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Plastics — Polyamide (PA) moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

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

Partie 2: Préparation des éprouvettes et détermination des propriétés

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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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).

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 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 16396-2 cancels and replaces ISO 1874-2:2012, which has been technically revised.

A list of all parts in the ISO 16396 series can be found on the ISO website.

Plastics — Polyamide (PA) 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 polyamide 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 given. Properties and test methods that are suitable and necessary to characterize polyamide 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 are the designatory properties viscosity number and tensile modulus of elasticity given in ISO 16396-1.

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 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 294-1, *Plastics — Injection moulding of test specimens of thermoplastic materials —Part 1: General principles, and moulding of multipurpose and bar test specimens*

ISO 294-4, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 4: Determination of moulding shrinkage*

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

ISO 527-2, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics*

ISO 1110, *Plastics — Polyamides — Accelerated conditioning of test specimens*

ISO 1133-2, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 2: Method for materials sensitive to time-temperature history and/or moisture*

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

ISO 8256, *Plastics — Determination of tensile-impact strength*

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

ISO 15512, *Plastics — Determination of water content*

ISO 16396-1, *Plastics — Polyamide (PA) moulding and extrusion materials — Part 1: Designation system, marking of products and basis for specifications*

ISO 20753, *Plastics — Test specimens.*

ISO 27547-1, *Plastics — Preparation of test specimens of thermoplastic materials using mouldless technologies — Part 1: General principles, and laser sintering of 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, *Electric 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*

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:

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

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

4 Preparation of test specimens

4.1 Treatment of the material before moulding or laser sintering

Before processing, the material sample shall have reached room temperature. Before processing, the moisture content of the material sample shall not exceed 0,2 % (mass fraction) in the case of PAs having a viscosity number ≤200 ml/g and not exceed 0,1 % (mass fraction) in the case of PAs having a viscosity

number >200 ml/g. For PA46, PA6T/66, PA6T/XT, PA6T/6I/66, PA6T/6I, PA6I/6T and PA NDT/INDT, the moisture content shall be less than 0,1 % (mass fraction).

The moisture content of filled or reinforced materials shall be expressed as a percentage of the total mass of the compound. The moisture content shall be determined in accordance with ISO 15512 and the viscosity number in accordance with ISO 307.

To ensure that the moisture content remains low, it is recommended that the sample material in the feed hopper of the injection-moulding machine be blanketed with a suitable gas (for example, dried air, nitrogen or argon).

Better results may be obtained using a dehumidifier hopper drier.

If test specimens are to be prepared by laser sintering (see 4.3 and Annex A), follow the instructions given in Annex A for treatment of the material before processing.

4.2 Injection moulding

For the acquisition and presentation of comparable data, injection-moulded specimens are used so that valid comparisons between materials can be made. These data represent the most basic approach to the designation of the properties of materials.

Injection-moulded specimens shall be prepared in accordance with ISO 294-1, using the conditions specified in Table 1. Such specimens shall be prepared by injection moulding from dry granules. It is essential that the specimens are always prepared by the same procedure using the same processing conditions. The material shall be kept in sealed, moisture-proof containers until it is required for use.

NOTE The ISO 294 series will be revised, defining only the injection-moulding conditions and will refer to ISO 20753 for the dimensions of the specimens. Other International Standards that have hitherto used different designations for the same specimen type will also be revised to bring the designations into line with those in ISO 20753. Many test standards, specifying test specimen or referring to other standards as ISO 3167 and the ISO 294 series, will be revised in due time and also refer to ISO 20753.

Besides injection moulding, other methods are also used to manufacture PA parts, e.g. extrusion, blow moulding and methods using mouldless technologies. Different methods of manufacture can lead to significantly different properties and it might be useful to measure these properties using test specimens prepared by the same method. For specimen preparation using laser sintering, see 4.3.

4.3 Laser sintering

Although injection-moulded specimens are the only ones to be used to measure designatory properties, it is sometimes useful to prepare specimens using the same techniques as are used in parts manufacture (see 4.2).

For specimen preparation using laser sintering, see Annex A.

