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Plastics — Unsaturated-polyester powder moulding compounds (UP-PMCs) —

Part 2: Preparation of test specimens and determination of properties

*Plastiques — Poudres à mouler à base de polyester non saturé
(UP-PMC) —*

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 3.

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 part of ISO 14530 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14530-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

ISO 14530 consists of the following parts, under the general title *Plastics — Unsaturated-polyester powder moulding compounds (UP-PMCs)*:

- *Part 1: Designation system and basis for specifications*
- *Part 2: Preparation of test specimens and determination of properties*
- *Part 3: Requirements for selected moulding compounds*

Plastics — Unsaturated-polyester powder moulding compounds (UP-PMCs) —

Part 2: Preparation of test specimens and determination of properties

1 Scope

This part of ISO 14530 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of unsaturated-polyester powder moulding compounds (UP-PMCs). Requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing are given here.

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 PMCs 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, PMCs are also included in this part of ISO 14530, as are the designatory properties found in ISO 14530-1.

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 herein. 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 normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 14530. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 14530 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 60:1977, *Plastics — Determination of apparent density of material that can be poured from a specified funnel*.

ISO 62:1999, *Plastics — Determination of water absorption*.

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

ISO 120:1977, *Plastics — Phenol-formaldehyde mouldings — Determination of free ammonia and ammonium compounds — Colorimetric comparison method*.

ISO 171:1980, *Plastics — Determination of bulk factor of moulding materials*.

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

ISO 179-1:—¹⁾, *Plastics — Determination of Charpy impact properties — Part 1: Non-instrumented impact test*.

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing*.

ISO 295:—²⁾, *Plastics — Compression moulding of test specimens of thermosetting materials*.

ISO 472:1999, *Plastics — Vocabulary*.

ISO 527-1:1993, *Plastics — Determination of tensile properties — Part 1: General principles*.

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

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

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

ISO 2039-1:1993, *Plastics — Determination of hardness — Part 1: Ball indentation method*.

ISO 2577:1984, *Plastics — Thermosetting moulding materials — Determination of shrinkage*.

ISO 2818:1994, *Plastics — Preparation of test specimens by machining*.

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

ISO 3671:1976, *Plastics — Aminoplastic moulding materials — Determination of volatile matter*.

ISO 4589-2:1996, *Plastics — Determination of burning behaviour by oxygen index — Part 2: Ambient-temperature test*.

ISO 4614:1977, *Plastics — Melamine-formaldehyde mouldings — Determination of extractable formaldehyde*.

ISO 6603-2:—³⁾, *Plastics — Determination of puncture impact behaviour of rigid plastics — Part 2: Instrumented impact testing*.

ISO 7808:1992, *Plastics — Thermosetting moulding materials — Determination of transfer flow*.

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

ISO 10350-1:1998, *Plastics — Acquisition and presentation of comparable single-point data — Part 1: Moulding materials*.

ISO 10724-1:1998, *Plastics — Injection moulding of test specimens of thermosetting powder moulding compounds (PMCs) — Part 1: General principles and moulding of multipurpose test specimens*.

ISO 10724-2:1998, *Plastics — Injection moulding of test specimens of thermosetting powder moulding compounds (PMCs) — Part 2: Small plates*.

ISO 11359-2:1999, *Plastics — Thermomechanical analysis (TMA) — Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*.

1) To be published. (Revision of ISO 179:1993)

2) To be published. (Revision of ISO 295:1991)

3) To be published. (Revision of ISO 6603-2:1989)

ISO 14530-1:1999, *Plastics — Unsaturated-polyester powder moulding compounds (UP-PMCs) — Part 1: Designation system and basis for specifications.*

ISO 14530-3:1999, *Plastics — Unsaturated-polyester powder moulding compounds (UP-PMCs) — Part 3: Requirements for selected moulding compounds.*

ISO 15062:^{—4)}, *Plastics — Determination of the thermal-flow and cure behaviour of thermosetting materials by torque rheometry.*

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 60167:1964, *Methods of test for the determination of the insulation resistance of solid insulating materials.*

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 — 50 W horizontal and vertical flame test methods.*

IEC 60695-11-20:1999, *Fire hazard testing — Part 11-20: Test flames — 500 W flame test methods.*

IEC 60707:1999, *Flammability of solid non-metallic materials when exposed to flame sources — List of test methods.*

3 Terms and definitions

For the purposes of this part of ISO 14530, the terms and definitions given in ISO 472 and ISO 14530-1 apply, plus the following:

3.1

thermal flow

parameter which characterizes the flow behaviour of plasticized thermosetting moulding compounds, e.g. when filling a mould cavity, and for which the minimum torque M_B measured in accordance with ISO 15062 may be taken as a measure

4 Preparation of test specimens

4.1 General

It is essential that specimens are always 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 (M = injection moulding, Q = compression moulding).

