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Plastics — Polypropylene (PP) moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Polypropylène (PP) pour moulage et extrusion —

Partie 2: Préparation des éprouvettes et détermination des propriétés

Reference number
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Foreword

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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 19069-2 cancels and replaces the third edition of ISO 1873-2:2007, which has been technically revised.

ISO 19069 consists of the following parts, under the general title *Plastics — Polypropylene (PP) moulding and extrusion materials:*

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

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

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Plastics — Polypropylene (PP) moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

1 Scope

This part of ISO 19069 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of polypropylene (PP) 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 which are suitable and necessary to characterize PP 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 19069, as are the designatory properties specified in ISO 19069-1.

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 tes*

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 16152, *Plastics — Determination of xylene-soluble matter in polypropylene*

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*

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ASTM D 5420, *Standard Test Method for Impact Resistance of Flat, Rigid Plastic Specimen by Means of a Striker Impacted by a falling Weight (Gardner Impact)*

3 Preparation of test specimens

3.1 General

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

3.2 Treatment of the material before moulding

Pre-treatment of the material sample is normally not necessary before moulding.

3.3 Injection moulding

Injection-moulded specimens shall be prepared in accordance with ISO 294-1 or ISO 294-3, under the conditions specified in Table 1. 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.

Table 1 — Conditions for injection moulding of test specimens

Material	Melt temperature	Mould temperature	Average injection velocity	Hold pressure time	Total cycle time
	°C	°C	mm/s	s	s
MFR < 1,5 g/10 min	255	40	200 ± 20	40	60
1,5 ≤ MFR < 7 g/10 min	230	40	200 ± 20	40	60
MFR ≥ 7 g/10 min	200	40	200 ± 20	40	60

NOTE 1 The uniformity of the mouldings shall be checked by weighing. Their masses shall not differ by more than 1 % from each other.

NOTE 2 Heat-sensitive polypropylenes may undergo molecular breakdown during moulding; therefore an increase in the melt flow rate (MFR) to > 1,5 times the original value shall be avoided with such materials. If the MFR increased by more than 1,5 times the original value, the melt temperature shall be lowered, 10°C at a time, until the increase in MFR is < 1,5 times the original value. This adjustment in melt temperature shall be reported.

3.4 Compression moulding

Compression-moulded sheets shall be prepared in accordance with ISO 293 under the conditions specified in Table 2.

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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	210	15 ± 5	≤ 40	5 or 10[a]	5 ± 1	contact	5 to 15

[a] 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. It is therefore desirable to use a moulding machine that is capable of maintaining a constant cooling rate.

If a frame mould is used, 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.

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.

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 thicknesses 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 test specimens shall be conditioned for a period of between 40 h and 96 h at (23 ± 2) °C, with no relative humidity requirement. Specimens containing fillers or additives that are susceptible to moisture uptake shall be conditioned in the same way but with an additional requirement for (50 ± 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 Tables 3 and 4, testing of unfilled test specimens shall be carried out at a standard temperature of (23 ± 2) °C, with no relative humidity requirement. Specimens made from materials containing fillers or additives that are susceptible to moisture uptake shall be tested in a standard atmosphere of (23 ± 2) °C and (50 ± 10) % relative humidity.

Table 3 of this part of ISO 10350 is compiled from ISO 10350-1 and the properties listed are those which are appropriate to polypropylene (PP) 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, which are in wide use or of particular significance in the practical characterization of polypropylene (PP) moulding and extrusion materials.

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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	Temperature 230 °C, load 2,16 kg
1.2	Melt volume-flow rate	MVR				cm^3/10 min	Temperature 230 °C, load 2,16 kg Use a value for the melt density of 738,6 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	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; Strain ≤ 0,5 %
2.10	Flexural modulus	E_f	ISO 178	80 × 10 × 4		MPa	Test speed 2 mm/min.
2.11	Charpy impact strength	α_{cU}	ISO 179-1 or ISO 179-2	80 × 10 × 4		kJ/m^2	Edgewise impact, method 1eU. Also record type of failure.
2.12	Charpy notched impact strength	α_{cA}		80 × 10 × 4 Machined V-notch, r = 0,25			Edgewise impact, method 1eA. Also record type of failure.
2.13	Tensile notched impact strength	α_{tI}	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.

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Table 3 *(continued)*

Property		Symbol	Standard	Specimen type (dimensions in mm)	Specimen preparation[a]	Unit	Test condition and supplementary instructions
2.14	Puncture energy	W_P	ISO 6603-2	60 × 60 × 2	M	J	Striker velocity 4,4 m/s. Striker diameter 20 mm.
2.15	Maximum puncture force	F_M				N	Support ring diameter 40 mm. Lubricate the striker. Clamp the specimen sufficiently to prevent any out-of-plane movement of its outer regions.
3 Thermal properties							
3.1	Melting temperature	T_m	ISO 11357-3	Moulding compound	—	°C	Record peak melting temperature. Use 10 °C/min heating/cooling rate.
3.2	Glass transition temperature	T_g	ISO 11357-2				Record midpoint temperature. Use 10 °C/min heating/cooling rate.
3.3	Temperature of deflection under load	T_f 1,8	ISO 75-2	80 × 10 × 4	M	°C	Max surface stress (MPa): 1,8
3.4		T_f 0,45					Max surface stress (MPa): 0,45 Use flatwise loading
3.5	Coefficient of linear thermal expansion	α_p	ISO 11359-2	Prepared from ISO 20753		°C–1	Parallel
3.6		α_n					Transverse Record the secant value over the temperature range 23 °C to 55 °C.
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 60250	≥ 60 × ≥ 60 × 2	M/Q	—	100 Hz Compensate for electrode edge effects.
4.2		ε_r 1M					1 MHz
4.3	Dissipation factor	tanδ 100				—	100 Hz
4.4		tanδ 1M					1 MHz

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Table 3 *(continued)*

Property		Symbol	Standard	Specimen type (dimensions in mm)	Specimen preparation[a]	Unit	Test condition and supplementary instructions	
4.5	Volume resistivity	ρ_e	IEC 60093	≥ 60 × ≥ 60 × 2	M/Q	Ω·m	Voltage 500 V	Measure value at 1 min.
4.6	Surface resistivity	σ_e				Ω		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 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	≥ 20 × ≥ 20 × 4	M	—	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	Use part of centre of multipurpose test specimen	M	kg/m^3		

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

[b] See Reference [1].

[c] Electrical properties are generally affected by the relative humidity, and should be measured in a standard atmosphere of (23 ± 2) °C and (50 ± 10) % relative humidity.

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

Property		Symbol	Standard	Specimen type (dimensions in mm)	Specimen preparation[a]	Unit	Test condition and supplementary instructions
1	Reduced viscosity	I	ISO 1628-3	Moulding compound	—	ml/g	
2	Gardner impact strength	MFE	ASTM D 5420	3,2 mm thick	M	J	Method GC
3	Xylene-soluble fraction	S_S	ISO 16152	Moulding compound	—	%	

[a] M = Injection moulding.

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

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