INTERNATIONAL STANDARD 3001

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Epoxy compounds — Determination of epoxy equivalent

Composés époxydiques — Détermination de l'équivalent époxyde

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

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Epoxy compounds — Determination of epoxy equivalent

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the epoxy equivalent and is applicable to all epoxy compounds with the exception of epoxyamines.

2 DEFINITION

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

3 PRINCIPLE

Reaction of the epoxy groups with nascent hydrogen bromide produced by the action of a standard 0,1 N solution of perchloric acid 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 acetic acid (4.1).

4.6 Perchloric acid, 0,1 N standard solution.

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

Standardize this solution by titrating it against 200,0 mg of potassium hydrogen phthalate (4.4) dissolved in 50 ml of 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 solution.

The normality T of the standard 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;

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

NOTES

1 If a potentiometric method is used for the determination of epoxy 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 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 perchloric acid standard solution (4.6).

NOTE — 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 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.

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