4) To be published.

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 Pretreatment of the material

Before processing by injection moulding, no treatment of the sample is normally necessary. If pretreatment is required, this shall be in accordance with the manufacturer's recommendations.

Before processing by compression moulding, treatment of the sample in accordance with ISO 295:—, subclause 5.2 (preforming), 6.2 (drying), 6.3 (high-frequency preheating) or 6.4 (preplastification), is allowed.

4.3 Injection moulding

Injection-moulded specimens shall be prepared in accordance with ISO 10724-1 and/or ISO 10724-2, using the conditions specified in Table 1.

Table 1 — Conditions for injection moulding of test specimens

Type of PMC	Melt temperature T_M (range) °C	Mould temperature T_C (range) °C	Average injection velocity, v_I (range) mm/s	Cure time t_{CR} (range) s
Injection UP-PMC	100 to 110	160 to 180	50 to 150	(see text)

The conditions may be selected from within the range specified in Table 1 provided that the statement in 4.1, first sentence, is observed and, in each specific case, definite values (not ranges) are laid down for

- the melt temperature T_M ;
- the mould temperature T_C and
- the cure time t_{CR} .

The cure time t_{CR} may be selected as a function of the curing behaviour and the type of pretreatment of the UP-PMC under test, provided that it is the same for all specimens of the same thickness moulded from any one type of UP-PMC and is stated together with the test results. The cure time selected shall ensure that all the specimens are cured as completely and homogeneously as possible.

NOTE For UP-PMCs with high thermal flow, the following situation may occur:

- injection moulding of certain mouldings with the intended quality is possible, but
- injection moulding of test specimens (e.g. ISO 3167 type A multipurpose specimens or ISO 10724-2 type D1/D2 small-plate specimens) is not possible.

In this case, and only in this case, it is recommended that the test specimens are:

- produced by compression moulding in accordance with ISO 295, or
- prepared from compression-moulded ISO 295 type E plates (120 mm × 120 mm × thickness) by machining in accordance with ISO 2818.

4.4 Compression moulding

Compression-moulded specimens shall be prepared in accordance with ISO 295, using the conditions specified in Table 2.

Table 2 — Conditions for compression moulding of test specimens

Type of PMC	Mould temperature T_C (range) °C	Mould pressure p_M (range) MPa	Cure time t_{CR} (range) s
Compression UP-PMC	155 to 175	10 to 30	20 to 60 per mm thickness

The conditions may be selected from within the range specified in Table 2 provided that the statement in 4.1, first sentence, is observed and, in each specific case, definite values (not ranges) are laid down for

- the mould temperature T_C ,
- the mould pressure p_M and
- the cure time t_{CR} .

The cure time t_{CR} may be selected as a function of the curing behaviour and the type of pretreatment of the UP-PMC under test, provided that it is the same for all specimens of the same thickness moulded from any one type of UP-PMC and is stated together with the test results. The cure time selected shall ensure that all the specimens are cured as completely and homogeneously as possible.

The test specimens required for the determination of the properties shall be machined from the moulded plates in accordance with ISO 2818, or ISO 3167 type A. Multipurpose specimens shall be moulded in accordance with ISO 295.

5 Conditioning of test specimens

Unless otherwise specified, the test specimens shall be conditioned as follows prior to determining the properties in Tables 3 and 4:

Method 1

Condition the test specimens in accordance with ISO 291 for at least 16 h at $(23 \pm 2)^\circ\text{C}$ and $(50 \pm 5)\%$ relative humidity.

This is the general test method, applicable in all cases where the use of method 2 is not stipulated. Method 1 is not explicitly mentioned in Tables 3 and 4.

Method 2

Condition the test specimens in distilled water at ambient temperature for 24 h and then in accordance with ISO 291 for 2 h at $(23 \pm 2)^\circ\text{C}$ and $(50 \pm 5)\%$ 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 10350-1 shall be applied.

All tests shall be carried out in the standard laboratory atmosphere of $(23 \pm 2)^\circ\text{C}$ and $(50 \pm 5)\%$ relative humidity unless specifically stated otherwise in Tables 3 and 4.

