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ISO 16396-2

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Plastics — Polyamide (PA) moulding and extrusion materials —

Part 2:

Preparation of test specimens and determination of properties

Plastiques — Matériaux à base de polyamide (PA) pour moulage et extrusion —

Partie 2: Préparation des éprouvettes et détermination des propriétés

ISO 16396-2:2022

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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 of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials.*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 16396-2:2017), which has been technically revised.

The main changes are as follows:

- the normative references clause has been updated;
- a footnote has been added in Table 1.

A list of all parts in the ISO 16396 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 — 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. It gives the requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing.

This document specifies 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. It lists the properties and test methods that are suitable and necessary to characterize polyamide moulding and extrusion materials.

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 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 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 pycnometer method and titration method

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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 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 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 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 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 https://www.electropedia.org/

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 2 g/kg in the case of PAs having a viscosity number ≤200 ml/g and not exceed 1 g/kg 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 1 g/kg.

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 can be obtained using a dehumidifier hopper drier.

If test specimens are to be prepared by laser sintering (see <u>4.3</u> and <u>Annex A</u>), follow the instructions given in <u>Annex A</u> 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 <u>Table 1</u>. 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, such as 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, <u>Annex A</u> shall apply.

Table 1 — Conditions for injection moulding of test specimens

| Material ^{a, b} | Viscosity number ^c ml/g | Filler content % | Plasticizer content % | Melt temperature ^d °C | Mould temperature ^e °C | |
|--------------------------|--|------------------|-----------------------------|--|---|--|
| | ≤160 | 0 | 0 | 250 | 80 | |
| | >160 but ≤200 | 0 | 0 | 260 | 80 | |
| PA6 | >200 | 0 | 0 | 270 | 80 | |
| | ≤120 | >0 but ≤70 | 0 | 270 | 80 | |
| | >120 but ≤200 | >0 but ≤70 | 0 | 290 | 80 | |
| | ≤160 | 0 | 0 | 280 | 80 | |
| PA6/66 | >160 | 0 | 0 | 280 | 80 | |
| | ≥100 | 0 | 0 | 290 | 80 | |
| PA66 | ≤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 | |
| DA 642 | ≤250 | 0 | 0 | 250 | 80 | |
| PA612 | ≤250 | >0 but ≤70 | ARO PR | 290 | 7 80 | |
| | ≤150 | 0 | ≥0 | 210 | 80 | |
| | >150 but ≤200 | (standa | ros≥oten | 230 | 80 | |
| | >200 but ≤240 | 0 | ≥0 | 250 | 80 | |
| PA11 | ≤130 | >0 but ≤30 | 6306 2.0022 | 220 | 80 | |
| | https:≤130 dards | >30 but ≤50 | andards 0 sist/c0d | 11a4h-230 _{h-41c3} | -he25- 80 | |
| | >130 but ≤240 | >0 but ≤20 /49 | b/iso-1 0 396-2-2 |)22 250 | 80 | |
| | >130 but ≤240 | >20 but ≤50 | 0 | 260 | 80 | |
| | ≤130 | ≤10 | ≤5 | 200 | 80 | |
| | >130 but ≤200 | ≤10 | ≤5 | 220 | 80 | |
| | >200 | ≤10 | ≤5 | 240 | 80 | |
| | ≤130 | 0 | >5 | 200 | 60 | |
| DA12 | >130 but ≤200 | 0 | >5 | 210 | 60 | |
| PA12 | >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/61, PA6I/6T, PA6T/61/66, PA66/61 and PA9T, the injection velocity shall be $200 \text{ mm/s} \pm 100 \text{ mm/s}$. For PA46, PA6T/66, PA6T/XT, PA6T/61, PA6I/6T, PA6T/61/66, PA66/61 and PA9T, the injection velocity shall be $300 \text{ mm/s} \pm 100 \text{ mm/s}$.

Preferred reference solvent according to ISO 307.

d Or the temperature and range recommended by the supplier.