Table 1 — Conditions for injection moulding of test specimens

Material[a, b]	Viscosity number[c] ml/g	Filler content %	Plasticizer content %	Melt temperature °C	Mould temperature °C
PA6	≤160	0	0	250	80
	>160 but ≤200	0	0	260	80
	>200	0	0	270	80
	≤120	>0 but ≤70	0	270	80
	>120 but ≤200	>0 but ≤70	0	290	80
PA6/66	≤160	0	0	280	80
	>160	0	0	280	80
PA66	≥100	0	0	290	80
	≤160	>0 but ≤50	0	290	80
	≤160	>50 but ≤70	0	300	100
PA66/6	≤200	≤70	0	290	80
PA46	≤260	≤70	0	315	120
PA69	≤200	0	0	270	80
PA610	≤200	0	0	270	80
PA612	≤250	0	0	250	80
	≤250	>0 but ≤70	0	290	80
PA11	≤150	0	≥0	210	80
	>150 but ≤200	0	≥0	230	80
	>200 but ≤240	0	≥0	250	80
	≤130	>0 but ≤30	0	220	80
	≤130	>30 but ≤50	0	230	80
	>130 but ≤240	>0 but ≤20	0	250	80
	>130 but ≤240	>20 but ≤50	0	260	80
PA12	≤130	≤10	≤5	200	80
	>130 but ≤200	≤10	≤5	220	80
	>200	≤10	≤5	240	80
	≤130	0	>5	200	60
	>130 but ≤200	0	>5	210	60
	>200	0	>5	220	60
	≤130	>10 but ≤30	0	230	80
	≤130	>30 but ≤70	0	250	80
	>130 but ≤240	>10 but ≤30	0	240	80
	>130 but ≤240	>30 but ≤70	0	260	80

[a] For the definition of PA and the symbols used to indicate the chemical structure of polyamide materials, see ISO 472 and ISO 16396-1, respectively.

[b] For the polyamides mentioned in this table, with the exception of PA46, PA6T/66, PA6T/XT, PA6T/6I, PA6I/6T, PA6T/6I/66, PA66/6I and PA9T, the injection velocity shall be 200 mm/s ± 100 mm/s. For PA46, PA6T/66, PA6T/XT, PA6T/6I, PA6I/6T, PA6T/6I/66, PA66/6I and PA9T, the injection velocity shall be 300 mm/s ± 100 mm/s.

[c] Preferred reference solvent according to ISO 307.

Table 1 *(continued)*

Material[a, b]	**Viscosity number**[c] ml/g	**Filler content** %	**Plasticizer content** %	**Melt temperature** °C	**Mould temperature** °C
PA MXD6	≤130	0	0	250	130
	>130 but ≤160	0	0	260	130
	≤130	>0 but ≤50	0	270	100
	≤130	>50 but ≤70	0	280	130
	>130 but ≤160	>0 but ≤50	0	280	130
	>130 but ≤160	>50 but ≤70	0	290	130
PA6T/XT	≤160	≤70	0	325	150
PA6T/66	≤160	≤70	0	330	100
PA6T/6I	≤200	≤70	0	325	130
PA6I/6T	≤200	≤70	0	325	130
PA6T/6I/66	≤200	≤70	0	325	130
PA66/6I	≤160	≤70	0	290	90
PA9T	≤200	≤70	0	320	140
PA NDT/INDT	≤160	0	0	280	80
	≤120	>0 but ≤50	0	300	80

[a] For the definition of PA and the symbols used to indicate the chemical structure of polyamide materials, see ISO 472 and ISO 16396-1, respectively.

[b] For the polyamides mentioned in this table, with the exception of PA46, PA6T/66, PA6T/XT, PA6T/6I, PA6I/6T, PA6T/6I/66, PA66/6I and PA9T, the injection velocity shall be 200 mm/s ± 100 mm/s. For PA46, PA6T/66, PA6T/XT, PA6T/6I, PA6I/6T, PA6T/6I/66, PA66/6I and PA9T, the injection velocity shall be 300 mm/s ± 100 mm/s.

[c] Preferred reference solvent according to ISO 307.

5 Conditioning of test specimens

5.1 Conditioning states of the test specimen

Separate sets of test specimens for determination of properties shall be conditioned in two different ways: one set dry-as-moulded and the other in the moist state.

