SLOVENSKI STANDARD
SIST EN ISO 3001:2000

01-maj-2000

Polimerni materiali - Epoksidne spojine - Določanje epoksidnega ekvivalenta (ISO 3001:1999)

Plastics - Epoxy compounds - Determination of epoxy equivalent (ISO 3001:1999)

Kunststoffe - Epoxid-Verbindungen - Bestimmung des Epoxid-Äquivalents (ISO 3001:1999)

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Plastiques - Compositions époxydiques - Détermination de l'équivalent époxy (ISO 3001:1999)

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Ta slovenski standard je istoveten z: **EN ISO 3001:1999**

ICS:

83.080.10 Duromeri Thermosetting materials

SIST EN ISO 3001:2000 **en**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 3001

February 1999

ICS 83.080.00

English version

Plastics - Epoxy compounds - Determination of epoxy equivalent (ISO 3001:1999)

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This European Standard was approved by CEN on 25 January 1999.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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Ref. No. EN ISO 3001:1999 E

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Foreword

The text of the International Standard ISO 3001:1999 has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 1999, and conflicting national standards shall be withdrawn at the latest by August 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 3001:1999 was approved by CEN as a European Standard without any modification.

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INTERNATIONAL STANDARD

ISO 3001

Fourth edition
1999-02-15

Plastics — Epoxy compounds — Determination of epoxy equivalent

Plastiques — Compositions époxydiques — Détermination de l'équivalent époxy

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Reference number
ISO 3001:1999(E)

SIST EN ISO 3001:2000

Foreword

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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.

International Standard ISO 3001 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This fourth edition cancels and replaces the third edition (ISO 3001:1997), which has been technically revised.

Annex A forms an integral part of this International Standard.

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Printed in Switzerland

SIST EN ISO 3001:2000

Plastics — Epoxy compounds — Determination of epoxy equivalent

1 Scope

This International Standard specifies a method for the determination of the epoxy equivalent and is applicable to all epoxy compounds. In the case of epoxyamines, it is necessary to apply the modification specified in annex A.

2 Definitions

For the purposes of this International Standard, the following definitions apply.

2.1
epoxy equivalent
the mass of resin, in grams, which contains one mole of epoxy groups

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2.2
epoxy index
the number of moles of epoxy groups contained in 1 kg of resin

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3 Principle

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

4 Reagents

During the analysis, use only reagents of recognized analytical grade.

4.1 Glacial acetic acid.

4.2 Acetic anhydride, purity > 96 %.

4.3 Chloroform.

4.4 Potassium hydrogen phthalate.

4.5 Crystal violet, indicator solution.

Dissolve 100 mg of crystal violet in 100 ml of glacial acetic acid (4.1).

4.6 Perchloric acid, 0,1 mol/l standard volumetric solution.

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4.6.1 Preparation

Add 8,5 ml of a 70 % (*m*/*m*) aqueous solution of perchloric acid to a solution consisting of a mixture of 500 ml of glacial acetic acid (4.1) and 30 ml of acetic anhydride (4.2). Make up to 1 000 ml with glacial acetic acid and mix thoroughly.

4.6.2 Standardization

WARNING — The use of safety goggles and a safety screen is recommended.

Standardize this solution by titrating it against 200 mg of potassium hydrogen phthalate (4.4) — previously dried, if necessary, for 2 h at 120 °C — dissolved in 20 ml of glacial acetic acid (4.1) and 10 ml of chloroform (4.3), using crystal violet indicator solution (4.5) or a potentiometric titration apparatus (5.1).

Carry out the end-point determination using 4 to 6 drops of crystal violet solution, titrating until a stable green colour is obtained, or carry out this procedure using a potentiometric titration apparatus. If a potentiometric method is used to determine the epoxy equivalent, it is necessary to use the same method for the standardization of the perchloric acid. Note the temperature t_s of the standard volumetric solution.

