INTERNATIONAL STANDARD

ISO 3673-2

Second edition
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Plastics — Epoxy resins —

Part 2: Preparation of test specimens and determination of properties

Plastiques — Résines époxydes —

Partie 2: Préparation des éprouvettes et détermination des propriétés

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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 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 3673-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 3673-2:1999), which has been technically revised.

ISO 3673 consists of the following parts, under the general title *Plastics — Epoxy resins*:

— *Part 1: Designation*

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

Introduction

The purpose of this part of ISO 3673 is to designate procedures for the determination of intrinsic properties of epoxy resins (EP). It specifies procedures and conditions for the preparation of test specimens of epoxy resins in a specified state, and methods for measuring their properties. Those properties and test methods which are suitable and necessary for characterizing epoxy resins are listed. Because of the specificity of thermosetting resins like epoxy 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 — Epoxy resins —

Part 2:
Preparation of test specimens and determination of properties

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, 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 regulatory requirements.

1 Scope

This part of ISO 3673 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of epoxy resins. Requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing are given.

Properties of crosslinked epoxy resins have been selected from the general test methods in ISO 10350-1:1998. Other test methods in wide use with, or of particular significance to, epoxy resins, particularly properties useful for the processing of non-crosslinked epoxy resins, are also included in this part of ISO 3673.

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

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

2 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, *Plastics — Determination of water absorption*

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

ISO 178, *Plastics — Determination of flexural properties*

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

ISO 179-2, *Plastics — Determination of Charpy impact properties — Part 2: Instrumented impact test*

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

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

ISO 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pyknometer method and titration method*

ISO 1523, *Determination of flash point — Closed cup equilibrium method*

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

ISO 2555, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method*

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

ISO 2592, *Determination of flash and fire points — Cleveland open cup method*

ISO 2719, *Determination of flash point — Pensky-Martens closed cup method*

ISO 2811 (all parts), *Paints and varnishes — Determination of density*

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

ISO 3001, *Plastics — Epoxy compounds — Determination of epoxy equivalent*

ISO 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity*

ISO 3105, *Glass capillary kinematic viscometers — Specifications and operating instructions*

ISO 3146, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods*

ISO 3167, *Plastics — Multipurpose test specimens*

ISO 3219, *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, *Plastics — Unsaturated polyesters and epoxy resins — Determination of overall volume shrinkage*

ISO 3679, *Determination of flash point — Rapid equilibrium closed cup method*

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

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

ISO 4625-1, *Binders for paints and varnishes — Determination of softening point — Part 1: Ring-and-ball method*

ISO 4630-1, *Clear liquids — Estimation of colour by the Gardner colour scale — Part 1: Visual method*

ISO 4895, *Plastics — Liquid epoxy resins — Determination of tendency to crystallize*

ISO 6271-1, *Clear liquids — Estimation of colour by the platinum-cobalt scale — Part 1: Visual method*

ISO 7142, *Binders for paints and varnishes — Epoxy resins — General methods of test*

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

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

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

ISO 12058-1, *Plastics — Determination of viscosity using a falling-ball viscometer — Part 1: Inclined-tube method*

ISO 21627-1, *Plastics — Epoxy resins — Determination of chlorine content — Part 1: Inorganic chlorine*

ISO 21627-2, *Plastics — Epoxy resins — Determination of chlorine content — Part 2: Easily saponifiable chlorine*

ISO 21627-3, *Plastics — Epoxy resins — Determination of chlorine content — Part 3: Total chlorine*

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

IEC 60112, *Method for the determination of the proof and the comparative tracking indices of solid insulating materials*

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

IEC 60250, *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, *Fluids for electrotechnical applications — Unused mineral insulating oils for transformers and switchgear*

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

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

3 Preparation of test specimens

3.1 General

This procedure is used only 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 of epoxy resins, the choice was made to prepare test specimens from resins not containing any filler or reinforcement in order to obtain the intrinsic properties of the crosslinked polymer, free of structural additives.

