INTERNATIONAL STANDARD

**ISO 18280**

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# Plastics — Epoxy resins — Test methods

*Plastiques — Résines époxydes — Méthodes d'essai*



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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

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 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 18280 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.



This second edition cancels and replaces the first edition (ISO 18280:2005), which has been revised to include the following additional test methods:



— determination of the softening point (see 3.1.2);

— determination of the 1,2-glycol content (see 3.2.3);

— determination of the electrical conductivity of aqueous resin extracts (see 3.2.4).

## Introduction

The purpose of this International Standard is to present an overview of ISO test methods for characterizing epoxy resins. Those test methods that are suitable and necessary for characterizing epoxy resins prior to polymerization are listed, along with brief explanations of the principles involved.

Because of the specificity of thermosetting resins like epoxy resins, a distinction is made between the presentation of properties before crosslinking (characteristics which are useful for processing) and after crosslinking (intrinsic characteristics). Procedures for determining intrinsic characteristics of crosslinked (or cured) epoxy resins are given in ISO 3673-2.

# Plastics — Epoxy resins — Test methods

## 1 Scope

Epoxy resins are a family of synthetic resins, including products, which range from viscous liquids to high-melting solids. The resin molecule contains as reactive sites one or more oxirane or epoxy groups, usually in the form of a glycidyl group. The most commercially important resin is the glycidyl ether of bisphenol A, produced by the condensation of epichlorohydrin and diphenylpropane (bisphenol A). Epoxy resins with different characteristics are also produced commercially by reacting epichlorohydrin with other materials. To be of use, the resins must be crosslinked with a curing agent or hardener. The choice of curing agent is of importance in designing an epoxy resin system for a given application. The major reactive groups in the resin, the epoxy and the hydroxyl groups, react with many other groups so that many types of chemical substance can be used as curing agents. These include acid anhydrides, aliphatic and aromatic amines and polyaminoamides. Some curing agents will crosslink the resin at ambient temperatures while others require the application of heat.

This International Standard provides an overview of the ISO test methods used to characterize epoxy resins. The listed property to be determined in any given case depends on agreement between the supplier and customer.



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

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

ISO 3146:2000, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods*

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 3251, *Paints, varnishes and plastics — Determination of non-volatile-matter content*

ISO 3451-1:2008, *Plastics — Determination of ash — Part 1: General methods*

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

ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method*

ISO 4615:1979, *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 5661, *Petroleum products — Hydrocarbon liquids — Determination of refractive index*

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

ISO 7327, *Plastics — Hardeners and accelerators for epoxide resins — Determination of free acid in acid anhydride*

ISO 9702, *Plastics — Amine epoxide hardeners — Determination of primary, secondary and tertiary amine group nitrogen content*

ISO 11357-3, *Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization*



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



ISO 21048, *Plastics — Epoxy resins — Determination of 1,2-glycol content*



ISO 21318, *Plastics — Epoxy resins — Determination of electrical conductivity of aqueous resin extracts*

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*

## 3 Description of test methods

### 3.1 Physical properties

#### 3.1.1 Determination of the melting range

##### 3.1.1.1 Determination of the melting range using capillary methods

The test shall be performed in accordance with ISO 3146:2000, method A.

**Principle of ISO 3146:2000, method A**

A specimen is heated in a capillary tube at a controlled rate, and monitored visually for change in shape.

NOTE Epoxy resins are not single chemical substances, but polymolecular compounds. This is evident from their melting behaviour — the process is not limited to one temperature but extends over a range of temperatures. The limits of the melting range consist of a "sinter point" (also known as the "stick point") and the "melted stage". The challenge is to establish exact definitions of these "points" for interpretation by various operators. The repeatability of the method is good, but reproducibility can be poor, hence the necessity for agreement on the definitions of these "points".

The melting range is the temperature interval between the sinter point and the melted stage.

The sinter point (stick point) is the temperature at which the first physical property change is observed in the powdered resin. The melted stage is the temperature at which the mass of the resin becomes totally fluid or (as opposed to the melting of partially crystalline substances) the temperature at which the sintered mass of the resin becomes translucent (not transparent) and/or when the resin starts to shrink and separate from the capillary tube wall.

The moisture content of the resin powder has an effect on the determination of the melting range. Since the melting range is to be determined on products as received, it is not permissible to subject the resin to drying before testing. However, for comparative tests, it might be desirable to take into account the moisture content. In such a case, it is possible to dry the resin powder to a constant mass, or at least for 48 h, using diphosphorus pentoxide or a similar dehydrating agent.

**Preparation of the samples**



Use a sample of resin in powder form. When the resin is in lump or flake form, crush it in a mortar and sift the pulverized resin through a 250 µm sieve. Take the sample from the material that passes through the 250 µm sieve.



### 3.1.1.2 Determination of the melting range using DSC



The test shall be performed in accordance with ISO 11357-3.

### 3.1.2 Determination of the softening point

The test shall be performed in accordance with ISO 4625-1 (ring-and-ball method).

**Principle of ISO 4625-1**

The softening point is defined as the temperature at which a disc of the sample held within a horizontal ring is forced downward a distance of 25,4 mm under the weight of a steel ball as the sample is heated at 5 °C/min in a distilled-water or glycerol bath.

### 3.1.3 Determination of density

The test shall be performed in accordance with ISO 1675 (pyknometer method) or ISO 3675 (hydrometer method). Unless otherwise indicated, the determination shall be carried out at 23 °C. Results are expressed in grams per cubic centimetre to three decimal places.

The pyknometer method may be used for all liquid resins; the hydrometer method is suitable for resins with a viscosity below 1 Pa·s.