative references to the latest version;
- added terms and definitions (see <u>Clause 3</u>);
- revised the contents and structures of <u>Table 2</u> and <u>Table 3</u> according to the revised ISO 10350-1;
- added new Annex A;
- revised the masses of weights used to load specimen in <u>Table B.1</u> (former Table A.1);
- revised Figure B.1, Figure B.2, Figure B.3 and Figure C.1 (former Figures A.1, A.2, A.3 and B.1).

A list of all parts in the ISO 21304 series can be found on the ISO website.

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 <a href="https://www.iso.org/members.html">www.iso.org/members.html</a>.

## 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 document 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. It gives the requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing.

This document gives the procedures and conditions for the preparation of test specimens and procedures for measuring properties of the materials from which these specimens are made. 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 document, as are the designatory properties specified in ISO 21304-1. This document specifies the materials with MFR less than 0,1 g/10 min at 190 °C /21,6 kg based on ISO 17855-1.

The methods of preparation and conditioning, the specimen dimensions and the test procedures specified in this document are used in order to obtain reproducible and comparable test results. 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 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 62, Plastics — Determination of water absorption

 ${\tt ISO~75.4, Plastics-Determination~of~temperature~of~deflection~under~load-Part~1:~General~test~method}$ 

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

ISO 178, Plastics — Determination of flexural properties

ISO 179-1, Plastics — Determination of Charpy impact properties — Part 1: Non-instrumented impact test

ISO 291, Plastics — Standard atmospheres for conditioning and testing

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

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

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

#### ISO 21304-2:2021(E)

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

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

ISO 1133-1, Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method

ISO 1183-1, Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pycnometer method and titration method

ISO 1183-2, Plastics — Methods for determining the density of non-cellular plastics — Part 2: Density gradient column method

ISO 1628-3, Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 3: Polyethylenes and polypropylenes

ISO 2818, Plastics — Preparation of test specimens by machining

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

ISO 8256, Plastics — Determination of tensile-impact strength

ISO 10350-1, Plastics — Acquisition and presentation of comparable single-point data — Part 1: Moulding materials

ISO 11357-2, Plastics — Differential scanning calorimetry (DSC)—Part 2: Determination of glass transition temperature and step height

ISO 11357-3, Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization

ISO 11359-2, Plastics — Thermomechanical analysis (TMA) — Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature

ISO 15527, Plastics — Compression-moulded sheets of polyethylene (PE-UHMW, PE-HD) — Requirements and test methods

ISO 20753, Plastics — Test specimens

ISO 21304-1, Plastics — Ultra-high-molecular-weight polyethylene (PE-UHMW) moulding and extrusion materials — Part 1: Designation system and basis for specifications

IEC 60112, Method for the determination of the proof and the comparative tracking indices of solid insulating materials

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

IEC 60296, Fluids for electrotechnical applications — Unused mineral insulating oils for transformers and switchgear

ISO/IEC 60695-11-10, Fire hazard testing — Part 11-10: Test flames — 50 W horizontal and vertical flame test methods

IEC 62631-2-1, Dielectric and resistive properties of solid insulating materials-Part 2-1:Relative permittivity and dissipation factor-Technical frequencies (0,1 Hz to 10 MHz)-AC Methods

IEC 62631-3-1, Dielectric and resistive properties of solid insulating materials — Part 3-1: Determination of resistive properties (DC methods) — Volume resistance and volume resistivity — General method

IEC 62631-3-2, Dielectric and resistive properties of solid insulating materials — Part 3-2: Determination of resistive properties (DC methods) — Surface resistance and surface resistivity

#### 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 <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at <a href="http://www.electropedia.org/">http://www.electropedia.org/</a>

#### 3.1

#### elongational stress

 $F_{(150/10)}$ 

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

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

#### 3.2

#### Charpy double-notched impact strength

impact energy absorbed in breaking a double-notched specimen, referred to the original cross-sectional area of the specimen, at double notches (see Figure C.1)

Note 1 to entry: It is expressed in kilojoules per square metre (kJ/m<sup>2</sup>).

