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ISO 15252-2

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Plastics — Epoxy powder moulding compounds (EP-PMCs) —

Part 2:

Preparation of test specimens and determination of properties

Plastiques — Poudres à mouler à base d'époxydes (EP-PMC) — Partie 2: Préparation des éprouvettes et détermination des propriétés



Reference number ISO 15252-2:1999(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 15252 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15252-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

ISO 15252 consists of the following parts, under the general title *Plastics* — *Epoxy powder moulding compounds* (*EP-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 — Epoxy powder moulding compounds (EP-PMCs) —

Part 2:

Preparation of test specimens and determination of properties

1 Scope

This part of ISO 15252 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of epoxy powder moulding compounds (EP-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 15252, as are the designatory properties found in ISO 15252-1.

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 15252. 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 15252 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.

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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

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)

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

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

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

ISO 15252-1:1999, Plastics — Epoxy powder moulding compounds (EP-PMCs) — Part 1: Designation system and basis for specifications.

ISO 15252-3:1999, Plastics — Epoxy powder moulding compounds (EP-PMCs) — Part 3: Requirements for selected moulding compounds.

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 15252, the terms and definitions given in ISO 472 and ISO 15252-1 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 $M_{\rm B}$ 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.

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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.

Type of PMC	$\begin{array}{c} \textbf{Melt temperature} \\ T_{\text{M}} \\ (\text{range}) \\ \\ ^{\circ}\text{C} \end{array}$	Mould temperature $T_{\rm C}$ (range) $^{\circ}{\rm C}$	Average injection velocity, $v_{\rm I}$ (range)	Cure time t _{CR} (range)
Injection EP-PMC	100 to 110	160 to 180	50 to 150	(see text)

Table 1 — Conditions for injection moulding of test specimens

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 $T_{\rm M}$,
- the mould temperature $T_{\mathbf{C}}$ and
- the cure time t_{CR} .

The cure time $t_{\rm CR}$ may be selected as a function of the curing behaviour and the type of pretreatment of the EP-PMC under test, provided that it is the same for all specimens of the same thickness moulded from any one type of EP-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 EP-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	$\begin{array}{c} \textbf{Mould temperature} \\ T_{\mathbb{C}} \\ (\text{range}) \end{array}$	$\begin{array}{c} \textbf{Mould pressure} \\ P_{\text{M}} \\ (\text{range}) \end{array}$	Cure time t _{CR} (range)
	°C	MPa	s
Compression EP-PMC	165 to 175	25 to 40	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 $p_{\mathbf{M}}$ and
- the cure time t_{CR} .

The cure time $t_{\rm CR}$ may be selected as a function of the curing behaviour and the type of pretreatment of the EP-PMC under test, provided that it is the same for all specimens of the same thickness moulded from any one type of EP-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 EP-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		ditions and ary instructions
1	Rheological and process	ing prope	erties					
1.1	Moulding shrinkage	S_{Mo}	ISO 2577	120 × 120 × 2 ISO 295 type E2	Q	%	Mean value of two	directions normal
1.2		S_{Mp}	See	60 × 60 × 2	M	,,,	Parallel to melt flo	w direction
1.3		S_{Mn}	footnote b	ISO 10724-2 type D2	•••		Normal to melt flo	w direction
2	Mechanical properties							
2.1	Tensile modulus	E_{t}				MPa	Test speed 1 mm	/min
2.2	Stress at break	$\sigma_{\! extsf{B}}$	ISO 527-1 and ISO 527-2	ISO 3167 type A or prepared from ISO 295 type E4	Q/M	IVII a	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 ≤ 0,5 %
2.5		$E_{\rm tc} 10^3$	130 033-1			IVII a	At 1000 h	_ Strain
2.6	Flexural modulus	E_{f}	ISO 178	80 × 10 × 4	Q/M	MPa	Test speed 2 mm	/min
2.7	Flexural strength	$\sigma_{\! extsf{fM}}$			٠	۵		
2.8	Charpy impact strength	a_{cU}	ISO 179-1	$80 \times 10 \times 4$			Edgewise impact	
2.9	Charpy notched impact strength	a_{cA}		$80 \times 10 \times 4$ machined V-notch r = 0,25	Q/M	kJ/m ²		
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 obtaine with notched Charpy impact test	
	Puncture impact behaviour							
2.11	— Force	F_{M}	100 0000 0	$60 \times 60 \times 2$ prepared	0/04	N	Maximum force	Striker velocity
2.12	— Energy	W_{P}	ISO 6603-2	from ISO 295 type E2 or ISO 10724-2 type D2	Q/M	J	Puncture energy at 50 % decrease in force after maximum	4,4 m/s. Striker diameter 20 mm. Lubricate striker. Clamp specimen to prevent any out-of- plane movement of its outer regions.

