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Plastics — Polyamide (PA) homopolymers for moulding and extrusion —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Homopolymères polyamides (PA) pour moulage et extrusion —

Partie 2: Préparation des éprouvettes et détermination des caractéristiques

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1874-2 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Together with ISO 1874-1 : 1985 it cancels and replaces ISO Recommendation R 1874 : 1971, of which the two parts of ISO 1874 constitute a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Plastics — Polyamide (PA) homopolymers for moulding and extrusion —

Part 2: Preparation of test specimens and determination of properties

1 Scope and field of application

This part of ISO 1874 specifies methods for the preparation of test specimens and the determination of characteristic properties of polyamide materials, including those properties used for designation purposes as described in ISO 1874-1.

Fixed test conditions are essential to allow direct comparison of results. Therefore, in specifications and agreements between interested parties, reference should be made to the methods specified in this part of ISO 1874.

The properties of articles produced from polyamide materials are governed by the nature of the moulding compound, the design and gating of the mould and the state of the moulding resulting from processing and post-treatment conditions. Therefore, the results of tests carried out by the methods in this part of ISO 1874 are valid only for the specified test specimens and not for articles of other shapes or for specimens of other dimensions and/or that are produced under other conditions.

2 References

ISO 75, *Plastics and ebonite — Determination of temperature of deflection under load.*

ISO 175, *Plastics — Determination of the effects of liquid chemicals, including water.*

ISO 178, *Plastics — Determination of flexural properties of rigid plastics.*

ISO 179, *Plastics — Determination of Charpy impact strength of rigid materials.*

ISO 180, *Plastics — Determination of Izod impact strength of rigid materials.*

ISO 291, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 294, *Plastics — Injection moulding test specimens of thermoplastic materials.*

ISO 307, *Plastics — Polyamides — Determination of viscosity number.*

ISO 527, *Plastics — Determination of tensile properties.* [1)]

ISO 537, *Plastics — Testing with the torsion pendulum.*

ISO 599, *Plastics — Polyamide homopolymers — Determination of matter extractable by boiling methanol.*

ISO 899, *Plastics — Determination of tensile creep.*

ISO 960, *Plastics — Polyamides — Determination of water content.* [1)]

ISO 1110, *Plastics — Polyamides — Accelerated conditioning of test specimens.* [1)]

ISO 1183, *Plastics — Methods for determining the density and relative density (specific gravity) of plastics excluding cellular plastics.*

ISO 1874-1, *Plastics — Polyamide (PA) homopolymers for moulding and extrusion — Part 1: Designation.*

ISO 2039-1, *Plastics — Determination of hardness — Part 1: Ball indentation method.*

ISO 2039-2, *Plastics — Determination of hardness — Part 2: Rockwell hardness.*

1) At present at the stage of draft.

ISO 3146, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers.*

ISO 3451-4, *Plastics — Determination of ash — Part 4: Polyamides.*

IEC Publication 93, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials.*

IEC Publication 112, *Method for determining the comparative and the proof tracking indices of solid insulating materials under moist conditions.*

IEC Publication 243, *Recommended methods of test for electric strength of solid insulating materials at power frequencies.*

IEC Publication 250, *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.*

3 Preparation of test specimens

Test specimens shall be prepared by injection moulding, observing the general instructions given in ISO 294 and the specific instructions and recommendations given below in 3.1 to 3.4.

3.1 Treatment of the moulding material

The test specimens shall be prepared from granules containing not more than 0,2 % moisture in the case of moulding material with a viscosity number up to 200, and not more than 0,1 % moisture in the case of moulding material with a viscosity number greater than 200.

For this reason, the granules shall be kept in moisture-proof containers until they are required for use. Before they are processed, they shall have attained room temperature.

The water content of filled and reinforced materials shall always refer to the matrix.

If the moisture content of the granules exceeds the appropriate limit given above, the material shall be dried at a temperature of 80 to 100 °C before the test specimens are produced, either in vacuo or in a stream of dry nitrogen, until the moisture content no longer exceeds the prescribed limit.

