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Plastics — Epoxy resins — Test methods

Plastiques — Résines époxydes — Méthodes d'essai

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Foreword

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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.

3.1.4 Determination of refractive index

The test shall be performed in accordance with ISO 5661.

3.1.5 Determination of viscosity

Three procedures for the determination of viscosity are proposed:

- A reference method: determination using a viscometer with a definite speed gradient, for example a plate/cone viscometer (ISO 3219).
- Two verification methods:
  - determination with a rotary viscometer (ISO 2555);
  - determination with a falling-ball viscometer (ISO 12058-1).

NOTE Since, in a number of cases, liquid epoxy resins or epoxy resin solutions are non-Newtonian fluids, the measured value might be dependent on the test method. When carrying out comparative tests, it is therefore necessary to ensure that the same method is always used.

3.1.6 Determination of tendency to crystallize

The test shall be performed in accordance with ISO 4895.

Principle of ISO 4895

Calcium carbonate powder is mixed with the liquid epoxy resin dissolved in ethanol. The mixture is kept at a specified low temperature and observed at specified time intervals to compare the change in fluidity and crystallization.

3.1.7 Determination of overall volume shrinkage

The test shall be performed in accordance with ISO 3521.

Principle of ISO 3521

The overall shrinkage is calculated from the specific gravity of a test specimen before and after curing.

First, the specific gravity of the resin composition is determined a) at the initial temperature of mixing of the components, excluding the initiators normally added to unsaturated polyesters, and b) at 23 °C after curing and conditioning of the test specimen.

The overall volume shrinkage is then calculated as a percentage of the change in the specific gravity before and after curing.

The specific gravity of the test specimen at the moment of mixing is determined for the mixed components at known intervals and the results extrapolated to zero time. For components that react at elevated temperature, the specific gravity of the mixture is determined by calculation from the individual specific gravities of the components. The specific gravity of the test specimen at 23 °C after curing and conditioning is determined by weighing in silicone oil.

3.1.8 Determination of the flash point

The test shall be performed in accordance with ISO 1523 or ISO 2592.

Principle of ISO 1523

A test portion is heated in a suitably designed closed cup by immersing it to the required level in a suitable bath. The temperature of the bath is slowly raised at such a rate that the difference in temperature between the liquid in the bath and the test portion in the cup never exceeds 2 °C, and the heating procedure ensures that the temperature of the test portion does not rise more quickly than about 0,5 °C in 1,5 min. During the heating period, ignition trials are carried out at intervals of not less than 1,5 min. The lowest temperature at which a flash occurs is noted and, from this and a duplicate determination, the flash point of the test portion is calculated, corrected to the standard atmospheric pressure of 101,3 kPa (1 013 mbar).

Principle of ISO 2592

The flash and fire points are determined by the Cleveland open cup method. The test cup is filled to a specified level with the test portion. The temperature of the test portion is increased rapidly at first and then at a slow constant rate as the flash point is approached. At specified temperature intervals, a small test flame is passed across the cup. The lowest temperature at which application of the test flame causes the vapour above the surface of the liquid to ignite is taken as the flash point at ambient barometric temperature.

3.1.9 Estimation of colour

The test shall be performed in accordance with ISO 4630-1 or ISO 6271-1.

Principle of ISO 4630-1 and ISO 6271

The colour of a sample under examination is viewed in a glass tube of standard diameter and visually compared with the colours of arbitrarily numbered colour standards. The standard that most closely matches the colour of the test sample is identified and the result is expressed in terms of a number on the Gardner colour scale (ISO 4630-1) or the Pt/Co colour scale (ISO 6271-1).

3.2 Chemical properties

3.2.1 Determination of epoxy equivalent

The test shall be performed in accordance with ISO 3001.

Principle of ISO 3001

The epoxy groups in an epoxy compound are reacted with nascent hydrogen bromide produced by the reaction of a 0,1 mol/l perchloric acid solution on tetraethylammonium bromide. The end point is determined either using crystal violet as an indicator or by a potentiometric method.

3.2.2 Determination of chlorine content

3.2.2.1 General

In producing epoxy resins based on epichlorohydrin, impurities containing chlorine can be formed. These are: inorganic chlorine (or ionic chlorine), 1,2-chlorohydrin, 1,3-chlorohydrin, 1-chloro-2-glycidylether. Their chemical activities differ significantly, so different analytical procedures are needed for their analysis.

3.2.2.2 Inorganic chlorine

The test shall be performed in accordance with ISO 21627-1.

Principle of ISO 21627-1

A test portion is dissolved in a suitable solvent. The chlorine ion content is determined by potentiometric titration with standardized silver nitrate solution.

3.2.2.3 Easily saponifiable chlorine

This mainly consists of chlorine that is present as 1,2-chlorohydrin. The test shall be performed in accordance with ISO 21627-2.

