BRITISH STANDARD

BS EN
1877-1:2000

Products and systems for the protection and repair of concrete structures — Test methods — Reactive functions related to epoxy resins —

Part 1: Determination of epoxy equivalent

The European Standard EN 1877-1:2000 has the status of a British Standard

ICS 91.080.40

National foreword

This British Standard is the official English language version of EN 1877-1:2000.

The UK participation in its preparation was entrusted by Technical Committee B/517, Concrete, to Subcommittee B/517/8, Protection and repair of concrete, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 8, an inside back cover and a back cover.

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English version

Products and systems for the protection and repair of concrete structures - Test methods - Reactive functions related to epoxy resins - Part 1: Determination of epoxy equivalent

Produits et systèmes pour la protection et la réparation des structures en béton - Méthodes d'essai - Fonctions réactives des résines époxydes - Partie 1: Détermination de l'équivalent époxyde

Produkte und Systeme für den Schutz und die Instandsetzung von Betontragwerken - Prüfverfahren - Reaktive, funktionelle Gruppen von Epoxidharzen - Teil 1: Bestimmung des Epoxid-Äquivalents

This European Standard was approved by CEN on 7 July 2000.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 1877-1:2000 E

Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 104 "Concrete (performance, production, placing and compliance criteria)", the secretariat of which is held by DIN.

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 December 2002, and conflicting national standards shall be withdrawn at the latest by December 2002.

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.

This European Standard describes several type of test methods. Specifications for the products and systems for the protection and repair of concrete structures will be subject of separate standards.

This European Standard is based on the ISO 3001 : 1997 standard.

1 Scope

This European Standard specifies a method for the determination of the epoxy equivalent and is applicable to all epoxy compounds, for products and systems for the protection and repair of concrete structure. This European standard does not apply to the measurement of epoxide equivalent of nitrogen-containing resins.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 21512 *Paints and varnishes – Sampling of products in liquid or paste form* (ISO 1512 : 1991)

3 Terms and definitions

For the purposes of this standard the following terms and definitions apply:

3.1
epoxide equivalent

The mass of resin, in grams, which contains one mole of epoxide group.

3.2
expoxide number

$$\frac{\text{Molar mass of epoxy - group} \times 100}{\text{epoxide equivalent}}$$

Content of epoxy groups in (%) in 100 g epoxy resin

3.3
epoxide index

$$\frac{1000}{\text{epoxide equivalent}}$$

Moles of epoxide per kilogram epoxy resin.

4 Principle

Reaction of the epoxide groups with nascent hydrogen bromide produced by the action of 0,1 mol/l standard volumetric perchloric acid solution on tetraethylammonium bromide. Determination of the endpoint either using crystal violet as indicator or by a potentiometric method.

5 Sampling

A representative and homogeneous sample of the material to be tested shall be taken according to EN 21512. Eliminate mineral filler and pigments with a suitable organic solvent before the determination of the epoxide equivalent. It should be checked, that any solvent has evaporated.

6 Apparatus

- Balance, accurate to within 0,1 mg.
- Calibrated burette, capacity 50 ml.
- Pipette, capacity 10 ml
- Conical flask, 100 ml or 200 ml, with ground glass neck and ground glass stopper.
- Instrument and electrodes for potentiometric titration.

In addition, when prepared the 0,1 mol/l perchloric acid in acetic acid.

- Magnetic stirrer with polytetrafluoroethylene-coated bar.
- Calibrated thermometer to permit temperature measurements within an accuracy of ± 0,1 K.
- Volumetric flask, 1 000 ml.
- Graduated cylinders, 50 ml and 500 ml.

7 Reagents

For the analysis, only reagents of analytical grade shall be used.

- Perchloric acid, 0,1 mol/l in acetic acid (Standard volumetric solution).
- Tetraethylammonium bromide.
- Tetrabuthylammonium iodide.
- Acetic acid 99 % to 100 %.
- Chloroform, methylenchloride, acetone.
- Crystal violet, 0,1 % in solution in acetic acid.
- Potassium hydrogen phthalate.

In addition, when prepared the 0,1 mol/l perchloric acid in acetic acid.

- Acetic acid 99 % to 100 %.
- Acetic anhydride, purity > 96 %.
- Phenolphthalein, 0,1 % solution.
- Perchloric acid, 70 %.
- Sodium hydroxide solution 0,1 mol/l.
- Distilled water.

