INTERNATIONAL STANDARD

ISO 3672-2

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Plastics — Unsaturated-polyester resins (UP-R) —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Résines de polyesters non saturés (UP-R) —

Partie 2: Préparation des éprouvettes et détermination des propriétés

Reference number
ISO 3672-2:2000(E)

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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 3672 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3672-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

ISO 3672 consists of the following parts, under the general title *Plastics — Unsaturated-polyester resins (UP-R)*:

— *Part 1: Designation system*

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

Introduction

The purpose of this part of ISO 3672 is to designate procedures for the determination of intrinsic properties of unsaturated-polyester (UP) resins. It specifies procedures and conditions for the preparation of test specimens of unsaturated-polyester resins in a specified state, and methods for measuring their properties. Those properties and test methods which are suitable and necessary for characterizing unsaturated-polyester resins are listed. Because of the specificity of thermosetting resins like unsaturated-polyester resins, contrary to other plastic products, a distinction is made between the presentation of the properties before crosslinking (characteristics which are useful for processing) and after crosslinking (intrinsic characteristics).

Plastics — Unsaturated-polyester resins (UP-R) —

Part 2: Preparation of test specimens and determination of properties

WARNING — Persons using this part of ISO 3672 should be familiar with normal laboratory practice. This part of ISO 3672 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 3672 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of unsaturated-polyester resins. Requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing are given here.

The properties of crosslinked unsaturated-polyester resins have been selected from the general test methods in ISO 10350-1. Other test methods in wide use for, or of particular significance to, unsaturated-polyester resins (particularly properties useful for the processing of non-crosslinked resins) are also included in this part of ISO 3672.

In order to obtain reproducible and comparable test results, it is necessary to use the test methods, sample preparation and conditioning procedures, and specimen dimensions specified herein. Values determined will not necessarily be identical to those obtained using specimens of different dimensions or prepared using different procedures.

Other standards exist concerning the determination of properties and preparation of test specimens for unsaturated-polyester-based products, to which reference will be made, if required.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 3672. 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 3672 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 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 178:1993, *Plastics — Determination of flexural properties.*

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

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

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

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 604:1993, *Plastics — Determination of compressive properties.*

ISO 760:1978, *Determination of water — Karl Fischer method (General method).*

ISO 1523:1983, *Paints, varnishes, petroleum and related products — Determination of flashpoint — Closed cup equilibrium method.*

ISO 1675:1985, *Plastics — Liquid resins— Determination of density by the pyknometer method.*

ISO 2114:—[2] , *Plastics (polyester resins) and paints and varnishes (binders) — Determination of partial acid value and total acid value.*

ISO 2535:—[3], *Plastics — Unsaturated-polyester resins — Measurement of gel time at ambient temperature.*

ISO 2554:1997, *Plastics — Unsaturated polyester resins — Determination of hydroxyl value.*

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

ISO 2719:—[4], *Determination of flash point — Pensky-Martens closed cup method.*

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

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

ISO 3219:1993, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate.*

ISO 3521:1997, *Plastics — Unsaturated polyester and epoxy resins — Determination of overall volume shrinkage.*

ISO 3672-1:2000, *Plastics — Unsaturated-polyester resins (UP-R) — Part 1: Designation system.*

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

ISO 4615:1979, *Plastics — Unsaturated polyesters and epoxide resins — Determination of total chlorine content.*

ISO 4630:1997, *Binders for paints and varnishes — Estimation of colour of clear liquids by the Gardner colour scale.*

ISO 4901:1985, *Reinforced plastics based on unsaturated polyester resins — Determination of residual styrene monomer content.*

ISO 6271:1997, *Clear liquids — Estimation of colour by the platinum-cobalt scale.*

ISO 6603-2:—[5], *Plastics — Determination of puncture impact behaviour of rigid plastics — Part 2: Instrumented impact testing.*

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

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2) To be published. (Revision of ISO 2114:1996)

3) To be published. (Revision of ISO 2535:1997)

4) To be published. (Revision of ISO 2719:1988)

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

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

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

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

ISO 14848:1998, *Plastics — Unsaturated-polyester resins — Determination of reactivity at 130 °C.*

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

IEC 60112:1979, *Methods 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 — 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.*

EN 59:1977, *Glass reinforced plastics — Measurement of hardness by means of a Barcol impressor.*

3 Preparation of test specimens

3.1 General

This procedure shall be used for the preparation of specimens for the determination of crosslinked-resin properties.