Principle of ISO 21627-2

Epoxy resins, excluding glycidyl esters, are reacted with NaOH solution at room temperature in butoxyethanol.

Glycidyl esters are reacted with NaOH at 50 °C in methanol. The mixture is acidified and the chloride ion concentration resulting from the saponification is determined by potentiometric titration with a standardized silver nitrate solution. A correction is made for the inorganic chlorine content of the sample, determined by the method specified in ISO 21627-1.

3.2.2.4 Total chlorine

There are two ISO methods for determining total chlorine: ISO 21627-3 and ISO 4615:1979.

Principle of ISO 21627-3

A test portion is dissolved in diethylene glycol monobutyl ether and the solution is saponified with an alcoholic solution of potassium hydroxide by heating under reflux. The total chlorine content is then determined by potentiometric titration of the solution with a standardized silver nitrate solution.

Principle of ISO 4615:1979

A test portion is oxidized with sodium peroxide (method A) or gaseous oxygen (method B), and the resulting chloride determined by electrometric or volumetric titration.

3.2.3 Determination of 1,2-glycol content

The test shall be performed in accordance with ISO 21048.

Principle of ISO 21048

1,2-glycol is oxidized to aldehyde compounds by an excess of orthoperiodic acid, then potassium iodide is added to the reaction mixture in the presence of sulfuric acid. The iodine generated is then titrated against standardized sodium thiosulfate solution. The 1,2-glycol content is calculated from the sodium thiosulfate consumption.

3.2.4 Determination of the electrical conductivity of aqueous resin extracts

The test shall be performed in accordance with ISO 21318.

Principle of ISO 21318

A test portion is extracted with water at 95 °C for 20 h ± 0,5 h. The electrical conductivity of the aqueous extract is measured. From this measurement, the overall content of ionic impurities, such as chloride, sodium ions and catalyst residues, in the sample can be assessed.

3.2.5 Determination of ash

The test shall be performed in accordance with ISO 3451-1:2008, method A.

Principle of ISO 3451-1:2008, method A

The ash is determined by direct calcination, i.e. by burning off the organic matter and heating the residue at high temperature until constant mass is reached.

3.2.6 Determination of primary, secondary and tertiary amine group nitrogen content in amine epoxy hardeners

The test shall be performed in accordance with ISO 9702.

Principle of ISO 9702

a) Aliphatic amines

Total amine group nitrogen content: The total alkalinity is determined by potentiometric titration with hydrobromic or perchloric acid in glacial acetic acid.

Tertiary amine group nitrogen content: The primary and secondary amine groups are converted into amide groups with acetic acid anhydride. The tertiary amine group alkalinity is determined by potentiometric titration with hydrobromic or perchloric acid in glacial acetic acid/acetic anhydride.

Primary amine group nitrogen content: The primary amine groups are reacted with a measured excess of 2,4-pentanedione (acetylacetone) in *N,N*-dimethylformamide to form imines. The excess acetylacetone is determined by potentiometric titration with potassium hydroxide (the reaction products of acetylacetone and primary amine groups are neutral under these conditions).

Secondary amine group nitrogen content: The secondary amine group nitrogen content is determined by calculating the difference between the total amine group nitrogen content and the sum of the primary and tertiary amine group nitrogen contents.

b) Aromatic amines

Total amine group nitrogen content: See aliphatic amines.

Tertiary amine group nitrogen content: See aliphatic amines.

Sum of the secondary and tertiary amine group nitrogen contents: The primary amine groups are converted to Schiff bases by salicylaldehyde in glacial acetic acid. With strongly basic amines, the secondary and tertiary amine group alkalinity is determined by potentiometric titration with hydrochloric acid in glacial acetic acid.

Primary amine group nitrogen content: The primary amine group nitrogen content is determined by calculating the difference between the total amine group nitrogen content and the sum of the secondary and tertiary amine group nitrogen contents.

Secondary amine group nitrogen content: The secondary amine group nitrogen content is determined by calculating the difference between the sum of the secondary and tertiary amine group nitrogen contents and the tertiary amine group nitrogen content.

3.2.7 Determination of free acid in acid anhydride hardeners and accelerators

The test shall be performed in accordance with ISO 7327.

Principle of ISO 7327

The small amount of free acid in a test portion of acid anhydride hardener or accelerator is reacted with rhodamine 6G to colour the test solution pink. The absorbance of the coloured solution is measured at a wavelength of about 510 nm using a spectrometer, and the free acid content is determined from a calibration curve.

3.2.8 Non-volatile-matter content

The test shall be performed in accordance with ISO 3251.

Principle of ISO 3251

Determination of the percentage residue by mass obtained by evaporation under the specific conditions of the test.