Properties shall be determined on specimens in the dry-as-moulded state, or on specimens in the moist state, or on specimens in both states. The state of the specimens shall be stated in the test report.

5.2 Dry-as-moulded (DAM) state

Test specimens are considered to be in the DAM state when they have been placed, immediately after moulding, in a moisture-proof container and stored at 23 °C ± 2 °C for at least 24 h. The moisture content of DAM specimens shall not exceed 0,2 % (mass fraction). The intentional addition of water to reach this moisture content is not allowed, nor is drying of specimens with moisture contents above this limit.

To maintain absorbed moisture at a low level, DAM specimens shall be tested in as short a time as possible (maximum 15 min) after removal from the moisture-proof container.

Annealing specimens prior to testing is not allowed.

5.3 Moist state

Test specimens are considered to be in the moist state when they have been conditioned at 23 °C and 50 % relative humidity until equilibrium has been reached (see ISO 291). The applied standard atmosphere class, or the applied tolerances, shall be stated in the test report.

NOTE The different classes of standard atmosphere correspond to different tolerance levels for the temperature and relative humidity (RH), as mentioned in ISO 291. The standard atmosphere classes for 23 °C/50 % RH are as follows:

— Class 1: (23 ± 1) °C/(50 ± 5) % RH;

— Class 2: (23 ± 2) °C/(50 ± 10) % RH.

The tolerances apply to the specimen-storage space in a test enclosure or conditioning enclosure. The relative-humidity tolerance includes real tolerances on dew points and allowance for the usual errors and drift in control equipment and hygrometers.

Test specimens which have been conditioned by the procedure for accelerated conditioning of polyamides specified in ISO 1110 are also considered to be in the moist state. The moisture content shall be reported.

6 Determination of properties

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 in this document. Values determined will not necessarily be identical to those obtained using specimens of different dimensions or prepared using different procedures.

For all ISO test methods applied in this document (Table 2 and Table 3), and in which is referred to the ISO 294 series and/or ISO 3167 for the designation and dimensions of the test specimen, ISO 20753 shall be used instead.

All tests shall be carried out in the standard atmosphere of 23 °C ± 2 °C and (50 ± 10) % relative humidity unless specifically stated otherwise in Tables 2 and 3.

Table 2 is compiled from ISO 10350-1, and the properties listed are those which are appropriate to polyamide 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 polyamide moulding and extrusion materials.

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

Property	Unit	Standard	Specimen type (dimensions in mm)	Specimen preparation	Test conditions and supplementary instructions
Rheological properties					
Moulding shrinkage of thermoplastics	%	ISO 294-4	60 × 60 × 2	M, DAM	Parallel and normal
Mechanical properties					
Tensile modulus	MPa	ISO 527-2	ISO 20753 Type A1	M, DAM + Moist	Test speed 1 mm/min
Charpy impact strength	kJ/m^2	ISO 179-1 or ISO 179-2	80 × 10 × 4	M, DAM + Moist	Method 1eU Edgewise impact Also record type of failure
Charpy notched impact strength	kJ/m^2	ISO 179-1 or ISO 179-2	80 × 10 × 4 Machined V-notch, r = 0,25	M, DAM + Moist	Method 1eA Edgewise impact Also record type of failure
Tensile notched impact strength	kJ/m^2	ISO 8256	80 × 10 × 4 Machined double V-notch, r = 1	M, DAM + Moist	Only to be quoted if fracture cannot be obtained with notched Charpy test
Thermal properties					
Temperature of deflection under load	°C	ISO 75-2	80 × 10 × 4	M, DAM	0,45 MPa and 1,80 MPa, Flatwise
Burning behaviour		IEC 60695-11-10	125 × 13 × 3 or alternative thicknesses <3	M, DAM	Record one of the classifications V-0, V-1, V-2, HB40 or HB75
Melting temperature	°C	ISO 11357-3	Moulding compound	—	Record peak melting temperature Use 10 °C/min or 20 °C/min
Electrical properties					
Relative permittivity	—	IEC 60250	≥60 × ≥60 × 2	M, DAM + Moist	Frequency 100 Hz and 1 MHz (compensate for electrode edge effects)
Dissipation factor	—	IEC 60250	≥60 × ≥60 × 2	M, DAM + Moist	Frequency 100 Hz and 1 MHz (compensate for electrode edge effects)
Volume resistivity	Ω·m	IEC 60093	≥60 × ≥60 × 2	M, DAM + Moist	Voltage 500 V
Surface resistivity	Ω	IEC 60093	≥60 × ≥60 × 2	M, DAM + Moist	Voltage 500 V
Electric strength	kV/mm	IEC 60243-1	≥60 × ≥60 × 1 and ≥60 × ≥60 × 2	M, DAM + Moist	Use 20 mm diameter spherical electrodes Immerse in transformer oil in accordance with IEC 60296 Use a voltage application rate of 2 kV/s
Comparative tracking index	—	IEC 60112	≥15 × ≥15 × 4	M, DAM	Use solution A