## 4 Preparation of test specimens

## 4.1 Treatment of the material before moulding

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

## 4.2 Compression moulding

Compression-moulded sheets shall be prepared in accordance with ISO 293 using the conditions specified in <u>Table 1</u>. 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	temperature pressure °C MPa		Preheating time	Full pressure MPa	Full- pressure time min	Average cooling rate	Demoulding temperature °C
ŀ	All grades	210	5	5 to 15	10	30 ± 1	15	≤ 40

A flash mould (see ISO 293) may be used, but it is necessary to start cooling while simultaneously applying the full pressure. This avoids the melt being pressed out of the frame and also avoids sink marks.

For thicker sheet (≥4 mm), a positive mould (see ISO 293) shall be used. 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.

#### 5 Conditioning of test specimens

Unfilled PE-UHMW test specimens shall be conditioned in accordance with ISO 291 for at least 40 h at 23 °C  $\pm$  2 °C, with no relative humidity requirement. Test specimens containing fillers and / or additives that are susceptible to moisture uptake shall be conditioned for at least 40 h at 23 °C  $\pm$  2 °C and 50 %  $\pm$  10 % relative humidity.

## 6 Determination of properties

In the determination of properties and the presentation of data, the standards, supplementary instructions and notes given in ISO 10350-1 shall be applied. Unless specifically stated in Table 2 and Table 3, testing of unfilled PE-UHMW test specimens shall be carried out at a standard temperature of 23 °C  $\pm$  2 °C with no relative humidity requirement. Specimens made from materials containing fillers and / or additives that are susceptible to moisture uptake shall be tested in a standard atmosphere of 23 °C  $\pm$  2 °C and 50 %  $\pm$  10 % relative humidity.

<u>Table 2</u> is compiled from ISO 10350-1, 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.

<u>Table 3</u> contains those properties, not found specifically in <u>Table 2</u>, 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 10350-1)

					XV			
	Property		Standard	Specimen type (dimensions in mm)	Specimen Prepara- tion a	Unit	Test conditions and supplementary instructions	
1 Me	chanical propertie	es		110				
1.1	Tensile modulus	$E_{t}$		1,10		MPa	Test speed 1 mm/min	
1.2	Yield stress	$\sigma_{_{ m V}}$		lickto		МРа	Failure with yielding	
1.3	Yield strain	$\varepsilon_{ m y}$					Test speed 50 mm/min	
1.4	Nominal strain at break	$arepsilon_{ m tb}$	ISO 527-1 ISO 527-2			%		
1.5	Stress at 50 % strain	$\sigma_{50}$	ISO 527-4				МРа	Failure without yielding $\varepsilon_h \leq 10 \%$ : test speed
1.6	Stress at break	$\sigma_{b}$						5 mm/min
1.7	Strain at break	$\epsilon_{\rm b}$					Q	%
1.8	Tensile creep mod-	$E_{\rm tc}1$	ICO 000 1			MD-	At 1 h	
1.9	ulus	$E_{\rm tc} 10^{3}$	ISO 899-1			MPa	At 1 000 h Strain ≤ 0,5 %	
1.10	Flexuralmodulus	$E_{\mathrm{f}}$	ISO 178	80 × 10 × 4		MPa	Test speed 2 mm/min	
				80 × 10 × 4				
1.11	Tensile-impact	$\alpha_{ m tN}$	ISO 8256	Machined dou- ble		kJ/m <sup>2</sup>		
	strength			V-notch,				
				r = 1				

a Q = Compression moulding.

b Electrical properties are generally affected by the relative humidity. Therefore, they shall be measured in a standard atmosphere of 23  $^{\circ}$ C ± 2  $^{\circ}$ C and 50  $^{\circ}$ C ± 10  $^{\circ}$ C relative humidity.

