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Plastics — Polyethylene (PE) moulding and extrusion materials —

Part 2:

Preparation of test specimens and determination of properties

Plastiques — Polyéthylène (PE) pour moulage et extrusion — Partie 2: Préparation des éprouvettes et détermination des propriétés



ISO 17855-2:2016(E)



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information.

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This first edition of ISO 17855-2 cancels and replaces ISO 1872-2:2007, which has been technically revised.

ISO 17855 consists of the following parts, under the general title *Plastics — Polyethylene (PE) moulding* and extrusion materials:

- Part 1: Designation system and basis for specifications
- Part 2: Preparation of test specimens and determination of properties

Plastics — Polyethylene (PE) moulding and extrusion materials —

Part 2:

Preparation of test specimens and determination of properties

1 Scope

This part of ISO 17855 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of polyethylene (PE) 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.

Procedures and conditions for the preparation of test specimens and procedures for measuring properties of the materials from which these specimens are made are also given. Properties and test methods that are suitable and necessary to characterize PE 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 17855, as are the designatory properties specified in ISO 17855-1.

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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

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 179-2, Plastics — Determination of Charpy impact properties — Part 2: Instrumented impact test

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

ISO 294–1, Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens

ISO 294-3, Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates

ISO 294–4, Plastics — Injection moulding of test specimens of thermoplastic materials — Part 4: Determination of moulding shrinkage

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- ISO 527–2, Plastics Determination of tensile properties Part 2: Test conditions for moulding and extrusion plastics
- 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 pyknometer method and titration method
- ISO 1183-2, Plastics Methods for determining the density of non-cellular plastics Part 2: Density gradient column method
- ISO 1183-3, Plastics Methods for determining the density of non-cellular plastics Part 3: Gas pyknometer 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 6603–2, Plastics Determination of puncture impact behaviour of rigid plastics Part 2: Instrumented impact testing
- 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 glass transition 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 16770, Plastics Determination of environmental stress cracking (ESC) of polyethylene Full-notch creep test (FNCT)
- ISO 17855-1:2014, Plastics Polyethylene (PE) moulding and extrusion materials Part 1: Designation system and basis for specifications
- ISO 20753, Plastics Test specimens
- IEC 60093, Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials
- 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 60250, 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, Fluids for electrotechnical applications Unused mineral insulating oils for transformers and switchgear

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

ASTM D638, Standard test method for tensile properties of plastics

ASTM D1693, Standard test method for environmental stress-cracking of ethylene plastics

3 Preparation of test specimens

3.1 General

It is essential that specimens are always prepared by the same procedure (either injection moulding or compression moulding), using the same processing conditions.

The procedure to be used for each test method is indicated in $\underline{\text{Table 3}}$ and $\underline{\text{Table 4}}$ (M = injection moulding, Q = compression moulding).

3.2 Treatment of the material before moulding

No pre-treatment of the material sample is normally necessary before moulding.

3.3 Injection moulding

Injection moulding of test specimens is used for PE moulding materials having a melt mass-flow rate (MFR) of ≥ 1 g/10 min, determined in accordance with ISO 1133-1 using set of test conditions D (190 °C/2,16 kg) specified in ISO 17855-1.

Injection-moulded specimens shall be prepared in accordance with ISO 294-1 or ISO 294-3, using the conditions specified in <u>Table 1</u>. It has been found that bar test specimens prepared in accordance with ISO 20753 give better precision than those injection-moulded directly to their final dimensions and so the use of this geometry is preferable.

An appropriate hold pressure, consistent with the production of blemish-free mouldings, shall be used.

Material	Melt Mould temperature		Average injection velocity	Cooling time	Total cycle time
	°C	°C	mm/s	S	S
$MFR \ge 1 g/10 min$	210	40	100 ± 20	35 ± 5	40 ± 5

Table 1 — Conditions for injection moulding of test specimens

3.4 Compression moulding

Compression moulding is used for materials with a melt mass-flow rate of <1 g/10 min, determined in accordance with ISO 1133-1 using set of test conditions D (190 °C/2,16 kg) specified in ISO 17855-1. For thinner specimens (\leq 2 mm thick) and where specifically prescribed in Table 3 and Table 4, compression moulding shall be used for all materials.