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 epoxy resin sample is normally necessary. If a pretreatment is required, this shall be in accordance with the manufacturer's recommendations.

3.3 Preparation of test sheets

3.3.1 Apparatus

3.3.1.1 **Plates**, having a thickness of 6 mm and approximate dimensions of 300 mm × 350 mm, for use in moulding sheets of resin:

3.3.1.1.1 **Two glass plates**.

or

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 3.3.3.4), preferably a centrifuge, or a vacuum dessicator allowing the plate/joint/shim assembly to be put under a static vacuum.

3.3.1.6 **Stirrer**, for mixing the resin and crosslinking agent (e.g. a 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 epoxy resin.

3.3.2 Reagents

3.3.2.1 **Crosslinking agent**, specific to epoxy resins (e.g. methyltetrahydrophthalic anhydride) (see warning in 3.3.3.5).

3.3.2.2 **Curing accelerator**, e.g. 2,4,6-tris(dimethylaminomethyl)phenol or *N*,*N*-dimethylbenzylamine.

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

3.3.3 Procedure

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

3.3.3.2 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.

3.3.3.3 Using a laboratory balance (3.3.1.8), weigh the following reagents into a glass beaker:

— 100 g of epoxy resin;

— methyltetrahydrophthalic anhydride at an anhydride/epoxy equivalent ratio of 0,9/1;

— 0,5 phr (parts per hundred parts of resin, by mass) of 2,4,6-tris(dimethylaminomethyl)phenol

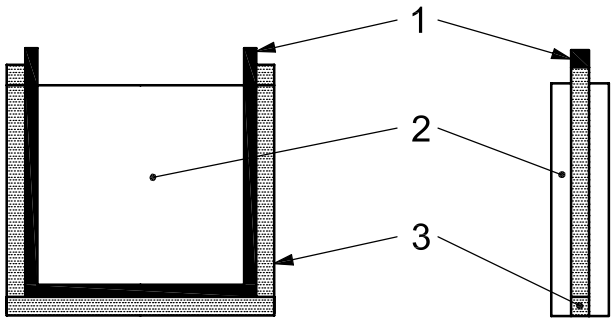
or

2 phr (parts per hundred parts of resin, by mass) of *N*,*N*-dimethylbenzylamine.

Using the stirrer, mix until homogeneous, avoiding the introduction of air bubbles as much as possible.

3.3.3.4 Remove any air bubbles from the mixture using the centrifuge (see 3.3.1.5), then carefully pour the mixture into 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.

When the viscosity of the mixture is high, air bubbles can be removed at an elevated temperature (e.g. 100 °C). In this case, the amount of accelerator has to be decreased. For example, use 0,25 phr of 2,4,6-tris(dimethylaminomethyl)phenol or 1 phr of *N*,*N*-dimethylbenzylamine.



Key

1 joint
2 glass or steel plates
3 shims

Figure 1 — Apparatus for preparing test sheets

3.3.3.5 Maintain the assembly in the vertical position while curing in the laboratory oven. If using methyltetrahydrophthalic anhydride with 2,4,6-tris(dimethylaminomethyl)phenol or *N*,*N*-dimethylbenzylamine for crosslinking, carry out the heat treatment for 2 h at 100 °C followed by 15 h at 150 °C.

WARNING — Fumes from the crosslinking agent (methyltetrahydrophthalic anhydride) irritate the respiratory system and skin. Use ventilation and wear protective masks, gloves and goggles.

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.

3.5 Conditioning test specimens

Unless otherwise specified, condition the test specimens 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.

4 Determination of properties

4.1 Properties are presented in the form of either Table 1 or Table 2, depending whether they concern

— non-crosslinked resins (properties useful for the processing of epoxy resins) (these properties are listed in Table 1);

— crosslinked resins (intrinsic properties of epoxy resins) (these properties are listed in Table 2).