It is essential that specimens are always prepared by the same procedure using the same processing conditions.

The specimens on which the properties are measured shall be cut from sheets of crosslinked resin, produced by a casting process. In view of the numerous possible fields of application for unsaturated-polyester resins, the choice was made to prepare specimens from resins not containing any filler or reinforcement in order to obtain the intrinsic properties of the crosslinked polymer, free of structural additives.

This procedure shall only be used for the determination of properties of crosslinked resins obtained from liquid unsaturated-polyester resins.

Test specimens from solid UP-R shall be prepared in accordance with the resin supplier's instructions.

Sheets of thermosetting resin shall be manufactured at 2 mm, 3 mm and 4 mm thickness, as required, for tests in Table 2. A sufficient number shall be produced to determine those properties required.

3.2 Pretreatment of materials

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

The resin shall be kept in moisture-proof containers until it is required for use.

3.3 Preparation of the sheets

3.3.1 Apparatus

3.3.1.1 Plates, having a thickness of 6 mm and approximate dimensions of 300 mm × 350 mm:

3.3.1.1.1 Two glass plates.

3.3.1.1.2 Two polished stainless-steel plates.

NOTE Alternatively, moulds may be made of other materials, such as steel or silicone.

3.3.1.2 Shims, having a thickness of 2 mm, 3 mm and 4 mm.

3.3.1.3 Silicone or latex joint, having a diameter of 5 mm.

3.3.1.4 Device for clamping and holding the plates.

3.3.1.5 Device for removing air bubbles from the reaction mixture (see 3.3.3), preferably a centrifuge, or a vacuum desiccator allowing the plate/joint/shim assembly to be put under a static vacuum.

3.3.1.6 Stirrer, for mixing the reaction mixture (e.g. glass rod).

3.3.1.7 Glass beaker, capacity 500 ml.

3.3.1.8 Laboratory balance, accurate to 0,1 g.

3.3.1.9 Laboratory oven, set at the temperature chosen for carrying out the post-treatment of the unsaturated-polyester resin.

3.3.2 Reagents

3.3.2.1 Crosslinking agent, specific to unsaturated-polyester resins, e.g. 2,4-pentanedione peroxide (acetylacetone peroxide) in a 34 % by mass solution in dimethyl phthalate and associated alcohols.

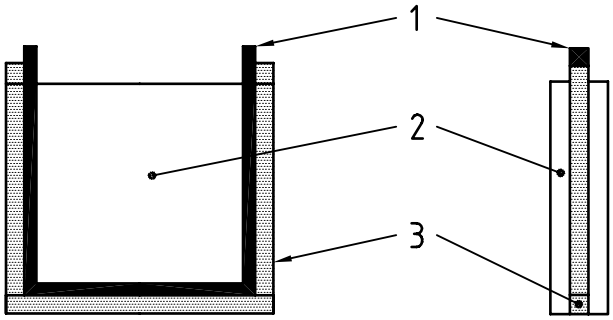
3.3.2.2 Polymerization accelerator, specific to unsaturated-polyester resins, e.g. cobalt(II) 2-ethylhexanoate in a solution containing 1 % by mass of cobalt.

3.3.2.3 External release agent, which does not modify the properties of the polymerized resin.

3.3.3 Procedure

Coat the plates (3.3.1.1.1 or 3.3.1.1.2) with a thin layer of release agent (3.3.2.3). Polish them until they shine in order to ensure that the cured-resin sheet produced has a high-quality surface finish.

Arrange the silicone or latex joint and the selected shim (2 mm, 3 mm or 4 mm) between the two plates as shown in Figure 1. Clamp the assembly with a suitable clamp and position vertically.



Key

1 Joint
2 Glass or steel plates
3 Shims

Figure 1 — Apparatus for preparing sheets

Using the laboratory balance (3.3.1.8), weigh 200 g of unsaturated-polyester resin into the glass beaker (3.3.1.7). Add 2 g of polymerization accelerator solution (3.3.2.2). Mix with the stirrer (3.3.1.6) until homogeneous, avoiding the introduction of air bubbles as much as possible.

