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Plastics — Epoxy compounds — Determination of epoxy equivalent

Plastiques — Compositions époxydiques — Détermination de l'équivalent époxyde

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Foreword

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

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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 Definition

For the purposes of this International Standard, the following definition applies:

2.1 epoxy equivalent: The mass of substance, in grams, which contains one mole of epoxy groups.

3 Principle

The epoxy groups in an epoxy compound are reacted with nascent hydrogen bromide produced by the action of a 0,1 mol/l 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, at least 96 % purity.

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.

WARNING — Perchloric acid can be explosive. The use of safety goggles and a safety screen is recommended.

4.6.1 Preparation

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

4.6.2 Standardization

Standardize this solution by titrating it, using 4 to 6 drops of crystal violet solution (4.5) as indicator, against a solution prepared by dissolving 200,0 mg of potassium hydrogen phthalate (4.4) in 50 ml of glacial acetic acid (4.1) (if necessary, dry the potassium hydrogen phthalate for 2 h at 120 °C before use). Titrate until a stable green colour is obtained. Note the temperature t_s of the standard volumetric perchloric acid solution.

4.6.3 Calculation of the concentration

The concentration c of the perchloric acid solution is given by the formula:

$$c = \frac{m}{V \times 0{,}20422}$$

where

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

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

4.7 Tetraethylammonium bromide solution

Dissolve 100 g of tetraethylammonium bromide in 400 ml of glacial acetic acid (4.1). Add a few drops of crystal violet indicator solution (4.5); if it changes colour, bring it back to the original colour with 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 Balance, accurate to within 0,1 mg.

5.2 Conical flask, capacity 100 ml or 200 ml, with ground-glass stopper.

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

5.4 Magnetic stirrer, with PTFE-coated bar.

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

5.6 Pipette, capacity 10 ml.

6 Procedure

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

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

Cool to room temperature, add 20 ml of glacial acetic acid (4.1) and then, with the pipette (5.6), 10 ml of tetraethylammonium bromide (or iodide) solution (4.7) and 4 to 6 drops of crystal violet indicator solution (4.5).

Titrate immediately, while stirring magnetically, with 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 epoxy equivalent EE is given, in grams per mole, by the equation

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

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;

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

c is the concentration of the perchloric acid solution (4.6) (usually 0,1 mol/l) at the time of standardization.

NOTES

1 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.

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

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

8 Precision

The precision of this method is not known because interlaboratory data are not available. Interlaboratory data are being obtained, and a precision statement will be added at the next revision.

9 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the sample;

c) a statement to the effect that tetraethylammonium iodide was used as the reagent, if this was used instead of tetraethylammonium bromide;

d) the result of the determination, expressed as given in clause 7;

e) the date of the test;

f) details of any operation not specified in this International Standard, as well as an incident which may have affected the results.

Annex A
(normative)
Modification applicable to epoxyamines

A.1 General

When determinations are carried out on nitrogen-containing epoxy resins using the method described in the body of this International Standard, the values determined for the epoxy equivalent are too low. This is due to a reaction between the perchloric acid and the 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 method can also be used to determine the epoxy equivalent of epoxyamines.

A.2 Principle

The amino nitrogen in the epoxyamine is determined by titrating against 0,1 mol/l standard volumetric perchloric acid solution. The second blank value thus obtained is used as a correction in the calculation of the epoxy equivalent, as described in clause A.4 of this annex.

A.3 Procedure

Determine the second blank value by following the procedure specified in clause 6 of this International Standard, but without adding tetraethylammonium bromide (or iodide) solution (4.7).

A.4 Expression of results

The epoxy equivalent EE of the epoxyamine is given, in grams per mole, by the equation

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

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 perchloric acid solution (4.6) used in the second blank test.

The other symbols are as defined in clause 7.

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