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Plastics — Acrylonitrile-styrene-acrylate (ASA), acrylonitrile-(ethylene-propylene-diene)-styrene (AEPDS) and acrylonitrile-(chlorinated polyethylene)-styrene (ACS) moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Matériaux pour moulage et extrusion à base d'acrylonitrile-styrène-acrylate (ASA), d'acrylonitrile-(éthylène-propylène-diène)-styrène (AEPDS) et d'acrylonitrile-(polyéthylène chloré)-styrène (ACS) —

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 6402-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 6402-2:1994), which has been technically revised.

ISO 6402 consists of the following parts, under the general title *Plastics — Acrylonitrile-styrene-acrylate (ASA), acrylonitrile-(ethylene-propylene-diene)-styrene (AEPDS) and acrylonitrile-(chlorinated polyethylene)-styrene (ACS) moulding and extrusion materials*:

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

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

Plastics — Acrylonitrile-styrene-acrylate (ASA), acrylonitrile-(ethylene-propylene-diene)-styrene (AEPDS) and acrylonitrile-(chlorinated polyethylene)-styrene (ACS) moulding and extrusion materials —

Part 2: Preparation of test specimens and determination of properties

1 Scope

1.1 This part of ISO 6402 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of acrylonitrile-styrene-acrylate (ASA), acrylonitrile-(ethylene-propylene-diene)-styrene (AEPDS) and acrylonitrile-(chlorinated polyethylene)-styrene (ACS) 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 here.

1.2 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 ASA, AEPDS and ACS moulding and extrusion materials are listed.

1.3 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 6402, as are the designatory properties specified in Part 1.

1.4 In order to obtain reproducible and comparable test results, it is necessary to use the methods of specimen 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 Conformance

In Clause 3, the year of publication of each normative reference has been specifically stated. In order to be able to claim conformity with this part of ISO 6402, it is essential that the user use only those editions given, and not earlier or more recent editions.

3 Normative references

The following referenced documents are indispensable for the application 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 62:1980, *Plastics — Determination of water absorption*

ISO 75-2:1993, *Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite*

ISO 178:1993, *Plastics — Determination of flexural properties*

ISO 179:1993, *Plastics — Determination of Charpy impact strength*

ISO 180:1993, *Plastics — Determination of Izod impact strength*

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials*

ISO 294-1:1996, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens*

ISO 306:1994, *Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST)*

ISO 527-2:1993, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics*

ISO 527-4:1997, *Plastics — Determination of tensile properties — Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites*

ISO 899-1:1993, *Plastics — Determination of creep behaviour — Part 1: Tensile creep*

ISO 1133:1997, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*

ISO 1183:1987, *Plastics — Methods for determining the density and relative density of non-cellular plastics*

ISO 1656:1996, *Rubber, raw natural, and rubber latex, natural — Determination of nitrogen content*

ISO 2561:1974, *Plastics — Determination of residual styrene monomer in polystyrene by gas chromatography*

ISO 2818:1980, *Plastics — Preparation of test specimens by machining*

ISO 3167:1993, *Plastics — Multipurpose test specimens*

ISO 4581:1994, *Plastics — Styrene/acrylonitrile copolymers — Determination of residual acrylonitrile monomer content — Gas chromatography method*

ISO 4589:1984, *Plastics — Determination of flammability by oxygen index*

ISO 6402-1, *Plastics — Acrylonitrile-styrene-acrylate (ASA), acrylonitrile-(ethylene-propylene-diene)-styrene (AEPDS) and acrylonitrile-(chlorinated polyethylene)-styrene (ACS) moulding and extrusion materials — Part 1: Designation system and basis for specifications*

ISO 8256:1990, *Plastics — Determination of tensile-impact strength*

ISO 10350:1993, *Plastics — Acquisition and presentation of comparable single-point data*

ISO 11357-2:1999, *Plastics — Differential scanning calorimetry (DSC) — Part 2: Determination of glass transition temperature*

IEC 60093:1980, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials*

IEC 60112:1979, *Method for determining the comparative and the proof tracking indices of solid insulating materials under moist conditions*

IEC 60243-1:1998, *Electrical strength of insulating materials — Test methods — Part 1: Tests at power frequencies*

IEC 60250:1969, *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 60296:1982, *Specification for unused mineral insulating oils for transformers and switchgear*

IEC 60695-11-10:1999, *Fire hazard testing — Part 11-10: Test flames — 50W horizontal and vertical flame methods*

4 Preparation of test specimens

4.1 General

It is essential that specimens always be prepared by the same procedure (either injection moulding or compression moulding), using the same processing conditions. The procedure to be used for each test method is indicated in Tables 3 and 4.