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 condition supplementary i			
3	Thermal properties									
3.1	Temperature of deflection under load	<i>T</i> _f 1,8	ISO 75-2	80 × 10 × 4	Q/M	°C	1,8	Max. surface	Use flatwise	
3.2		T _f 8,0	100 70 2	30 × 10 × 4	Q/W		8,0	stress (MPa)	loading	
3.3	Coefficient of linear thermal expansion	$\alpha_{\rm o}$		$60 \times 10 \times 2$ prepared from $120 \times 120 \times 2$ ISO 295 type E2	Q		_	-	Record secant value over temperature range from 23 °C to 55 °C	
3.4		α_{p}	ISO 11359-2	60 × 10 × 4 prepared from ISO 3167 type A		°C ⁻¹	Parallel to direction	melt flow		
3.5	3	α_{p}		$60 \times 10 \times 2$ prepared from $60 \times 60 \times 2$	М		Parallel to melt flow direction			
3.6		α_{n}		ISO 10724-2 type D2			Normal to direction	melt flow		
3.7	Burning behaviour	B _{50/3,0}	IEC	125 × 13 × 3	Q	_	Record on	e of classit	ications:	
3.8		B _{50/x}	60695-11-10	Additional specimen of different thickness <i>x</i>	-		V-0; V-1; HB40 or HB75 (V-2 not applicable to thermosets)			
3.9		B _{500/3,0}	IEC	$\geqslant 150 \times \geqslant 150 \times 3$	Q	_	Record on	e of classif	ications:	
3.10		B _{500/x}	60695-11-20	Additional specimen of different thickness <i>x</i>	•		Record one of classifications: 5VA; 5VB or N			
3.11	Oxygen index	O/23	ISO 4589-2	80 × 10 × 4	Q/M	%	Use proced	dure A: top	surface ignition	
	Q = Compression moulding	ding								

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 properti	es									
4.1	Relative permittivity	<i>€</i> _r 100				_	100 Hz				
4.2		$\varepsilon_{\rm r}$ 1M	IEC 60250	$\geqslant 60 \times \geqslant 60 \times 1$	Q/M		1 MHz	Compensate for electi	rode edge		
4.3	Dissipation factor	$\tan\delta$ 100	120 00200	or $\geqslant 60 \times \geqslant 60 \times 2$	S, III	_	100 Hz	effects. 1-minute value.			
4.4		$\tan\delta$ 1M					1 MHz				
4.5	Volume resistivity	$ ho_{e}$		≥ 60 × ≥ 60 × 1		Ω-cm	Voltage	1-minute value			
4.6	Surface resistivity	$\sigma_{\! m e}$	IEC 60093	or $\geqslant 60 \times \geqslant 60 \times 2$	Q/M	Ω	500 V	Use contacting line electrodes 50 mm long, 1 mm to 2 mm wide and 5 mm apart	1-minute value		
4.7	Electric strength	$E_{\rm s}$ 1	IEC 60243-1	≥ 60 × ≥ 60 × 1	Q/M	kV/mm		n-diameter spherical el			
4.8		$E_{\rm s}$ 2	120 00240 1	$\geqslant 60 \times \geqslant 60 \times 2$	Q/W	KV/IIIII	Immerse in transformer oil in accordance with IEC 60296. Use a voltage application rate of 2 kV/s.				
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	on A			

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 5.2	Water absorption	W _w 24 W _w 24	ISO 62	60 × 60 × 1 prepared from 120 × 120 × 1 ISO 295 type E1 or 60 × 60 × 1 ISO 10724-2 type D1	Q/M	mg % by mass	24 h immersion in water at 23 °C
5.3	Density	$ ho_{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 15252

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		ditions and ary instructions
1	Rheological and processi	ng properti	ies					
1.1	Apparent density	$ ho_{u}$	ISO 60			g/cm ³		_
1.2	Bulk factor	γ	ISO 171	Moulding compound	_	_	Bulk factor $\gamma = \rho$ (for $\rho_{\rm m}$ see Tabl	$\rho_{\rm m}/\rho_{\rm u}$ le 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	
3	Thermal properties							
3.1	Flammability (glow bar)	ВН	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	≥ 50 × ≥ 75 × 4	Q	Ω	Voltage 500 V	Dry, method 1
4.2		R ₂₅ W	120 00107	> 00 X > 10 X +	3	32	1-minute value	Wet, method 2
5	Other properties							
5.1	Free ammonia	$m_{E}AM$	ISO 120		No	t relevant	į	
5.2	Volatile matter	m_{\bigvee}	ISO 3671		No	t relevant	İ	
	Extractable formaldehyde							
5.3	— with water	$m_{E/W}F$	ISO 4614		No	t relevant	t	
5.4	 with acetic acid 	$m_{E/AA}F$						
5.5	— with ethyl alcohol	$m_{E/AL}F$						
	Q = Compression moulding M = Injection moulding							

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