Table 2 (continued)

	Property	Symbol	Standard	Specimen type (dimensions in mm)	Specimen Preparation a	Unit		itions and sup- ry instructions	
2 Th	ermal properties								
2.1	Melting tempera-	$T_{ m m}$	ISO 11357-				Record per perature.	ak melting tem-	
2.1	ture	<sup>1</sup> m	3	Powder	_	°C	Use 10 K, cooling rat	/min heating/ e.	
2.2	Glass transition	$T_{ m g}$	ISO 11357- 2				Record the method used for determination of $T_{\rm g}$ .		
	temperature	J					Use 10 K/min heating rate.		
2.3	Temperature of	$T_{\rm f}$ 1,8	ISO 75-1				1,8 MPa	Use flatwise	
2.4	deflection under load	T <sub>f</sub> 0,45	ISO 75-2	80 × 10 × 4		°C	0,45 MPa	loading	
2.5		$\alpha_{\mathrm{p}}$		D 16		O	Parallel	Record the	
2.6	Coefficient of linear thermal expansion	$\alpha_{\mathrm{n}}$	ISO 11359- 2	Prepared from ISO 20753 type A2	OF	°C <sup>-1</sup>	Transverse	secant value over the tem- perature range 23 °C to 55 °C.	
2.7	Flammabili-	B50/3	ISO/	125 × 13 × 3	Α,		Record one of the classi-		
	ty-Burning behav-		IEC 60695-	Additional		_	fications		
2.8	iour	B50/h	11–10	thickness, h			V-0, V-1, V-2, HB, HB40 or HB75.		
2.9	Ignitability-0xy-gen index	OI	ISO 4589-2	80 × 10 × 4		%	Use proced	lure A ce ignition).	

a Q = Compression moulding.

b Electrical properties are generally affected by the relative humidity. Therefore, they shall be measured in a standard atmosphere of 23 °C  $\pm$  2 °C and 50 %  $\pm$  10 % relative humidity.

Table 2 (continued)

			Specimen type	Specimen		Test conditions and sup-		
Property	Symbol	Standard	(dimensions in mm)	Prepara- tion <sup>a</sup>	Unit	plementary instructions		
ctrical properties	b							
Relative permit- tivity	$\varepsilon_{\rm r} 100$ $\varepsilon_{\rm r} 1{\rm M}$				_	100 Hz 1 MHz		
Dissipation factor	tan δ 100	IEC 62631- 2-1				Compensate for electrode edge effects.		
Dissipation factor	tan δ1M				_	1 MHz		
Volume resistivity	$ ho_{ m e}$	IEC 62631- 3-1	> 60 x > 60 x 2		Ω•m	Measure value at 1 min.		
Surface resistivity	$\sigma_{ m e}$	IEC 62631- 3-2		Q Q	Ś Ś.	Voltage electrodes 1 mm to 2 mm wide, 50 mm long and 5 mm apart.		
	E <sub>B</sub> 1		≥ 60 × ≥ 60 × 1	~e`		Use 20 mm diameter spher-		
Electric strength	E <sub>B</sub> 2	IEC 60243- 1	≥60 × ≥ 60 × 2	<b>.</b>	kV/ mm	ical electrodes.  Immerse in transformer oil in accordance with IEC 60296.  Use a voltage application rate of 2 kV/s.		
Comparative tracking index		IEC 60112	≥ 20 × ≥ 20 × 4		-	Use solution A.		
ier properties		CO,						
		150 62	60 × 60 × 1		0%	Saturation value in water at 23 °C.		
water absorption	WH	150 62	00 × 00 × 1	Q	70	Equilibrium value at 23 °C, 50 % RH.		
Density	ρ	ISO 1183-1 ISO 1183-2	10 × 10 × 4 5 × 5 × 4		kg/m <sup>3</sup>	Test specimen to be taken from moulded specimen.		
	Ctrical properties Relative permittivity  Dissipation factor  Volume resistivity  Surface resistivity  Electric strength  Comparative tracking index  er properties  Water absorption	$\begin{array}{c} \textbf{ctrical properties} \ \textbf{b} \\ \textbf{Relative permittivity} & \boldsymbol{\varepsilon_r} \ 100 \\ \boldsymbol{\varepsilon_r} \ 1M \\ \textbf{Dissipation factor} & \begin{array}{c} \boldsymbol{tan} \\ \boldsymbol{\delta} \ 100 \\ \hline \boldsymbol{tan} \\ \boldsymbol{\delta} \ 1M \\ \end{array} \\ \textbf{Volume resistivity} & \boldsymbol{\rho_e} \\ \\ \textbf{Surface resistivity} & \boldsymbol{\sigma_e} \\ \\ \textbf{Electric strength} & \boldsymbol{E_B} \ 1 \\ \\ \textbf{Electric strength} & \boldsymbol{E_B} \ 2 \\ \\ \textbf{Comparative tracking index} & \textbf{CTI} \\ \\ \textbf{er properties} \\ \\ \textbf{Water absorption} & \boldsymbol{W_w} \\ \\ \textbf{Water absorption} \\ \end{array}$	$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	Property Symbol Standard (dimensions in mm)  Ctrical properties b  Relative permititivity $\frac{\varepsilon_r \ 100}{\varepsilon_r \ 1M}$ Dissipation factor $\frac{\delta 100}{\delta 1M}$ Volume resistivity $\frac{\varepsilon_r \ 100}{\delta 1M}$ Electric strength $\frac{E_B \ 1}{E_B \ 2}$ Electric strength $\frac{E_B \ 2}{E_B \ 2}$ Electr	Property Symbol Standard (dimensions in mm) Preparation a Relative permittivity $\frac{\varepsilon_r \ 100}{\varepsilon_r \ 1M}$ Relative perm	Property Symbol Standard (dimensions in mm) Preparation a Unit ctrical properties b  Relative permitivity $\frac{\varepsilon_{r} \ 100}{\varepsilon_{r} \ 1M}$ Dissipation factor $\frac{\delta 100}{\delta 1M}$ Volume resistivity $\rho_{e}$ IEC 62631- $\frac{\delta 100}{\delta 1M}$ Surface resistivity $\sigma_{e}$ IEC 62631- $\frac{\delta 100}{\delta 1M}$ Electric strength $\frac{E_{B} \ 1}{\delta 1M}$ Electric strength $\frac{E_{B} \ 1}{\delta 1M}$ Electric strength $\frac{E_{B} \ 1}{\delta 1M}$ Electric strength $\frac{\delta 1}{\delta 1M}$ Elect		

