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Polimerni materiali - Epoksidne spojine - Določanje epoksidnega ekvivalenta

Plastics -- Epoxide compounds -- Determination of epoxide equivalent

Plastiques -- Compositions époxydiques -- Détermination de l'équivalent époxyde

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INTERNATIONAL STANDARD ISO 3001

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Plastics — Epoxide compounds — Determination of epoxide equivalent

Plastiques — Compositions époxydiques — Détermination de l'équivalent époxyde

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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.

International Standard ISO 3001 was developed by Technical Committee ISO/TC 61, *Plastics.* The first edition (ISO 3001-1975) had been approved by the member bodies of the following countries :

Australia	Germany	Romania
Austria	Hungary	South Africa, Rep. of
Belgium	Ireland	Spain
Brazil	Israel	Sweden
Bulgaria	Italy	Switzerland
Canada	Japan	Thailand
Czechoslovakia	New Zealand	Turkey
Egypt, Arab Rep. of	Poland	U.S.A.
France	Portugal	U.S.S.R.

The member body of the following country had expressed disapproval of the document on technical grounds :

United Kingdom

This second edition, which supersedes ISO 3001-1975, incorporates draft Addendum 1, which features at present as an annex, and which was circulated to the member bodies in January 1977. This draft addendum has been approved by the member bodies of the following countries :

Austria	India	Romania
Belgium	Iran	South Africa, Rep. of
Brazil	Israel	Spain
Bulgaria	Italy	Switzerland
Canada	Korea, Rep. of	Turkey
Czechoslovakia	Mexico	U.S.A.
France	Poland	Yugoslavia
Germany	Portugal	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Netherlands
United Kingdom

Printed in Switzerland

Plastics — Epoxide compounds — Determination of epoxide equivalent

1 SCOPE AND FIELD OF APPLICATION

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

2 DEFINITION

epoxide equivalent: The mass of substance, in grams, which contains one mole of epoxide group.

3 PRINCIPLE

Reaction of the epoxide groups with nascent hydrogen bromide produced by the action of a 0,1 N standard volumetric perchloric acid solution on tetraethylammonium bromide. Determination of the end-point either using crystal violet as indicator or, for dark-coloured products, by a potentiometric method.

4 REAGENTS

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

4.1 Acetic acid.

4.2 Acetic anhydride.

4.3 Chloroform.

4.4 Potassium hydrogen phthalate.

4.5 Crystal violet, indicator solution.

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

4.6 Perchloric acid, 0,1 N standard volumetric solution.

4.6.1 *Preparation*

To 8,5 ml of a 70 % (*m/m*) aqueous solution of perchloric acid, add 300 ml of the acetic acid (4.1) followed by 20 ml of the acetic anhydride (4.2). Dilute to 1 litre with the acetic acid (4.1) and mix thoroughly.

4.6.2 *Standardization*

Standardize this solution by titrating it against 200,0 mg of potassium hydrogen phthalate (4.4) dissolved in 50 ml of the acetic acid (4.1), using the crystal violet indicator solution (4.5).

(If necessary, dry the potassium hydrogen phthalate for 2 h at 120 °C before use.) Carry out the end-point determination using 4 to 6 drops of the crystal violet indicator solution (see notes), titrating until a stable green colour is obtained. Note the temperature t_s of the standard volumetric solution.

4.6.3 *Calculation of the concentration*

The normality T of the perchloric acid solution is given by the formula

$$T = \frac{m}{V \times 0{,}204\ 22}$$

where

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

V is the volume, in millilitres, of the perchloric acid solution (4.6.1) used in the titration.

NOTES

1 If a potentiometric method is used for the determination of epoxide equivalent, it is necessary to use the same method for the standardization of the perchloric acid.

2 The use of safety goggles and a safety screen is recommended.

4.7 Tetraethylammonium bromide reagent solution.

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

NOTE — For some epoxide 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 should be excluded as much as possible. Solutions of tetrabutylammonium iodide in chloroform are unstable and must be freshly prepared for each titration.