4.6.3 Calculation of the concentration

Calculate the concentration c of the perchloric acid solution (4.6.1), in moles per litre, by the following equation, rounding to four places of decimals:

$$c = \frac{m}{(V_1 - V_0) \times 0{,}204\,22}$$

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where

m is the mass, in grams, of potassium hydrogen phthalate used;

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V_0 is the volume, in millilitres, of perchloric acid solution (4.6.1) used in the blank test;

V_1 is the volume, in millilitres, of perchloric acid solution (4.6.1) used in the determination.

4.7 Tetraethylammonium bromide, reagent solution.

Dissolve 100 g of tetraethylammonium bromide in 400 ml of glacial acetic acid (4.1). Add a few drops of crystal violet solution (4.5). If it changes colour, bring it back to the original (blue-green) colour with standard volumetric perchloric acid solution (4.6).

For some epoxy compounds of low reactivity, the use of tetrabutylammonium iodide is advised, either as the solid or as a 10 % solution in chloroform. In this case, light shall be excluded as much as possible. Solutions of tetrabutylammonium iodide in chloroform are unstable and shall be freshly prepared for each titration.

5 Apparatus

5.1 Potentiometric titration apparatus, equipped with a silver electrode and a silver chloride or mercury sulfate electrode.

5.2 Balance, accurate to within 0,1 mg.

5.3 Conical flask, capacity 100 ml or 200 ml, with ground-glass neck and ground-glass stopper.

5.4 Microburette, with closed reservoir, or **calibrated burette**, capacity 10 ml.

5.5 Glass apparatus, with ground joints, vents being protected from moisture by calcium chloride tubes.

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5.6 Magnetic stirrer, with polytetrafluoroethylene-coated bar.

5.7 Thermometer, calibrated to permit temperature measurements to within ± 0,1 °C.

5.8 Pipette, capacity 10 ml.

5.9 Volumetric flask, capacity 1 000 ml.

5.10 Measuring cylinders, capacity 50 ml and 500 ml.

6 Procedure

6.1 Weigh into the flask, to the nearest 0,1 mg, a test portion containing 0,6 mmol to 0,9 mmol of epoxy groups (this corresponds to a mass of between 0,6 × EE mg and 0,9 × EE mg, where EE is the estimated epoxy equivalent).

6.2 Add 10 ml of chloroform (4.3), then dissolve the test portion using the magnetic stirrer (5.6) and, if necessary, by heating slightly.

Cool to room temperature, add 20 ml of glacial acetic acid (4.1) and then, with the pipette (5.8), 10 ml of tetraethylammonium bromide solution (4.7).

In the case of high-molecular-mass epoxy resins, increase the chloroform volume to 30 ml.

6.3 If using the indicator method, add 4 to 6 drops of crystal violet solution (4.5) and titrate the solution on the magnetic stirrer with perchloric acid solution (4.6).

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Carry out the titration until a stable green colour is obtained.

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6.4 If using the potentiometric method, place the electrodes in the test solution. Adjust the speed of the magnetic stirrer to give vigourous stirring without splattering. Titrate the solution with perchloric acid solution (4.6).

6.5 Note the temperature t of the perchloric acid solution in order to be able to allow for expansion of the solution with increasing temperature.

6.6 Carry out a blank test at the same time as the determination, following the same procedure and using the same reagents but omitting the test portion.

7 Expression of results

Calculate the epoxy equivalent EE, in grams per mole, by the following equation, rounding to three places of decimals:

$$\mathrm{EE} = \frac{1\,000\, m}{(V_1 - V_0)\left(1 - \frac{t - t_s}{1\,000}\right) c}$$

where

m is the mass, in grams, of the test portion;

V_0 is the volume, in millilitres, of perchloric acid solution (4.6) used in the blank test;

V_1 is the volume, in millilitres, of perchloric acid solution (4.6) used in the determination;

t is the temperature, in degrees Celsius, of the perchloric acid solution (4.6) at the time of the determination and blank test;