<sup>&</sup>lt;sup>a</sup> Q = Compression moulding.

Electrical properties are generally affected by the relative humidity. Therefore, they shall be measured in a standard atmosphere of 23  $^{\circ}$ C  $\pm$  2  $^{\circ}$ C and 50  $^{\circ}$ E  $\pm$  10  $^{\circ}$ C relative humidity.

	Property	Symbol	Standard	Specimen type (dimensions in mm)	Specimen prepara- tion <sup>a</sup>	Unit	Test condition and supplemen- tary instructions
1 Rhe	ological properties						
1.1	Melt Volume-flow rate b	MVR (230/21,6 <sub>T</sub> )	ISO 21304-2	Powder, granules	_	cm <sup>3</sup> /10min	See <u>Annex A</u> .
2 Me	chanical properties						
2.1	Elongational stress	$F_{(150/10)}$	ISO 21304-2	Figure B.3		МРа	See <u>Annex B</u> .
				120 × 15 × 10	0	2	
2.2	Charpy double-notched impact strength	$\alpha_{cN}$	ISO 21304-2	Double V-notch, 14° ± 2°	Q	kJ/m²	See <u>Annex C</u> .
3 Oth	er properties			<u> </u>	1	OX	I
3.1	Viscosity number	I	ISO 1628-3	Powder	^	mL/g	_
3.2	Abrasion properties - Degree of abrasion	$W_s$			col	%	Rate of rotation: 1 200 min <sup>-1</sup>
3.3	Abrasion properties- Index of abrasion	$\eta_r$	ISO 15527	76,2 × 25,4 × 6,35	of 150	%	Temperature of slurry: not exceed 23 °C
a Ç	ee A.1 note.			Le full POR			
	ee A.1 note.	COM.	Click to vi	2 <sup>M</sup>			
	NDARDSIE	0.					

## Annex A

(normative)

## Method for determining the melt volume-flow rate of PE-UHMW materials

#### A.1 Overview

This annex specifies a method for determining the melt volume-flow rate (MVR) of some PE-UHMW materials under the test condition of 230 °C /21,6 kg using a triple bore area die (see A.2).