Compression-moulded sheets shall be prepared in accordance with ISO 293 using the conditions specified in <u>Table 2</u>.

Table 2 — Conditions for compression moulding of test specimens

Material	Moulding temperature	Average cooling rate	Demoulding temperature	Full pressure	Full- pressure time	Preheating pressure	Preheating time
	°C	°C /min	°C	MPa	min	MPa	min
All grades	180	15	≤ 40	5 or 10 ^a	5 ± 1	contact	5 to 15

Use 5 MPa for a frame mould and 10 MPa for a positive mould.

NOTE Inconsistent cooling rates can lead to significant deviations in measured properties due to the effect on the crystallinity of the specimens. So, it is desirable to use a moulding machine that can keep a constant cooling rate.

If a frame mould is used, it is necessary to start the cooling cycle while simultaneously applying the full pressure. This avoids the melt being pressed out of the frame and also avoids sink marks.

With the frame mould, the full pressure is only applied to the frame and thus, the sheets produced may suffer from insufficient homogeneity and pellet boundaries may be preserved.

For thicker sheet (approximately 4 mm), a 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 up to 15 min can be necessary, especially if electric heating is used.

The test specimens required for the determination of the properties shall be machined or stamped from the compression-moulded sheets in accordance with ISO 2818.

NOTE Stamping is suitable for specimens of lower thickness up to 4 mm. Compared with milling or sawing, it gives less stress to the specimens and deforms them less.

4 Conditioning of test specimens

Unfilled PE test specimens shall be conditioned for at least 16 h at 23 °C \pm 2 °C, with no relative humidity requirement. Specimens containing fillers or additives that are susceptible to moisture uptake shall be conditioned for at least 16 h at 23 °C \pm 2 °C and (50 \pm 10) % 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 10350-1 shall be applied. Unless specifically stated in <u>Table 3</u> and <u>Table 4</u>, testing of unfilled PE 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 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.

Table 3 is compiled from ISO 10350-1, and the properties listed are those that are appropriate to polyethylene (PE) moulding and extrusion materials. These properties are those considered useful for comparisons of data generated for different thermoplastics.

<u>Table 4</u> contains those properties not found specifically in <u>Table 3</u>, which are in wide use or of particular significance in the practical characterization of polyethylene (PE) moulding and extrusion materials.

Table 3 — General properties and test conditions (selected from ISO 10350-1)

Property		Symbol	Standard	Specimen type (dimensions in mm)	Specimen prepara- tion ^a	Unit	Test condition and supplementary instructions		
1 Rh	eological properties								
1.1	Melt mass-flow rate	MFR				g/ 10min	See condition given in ISO 17855-1.		
1.2	Melt volume-flow rate	MVR	ISO 1133-1	Moulding compound	_	cm ³ / 10min	See condition given in ISO 17855-1. Use a value for the melt density of 763,6 kg/m³ to calculate the mass-flow rate of unfilled materials.b		
1.3	Moulding	S_{Mp}	ISO 294-4	60 × 60 × 2	M	%	Parallel		
1.4	shrinkage	S_{Mn}	130 294-4	00 × 00 × 2	IVI	90	Normal		
2 Me	chanical properties								
2.1	Tensile modulus	E_{t}				MPa	Test speed 1 mm/min.		
2.2	Yield stress	$\sigma_{ m y}$				МРа			
2.3	Yield strain	$arepsilon_{ ext{y}}$					Failure with yielding.		
2.4	Nominal strain at break	$arepsilon_{ ext{tB}}$	ISO 527-2			% MPa	test speed 50 mm/min.		
2.5	Stress at 50 % strain	σ_{50}		ISO 20753 Type A1 or A2			Failure without yielding. $\varepsilon_{\rm B} \le 10$ %: test speed 5 mm/		
2.6	Stress at break	$\sigma_{ m B}$					min.		
2.7	Strain at break	$arepsilon_{ m B}$				%	$\varepsilon_{\rm B}$ > 10 %: test speed 50 mm/min.		
2.8	Tensile creep modulus	$\frac{E_{\rm tc}1}{E_{\rm tc}10^3}$	ISO 899-1				MPa	At 1 h At 1 000 h Strain ≤ 0,5 %	
2.10	Flexural modulus	$E_{\rm f}$	ISO 178	80 × 10 × 4		MPa	Test speed 2 mm/min.		
2.11	Charpy notched impact strength	$lpha_{ m cA}$	ISO 179-1 or ISO 179-2	$80 \times 10 \times 4$ Machined V-notch, $r = 0.25$	M/Q	kJ/m ²	Edgewise impact, method		
2.12	Tensile notched impact strength	$lpha_{ m tl}$	ISO 8256	$80 \times 10 \times 4$ Machined double V-notch, $r = 1$		kJ/m²	Only to be quoted if fracture cannot be obtained with notched Charpy test.		
2.13	Puncture energy	W_{P}				J	Striker velocity 4,4 m/s.		
							Striker diameter 20 mm.		
				6063		N	Support ring diameter 40 mm.		
2.14	Maximum puncture force	F_{M}	ISO 6603-2	60 × 60 × 2			Lubricate the striker. Clamp the specimen suffi-		
							ciently to prevent any out of plane movement of its outer regions.		

