INTERNATIONAL STANDARD

ISO 14526-2

First edition
1999-11-15

Plastics — Phenolic powder moulding compounds (PF-PMCs) —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Poudres à mouler phénoliques (PF-PMC) —

Partie 2: Préparation des éprouvettes et détermination des propriétés

Reference number
ISO 14526-2:1999(E)

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Foreword

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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 14526 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14526-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

After a transition period of, at the most, four years, the three parts of ISO 14526 (see below) will replace ISO 800:1992, of which they constitute a technical revision.

ISO 14526 consists of the following parts, under the general title *Plastics — Phenolic powder moulding compounds (PF-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 — Phenolic powder moulding compounds (PF-PMCs) —

Part 2:
Preparation of test specimens and determination of properties

1 Scope

This part of ISO 14526 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of phenolic powder moulding compounds (PF-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 14526, as are the designatory properties found in ISO 14526-1.

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 14526. 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 14526 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 800:1992, *Plastics — Phenolic moulding materials — Specification.*

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.*

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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 14526-1:1999, *Plastics — Phenolic powder moulding compounds (PF-PMCs) — Part 1: Designation system and basis for specifications.*

ISO 14526-3:1999, *Plastics — Phenolic powder moulding compounds (PF-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 14526, the terms and definitions given in ISO 472 and ISO 14526-1 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 PF-PMC	110 to 120	165 to 175	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 PF-PMC under test, provided that it is the same for all specimens of the same thickness moulded from any one type of PF-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 PF-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 PF-PMC with fine filler	165 to 175	25 to 40	20 to 60 per mm thickness
Compression PF-PMC with coarse filler	165 to 175	40 to 60	

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 PF-PMC under test, provided that it is the same for all specimens of the same thickness moulded from any one type of PF-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 ± 2) °C and (50 ± 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 ± 2) °C and (50 ± 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 ± 2) °C and (50 ± 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 PF-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 PF-PMCs. Comparisons of different materials using these properties may well be restricted to those thermosets in the same generic families.

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	$120 \times 120 \times 2$ ISO 295 type E2	Q	%	Mean value of two directions normal to each other	
1.2		S_{Mp}	See footnote b	$60 \times 60 \times 2$ 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	$E_{tc}1$	ISO 899-1			MPa	At 1 h	Strain $\leqslant$ 0,5 %
2.5		$E_{tc}10^3$					At 1000 h	
2.6	Flexural modulus	E_f	ISO 178	$80 \times 10 \times 4$	Q/M	MPa	Test speed 2 mm/min	
2.7	Flexural strength	σ_{fM}						
2.8	Charpy impact strength	a_{cU}	ISO 179-1	$80 \times 10 \times 4$	Q/M	kJ/m^2	Edgewise impact	
2.9	Charpy notched impact strength	a_{cA}		$80 \times 10 \times 4$ machined V-notch r = 0,25				
2.10	Tensile-impact strength	a_{t1}	ISO 8256	$80 \times 10 \times 4$ machined double V-notch r = 1			Record if fracture cannot be obtained with notched Charpy impact test	
	Puncture impact behaviour							
2.11	— Force	F_M	ISO 6603-2	$60 \times 60 \times 2$ prepared from ISO 295 type E2 or ISO 10724-2 type D2	Q/M	N	Maximum force	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	— Energy	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	$T_f1,8$	ISO 75-2	$80 \times 10 \times 4$	Q/M	°C	1,8	Max. surface stress (MPa)	Use flatwise loading
3.2		$T_f8,0$					8,0		
3.3	Coefficient of linear thermal expansion	α_o	ISO 11359-2	$60 \times 10 \times 2$ prepared from $120 \times 120 \times 2$ ISO 295 type E2	Q	$°C^{-1}$	—		Record secant value over temperature range from 23 °C to 55 °C
3.4		α_p		$60 \times 10 \times 4$ prepared from ISO 3167 type A	M		Parallel to melt flow direction		
3.5		α_p		$60 \times 10 \times 2$ prepared from $60 \times 60 \times 2$ ISO 10724-2 type D2			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	$125 \times 13 \times 3$	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	$\geqslant 150 \times \geqslant 150 \times 3$	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	$80 \times 10 \times 4$	Q/M	%	Use procedure A: top surface ignition		