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, the material shall be dried under appropriate conditions to produce samples without surface defects such as splay marks.

4.3 Injection moulding

Injection-moulded specimens shall be prepared in accordance with ISO 294-1, using the conditions specified in Table 1, in which the temperature values given are target values (see ISO 294-1 for tolerances).

Table 1 — Conditions for injection moulding of test specimens

Material	Melt temperature °C	Mould temperature °C	Injection velocity mm/s
ACS	200	60	200 ± 100
ASA and AEPDS, general and high-heat grades	250	60	200 ± 100

4.4 Compression moulding

Compression-moulded sheets shall be prepared in accordance with ISO 293, using the conditions specified in Table 2, in which the moulding temperatures given are target values (see ISO 293 for tolerances).

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

Table 2 — Conditions for compression moulding of test specimens

Material	Moulding temperature °C	Average cooling rate °C/min	Demoulding temperature °C	Full pressure MPa	Full pressure time min	Preheating time min
ACS	180	10	⩽ 60	4 ± 0,5	5 ± 1	5 ± 1
ASA and AEPDS, general and high-heat grades	220	10	⩽ 60	4 ± 0,5	5 ± 1	5 ± 1

5 Conditioning of test specimens

Test specimens for rheological and thermal properties shall be dried and stored in a desiccator at (23 ± 2) °C until tested. Test specimens for other properties shall be conditioned for at least 16 h at (23 ± 2) °C and (50 ± 10) % relative humidity.

6 Determination of properties

In the determination of properties and the presentation of data, the standards, supplementary instructions and notes given in ISO 10350 shall be applied. All tests shall be carried out in the standard atmosphere of (23 ± 2) °C and (50 ± 10) % relative humidity unless specifically stated otherwise in Tables 3 and 4.

Table 3 is compiled from ISO 10350, and the properties listed are those which are appropriate to ASA, AEPDS and ACS 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 ASA, AEPDS and ACS moulding and extrusion materials.

Table 3 — General properties and test conditions (selected from ISO 10350)

Property	Unit	Test method	Specimen type (dimensions in mm)	Specimen preparation	Test conditions and supplementary instructions	
Rheological properties						
Melt mass-flow rate	g/10 min	ISO 1133	Moulding compound	—	220 °C, load 10 kg.[a]	
Melt volume-flow rate	cm^3/10 min					
Mechanical properties						
Tensile modulus	MPa	ISO 527-2, ISO 527-4	ISO 3167	Injection moulding	Test speed 1 mm/min.	
Yield stress					Test speed 50 mm/min.	
Yield strain	%				Test speed 50 mm/min.	
Strain at break					Test speed 50 mm/min.	
Stress at 50 % strain	MPa				Test speed 50 mm/min. Only to be quoted if no yielding is observed up to 50 % nominal strain.	
Tensile creep modulus	MPa	ISO 899-1			At 1 h At 1 000 h	Strain $\leqslant$ 0,5 %
Flexural modulus	MPa	ISO 178	80 × 10 × 4		Test speed 2 mm/min.	
Flexural strength						
Charpy impact strength	kJ/m^2	ISO 179	80 × 10 × 4		Edgewise impact. Also record type of failure.	
Charpy notched impact strength			80 × 10 × 4 V-notch, r = 0,25			
Tensile notched impact strength		ISO 8256	80 × 10 × 4 double V-notch, r = 1		Only to be quoted if fracture cannot be obtained with notched Charpy impact test.	
Thermal properties						
Glass transition temperature	°C	ISO 11357-2	Moulding compound	—	Record midpoint temperature. Use 10 °C/min.	
Temperature of deflection under load	°C	ISO 75-2	80 × 10 × 4	Injection moulding	0,45 MPa and 1,8 MPa.	
Vicat softening temperature	°C	ISO 306	10 × 10 × 4		Heating rate 50 °C /h, load 50 N.	
Burning behaviour	mm/min	IEC 60695-11-10	125 × 13 × 3		Record one of classifications V-0, V-1, V-2, HB40, HB75.	
Oxygen index	%	ISO 4589	80 × 10 × 4		Procedure A — top surface ignition.	