M = Injection moulding, Q = Compression moulding.

b See Reference [1].

Electrical properties are generally affected by the relative humidity. They shall therefore be measured in a standard atmosphere of 23 °C \pm 2 °C and (50 \pm 10) % relative humidity.

 Table 3 (continued)

				Table 5 (continued	,					
3 Tł	nermal properties									
3.1	Melting temperature	$T_{ m m}$	ISO 11357-3	Mauldina			Record peak melting temperature. Use 10 °C/min heating ing rate.			_
3.2	Glass transition temperature	$T_{ m g}$	ISO 11357-2	Moulding compound	_	°C	Record midpoir ture. Use 10 °C/min l		-	
3.3		T _f 1,8					ing rate. Maxi- 1,8			
3.4	Temperature of de- flection under load	T _f 0,45	ISO 75-2	80 × 10 × 4		°C	mum surface stress (MPa)			Use flatwis loading
3.5		$\alpha_{ m p}$					Parallel]	Reco	ord the
3.6	Coefficient of linear thermal expansion	$\alpha_{ m n}$	ISO 11359-2	Prepared from ISO 20753	M/Q	°C-1	Transverse		seca over cemp	cant value ver the mperature inge 23 °C
3.7		B50/1,5		125 × 13 × 1,5			Record one of the classifica-			
3.8	Burning behaviour	B50/h	IEC 60695- 11-10	Thickness h greater than 1,5 mm			tions V-0, V-1, V-2, HB, HB40 of HB75.			40 or
3.9	Oxygen index	_	ISO 4589-2	80 × 10 × 4		%	Use procedure A (top surfaignition).			p surfac
4 El	ectrical properties ^c									
4.1	Relative permit-	ε _r 100				_	100 Hz			
4.2	tivity	ε _r 1M	IEC 60250				1 MHz		mpensate for ctrode edge	
4.3	Dissipation factor	tanδ 100	120 00230			_	100 Hz		effects.	
4.4	Dissipation factor	tanδ 1M					1 MHz			
4.5	Volume resistivity	$ ho_{ m e}$		≥60 × ≥60 × 2	Q	Ω·m	_	Mea 1 mi		e value a
4.6	Surface resistivity	$\sigma_{ m e}$	IEC 60093			Ω	Voltage 500V	line 1 m wid long	Use contacting line electrodes 1 mm to 2 mm wide, 50 mm long and 5 mm apart.	
4.7	Electric strength	E _B 1	IEC 60243- 1	≥60 × ≥60 × 1		kV/ mm	cal electr Immerse er oil in a IEC 6029	se in transform- accordance with 196. oltage application rate		
4.8	Comparative tracking index	CTI-A	IEC 60112	≥20 × ≥20 × 4		_	Use solut	e solution A.		

a M = Injection moulding, Q = Compression moulding.

b See Reference [1].