Table 3 is compiled from ISO 10350-1, and the properties listed are those which are appropriate to UP-PMCs for compression and injection moulding. These properties are those considered useful for comparisons of data generated for different thermosets and thermoplastics.

Table 4 contains those properties, not found specifically in Table 3, which may be of interest for the practical characterization of UP-PMCs. Comparisons of different materials using these properties may well be restricted to those thermosets in the same generic families.

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Table 3 — Properties and test conditions

1	2	3	4	5	6	7			
Property	Symbol	Standard	Specimen type (dimensions in mm)	Processing ^a	Unit	Test conditions and supplementary instructions			
1 Rheological and processing properties									
1.1	Moulding shrinkage	S_{Mo}	ISO 2577	120 × 120 × 2 ISO 295 type E2	Q	%	Mean value of two directions normal to each other		
1.2		S_{Mp}	See footnote b	60 × 60 × 2 ISO 10724-2 type D2	M		Parallel to melt flow direction		
1.3		S_{Mn}					Normal to melt flow direction		
2 Mechanical properties									
2.1	Tensile modulus	E_t	ISO 527-1 and ISO 527-2	ISO 3167 type A or prepared from ISO 295 type E4	Q/M	MPa	Test speed 1 mm/min		
2.2	Stress at break	σ_B				%	Test speed 5 mm/min		
2.3	Strain at break	ε_B							
2.4	Tensile creep modulus	E_{tc1}	ISO 899-1	80 × 10 × 4	Q/M	MPa	At 1 h		
2.5		$E_{tc}10^3$				MPa	At 1000 h		
2.6	Flexural modulus	E_f	ISO 178	80 × 10 × 4	Q/M	MPa	Test speed 2 mm/min		
2.7	Flexural strength	σ_{fM}							
2.8	Charpy impact strength	a_{cU}	ISO 179-1	80 × 10 × 4	Q/M	kJ/m ²	Edgewise impact		
2.9	Charpy notched impact strength	a_{cA}		80 × 10 × 4 machined V-notch $r = 0,25$					
2.10	Tensile-impact strength	a_{t1}	ISO 8256	80 × 10 × 4 machined double V-notch $r = 1$	Q/M	N	Record if fracture cannot be obtained with notched Charpy impact test		
2.11	Puncture impact behaviour	F_M	ISO 6603-2	60 × 60 × 2 prepared from ISO 295 type E2 or ISO 10724-2 type D2			Striker velocity 4,4 m/s. Striker diameter 20 mm. Lubricate striker. Clamp specimen to prevent any out-of-plane movement of its outer regions.		
2.12	— Force	W_P		J		Puncture energy at 50 % decrease in force after maximum			
^a Q = Compression moulding M = Injection moulding									
^b International Standard to be prepared.									

Table 3 — Properties and test conditions (continued)

1	2	3	4	5	6	7			
Property	Symbol	Standard	Specimen type (dimensions in mm)	Processing ^a	Unit	Test conditions and supplementary instructions			
3 Thermal properties									
3.1	Temperature of deflection under load	T_f 1,8	ISO 75-2	80 × 10 × 4	Q/M	°C	1,8	Max. surface stress (MPa)	Use flatwise loading
3.2		T_f 8,0					8,0		
3.3	Coefficient of linear thermal expansion	α_o	ISO 11359-2	60 × 10 × 2 prepared from 120 × 120 × 2 ISO 295 type E2	Q	°C ⁻¹	—	Record secant value over temperature range from 23 °C to 55 °C	
3.4		α_p		60 × 10 × 4 prepared from ISO 3167 type A			Parallel to melt flow direction		
3.5		α_p		60 × 10 × 2 prepared from 60 × 60 × 2 ISO 10724-2 type D2	M		Parallel to melt flow direction		
3.6		α_n					Normal to melt flow direction		
3.7	Burning behaviour	$B_{50/3,0}$	IEC 60695-11-10	125 × 13 × 3	Q	—	Record one of classifications: V-0; V-1; HB40 or HB75 (V-2 not applicable to thermosets)		
3.8		$B_{50/x}$		Additional specimen of different thickness x					
3.9		$B_{500/3,0}$	IEC 60695-11-20	≥ 150 × ≥ 150 × 3	Q	—	Record one of classifications: 5VA; 5VB or N		
3.10		$B_{500/x}$		Additional specimen of different thickness x					
3.11	Oxygen index	O/23	ISO 4589-2	80 × 10 × 4	Q/M	%	Use procedure A: top surface ignition		

^a Q = Compression moulding
M = Injection moulding