5 APPARATUS

5.1 Balance, accurate to within 0,1 mg.

5.2 Conical flask, 100 or 200 ml, with ground glass neck and ground glass stopper.

5.3 Micro-burette with closed reservoir or **calibrated burette,** capacity 10 ml.

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

5.5 **Magnetic stirrer** with polytetrafluorethylene-coated bar.

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

5.7 **Pipette,** capacity 10 ml.

6 PROCEDURE

Weigh into the flask, to the nearest 0,2 mg, a test portion containing from 0,6 to 0,9 millimole of epoxide. (This corresponds to a mass of between 0,6 and 0,9 × EE mg, where EE is the estimated epoxide equivalent.)

Add 10 ml of the chloroform (4.3), then dissolve the test portion by stirring and, if necessary, heating slightly.

Cool to room temperature, add 20 ml of the acetic acid (4.1) and then, with the pipette (5.7), 10 ml of the reagent solution (4.7) and 4 to 6 drops of the crystal violet indicator solution (4.5).

Titrate immediately, while stirring magnetically, with the perchloric acid solution (4.6) until a stable green colour is obtained.

Note the temperature t of the perchloric acid solution.

At the same time carry out a blank test, omitting the test portion.

7 EXPRESSION OF RESULTS

The epoxide equivalent EE is given, in grams per mole, by the formula :

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

where

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

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

V_1 is the volume, in millilitres, of the 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;

t_s is the temperature, in degrees Celsius, of the perchloric acid solution (4.6) at the time of standardization;

T is the normality of the solution (4.6) (usually 0,1 N) at the time of standardization.

NOTE — The use of the correction factor is necessary because of the significant coefficient of expansion of the perchloric acid solution ($1{,}07 \times 10^{-3}\,°C^{-1}$), which corresponds to a volume variation of 0,1 % per degree Celsius. Use of this factor can be avoided by working in a temperature-controlled room.

The result is sometimes expressed as epoxide index, expressed in moles of epoxide per kilogram, calculated as follows :

$$\text{Epoxide index} = \frac{1\,000}{EE}$$

8 TEST REPORT

The test report shall include the following particulars :

a) the identification of the sample;

b) the epoxide equivalent;

c) the reagent used if it is not tetraethylammonium bromide;

d) any other factor likely to have affected the result.

ANNEX

MODIFICATION APPLICABLE TO EPOXYAMINES

A.1 SCOPE AND FIELD OF APPLICATION

When determinations are carried out on nitrogen-containing epoxide resins according to the method described in the body of this International Standard, the values determined for the epoxide equivalent are too low. This is due to a reaction between perchloric acid and amino nitrogen, which leads to the formation of a salt.

If account is taken of the perchloric acid involved in the formation of the salt, then the standard can also be used for determining the epoxide equivalent of epoxyamines.

A.2 PRINCIPLE

Titration of the amino nitrogen of the epoxyamine with 0,1 N standard volumetric perchloric acid solution. The second blank value thus obtained is used as a correction in the calculation of the epoxide equivalent according to clause A.4 of this annex.

A.3 PROCEDURE

Determine the second blank value according to clause 6 of this International Standard, but without addition of tetraethylammonium bromide solution (4.7).

A.4 EXPRESSION OF RESULTS

The epoxide equivalent, EE, of epoxyamines is given, in grams per mole, by the formula

$$EE = \frac{1\,000 \times m}{\left(V_1 - V_0 - V_2 \frac{m}{m_1}\right)\left(1 - \frac{t - t_s}{1\,000}\right) \times T}$$

where

m_1 is the mass, in grams, of the test portion used in the second blank test;

V_2 is the volume, in millilitres, of the perchloric acid solution (4.6) used in the second blank test;

the other symbols have the same meaning as in clause 7 of this International Standard.