3.2 Injection moulding machine

Specimens with comparable properties can be produced on commercial injection moulding machines using screws that are suitable for processing polyamides. Shut-off nozzles shall be used or open nozzles on machines equipped with melt decompression.

The following example lists some specifications for a typical injection moulding machine that is suitable for the production of test specimens.

Clamping force	350 kN
Screw diameter D	about 30 mm
Screw length	about 18 D
Depth of flights	5,5 to 3,0 mm
Ratio of stroke volume to shot volume (see ISO 294)	not greater than 6:1

The residence time shall not exceed 15 min.

In order to avoid uptake of moisture, the feed hopper should be closed by suitable means and contact between the granules and the open air should be less than 30 min.

3.3 Processing temperatures

Whenever possible, the plastic melt temperature (see ISO 294) shall be chosen from those recommended in table 1.

Table 1

Polymer	Viscosity number[1] ml/g	Glass content %	Plastic melt temperature °C
PA 6	130 to 160	0	250
	200 to 240	0	270
	130 to 160	< 30	280[2]
	130 to 160	30 to 50	290
PA 66	130 to 160	0	280
	160 to 200	0	290
	130 to 160	10 to 50	290
PA 69, PA 610	130 to 160	0	270
PA 612	160 to 200	0	270
PA 11, PA 12	110 to 150	0	210
	150 to 200	0	230
	200 to 240	0	250
	110 to 130	30 to 50	230
	130 to 240	10 to 20	250
	130 to 240	> 20 to 50	260
PA MXD 6	110 to 130	0	250
	130 to 160	0	260
	110 to 130	20 to 60	270
	130 to 160	20 to 60	280

1) Except for PA 11 and PA 12, the values presented relate to measurements in 96 % sulfuric acid. The values obtained in other solvents can be converted to the appropriate values in 96 % sulfuric acid using the formulae or graphs presented in ISO 307.

2) This temperature is also applicable to PA 6 with mineral fillers.

The temperature of the mould cavity shall be kept at 80 ± 3 °C, except for glass-fibre-reinforced PA MXD 6 when it shall be 130 ± 10 °C.

The plastic melt temperature and the mould temperature shall be kept constant in order to ensure that specimens with reproducible properties are obtained.

3.4 Processing conditions

Accurate, reproducible settings of the processing parameters are essential to obtain specimens with comparable properties. The following example lists processing conditions suitable for a specimen and sprue having a volume of approximately 15 cm^3.

Plastic melt temperature	see 3.3
Injection time	$<$ 1,0 s
Hold pressure time	about 15 s
Total cycle time	about 30 s
Maximum injection pressure	80 MPa
Hold pressure	80 MPa
Average injection velocity (cross-section 10 mm × 4 mm)	$>$ 400 mm/s

If the shot volume is different, the injection conditions shall be altered accordingly.

The hold pressure shall be maintained until the supply of molten material has frozen.

The application of a certain back-pressure (5 to 10 MPa) is recommended to obtain a homogeneous and bubble-free melt.

The uniformity of the test specimens shall be checked by weighing. The variation in mass shall not exceed 0,5 %. The specimens shall have smooth surfaces and shall be free from streaks, sink marks and voids. There shall be no irregularities visible to the naked eye or under the microscope. It is recommended that a microtome section (approximately 10 µm thick) be taken over the entire central section perpendicular to the axis and examined under a polarizing microscope.

4 Conditioning of test specimens

The properties shall be determined on specimens in the dry, as-moulded state and/or the moist state, according to requirements. The condition applied shall be reported.

4.1 Dry, as-moulded state

Test specimens are considered to be in the dry, as-moulded state when they have been stored in a moisture-proof container for 2 ± 1 days at 23 ± 2 °C after moulding and their moisture content is not greater than 0,2 % (*m*/*m*).

Drying of specimens with moisture contents above 0,2 % (*m*/*m*) is not permitted. Specimens shall be moulded from dry granules (see 3.1). Unless otherwise specified in the relevant International Standard, dry, as-moulded specimens shall be tested at 23 ± 2 °C. Specimens shall be tested in as short a time as possible (maximum 30 min) after removal from the moisture-proof container, to keep moisture absorption at a low level.

