
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



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

STANDARDSISO.COM : Click to view the full PDF of ISO 3673-2:1999

© ISO 1999

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 734 10 79
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Foreword.....	iv
Introduction.....	v
1 Scope	1
2 Normative references	1
3 Preparation of test specimens	3
3.1 General.....	3
3.2 Pretreatment of materials	4
3.3 Preparation of test sheets.....	4
3.4 Cutting out test specimens.....	5
3.5 Conditioning test specimens.....	6
4 Determination of properties.....	6

STANDARDSISO.COM : Click to view the full PDF of ISO 3673-2:1999

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 3673 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

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 the 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).

STANDARDSISO.COM : Click to view the full PDF of ISO 3673-2:1999

Plastics — Epoxy resins —

Part 2:

Preparation of test specimens and determination of properties

WARNING — Persons using this part of ISO 3673 should be familiar with normal laboratory practice. This part of ISO 3673 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 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 here.

Properties of crosslinked epoxy resins have been selected from the general test methods in ISO 10350:1993. Other test methods in wide use, 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 normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 3673. 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 3673 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 75-1:1993, *Plastics — Determination of temperature of deflection under load — Part 1: General test method*.

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 3673-2:1999(E)

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

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

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 pycnometer method.*

ISO 2555:1989, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method.*

ISO 2592:—²⁾, *Petroleum products — Determination of flash and fire points — Cleveland open cup method.*

ISO 2719:1988, *Petroleum products and lubricants — Determination of flash point — Pensky-Martens closed cup method.*

ISO 2811:1997 (all parts), *Paints and varnishes — Determination of density.*

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

ISO 3001:1999, *Plastics — Epoxy compounds — Determination of epoxy equivalent.*

ISO 3104:1994, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity.*

ISO 3105:1994, *Glass capillary kinematic viscometers — Specifications and operating instructions.*

ISO 3146:—³⁾, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods.*

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 polyesters and epoxy resins — Determination of overall volume shrinkage.*

ISO 3679:1983, *Paints, varnishes, petroleum and related products — Determination of flashpoint — Rapid equilibrium method.*

ISO 4583:1998, *Plastics — Epoxy resins and related materials — Determination of easily saponifiable chlorine.*

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 4625:1980, *Binders for paints and varnishes — Determination of softening point — Ring-and-ball method.*

2) To be published. (Revision of ISO 2592:1973)

3) To be published. (Revision of ISO 3146:1985)

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

ISO 4895:1997, *Plastics — Liquid epoxy resins — Determination of tendency to crystallize.*

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

ISO 7142:1982, *Binders for paints and varnishes — Epoxy resins — General methods of test.*

ISO 10350:1993⁴⁾, *Plastics — Acquisition and presentation of comparable single-point data.*

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 11376:1997, *Plastics — Epoxy resins and glycidyl esters — Determination of inorganic chlorine.*

ISO 13651:1996, *Plastics — Epoxy resins and related materials — Determination of total saponifiable chlorine.*

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 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.*

ASTM D 1545-98, *Standard Test Method for Viscosity of Transparent Liquids by Bubble Time Method.*

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 for 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.

4) ISO 10350:1993 is the normative reference, although it has now been replaced by ISO 10350-1:1998.

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:

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 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. 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 (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.

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 (3.3.1.7):

100 g of epoxy resin;

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

0,5 phr (mass ratio based on epoxy resin) of 2,4,6-tris(dimethylaminomethyl)phenol;

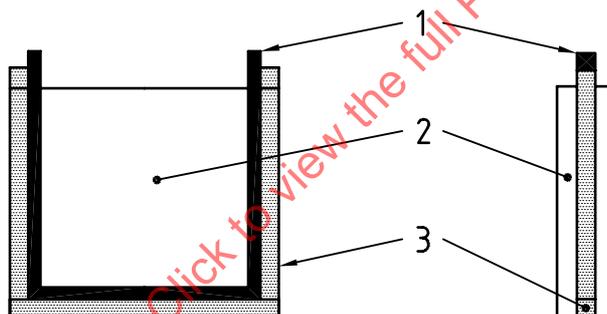
or

2 phr (mass ratio based on epoxy resin) of *N,N*-dimethylbenzylamine.

Using the stirrer (3.3.1.6), 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.

NOTE 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 (3.3.1.9). 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 in accordance with ISO 291 for at least 16 h at $23\text{ °C} \pm 2\text{ °C}$ and $(50 \pm 5)\%$ 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).

4.2 In the determination of intrinsic properties, the presentation of results, the standards, and the supplementary instructions and notes given in ISO 10350:1993 shall be applied. All the tests shall be carried out at $23\text{ °C} \pm 2\text{ °C}$ and $50\% \pm 5\%$ relative humidity in accordance with ISO 291 unless specifically stated otherwise in Table 2. Table 2 is taken from ISO 10350:1993 and the properties listed are those which are appropriate to epoxy-based products. These properties are those considered useful for comparisons of data generated for different thermosets and thermoplastics.

STANDARDSISO.COM : Click to view the full PDF of ISO 3673-2:1999