NOTE 1 There is no need to add the accelerator if the unsaturated-polyester resin is already pre-accelerated.

Then add 3 g of peroxide solution (3.3.2.1) to the glass beaker. Mix with the stirrer until homogeneous, avoiding the introduction of air bubbles as much as possible.

It is desirable to store the peroxide solution at low temperature (5 °C), for example in a refrigerator. In this case, the solution shall be maintained at room temperature for 6 h before use.

WARNING — Under no circumstances should the acetylacetone peroxide and cobalt octoate solutions be mixed together, as an explosive mixture is formed. Mix each separately into the polyester resin.

NOTE 2 The type and proportions of accelerator and initiator given in this part of ISO 3672 are the reference conditions. In particular cases, other conditions may be agreed between the interested parties.

Remove the air bubbles from the reaction mixture using the centrifuge or the vacuum desiccator (3.3.1.5), then carefully pour it into the container formed by the plate/joint/shim assembly, without trapping any air bubbles in the resin. In the absence of a centrifuge, once the assembly is filled with the reactant mixture place it vertically in a vacuum desiccator (see 3.3.1.5) and apply a static vacuum for the time required to remove all air bubbles.

Maintain the assembly in the vertical position for 24 h to allow the crosslinking to take place.

Then open the mould and take out the sheet.

In order to determine the intrinsic properties of the unsaturated-polyester resin, a complementary heat treatment is carried out (post-cure).

For ambient applications (unheated and/or non-thermoregulated mould), carry out this complementary treatment as follows:

— place the unsaturated-polyester sheet between two polished stainless-steel plates (3.3.1.1.2);

— allow to stand for 24 h at ambient temperature, then heat for 16 h at 40 °C in the laboratory oven (3.3.1.9).

For high-temperature applications (> 60 °C), carry out the complementary treatment as follows:

— place the unsaturated-polyester sheet between two polished stainless-steel plates;

— allow to stand for 24 h at ambient temperature, then heat for 16 h at 40 °C followed by 2 h at 120 °C in the laboratory oven.

3.4 Cutting out test specimens

Cut test specimens from the prepared sheets (thickness 2 mm, 3 mm or 4 mm) in accordance with ISO 2818.

4 Conditioning of test specimens

Unless otherwise specified, condition the test specimens in accordance with ISO 291 for at least 16 h at 23 °C ± 2 °C and (50 ± 5) % relative humidity prior to determining the properties in Tables 1 and 2.

5 Determination of properties

5.1 Properties are presented in the form of three tables, Tables 1, 2 and 3, depending whether they concern:

— non-crosslinked resins (properties useful for the processing of unsaturated-polyester resins) (these properties are listed in Table 1).

— crosslinked resins (intrinsic properties of unsaturated-polyester resins) (these properties are listed in Table 2 and Table 3).

5.2 In the determination of intrinsic properties, the presentation of results, the standards, and the supplementary instructions and notes given in ISO 10350-1 shall be applied. All the tests shall be carried out at 23 °C ± 2 °C and (50 ± 5) % relative humidity in accordance with ISO 291, unless specifically stated otherwise in Table 2.

Table 2 is taken from ISO 10350-1 and the properties listed are those which are appropriate to unsaturated-polyester-based products. These properties are considered useful for comparisons of data generated for different thermosets and thermoplastics.

Table 3 contains those properties, not found specially in Table 2, which may be of interest for the practical characterisation of unsaturated-polyester resins.

Table 1 — Processing properties

	Property	Standard	Unit	Test conditions and supplementary instructions
1	**Rheological properties**			
1.1	Viscosity	ISO 3219	Pa·s	Measured at known shear rate (liquid UP)
2	**Physical properties**			
2.1	Density	ISO 1675	g/ml	
2.2	Colour	ISO 4630 ISO 6271	Gardner scale Pt-Co scale	Gardner colour Pt-Co colour (Hazen)
2.3	Flash point	ISO 1523 ISO 2719	°C °C	Applicable to resins with flash point up to 65 °C Applicable to resins with flash point over 65 °C
2.4	Softening point	ISO 4625	°C	Ring-and-ball method (solid UP)
3	**Chemical properties**			
3.1	Acid value Hydroxyl value	ISO 2114 ISO 2554	mg KOH/g	
3.2	Volatile-matter content	To be agreed between the interested parties	mass %	
3.3	Water content	ISO 760	mass %	
3.4	Halogen content	ISO 4615	mass %	
4	**Crosslinking-related properties**			
4.1	Gel time Reactivity	ISO 2535 ISO 14848	min °C, min	Measure gel time at 25 °C
4.2	Volume shrinkage	ISO 3521	volume %	