4.2 Moist state

Test specimens are considered to be in the moist state when they have been conditioned at 23 ± 2 °C and (50 ± 5) % relative humidity until equilibrium has been reached (see annex to ISO 291).

Test specimens conditioned by the procedure for accelerated conditioning of polyamides specified in ISO 1110 are also considered to be in the moist state.

The method of conditioning shall be stated with the test results because effects of the conditioning method on the state of the specimens cannot be excluded.

Unless otherwise specified in the relevant International Standard, specimens in the moist state shall be tested in the standard atmosphere 23/50 according to ISO 291.

5 Determination of properties

A list of the relevant test methods for polyamide materials is given in table 2.

Table 2

Property	Unit	Method	Specimen dimensions mm	Particular test conditions
Mechanical properties				
Shear modulus	MPa	ISO 537	60 × 10 × 1[1]	} ~70 °C to the softening temperature
Logarithmic decrement of damping		ISO 537	60 × 10 × 1[1]	
Elastic modulus in tension	MPa	ISO 527	Type 2 150 × 10 × 4	Test speed: 1 mm/min ± 50 %
Tensile properties:				
a) Tensile stress at yield	MPa			Test speed: a) and b) 50 mm/min ± 10 % c) 5 mm/min ± 20 % Values under b) to be given only if values under a) cannot be determined. Values under c) to be given only if values under a) and b) cannot be determined. Elongations greater than 50 % may be presented as "greater than 50 %".
Elongation at yield	%			
Elongation at break	%			
b) 1 % offset yield stress	MPa	ISO 527	Type 2 150 × 10 × 4	
Elongation at 1 % offset yield	%			
Elongation at break	%			
c) Tensile strength	MPa			
Elongation at maximum load	%			
Flexural stress at the conventional deflection[2]	MPa	ISO 178	80 × 10 × 4	Test speed: 2 mm/min
Impact strength				
— Charpy notched[2]	$kJ \cdot m^{-2}$	ISO 179	80 × 10 × 4	1A } Distance between supports: 40 mm
— Charpy unnotched[2]	$kJ \cdot m^{-2}$	ISO 179	80 × 10 × 4	1D }
— Izod	$kJ \cdot m^{-2}$	ISO 180	80 × 10 × 4	1A
Creep modulus	MPa	ISO 899	150 × 10 × 4	At 1 000 h. Stress to be chosen so that elongation at 1 000 h does not exceed 0,5 %.
Rockwell hardness		ISO 2039-2		
Ball indentation hardness		ISO 2039-1	> 4	
Thermal properties				
Temperature of deflection under load	°C	ISO 75	110 × 10 × 4	Methods A and B
Melting temperature	°C	ISO 3146	—	DTA or DSC method
Electrical properties				
Surface resistance	Ω	IEC Publication 93	100 × 100 × 1,5[3] (min.)	
Volume resistivity	Ω · cm	IEC Publication 93	100 × 100 × 1,5[3]	Test voltage: 1 000 V
Electric strength	kV/mm	IEC Publication 243	100 × 100 × 1,0	Insulating liquid
Relative permittivity		IEC Publication 250	100 × 100 × 1,5[3] (min.)	50 Hz and 1 MHz
Dissipation factor tan δ		IEC Publication 250	100 × 100 × 1,5[3] (min.)	50 Hz and 1 MHz
Comparative tracking index		IEC Publication 112	50 × 50 × 3[4] (min.)	50 to 60 Hz. Solution A
Miscellaneous properties				
Effect of liquid chemicals		ISO 175	Disc ϕ 50 × 3	Immersion time: 7 days
Moisture content	%	ISO 960		
Ash content	%	ISO 3451-4		
Viscosity number	ml/g	ISO 307		
Density	g/cm^3	ISO 1183		
Extractable matter	%	ISO 599		

1) Other dimensions may be employed provided that they ensure sufficiently rapid transfer of heat.

2) It is proposed to delete these tests from the table at the next review of this standard.

3) Preferred specimen sizes. 100 mm discs are an acceptable alternative.

4) Preferred specimen size. 50 mm discs are an acceptable alternative.

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