NOTE It has been confirmed that the melt volume-flow rate (MVR) is useful for characterizing some PE-UHMW materials (e.g. PE-UHMW pipe materials) under the test condition of 230 °C /21,6 kg and bore diameter of the die with 3,628 mm, although PE-UHMW materials are polyethylene materials having a melt mass-flow rate (MFR) of less than 0,1 g/10 min, measured at 190 °C / 21,6 kg (see ISO 21304-1).

## A.2 Apparatus

Requirements of apparatus shall be in accordance with ISO 11331, but use a triple bore area die with bore diameter 3,628 mm.

NOTE The bore area of the triple bore area die is nearly triple area as big as the bore area of standard die of ISO 1133-1.

## A.3 Test sample

- **A.3.1** The test sample may be in any form that can be introduced into the bore of the cylinder, e.g. powder, granules, strips of film or sections of moulded or extruded parts, see ISO 1133-1.
- **A.3.2** The stabilizer could be mixed homogeneously into the PE-UHMW moulding powder at a concentration capable of reducing crosslinking. If the PE-UHMW resin or the stabilizer is in granular or pellet form, grind or pulverize to so that a homogeneous mixture is obtained.

NOTE It has been proved that adding a mass fraction of 0,5 % hindered phenolic antioxidant or phenolic/phosphite blend of 1/1 is feasible.

## A.4 Procedure

- **A.4.1** Make the apparatus temperature to 230 °C and stabilize it for not less than 15 min.
- **A.4.2** Weigh the test sample according to MVR field (see <u>Table A.1</u>).

MVR	Mass of test sample in cylinder	Piston move distance		
cm <sup>3</sup> /10min	g	mm		
> 0,1, but ≤ 0,15	3	0,5		
> 0,15, but ≤ 0,4	3	1		
> 0,4, but ≤ 1	3	2		
> 1, but ≤ 20	4	5		
> 20	5	10		

Table A.1 — Guidelines for experimental parameters

**A.4.3** Ensure that the piston, cylinder and die are cleaned. In 20 s, quickly charge the test sample into the cylinder. During charging, compress the material by the packing rod with hand pressure, place the total 21,6 kg weight on the piston and start timing. The procedure should be completed within a period of 1 min. If needed, cut off the extrudate with the cutting tool and discard. Continue to allow the loaded piston to descend under gravity.

**A.4.4** Start to automatically measure the time required by the piston to move as specified distance shown in Table A.1.

**A.4.5** Calculate (or record) the value of MVR, and express the value of MVR to three significant figures but with a maximum of two decimal places (see <u>A.5</u>).

## A.5 Calculation of results

Calculate the melt volume-flow rate (MVR) expressed in cubic centimetres per 10 min (cm<sup>3</sup>/10min), using Formula (A.1):

$$MVR(230/21,6_{\rm T}) = \frac{A \times 600 \times l}{t} \tag{A.1}$$

where

(230/21,6 $_{\rm T}$ ) is the test condition with 230 °C and 21,6 kg load using triple bore area die with bore diameter 3,628 mm;

A is the mean of the nominal cross-sectional areas of the cylinder and the piston head, in square centimetres (cm<sup>2</sup>) and is equal to 0,711 cm<sup>2</sup> (see Note);

is the factor used to convert cubic centimeters per second into cubic centimeters per 10 min (600 s);

*l* is the predetermined distance moved by the piston or the mean value of the individual distance measurements, in centimeters (cm) (see <u>Table A.1</u>).

is the predetermined time of measurement or the mean value of the individual time measurements, in seconds (s).

NOTE Due to the tolerances permitted on the cylinder and piston diameters, the mean of the actual cross-sectional areas of the cylinder and the piston head varies by less than  $\pm$  0,5 %. This effect is considered negligible and for simplicity of operation the nominal value of 0,711 cm<sup>2</sup> is used.

#### A.6 Precision

The precision of the method is not known because interlaboratory data are not available at the time of publication. However, earlier test data by PE-UHMW producers indicated a coefficient of variation of ± 10 % within a laboratory and ± 15 % inter laboratories can be expected.