Electrical properties are generally affected by the relative humidity. They shall therefore be measured in a standard atmosphere of 23 °C \pm 2 °C and (50 \pm 10) % relative humidity.

Table 3 (continued)

5 Otl	5 Other properties											
5.1	TATatanahaanstian	w_{w}	100.62	ISO 62 60 × 60 × 1	M/0	%	Saturation value in water at 23 °C.					
5.2	Water absorption	$w_{ m H}$	150 62		M/Q		Equilibrium value at 23 °C, 50 % RH.					
5.3	Density	ρ	ISO 1183-1 or ISO 1183-2 or ISO 1183-3	_	Q	kg/ m ³	For comparison purposes only. Not to be used for specifications.					

a M = Injection moulding, Q = Compression moulding.

Table 4 — Additional properties and test conditions of particular utility to PE moulding and extrusion materials

Property Symbo		Symbol	Standard	Specimen type (dimensions in mm)	Specimen preparation ^a	Unit	Test condition and supplementary instructions	
1 Me	chanical properti	es						
1.1	Yield stress	d stress $\sigma_{ m y}$				MPa	Thickness 1 mm or	
1.2	Yield strain	$\varepsilon_{ m y}$	ASTM D638 ^b	True o IV		%	2 mm.	
1.3	Stress at break	$\sigma_{ m B}$	ASTM D6385	Type IV	Q	MPa	Test speed 50 mm/ min.	
1.4	Strain at break	$arepsilon_{ m B}$				%		
2 Ot	her properties							
2.1	Reduced viscosity	I	ISO 1628-3	Moulding compound	_	ml/g		
2.2	Environmental stress-cracking ^c	F ₅₀	ASTM D1693	38 × 13 × h	Q	h	Determine the 50 % failure rate F_{50} using Cond. A, $\rho \le 0.925$ (h: 3,00 to 3,30).	
2.3	Stress-cracking	$t_{ m f}$	ISO 16770	To be selected from ISO 16770			1,84 to 1,97). Use conditions from ISO 16770 according to polymer end-use application.	
2.4	Density	ρ	ISO 1183-1 or ISO 1183-2 or ISO 1183-3	Extrudate	From ISO 1133-1 determination	kg/m³	ISO 17855-1:2014, 3.4.2 This is the designatory density value.	

a Q = Compression moulding.

b See Reference [1].

^c Electrical properties are generally affected by the relative humidity. They shall therefore be measured in a standard atmosphere of 23 °C \pm 2 °C and (50 \pm 10) % relative humidity.

b Use of small tensile bar is allowed when the elongation of multipurpose test specimen is too large to obtain stress or strain at break.

^c Stress-cracking tests give relative comparisons, especially for extrusion types of PE material, and are untypical for many applications. However, tests give good characterization of material suitability for certain applications. Performance tests on products are nevertheless needed for full assessment of material suitability for a given application.

Annex A

(informative)

Interlaboratory trials for density: Immersion method in accordance with ISO 1183-1

Finland arranged an interlaboratory trial for density. The data accumulated showed a significant level of variability in the lab-to-lab reproducibility. In 2005, Finland arranged a new smaller trial in order to find the critical sources of error. For the new trial, the laboratories used all the correction factors recommended by balance manufacturers. The results were good and at an acceptable level. Special attention was paid to the following:

- a) compression moulding: 30 min ageing at 100 °C in the press or in boiling water;
- b) time: measurements after conditioning for 1 h at 23 °C;
- c) cutting of specimens: a sharp cutting tool was used; no voids on the surfaces (at least with PE-HD this is critical);
- d) immersion liquid used: kerosene-based liquid, ethanol;
- e) temperature of bath: 23 °C ± 2 °C;
- f) measurement of liquid density: with a glass float before measurements (this is critical with ethanol);
- g) reading the mass: 2 min after immersion.

Bibliography

[1] ZOLLER P. J. Appl. Polym. Sci. 1979, **23** pp. 1051–1061