8 Preparation of the perchloric acid (0,1 mol/l) in acetic acid

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

Determination of the true percentage of the approximately 70 % (m/m) perchloric acid. Weigh approximately 0,3 g of the perchloric acid (E_1) in a 200 ml conical flask, add 50 ml distilled water and mix thoroughly. Titrate it against 0,1 mol/l sodium hydroxide solution (V_1) using the phenolphthalein indicator solution. The content of perchloric acid is given by the equation :

$$P = \frac{E_1 \cdot 1000}{V_1 \cdot F_{NaOH}}$$

where

P = content of the perchloric acid ;
E_1 = is the mass in (g), of the approximate 70 % perchloric acid ;
V_1 = is the volume, in (ml), of the 0,1 mol/l sodium hydroxide solution ;
F_{naOH} = is the factor of the 0,1 mol/l sodium hydroxide solution.

Weigh the calculated mass of the 70 % aqueous solution of the perchloric acid to an accuracy of 0,1 mg into a 1 000 ml volumetric flask. Add 300 ml of the acetic acid followed by 50 ml of the acetic anhydride under cooling. The temperature of the solution shall be kept at 20 °C. Dilute to 1000 ml with the acetic acid and mix thoroughly. The solution should not show any discoloration to yellow for a period of 12 h. Then carry out the standardization.

9 Standardization

a) Standardize this solution by titrating it in a 200 ml conical flask to 300 mg to 500 mg potassium hydrogen phthalate dissolved in 15 ml acetic acid, using the crystal violet indicator solution. If necessary, dry the potassium hydrogen phthalate for 24 hours at 120 °C before use. Carry out the end-point determination using 4 to 6 drops of the crystal violet indicator solution, titrating until a stable green colour is obtained. Record the temperature t_s of the standard volumetric solution. Carry out a double determination.

NOTE: If the potentiometric method is used for the determination of epoxide equivalent, it is recommended to use the same method for the standardization of the perchloric acid.

b) Alternative: Use the manufacturers concentration of the solution.

10 Calculation of the concentration

The concentration c of the perchloric acid solution in moles per litre is given by the equation :

$$c = \frac{E_2}{V_2 \cdot 0{,}2042}$$

where

E_2 : is the mass, in (g), of potassium hydrogen phthalate used ;
V_2 : is the volume, in (ml), of the perchloric acid solution used in the titration.

11 Preparation of the Tetraethylammonium bromide reagent solution

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

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 should be freshly prepared for each titration.

12 Procedure

- Weigh into the flask, to the nearest 0,1 mg, a test portion containing from 0,6 mmol to 0,9 mmol of epoxy groups (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 solvent, then dissolve the test portion using the magnetic stirrer and, if necessary, by heating slightly.
- Cool to room temperature, add 20 ml of acetic acid and then, with the pipette, 10 ml of tetraethylammonium bromide solution.
- If using the indicator method, add 4 to 6 drops of crystal violet solution and titrate the solution on the magnetic stirrer with the perchloric acid solution.
- Carry out the titration until a stable green colour is obtained.
- If using the potentiometric method, place the electrodes in the test solution and titrate the solution on the magnetic stirrer with the perchloric acid solution.
- Note the temperature t of the perchloric acid solution in order to be able to allow for expansion of the solution with increasing temperature.
- Carry out a blank test an the same time as the determination, following the same procedure and using the same reagents but omitting the test portion.

13 Calculation

The epoxide equivalent *EE* is given by the equation :

$$EE = \frac{1000 \cdot m}{(V_1 - V_0) \cdot \left(1 - \frac{t - t_s}{1000}\right) \cdot c}$$

where

m is the mass, in (g), of the test portion ;
V_0 is the volume, in (ml), of the perchloric acid solution used in the blank test ;
V_1 is the volume, in (ml), of the perchloric acid solution used in the determination ;
t is the temperature, in (°C), of the perchloric acid solution at the time of the determination and blank test ;
t_S is the temperature, in (°C), of the perchloric acid solution at the time of standardization ;
c is the concentration of the perchloric acid solution (usually 0,1 mol/l) at the time of standardization.

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

14 Precision

Repeatability limit : 0,5 %

Reproducibility limit : 1 %.

15 Test report

The test report shall include the following information :

a) a reference to this European Standard ;

b) all details necessary to identify the product tested ;

c) the epoxide equivalent and the way in which it is expressed;

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

e) any other factor likely to have affected the result ;

f) any deviations, by agreement or otherwise, from the procedure described ;

g) the date of test.

Bibliography

ISO 3001 : 1997 *Plastics – Epoxy compounds – Determination of epoxy equivalent.*

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