### A.7 Test report

The test report shall include the following information:

- a reference to Annex A of this document, i.e. ISO 21304-2:2021, Annex A;
- all details necessary for the complete identification of the test sample, including the physical form of 1502/30A.2 of the material with which the cylinder was charged;
- c) the details of any stabilization;
- d) the triple bore area die used in the test;
- the temperature and load used in the test of 230 °C/21,6 kg;
- the predetermined distance moved by the piston;
- the melt volume-flow rate, in cubic centimetres per 10 min, expressed to three significant figures but with a maximum of two decimal places. standards is o.com. circk to view the full but with a maximum of two decimal places;
- the date of the test.

## **Annex B**

(normative)

## Method for determining the elongational stress of PE-UHMW moulding materials

#### **B.1** Overview

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

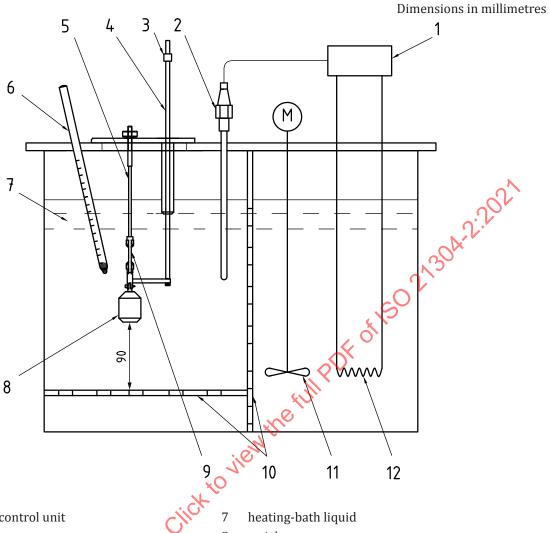
## **B.2** Apparatus

The apparatus of determining the elongational stress contains the following. See Figures B.1 and B.2.

- a) a temperature-control unit;
- b) a contact thermometer;
- c) a holding device with lower clamp and upper clamp, in accordance with Figure B.1, with arresting device;
- d) a mercury-in-glass or equivalent thermometer, graduated in intervals of 0,5 °C, suitable for measuring temperatures within the range 150 °C ± 2 °C;
- e) a heating-bath liquid;
- f) a set of weights, 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 <a href="Table B.1">Table B.1</a>);
- g) the test specimen;
- h) perforated plates, one fitted near the bottom of the bath, the other separating the mixer and the heating coil from the specimen;
- i) a mixer with motor;
- j) a heating coil;
- k) a stopwatch, accurate to 0,1 s;
- l) measuring instruments, accurate to 0,02 mm, for measuring the width and thickness of the narrow parallel-sided section of the test specimen.

#### Table B.1 — Masses<sup>a</sup> of weights used to load specimen

80	100	120	150	180	200	250	300	350	400	450	500	550	600	650	700	750	800
<sup>a</sup> In grams.																	



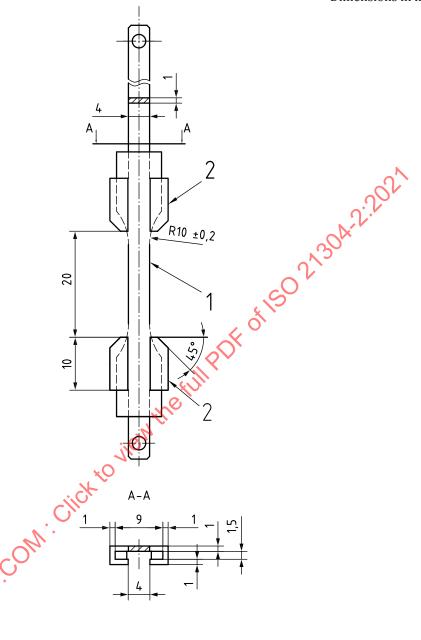
1 temperature-control unit

Key

- 2 contact thermometer
- 3 arresting device
- 4 holding device of lower clamp
- 5 holding device of upper clamp
- 6 mercury-in-glass thermometer or equivalent
- 8 weight
- 9 test specimen
- 10 perforated plates
- 11 mixer with motor (M)
- 12 heating coil

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

Dimensions in millimetres



Key

- 1 specimen
- 2 clamp

Figure B.2 — Specimen holder

Dimensions in millimetres

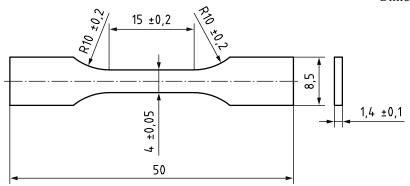


Figure B.3 — Test specimen

## **B.3** Compression moulding of sheet

Use the conditions specified in <u>Table 1</u>. 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.