[a] Q = Compression moulding
M = Injection moulding

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**		
4	**Electrical properties**								
4.1	Relative permittivity	$\varepsilon_r 100$	IEC 60250	$\geqslant 60 \times \geqslant 60 \times 1$ or $\geqslant 60 \times \geqslant 60 \times 2$	Q/M	—	100 Hz	Compensate for electrode edge effects. 1-minute value.	
4.2		$\varepsilon_r 1M$					1 MHz		
4.3	Dissipation factor	$\tan\delta 100$				—	100 Hz		
4.4		$\tan\delta 1M$					1 MHz		
4.5	Volume resistivity	ρ_e	IEC 60093	$\geqslant 60 \times \geqslant 60 \times 1$ or $\geqslant 60 \times \geqslant 60 \times 2$	Q/M	Ω·cm	Voltage 500 V	1-minute value	
4.6	Surface resistivity	σ_e				Ω		Use contacting line electrodes 50 mm long, 1 mm to 2 mm wide and 5 mm apart	1-minute value
4.7	Electric strength	$E_s 1$	IEC 60243-1	$\geqslant 60 \times \geqslant 60 \times 1$	Q/M	kV/mm	Use 20-mm-diameter spherical electrodes. Immerse in transformer oil in accordance with IEC 60296. Use a voltage application rate of 2 kV/s.		
4.8		$E_s 2$		$\geqslant 60 \times \geqslant 60 \times 2$					
4.9	Proof tracking index	PTI	IEC 60112	$\geqslant 15 \times \geqslant 15 \times 4$ prepared from $120 \times 120 \times 4$ ISO 295 type E4 or ISO 3167 type A	Q/M	—	Use solution A		

[a] Q = Compression moulding
M = Injection moulding

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**
5	**Other properties**						
5.1	Water absorption	W_w24	ISO 62	$60 \times 60 \times 1$ prepared from $120 \times 120 \times 1$ ISO 295 type E1 or $60 \times 60 \times 1$ ISO 10724-2 type D1	Q/M	mg	24 h immersion in water at 23 °C
5.2		W_w24				% by mass	
5.3	Density	ρ_m	ISO 1183	$\geqslant 10 \times \geqslant 10 \times 4$ prepared from $120 \times 120 \times 4$ ISO 295 type E4 or use part of centre of ISO 3167 type A	Q/M	g/cm^3	The four methods specified in ISO 1183 are regarded as equivalent for the purposes of this part of ISO 14526

[a] Q = Compression moulding
M = Injection moulding

Table 4 — Additional 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	Apparent density	ρ_u	ISO 60	Moulding compound	—	g/cm^3	—	
1.2	Bulk factor	γ	ISO 171			—	Bulk factor $\gamma = \rho_m/\rho_u$ (for ρ_m see Table 3, property 5.3)	
1.3	Transfer flow	F_{tr}	ISO 7808			%	—	
2	**Mechanical properties**							
2.1	Ball indentation hardness	H961/30	ISO 2039-1	$\geqslant 20 \times \geqslant 20 \times 4$	Q/M	MPa	Use an indenting load of 961 N and an indenting time of 30 s	
3	**Thermal properties**							
3.1	Flammability (glow bar)	BH	IEC 60707	$(125 \pm 5) \times 10 \times 4$ prepared from ISO 3167 type A or $\geqslant 120 \times \geqslant 120 \times 4$ ISO 295 type E4	Q/M	—	Method BH	
4	**Electrical properties**							
4.1	Insulation resistance	R_{25}d	IEC 60167	$\geqslant 50 \times \geqslant 75 \times 4$	Q	Ω	Voltage 500 V 1-minute value	Dry, method 1
4.2		R_{25}w						Wet, method 2
5	**Other properties**							
5.1	Free ammonia	m_EAM	ISO 120	$\geqslant 120 \times \geqslant 120 \times 4$ ISO 295 type E4	Q	% by mass	Reduce a representative sample of the moulding to powder	
				ISO 3167 type A	M			
5.2	Volatile matter	m_V	ISO 3671	Not relevant				
	Extractable formaldehyde							
5.3	— with water	$m_{E/W}$F	ISO 4614	Not relevant				
5.4	— with acetic acid	$m_{E/AA}$F						
5.5	— with ethyl alcohol	$m_{E/AL}$F						

[a] Q = Compression moulding
M = Injection moulding

ICS 83.080.10
Price based on 11 pages