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Plastics — Polybutene-1 (PB-1) moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Matériaux à base de polybutène-1 (PB-1) 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 www.iso.org/directives).

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 www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This first edition of ISO 21302-2 cancels and replaces ISO 8986-2:2009, which has been technically revised. The main changes compared to the previous edition are as follows:

— the symbol of general properties and additional properties has been added;

— the type and size of tensile sample ISO 527-4 (1B type test) has been modified to ISO 20753 (A1 or A2 type test);

— the type and size of the relative permittivity, dissipation factor, volume resistivity and surface resistivity samples have been modified from ≥ 80 × ≥ 80 × 1 to ≥ 60 × ≥ 60 × 2;

— the type and size of the electric strength sample has been modified from ≥ 80 × ≥ 80 × 1 or ≥ 80 × ≥ 80 × 3 to ≥ 60 × ≥ 60 × 1;

— the type and size of the water absorption sample has been modified from 50 × 50 × 3 or ø50 × 3 to 60 × 60 × 1;

— the test conditions of the density have been added. (the sample was regulated in a standard atmosphere of 23 °C ± 2 °C and 50 % ± 10 % relative humidity).

A list of all parts in the ISO 21302 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 — Polybutene-1 (PB-1) 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 polybutene-1 (PB-1) moulding and extrusion materials. For the sake of simplicity, the designation polybutene-1 and the abbreviation PB are used in this document. Requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing are also specified.

Procedures and conditions for the preparation of test specimens and procedures for measuring properties of the materials from which these specimens are made are given. Properties and test methods which are suitable and necessary to characterize PB-1 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 is the designatory property specified in ISO 21302-1.

In order to obtain reproducible and comparable test results, it is intended to use the methods of specimen preparation and conditioning, the specimen dimensions and the test procedures specified in this document. 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 291, *Plastics — Standard atmospheres for conditioning and testing*

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

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

ISO 20753, *Plastics — Test specimens*

ISO 21302-1, *Plastics — Polybutene-1 (PB-1) moulding and extrusion materials — Part 1: Designation system and basis for specifications*

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*

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 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 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*

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 http://www.electropedia.org/

4 Preparation of test specimens

4.1 General

The test specimens shall be prepared by compression moulding.

It is essential that the specimens are always prepared by the same procedure using the same processing conditions.

The material shall be kept in moisture-proof containers until it is required for use.

The moisture content of filled or reinforced materials shall be expressed as a percentage of the total mass of the compound.

4.2 Treatment of the material before moulding

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

4.3 Compression moulding

Compression-moulded sheets shall be prepared in accordance with ISO 293, using the conditions specified in Table 1.

Table 1 — 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	200	30	30 ± 5	5 or 10[a]	5 ± 1	Contact	5 to 15
[a] Use 5 MPa with a frame mould and 10 MPa with a positive mould.							

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

A type 1 (frame) mould 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 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 or 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.

NOTE 1 Since only the average cooling rate is specified, the actual cooling rate during crystallization is not fixed. This can lead to significant deviations in properties related to crystallinity, such as density and mechanical properties.

NOTE 2 Since for frame moulds full pressure is only applied upon cooling, compression-moulded sheets can suffer from insufficient homogeneity and pellet boundaries can be preserved if the heating time or the pressure is insufficient.

5 Conditioning of test specimens

Before testing, test specimens shall be conditioned at atmospheric pressure in one of the standard atmospheres specified in ISO 291 for the length of time recommended by the material manufacturer. The conditioning atmosphere and conditioning time used shall be reported along with the test conditions in the test report.

Due to the temperature dependence of the conditioning process, conditioning of test specimens at 27 °C may require longer or lead to unsatisfactory test results. For the sake of the reproducibility of the results, conditioning at 23 °C is therefore preferred.

Because of the slowness of the crystalline-phase transition which takes place after PB compounds have solidified from the melt and which results in significant changes in characteristics such as shrinkage and tensile properties, it is necessary to delay physical testing after moulding until this phase transition is complete.

The use of accelerated ageing at elevated pressure is allowed if it can be demonstrated that the test results are reproducible and equivalent to those obtained on specimens aged at atmospheric pressure.

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. All tests shall be carried out in one of the standard atmospheres specified in ISO 291 unless specifically stated otherwise in Tables 2 and 3.

NOTE Comparison of data obtained under different test conditions can lead to erroneous conclusions.

Table 2 is compiled from ISO 10350-1, and the properties listed are those which are appropriate to polybutene (PB) moulding and extrusion materials. These properties are those considered useful for comparisons of data generated for different thermoplastics.

Table 3 contains those properties, not found specifically in Table 2, which are in wide use or of particular significance in the practical characterization of polybutene (PB) moulding and extrusion materials.

Table 2 — General properties and test conditions (selected from ISO 10350-1)

Property		Symbol	Standard	Specimen type (dimensions in mm)	Specimen preparation[a]	Unit	Test conditions and supplementary instructions
1. Rheological properties							
1.1	Melt volume-flow rate	MVR	ISO 1133-1	Moulding compound	—	$cm^3/10$ min	See conditions given in ISO 21302-1.
1.2	Melt mass-flow rate	MFR				g/10 min	Use melt density of 776,5 kg/m^3 to calculate the melt mass-flow rate.
2. Mechanical properties							
2.1	Tensile modulus	E_t	ISO 527-2	ISO 20753 Type A1 or A2	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		Q	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	Q	MPa	Test speed 2 mm/min
2.11	Charpy impact strength	α_{cU}		80 × 10 × 4	Q	kJ/m^2	Method 1eU (edgewise impact)
2.12	Charpy notched impact strength	α_{cA}	ISO 179-1 or ISO 179-2	80 × 10 × 4 V-notch, r = 0,25	Q		Method 1eA (edgewise impact)
2.13	Tensile notched impact strength	α_{tI}	ISO 8256	80 × 10 × 4 double V-notch, r = 1	Q		Only to be quoted if fracture cannot be obtained with notched Charpy test

a Q = Compression moulding.

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