#### **B.4** Procedure

#### B.4.1 Test specimens (see Figure B.3)

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

NOTE All surfaces of the test specimens are free from visible flaws, scratches or other imperfections.

#### **B.4.1.1** 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.

#### **B.4.2 Determination**

- **B.4.2.1** Fill the heating bath with a suitable liquid (e.g. silicone oil) and raise the temperature to  $150 \,^{\circ}\text{C} \pm 2 \,^{\circ}\text{C}$ .
- **B.4.2.2** Clamp one of the test specimens in the holder as shown in <u>Figure B.2</u>, hook a weight to the holder and suspend the specimen and weight in the bath liquid as shown in <u>Figure B.1</u> 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.
- **B.4.2.3** Five minutes after the specimen has entered the bath liquid, free the holder from the arresting device and simultaneously start the stopwatch.
- **B.4.2.4** 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).

**B.4.2.5** Repeat the operations described in <u>B.4.2.2</u> to <u>B.4.2.4</u> 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 18 weights listed in <u>Table B.1</u> 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.

#### **B.5** Calculation of results

For each of the six separate determinations, the tensile stress ( $\sigma$ ), expressed in megapascals (MPa), is given by Formula (B.1):

$$\sigma = \frac{m \times 9.81}{b \times s} \times \left(1 - \frac{\rho_{\rm m}}{\rho_{\rm w}}\right) \tag{B.1}$$

where

*m* is the mass, in grams (g), of the weight used;

9,81 is the acceleration due to gravity, in metres per square second  $(m/s^2)$ .

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

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

 $\rho_{\rm m}$  is the density of the heating-bath liquid at 150 °C, in grams per cubic centimetre (g/cm<sup>3</sup>);

 $\rho_{\rm w}$  is the density of the weight at 150 °C, in grams per cubic centimetre (g/cm<sup>3</sup>).

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 buoyance.

Using a log/log scale, plot the tensile stress for the six specimens against the corresponding times for 600 % elongation recorded in 8.4.2.4 and 8.4.2.5. 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 8.4). This value represents the elongational stress  $F_{(150/10)}$  in megapascals (MPa).

The six points plotted shall lie in a straight line. A linear regression of the log of tensile stress against the log of time for 600 % elongation gives the correlation coefficient, R. A value of  $R^2 < 0.95$  indicates that partial crosslinking has occurred in the test specimens. In such a case, prepare further specimens using an increased amount of stabilizer (see B.3), 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.

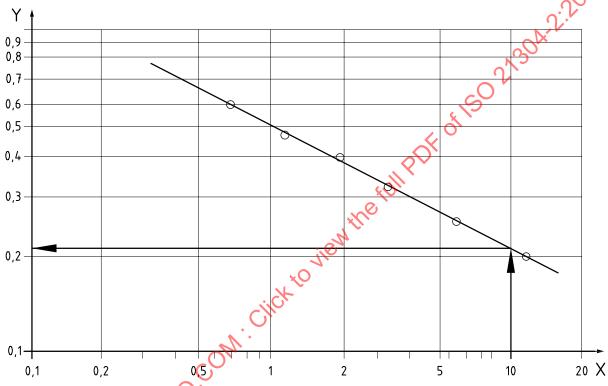
#### **B.6 Precision**

The precision of this method is not known because interlaboratory data are not available. However, a coefficient of variation of about  $\pm 5$  % could be expected.

## **B.7** Test report

The test report shall include the following information:

- a) all details necessary for identification of the PE-UHMW moulding materials tested;
- b) the elongational stress  $F_{(150/10)}$ , in megapascals (MPa);
- c) details of any departures from the standard method specified herein, plus the reasons why;
- d) the date of the test.



Key

X time to reach 600 % elongation(min)

Y tensile stress σ (MPa)

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