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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Plastics — Liquid epoxide resins — Determination of tendency to crystallize

Plastiques — Résines époxydes liquides — Détermination de tendance à la cristallisation

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ISO 4895:1987

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4895 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Plastics — Liquid epoxide resins — Determination of tendency to crystallize

1 Scope and field of application

This International Standard specifies a method for determining the tendency of liquid epoxide resins of the bisphenol A type to crystallize.

NOTE — The tendency of a liquid epoxide resin to crystallize depends on the ambient temperature, its purity (homogeneity and contaminants), its moisture content and other factors.

2 Reference

ISO 3001, *Plastics — Epoxide compounds — Determination of epoxide equivalent*.

3 Principle

Dilution of a sample of the epoxide resin with *n*-butyl glycidyl ether (NBGE) and seeding with crystals of the diglycidyl ether of 2,2-diphenolpropane (DGE chemically pure). Cooling to 10 ± 1 °C and examination at regular intervals until crystallization occurs.

4 Reagents

4.1 Diglycidyl ether of 2,2-diphenolpropane (DGE), pure and finely ground crystals (passing through a 250 µm aperture screen).

NOTES

1 The pure DGE can be obtained by precipitation of a commercial epoxide resin with a high content of 4,4' isomer of DGE. This may be prepared as follows :

- Place 250 g of the epoxide resin in a 1 000 ml beaker and add 200 to 250 ml of a solvent mixture which consists of 1 part by volume of butanone (methyl ethyl ketone) and 4 parts by volume of methanol. Stir the mixture and allow the crystalline phase to separate.
- Centrifuge the crystalline phase and remove the remaining mother liquor by decantation.
- Wash the crystals four times in a 1 000 ml beaker using 200 to 250 ml volume of methanol and stir for 1 min. Allow the mixture to stand until the crystals settle and then separate the crystals by decantation.
- Add to the crystals 100 ml of methanol, stir for 1 min and filter under vacuum on a Buchner funnel using a filter paper.

- Dry the crystals under a vacuum of 1,33 kPa at 23 °C for 4 to 6 h.
- Check the purity of the DGE by measuring :
 - the refractive index, (n_D at 65 °C): $1,554 \pm 0,004$;
 - the epoxide equivalent, determined in accordance with ISO 3001: 170 ± 2 ;
 - the melting point: 44 ± 1 °C.

If the material does not meet these requirements, it shall be washed again and re-dried as described in c), d) and e) and then checked for purity again as described above.

2 It is preferable to use the diglycidyl ether to start the crystallization prepared as indicated in 4.1.

However, any other method of obtaining DGEBA of equivalent crystallinity and purity can be used.

4.2 *n*-Butyl glycidyl ether (NBGE), of technical quality.

WARNING — NBGE is toxic. Avoid inhaling vapours and all contact with skin and eyes. Work under a hood or in a ventilated area. Threshold of toxicity : 50 mg/m³.

5 Apparatus

5.1 Refrigerator or cooling chamber, maintained at 10 ± 1 °C.

5.2 Test tubes, approximately 18 mm in diameter and 180 mm long, with ground glass necks and ground glass stoppers.

6 Procedure

6.1 Mix the liquid epoxy resin to be tested with *n*-butyl glycidyl ether (4.2) according to the following mass ratio :

- 90 % (*m/m*) liquid epoxide resin to be tested;
- 10 % (*m/m*) *n*-butyl glycidyl ether.

NOTE — A liquid epoxide resin already diluted with low viscous mono- or polydiglycidyl ether should not further be diluted with the NBGE.

6.2 Half fill a test tube (5.2) with the mixture. Close the test tube with a glass stopper and place it in an oven for 16 h at 60 °C. After this period, remove the test tube with the mixture and cool it to 23 ± 1 °C.

6.3 Add 10 mg of pure DGE crystals (4.1) to the mixture and stir with a glass rod. Close the test tube with the glass stopper and place it in the refrigerator (5.1) at 10 ± 1 °C.

6.4 Examine the appearance of the mixture in the test tube every day. Record the elapsed time at which the following are observed:

- a) the first slight opalescence;
- b) greater opalescence than in a);
- c) milkiness, i.e. complete opacity;
- d) about 50 % (V/V) solid phase in the mixture;
- e) a fully crystallized mass.

7 Expression of results

7.1 The tendency of a liquid epoxide resin to crystallize is expressed as the number of days necessary to reach the appearance conditions a) to e) specified in 6.4.

7.2 The resin may be reported as having a reduced tendency to crystallize if it complies with one of the following conditions:

- 1) no cloudiness is detected in the test mixture after 72 h storage at 10 ± 1 °C;
- 2) the first slight cloudiness is detected after 48 h storage at 10 ± 1 °C, but the test mixture is not completely opaque or milky after 5 days storage.
- 3) limit the test to 1 week.

8 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) complete identification of the product tested;
- c) results of the test;
- d) any deviation, by agreement or otherwise, from the procedure specified.

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