For low crystalline PA6/66 grades a mould temperature of 80 $^{\circ}$ C can cause demoulding problems. For these PA grades, a mould temperature of 50 $^{\circ}$ C, or the temperature recommended by the supplier, is recommended. The mould temperature shall be reported.

90

120

80

80

| Material ^{a, b} | Viscosity number ^c ml/g | Filler content % | Plasticizer content % | Melt temperature ^d °C | Mould temperature ^e °C |
|--------------------------|--|------------------|-----------------------------|--|---|
| | ≤130 | 0 | 0 | 250 | 130 |
| | >130 but ≤160 | 0 | 0 | 260 | 130 |
| DA MVD6 | ≤130 | >0 but ≤50 | 0 | 270 | 100 |
| PA MXD6 | ≤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 | 120 |
| PA6T/6I | ≤200 | ≤70 | 0 | 340 | 140 |
| PA6I/6T | ≤200 | ≤70 | 0 | 325 | 130 |
| PA6T/6I/66 | ≤200 | ≤70 | 0 | 325 | 130 |
| | | | | | |

Table 1 (continued)

n

0

0

0

290

320

280

300

≤70

≤70

0

>0 but ≤50

PA66/6I

PA9T

PA NDT/INDT

≤160

≤200

≤160

≤120

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 \pm 2 °C for at least 24 h. The moisture content of DAM specimens shall not exceed 2 g/kg . 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.

^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/61, PA6I/6T, PA6T/6I, PA6I/66, PA66/6I and PA9T, the injection velocity shall be 200 mm/s ± 100 mm/s. For PA46, PA6T/66, PA6T/XT, PA6T/61, PA6I/6T, PA6T/6I, PA6I/6T, PA6T/6I, PA6I/6T, PA6T/6I, PA6I/6T, PA6T/6I, PA6I/6T, PA6I/6T, PA6I/6I, P

Preferred reference solvent according to ISO 307.

d Or the temperature and range recommended by the supplier.

^e For low crystalline PA6/66 grades a mould temperature of 80 $^{\circ}$ C can cause demoulding problems. For these PA grades, a mould temperature of 50 $^{\circ}$ C, or the temperature recommended by the supplier, is recommended. The mould temperature shall be reported.

5.3 Moist state

Test specimens are considered to be in the moist state when they have been conditioned at $23\,^{\circ}$ C and $50\,^{\circ}$ C 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 $23 \, ^{\circ}\text{C}/50 \, \%$ 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, the methods of preparation and conditioning, the specimen dimensions and the test procedures specified in this document, and in particular, <u>Table 2</u> and <u>Table 3</u>, shall be used. 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 (see <u>Table 2</u> and <u>Table 3</u>), 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 \pm 2 °C and (50 \pm 10) % relative humidity unless specifically stated otherwise in <u>Table 2</u> and <u>Table 3</u>.

<u>Table 2</u> 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.

<u>Table 3</u> contains those properties, not found specifically in <u>Table 2</u>, which are in wide use or of particular significance in the practical characterization of polyamide moulding and extrusion materials.

| Table 2 — Standard | properties and | l test conditions | (selected from) | (SO 10350-1) |
|--------------------|----------------|-------------------|------------------|--------------|
| | | | | |

| Property | Unit | Standard | Specimen type (dimensions in mm) | Specimen preparation | Test conditions and supplementary instructions | |
|------------------------|--------|-----------|----------------------------------|----------------------|--|--|
| Rheological properties | | | | | | |
| Moulding shrinkage | % | ISO 294-4 | 60 × 60 × 2 | M, | Parallel and normal | |
| of thermoplastics | | | | DAM | | |
| Mechanical properties | | | | | | |
| Tensile modulus | MDo 10 | ISO 527-2 | ISO 20753 | M, | To at an a a d 1 mm /min | |
| Tensile modulus | MPa | 150 527-2 | Type A1 | DAM + Moist | Test speed 1 mm/min | |