

Plastics — Polyethylene (PE) moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee PRI/82, Thermoplastic materials.

A list of organizations represented on PRI/82 can be obtained on request to its secretary.

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Part 2: Preparation of test specimens and determination of
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Plastiques - Polyéthylène (PE) pour moulage et extrusion -
Partie 2: Préparation des éprouvettes et détermination des
propriétés (ISO 1872-2:2007)

Kunststoffe - Polyethylen (PE)-Formmassen - Teil 2:
Herstellung von Probekörpern und Bestimmung von
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EN ISO 1872-2:2007

Foreword

This document (EN ISO 1872-2:2007) has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2007, and conflicting national standards shall be withdrawn at the latest by August 2007.

This document supersedes EN ISO 1872-2:1997.

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Endorsement notice

The text of ISO 1872-2:2007 has been approved by CEN as EN ISO 1872-2:2007 without any modifications.

INTERNATIONAL
STANDARD

ISO
1872-2

Third edition
2007-02-01

**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*



Reference number
ISO 1872-2:2007(E)

Foreword

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ISO 1872-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This third edition cancels and replaces the second edition (ISO 1872-2:1997), which has been technically revised, and incorporates Amendment Amd.1:2000.

ISO 1872 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 1872 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 1872, as are the designatory properties specified in ISO 1872-1.

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 referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 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*

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- 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 1133:2005, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*
- 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 1183-3, *Plastics — Methods for determining the density of non-cellular plastics — Part 3: Gas pycnometer method*
- ISO 1628-3, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 3: Polyethylenes and polypropylenes*
- ISO 1872-1:1993, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 1: Designation system and basis for specifications*
- ISO 2818, *Plastics — Preparation of test specimens by machining*
- ISO 3167, *Plastics — Multipurpose test specimens*
- 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 (Under revision)*
- ISO 11357-2, *Plastics — Differential scanning calorimetry (DSC) — Part 2: Determination of glass transition temperature*
- 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)*
- 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 switchgears*

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

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 Preparation of test specimens

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 Tables 3 and 4 (M = injection moulding, Q = compression moulding).

3.1 Treatment of the material before moulding

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

3.2 Injection moulding

Injection moulding of test specimens is used for PE moulding materials having a melt mass-flow rate of ≥ 1 g/10 min, determined in accordance with ISO 1133:2005 using set of test conditions D (190 °C/2, 16 kg).

Injection-moulded specimens shall be prepared in accordance with ISO 294-1 or ISO 294-3, using the conditions specified in Table 1. It has been found that type A bar test specimens (prepared in accordance with ISO 3167) give better precision than type B (injection-moulded directly to their final dimensions) and so the use of this geometry is preferable.

NOTE Details of the work can be found at: <http://isotc.iso.ch/livelink/livelink?func=ll&objid=927134&objAction=browse&> on the SC9 server. (11 Round-robin test results: Technical report. Comparison of precision data for two types of bar test specimens formed in PP and PE.)

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 mm/s	Cooling time s	Total cycle time s
MFR ≥ 1 g/10 min	210	40	100 \pm 20	35 \pm 5	40 \pm 5

3.3 Compression moulding

Compression moulding is used for materials with a melt mass-flow rate of < 1 g/10 min, determined in accordance with ISO 1133:2005 using set of test conditions D (190 °C/2,16 kg). 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. The test specimens required for the determination of the properties shall be machined from the compression-moulded sheets in accordance with ISO 2818, or stamped

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

Table 2 — Conditions for compression moulding of test specimens

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	180	15	≤ 40	5/10 ^a	5 ± 1	Contact	5 to 15
^a Use 5 MPa for a frame mould and 10 MPa for a positive mould. NOTE 1 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. NOTE 2 For a type 1 mould, since full pressure is only applied upon the frame, compression-moulded sheet may suffer from insufficient homogeneity and pellet boundaries may be preserved.							

A type 1 (frame) mould may be used, but it is necessary to start the cooling cycle whilst 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 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 up to 15 min may be necessary, especially if electric heating is used.

4 Conditioning of test specimens

Unfilled PE test specimens shall be conditioned for at least 16 h at $23 \text{ °C} \pm 2 \text{ °C}$, with no relative humidity requirement. Specimens made from materials containing fillers or additives that are susceptible to moisture uptake shall be conditioned for at least 16 h at $23 \text{ °C} \pm 2 \text{ °C}$ 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 Table 3 and 4, testing of unfilled PE test specimens shall be carried out in the standard atmosphere of $23 \text{ °C} \pm 2 \text{ °C}$ with no relative humidity requirement. Specimens made from materials containing fillers and additives that are susceptible to moisture uptake shall be tested in the standard atmosphere of $23 \text{ °C} \pm 2 \text{ °C}$ 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.

