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Plastics — Polyethylene (PE) moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Matériaux à base de polyéthylène (PE) pour moulage et extrusion —

Partie 2: Préparation des éprouvettes et détermination des propriétés

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Contents

Page

Foreword

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This document was prepared by Technical Committee Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC9, *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 second edition cancels and replaces the first edition (ISO 17855-2:2016), which has been technically revised.

The main changes are as follows:

— ISO 16241, ISO 18488, ISO 18489 and ISO 22088-2 have been integrated to Clause 2;

— Table 1 Conditions for injection moulding of test specimens have been changed;

— Table 2 Conditions for compression moulding of test specimens have been changed;

— properties from ISO 16241, ISO 18488, ISO 18489 and ISO 22088-2 have been integrated in Table 4;

— Annex A (informative) has been deleted.

A list of all parts in the ISO 17855 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 www.iso.org/members.html.

Plastics — Polyethylene (PE) 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 for determining the properties of polyethylene (PE) moulding and extrusion materials. It gives requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing.

This document specifies 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 that are suitable and essential to characterize PE moulding and extrusion materials are listed.

The properties in this document have been selected from the general test methods in ISO 10350-1. Other test methods in wide use for or of particular significance to PE moulding and extrusion materials are also included in this document, as are the designatory properties specified in ISO 17855-1. Properties of slow crack growth, etc. are specified in documents of polyethylene (PE) materials for piping systems.

The methods of preparation and conditioning, the specimen dimensions and the test procedures specified herein are used 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*

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*

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 step height*

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

ISO 11357-6, *Plastics — Differential scanning calorimetry (DSC) — Part 6: Determination of oxidation induction time(isothermal OIT)and oxidation induction temperature (dynamic OIT)*

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

ISO 16241, *Notch tensile test to measure the resistance to slow crack growth of polyethylene materials for pipe and fitting products (PENT)*

ISO 16770, *Plastics — Determination of environmental stress cracking (ESC) of polyethylene — Full-notch creep test (FNCT)*

ISO 17855-1, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 1: Designation system and basis for specifications*

ISO 18488, *Polyethylene (PE) materials for piping systems — Determination of Strain Hardening Modulus in relation to slow crack growth — Test method*

ISO 18489, *Polyethylene (PE) materials for piping systems — Determination of resistance to slow crack growth under cyclic loading — Cracked Round Bar test method*

ISO 20753, *Plastics — Test specimens*

ISO 22088-2, *Plastics — Determination of resistance to environmental stress cracking (ESC) — Part 2: Constant tensile load method*

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 — Mineral insulating oils for electrical equipment*

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 - 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*

ASTM D 638, *Standard test method for tensile properties of plastics*

ASTM D 1693, *Standard test method for environmental stress-cracking of ethylene plastics*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Preparation of test specimens

4.1 General

Specimens shall 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 shall be in accordance with those indicated in Tables 3 and 4 (M = injection moulding, Q = compression moulding).

4.2 Treatment of the material before moulding

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

4.3 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 Table 1. The preparation of test specimens by machining is used as agreed upon by the interested parties.

NOTE It has been determined that bar test specimens prepared by cutting from type A1 specimens according to ISO 20753 give better precision than those injection-moulded specimens.

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

Table 1 — Conditions for injection moulding of test specimens

Material	Melt temperature °C	Mould temperature °C	Average Injection velocity[a] mm/s	Hold time s	Cycle time s
MFR ≥ 1 g/10 min	210	40	100 ± 10	30 ± 5	50 ± 5
[a] Average injection velocity shall be calculated according to ISO 294-1					

4.4 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 (≤ 2 mm thick) and where specifically prescribed in Tables 3 and 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 Table 2.

Table 2 — Conditions for compression moulding of test specimens

Material	Moulding temperature °C	Cooling rate k /min	Demoulding temperature °C	Preheating pressure MPa	Preheating time min	Full pressure MPa	Full-pressure time min
All grade [a]	180	15 ± 2[b]	≤ 40	<0,1 [c]	5 to 15	5 or 10[d]	5 ± 1
[a] Different compression moulding conditions apply to specimens for SCR and ESCR measurements, in particular for specimen thicknesses of 10 mm and above. Appropriate compression moulding conditions for these applications shall be taken from ISO 16241, ISO 16770 and ISO 18489.							
[b] Cooling method B1 of ISO 293 can keep a constant (linear) cooling rate.							
[c] According to ISO 293, preheating pressure is contact pressure which less than 0,1 MPa.							
[d] Use 5 MPa for a frame mould and 10 MPa for a positive mould.							