M = Injection moulding.

DAM = Dry-as-moulded state.

Moist = Moist state.

Table 2 *(continued)*

Property	Unit	Standard	Specimen type (dimensions in mm)	Specimen preparation	Test conditions and supplementary instructions
Other properties					
Water absorption	%	ISO 62	60 × 60 × 2	M, DAM	24 h immersion in water at 23 °C
Density	kg/m³	ISO 1183-1 or ISO 1183-2 or ISO 1183-3	ISO 20753 Centre Type A1	M, DAM	
M = Injection moulding. DAM = Dry-as-moulded state. Moist = Moist state.					

Table 3 — Specialized properties and test conditions

Property	Unit	Standard	Specimen type (dimensions in mm)	Specimen preparation	Test conditions and supplementary instructions
Mechanical properties					
Yield stress	MPa	ISO 527-2	ISO 20753 Type A1	M, DAM + Moist	Test speed: Unfilled materials: 50 mm/min Reinforced/filled materials: 5 mm/min
Yield strain	%				
Nominal strain at break	%				
Stress at break	MPa				
Strain at break	%				
Other properties					
Ash yield	%	ISO 3451-4	Moulding compound	—	Only for filled or reinforced grades
Moisture content	%	ISO 15512			
Viscosity number	ml/g	ISO 307	Moulding compound	—	See conditions given in ISO 16396-1
Melt volume-flow rate (MVR) or melt mass- flow rate (MFR)[a]	cm³/ 10 min or g/10 min	ISO 1133-2	—	—	Moisture content ≤0,02 % Test temperature: 225 °C, 250 °C, 275 °C or 300 °C Load (kg): 1,2 kg, 2,16 kg, 5 kg, 10 kg or 21,6 kg
[a] Drying conditions: — Vacuum oven with N_2 flow: temperature 75 °C to 85 °C, pressure <200 mbar, time ≤48 h. — Vacuum oven without N_2 flow: temperature <100 °C, pressure <50 mbar, time ≤48 h. Other drying conditions may be used or might even be necessary to achieve a moisture content ≤ 0,02 %. Sample storage: moisture-proof container or container with a non-hermetically closing lid stored in a desiccator. M = Injection moulding. DAM = Dry-as-moulded state. Moist = Moist state.					

Annex A
(normative)

Specimen preparation using laser sintering

Before starting the laser-sintering process, condition the powder for at least 16 h at 23 °C and refer to 4.1 regarding the maximum permissible moisture content of the laser-sintering powder.

Specimens produced by laser sintering shall be prepared in accordance with ISO 27547-1, using the temperatures given in Table A.1 and the following sintering conditions:

— layer thickness: 150 µm;

— laser power used when producing the contour: 15 W;

— laser power used when hatching: 20 W;

— laser beam travel speed when producing the contour: 700 mm/s;

— laser beam travel speed when hatching: 1 100 mm/s.

Table A.1 — Temperatures for the laser sintering of test specimens from different materials

Material	**Minimum polymer temperature** (at beginning of laser sintering) °C	**Temperature of specimen-preparation chamber** °C
PA6	218	120
PA612	212	120
PA1012	190	110
PA11	188	120
PA12	178	110
TPA-EE (formerly PEBA)	135	110

Bibliography

[1] ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

[2] ISO 294-3, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates*

[3] ISO 472, *Plastics — Vocabulary*

[4] ISO 10350-1, *Plastics — Acquisition and presentation of comparable single-point data — Part 1: Moulding materials*

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