Table 4 contains those properties not found specifically in Table 3 that 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	International Standard	Specimen type (dimensions in mm)	Specimen preparation ^a	Unit	Test conditions and supplementary instructions	
1 Rheological properties							
1.1	Melt mass-flow rate	MFR	ISO 1133	Moulding compound	—	g/10 min	See conditions given in ISO 1872-1.
1.2	Melt volume-flow rate	MVR				cm ³ /10 min	See conditions given in ISO 1872-1. 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 3167	M/Q	MPa	Test speed 1 mm/min
2.2	Yield stress	σ_y					%
2.3	Yield strain	ε_y				MPa	
2.4	Nominal strain at break	ε_{tB}					%
2.5	Stress at 50 % strain	σ_{50}				MPa	
2.6	Stress at break	σ_B					%
2.7	Strain at break	ε_B				MPa	
2.8	Tensile creep modulus	$E_{tc} \cdot 1$	ISO 899-1	At 1 000 h			
2.9		$E_{tc} \cdot 10^3$					
2.10	Flexural modulus	E_f	ISO 178	80 × 10 × 4	MPa	Test speed 2 mm/min	
2.11	Charpy notched impact strength	a_{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.	
2.12	Tensile notched impact strength	a_{tI}	ISO 8256	80 × 10 × 4 Machined double V-notch, $r = 1$		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.	
2.14	Maximum puncture force	F_M			N	Clamp the specimen sufficiently to prevent any out of plane movement of its outer regions.	

Table 3 (continued)

Property		Symbol	International Standard	Specimen type (dimensions in mm)	Specimen preparation ^a	Unit	Test conditions and supplementary instructions		
3 Thermal properties									
3.1	Melting temperature	T_m	ISO 11357-3	Moulding compound	—	°C	Record peak melting temperature. Use 10 °C/min rise and fall.		
3.2	Glass transition temperature	T_g	ISO 11357-2				Record midpoint temperature. Use 10 °C/min rise and fall.		
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 3167	M/Q	°C ⁻¹	Parallel	Record the secant value over the temperature range 23 °C to 55 °C.	
3.6		α_n					Transverse		
3.7	Burning behaviour	B50/1,5	IEC 60695-11-10	125 × 13 × 1,5	M/Q		Record one of the classifications V-0, V-1, V-2, HB, HB40 or HB75.		
3.8		B50/h		Other 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 60250	$\geq 60 \times \geq 60 \times 2$	Q		—	100 Hz	Compensate for electrode edge effects.
4.2		ϵ_r 1M					—	1 MHz	
4.3	Dissipation factor	$\tan \delta$ 100					—	100 Hz	
4.4		$\tan \delta$ 1M					—	1 MHz	
4.5	Volume resistivity	ρ_e	IEC 60093		Q		$\Omega \cdot m$	Value at 1 min	
4.6	Surface resistivity	σ_e					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	$\geq 60 \times \geq 60 \times 1$		kV/mm	Use 20 mm diameter spherical electrodes. Immerse in transformer oil in accordance with IEC 60296. Use a voltage application rate of 2 kV/s.		
4.8	Comparative tracking index	CTI-A	IEC 60112	$\geq 20 \times \geq 20 \times 4$		—	Use solution A.		
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 Reference: P. Zoller, *Journal of Applied Polymer Science*, **23**, 1979, pp. 1051-1061.

^c Electrical properties are generally affected by the relative humidity. So, they must 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	Inter-national Standard	Specimen type (dimensions in mm)	Specimen preparation ^a	Unit	Test conditions and supplementary instructions	
1 Mechanical properties							
1.1	Yield stress	σ_y	ASTM D 638 ^b	Type IV	Q	Thickness 1 mm or 2 mm Test speed 50 mm/min	
1.2	Yield strain	ε_y					MPa
1.3	Stress at break	σ_B					%
1.4	Strain at break	ε_B					MPa
2 Other properties							
2.1	Reduced viscosity	l	ISO 1628-3	Moulding compound	—	ml/g	
2.2	Environmental stress-cracking ^c	F_{50}	ASTM D 1693	$38 \times 13 \times h$	Q	h	Determine the 50 % failure rate F_{50} using Cond. A, $\rho \leq 0,925$ (h : 3,00 to 3,30) Cond. B, $\rho > 0,925$ (h : 1,84 to 1,97)
2.3		t_f	ISO 16770	To be selected from ISO 16770			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 determination	kg/m ³	ISO 1872-1:1993, 3.3.1 This is the designatory density value.
<p>^a M = Injection moulding Q = Compression moulding</p> <p>^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.</p> <p>^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.</p>							

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.

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