If the preheating of the material is insufficient, the sheets produced will suffer from insufficient homogeneity and pellet boundaries. 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 If some materials need to change the moulding temperature, such as 210 °C, it need to be negotiated by relevant parties.

If the tested PE material is a powder, it may be necessary to calendar or compound the material prior to the compression moulding step. The powder shall be heat-stabilized when this is done.

Whether it is frame or positive mould, it is necessary to start the cooling cycle whilst simultaneously applying the full pressure. This avoids sink marks for frame mould and avoids the physical properties are influenced by different pressure when positive mould is used.

For 4 mm thick sheets, the positive mould has been found to work satisfactorily, while for the thicker sheets (>4 mm) use of the positive mould is necessary.

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 for 24 h after moulding. Annealing of the sheets would be carried out if it is required by relevant standard.

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.

5 Conditioning of test specimens

Unfilled PE test specimens shall be conditioned for at least 16 h at 23 °C ± 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 ± 2 °C and (50 ± 10) % relative humidity unless sufficient testing has been conducted that indicates that specific material properties are not affected by 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 3 and 4, testing of unfilled PE test specimens shall be carried out at a standard temperature of 23 °C ± 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 ± 2 °C and (50 ± 10) % relative humidity unless sufficient testing has been conducted that indicates that specific material properties are not affected by humidity.

Table 3, compiled from ISO 10350-1, lists properties which are appropriate to polyethylene (PE) moulding and extrusion materials. These properties shall be used for comparisons of data generated for different thermoplastics.

Table 4 contains properties, not found specifically in Table 3, which shall be used 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 preparation[a]	Unit	Test condition and supplementary instructions
1 Rheological properties							
1.1	Melt mass-flow rate	MFR	ISO 1133-1	Moulding compound	--	g/10 min	See condition given in ISO 17855-1.
1.2	Melt volume-flow rate	MVR				cm^3/10 min	See condition given in ISO 17855-1. Use a value for the melt density of 763,6 kg/m^3 to calculate the mass-flow rate of unfilled materials.[b]
1.3	Moulding shrinkage	S_{Mp}	ISO 294-4	60 × 60 × 2	M	%	Parallel
1.4		S_{Mn}					Normal
2 Mechanical properties							
2.1	Tensile modulus	E_t	ISO 527-2	ISO 20753 Type A1 or A2	M/Q	MPa	Test speed 1 mm/min.
2.2	Yield stress	σ_y					Failure with yielding. test speed 50 mm/min.
2.3	Yield strain	ε_y				%	
2.4	Nominal strain at break	ε_{tb}					
2.5	Stress at 50 % strain	σ_{50}				MPa	Failure without yielding. $\varepsilon_b \leq 10$ %: test speed 5 mm/min. $\varepsilon_b > 10$ %: test speed 50 mm/min.
2.6	Stress at break	σ_b					
2.7	Strain at break	ε_b				%	
2.8	Tensile creep modulus	$E_{tc}1$	ISO 899-1			MPa	At 1 h; Strain ≤ 0,5 %.
2.9		$E_{tc}10^3$					At 1 000 h
2.10	Flexural modulus	E_f	ISO 178	80 × 10 × 4		MPa	Test speed 2 mm/min.
2.11	Charpy notched impact strength	α_{cA}	ISO 179-1 or ISO 179-2	80 × 10 × 4 Machined V-notch, r = 0,25		kJ/m^2	Edgewise impact, method 1eA. Also record type of failure.

[a] M = Injection moulding, 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 ± 2 °C and (50 ± 10) % relative humidity.