Table 3 (*continued*)

Property	Unit	Test method	Specimen type (dimensions in mm)	Specimen preparation	Test conditions and supplementary instructions	
Electrical properties						
Relative permittivity	—	IEC 60250	⩾ 80 × ⩾ 80 × 1	Compression moulding	100 Hz	Compensate for electrode edge effects.
					1 MHz	
Dissipation factor	—				100 Hz	
					1 MHz	
Volume resistivity	Ω·m	IEC 60093			Voltage 100 V	1-minute value.
Surface resistivity	Ω					Use contacting line electrodes 1 mm to 2 mm wide, 50 mm long and 5 mm apart.
Electric strength	kV/mm	IEC 60243-1	⩾ 80 × ⩾ 80 × 1	Injection moulding	Use 25 mm/75 mm coaxial-cylinder electrodes. Immerse in transformer oil in accordance with IEC 60296. Use a 20 s step-by-step test.	
			⩾ 80 × ⩾ 80 × 3			
Comparative tracking index	—	IEC 60112	⩾ 15 × ⩾ 15 × 4	Injection moulding	Use solution A.	
Other properties						
Water absorption	%	ISO 62	Thickness ⩽ 1	Compression moulding	Saturation value in water at 23 °C.	
					Equilibrium value at 23 °C, 50 % relative humidity.	
Density	kg/m^3	ISO 1183	10 × 10 × 4	Injection moulding	Specimen to be taken from moulded product.	

[a] In the case of ASA and AEPDS, 240 °C at 10 kg load is recommended for high-heat grades with a low content of *N*-phenylmaleimide when polymer residue adheres to the cylinder wall or MFR/MVR value is not reproducible at 220 °C, 10 kg load. Likewise, 265 °C, 10 kg load is recommended for high-heat grades with a high content of *N*-phenylmaleimide when polymer residue adheres to the cylinder wall or MFR/MVR value is not reproducible at 240 °C, 10 kg load.

Table 4 — Additional properties and test conditions of particular utility to ASA, AEPDS and ACS moulding and extrusion materials

Property	Unit	Test method	Specimen type (dimensions in mm)	Specimen preparation	Test conditions and supplementary instructions
Mechanical properties					
Izod impact strength	kJ/m^2	ISO 180	80 × 10 × 4	Injection moulding	Also record type of failure.
Other properties					
Residual styrene monomer content	%	ISO 2561	Moulding compound	—	
Residual acrylonitrile content	%	ISO 4581			
Bound acrylonitrile	%	ISO 1656			See Annex A.

Annex A
(normative)

Determination of the bound-acrylonitrile content in the continuous phase

A.1 Principle

The unbound resin in the continuous phase is separated from the dispersed elastomeric phase, the nitrogen content of this resin is determined and the acrylonitrile content of the continuous phase calculated.

A.2 Procedure

A.2.1 Pre-extraction with *n*-hexane

Extract the dried particles (approximately 3 mm × 3 mm × 3 mm) with *n*-hexane for about 80 h in a Soxhlet apparatus. During this time, additives such as antioxidants and lubricants will be removed. Dry the residue under vacuum at 60 °C for at least 2 h.

A.2.2 Extraction with acetone

Extract 1,2 g of residue obtained in A.2.1 with 50 cm^3 of acetone, with occasional stirring, for 24 h at room temperature. Centrifuge the resin from the insoluble residue (20 000 rev/min for 40 min is satisfactory). Extract the residue several times with acetone and separate by centrifuging. The combined acetone extracts contain all the unbound resin, which can be precipitated by pouring it into a tenfold volume of methanol at –10 °C. Dry the precipitated resin under vacuum at 60 °C.

A.2.3 Acrylonitrile content

Determine the nitrogen content of the precipitated resin by the Kjeldahl semi-micro method specified in ISO 1656. Calculate the acrylonitrile content from the nitrogen content using the equation:

$$AN = 3{,}79 \times N$$

where

AN is the acrylonitrile content, expressed as a percentage by mass;

N is the nitrogen content, expressed as a percentage by mass;

3,79 is the ratio of the relative molecular masses of acrylonitrile (C_2H_3CN) and nitrogen.

A.3 Alternative procedure

The percentage acrylonitrile content may also be determined by a pyrolysis/thermal-conductivity method.

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