Table 2 — Intrinsic properties and test conditions

	Property	Symbol	Standard	Specimen type (dimensions in mm)	Unit	Test conditions and supplementary instructions	
1	**Rheological properties**						
1.1	Moulding shrinkage	S_{Mo}	ISO 2577	120 × 120 × 2	%	Record mean of two directions normal to each other	
2	**Mechanical properties**						
2.1	Tensile modulus	E_t	ISO 527-1 ISO 527-2	ISO 3167, type A	MPa	Test speed 1 mm/min	
2.2	Stress at break	σ				Test speed 5 mm/min	
2.3	Strain at break	ε_B			%	Test speed 5 mm/min	
2.4 2.5	Tensile creep modulus	$E_{tc}1$ $E_{tc}10^3$	ISO 899-1		MPa	At 1 h At 1 000 h	Strain < 0,5 %
2.6	Flexural modulus	E_f	ISO 178	80 × 10 × 4	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 machined V-notch r = 0,25	kJ/m^2	Edgewise impact	
2.9	Charpy notched impact strength	a_{cA}					
2.10 2.11	Multi-impact behaviour	F_M W_P	ISO 6603-2	60 × 60 × 4	N J	Max. force Total energy	Striker velocity 4,4 m/s Striker diameter 20 mm Lubricate striker
3	**Thermal properties**						
3.1	Glass transition temperature	T_g	ISO 11357-2		°C	Use 10 °C/min	
3.2 3.3	Temperature of deflection under load (HDT)	$T_f1{,}8$ $T_f0{,}45$	ISO 75-2	80 × 10 × 4	°C	1,8 0,45	Maximum surface stress (MPa)
3.4	Coefficient of linear expansion	α_p α_n	ISO 11359-2	Prepared from ISO 3167	°C^{-1}	Record secant value over temperature range 23 °C to 55 °C	
3.5	Burning behaviour	B50/3	IEC 60695-11-10	125 × 13 × 3		Record one of classifications: V-0, V-1, V-2, HB 40 or none	
3.6		B500/3	IEC 60695-11-20	⩾150 × ⩾150 × 3		Record one of classifications: 5 VA, 5 VB or none	
3.7	Oxygen index		ISO 4589-2	80 × 10 × 4		Use procedure A: top surface ignition	
4	**Electrical properties**						
4.1 4.2	Relative permittivity	ε_r100 ε_r1M	IEC 60250	⩾60 × ⩾60 × 2		100 Hz 1 MHz	Compensate for electrode edge effects
4.3 4.4	Dissipation factor	$\tan\delta100$ $\tan\delta1M$				100 Hz 1 MHz	
4.5	Volume resistivity	ρ_e	IEC 60093		Ω·m		
4.6	Surface resistivity	σ_e			Ω		
4.7	Electric strength	E_B	IEC 60243-1		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	Comparative tracking index	CTI	IEC 60112	⩾15 × ⩾15 × 4 prepared from ISO 3167		Use solution A	
5	**Other properties**						
5.1 5.2	Water absorption	w_w w_H	ISO 62	Thickness ⩾1	%	Saturation value in water at 23 °C Saturation value at 23 °C, 50 % R.H.	
5.3	Density	ρ	ISO 1183	4-mm-thick plate	kg/m^3	The four methods specified in ISO 1183 are regarded as equivalent for the purposes of this part of ISO 3672	

Table 3 — Additional ("non-standard") properties and test conditions

	Property	Symbol	Standard	Specimen type (dimensions in mm)	Unit	Test conditions and supplementary instructions
1	**Rheological and processing properties**					
1.1	Barcol hardness		EN 59		Barcol units	
2	**Mechanical properties**					
2.1	Compressive strength		ISO 604		MPa	
2.2	Compressive modulus	E_Q	ISO 604		GPa	
3	**Other properties**					
3.1	Residual styrene monomer content		ISO 4901		mass %	CPG method

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