Table 3 *(continued)*

Property		Symbol	Standard	Specimen type (dimensions in mm)	Specimen preparation[a]	Unit	Test condition and supplementary instructions
2.12	Tensile notched impact strength	α_{tl}	ISO 8256	80 × 10 × 4 Machined double V-notch, r = 1		kJ/m^2	Only to be quoted if fracture cannot be obtained with notched Charpy test.
2.13	Puncture energy	W_p	ISO 6603-2	60 × 60 × 2		J	Striker velocity 4,4 m/s. Striker diameter 20 mm. Support ring diameter 40 mm. Lubricate the striker. Clamp the specimen sufficiently to prevent any out of plane movement of its outer regions.
2.14	Maximum puncture force	F_M				N	
3 Thermal properties							
3.1	Melting temperature	T_m	ISO 11357-3	Moulding compound	—	°C	Record peak melting temperature. Use 10 K/min heating/cooling rate
3.2	Glass transition temperature	$T_{1/2,g}$	ISO 11357-2				Use 10 K /min heating rate. Half-step-height method
3.3	Temperature of deflection under load	T_f 1,8	ISO 75-2	80 × 10 × 4	M/Q	°C	Maximum surface stress (MPa): 1,8; 0,45. Use flatwise loading.
3.4		T_f 0,45					
3.5	Coefficient of linear thermal expansion	α_p	ISO 11359-2	Prepared from ISO 20753		°C^{-1}	Parallel; Transverse. Record the secant value over the temperature range 23 °C to 55 °C.
3.6		α_n					
3.7	Burning behaviour	B50/1,5	IEC 60695-11-10	125 × 13 × 1,5			Record one of the classifications V-0, V-1, V-2, HB, HB40 or HB75.
3.8		B50/h		Thickness h greater than 1,5 mm			
3.9	Oxygen index	—	ISO 4589-2	80 × 10 × 4		%	Use procedure A (top surface ignition).
4 Electrical properties[c]							
4.1	Relative permittivity	ε_r 100	IEC 62631-2-1	≥ 60×≥ 60× 2	Q	—	100 Hz; 1 MHz. Compensate for electrode edge effects.
4.2		ε_r 1 M					
4.3	Dissipation factor	tanδ 100				—	100 Hz; 1 MHz.
4.4		tanδ 1 M					
4.5	Volume resistivity	ρ_e	IEC 62631-3-1	≥ 60 × ≥ 60 × 2		Ω•m	Measure value at 1 minute.
4.6	Surface resistivity	σ_e	IEC 62631-3-2			Ω	Voltage 500 V. 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	Use 20 mm diameter spherical electrodes. Immerse in mineral insulating oils in accordance with IEC 60296. Use a voltage application rate of 2 kV/s.
4.8	Comparative tracking index	CTI-A	IEC 60112	≥ 20 × ≥ 20 × 4		—	Use solution A.

[a] M = Injection moulding, 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 ± 2 °C and (50 ± 10) % relative humidity.

Table 3 *(continued)*

Property		Symbol	Standard	Specimen type (dimensions in mm)	Specimen preparation[a]	Unit	Test condition and supplementary instructions
5 Other properties							
5.1	Water absorption	w_w	ISO 62	60 × 60 × 1	M/Q	%	Saturation value in water at 23 °C.
5.2		w_H					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.

[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 ± 2 °C and (50 ± 10) % relative humidity.

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

Property		Symbol	Standard	Specimen type (dimensions in mm)	Specimen preparation[a]	Unit	Test condition and supplementary instructions
1 Mechanical properties							
1.1	Yield stress	σ_y	ASTM D 638 [b]	Type IV	Q	MPa	Thickness 1 mm or 2 mm. Test speed 50 mm/min.
1.2	Yield strain	ε_y				%	
1.3	Stress at break	σ_B				MPa	
1.4	Strain at break	ε_B				%	
2 Other properties							
2.1	Reduced viscosity	I	ISO 1628-3	Moulding compound	—	ml/g	
2.2	Environmental stress-cracking c	F50	ASTM D 1693	38 × 13 × h	Q	h	Determine the 50 % failure rate F50 using Cond. A, ρ≤ 0,925 (h: 3,00 to 3,30). Cond. B, ρ> 0,925 (h: 1,84 to 1,97).
2.3		t_f	ISO 16770	To be selected from ISO 16770			Sheet annealing procedure according to ISO 16770 Use conditions from ISO 16770 according to polymer end-use application.
		t	ISO 22088-2	Type 1BA of ISO 527-2	M / Q	MPa	Thickness(2 ± 0,2) mm is recommended. 3 mm to 4 mm or products thickness may be used.
		σ				h	
2.4	Strain hardening modulus	$<G_p>$	ISO 18488	To be selected from ISO 18488	Q	MPa	Sheet annealing procedure according to ISO 18488.

[a] M = Injection moulding, Q = Compression moulding.

[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. Tests give good characterization of material suitability for certain applications, however. Performance tests on products are nevertheless needed for full assessment of material suitability for a given application.