SLOVENSKI STANDARD
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Polimerni materiali - Epoksidne smole in sorodni materiali - Določanje lahko umiljivega klora

Plastics -- Epoxy resins and related materials -- Determination of easily saponifiable chlorine

Plastiques -- Résines époxydes et matières apparentées -- Dosage du chlore facilement saponifiable

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INTERNATIONAL STANDARD ISO 4583

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Plastics — Epoxide resins and related materials — Determination of easily saponifiable chlorine

Plastiques — Résines d'époxydes et matières apparentées — Dosage du chlore facilement saponifiable

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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 4583 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in December 1976.

It has been approved by the member bodies of the following countries :

Australia	India	Poland
Austria	Iran	Portugal
Belgium	Israel	Romania
Brazil	Italy	Switzerland
Bulgaria	Japan	Turkey
Canada	Korea, Rep. of	United Kingdom
Czechoslovakia	Mexico	U.S.A.
Finland	Netherlands	U.S.S.R.
France	New Zealand	Yugoslavia

No member body expressed disapproval of the document.

Printed in Switzerland

Plastics — Epoxide resins and related materials — Determination of easily saponifiable chlorine

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of easily saponifiable chlorine in epoxide resins (see 6.1), glycidyl esters and 1-chloro-2,3-epoxypropane (see 6.2). The values obtained are indicative of the concentration of easily saponifiable chlorine of chlorohydrin groups in the compounds.

NOTE — For quality control purposes, a shorter reflux time of 30 min is permissible if it can be shown to give similar results. This should be recorded in the test report.

2 REFERENCE

ISO 4573, *Plastics — Epoxide resins and glycidyl esters — Determination of inorganic chlorine — Direct potentiometric method.*

3 PRINCIPLE

Reaction of a test portion with a sodium hydroxide solution in 2-butoxyethanol for 2 h at room temperature for epoxide resins, or with a methanolic sodium hydroxide solution at 50 °C for glycidyl esters and epichlorohydrin. Acidification of the mixture and determination of the chloride ion resulting from the saponification by potentiometric titration with a standard volumetric silver nitrate solution. A correction is made for the inorganic chlorine content of the sample determined according to the method specified in ISO 4573.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade (unless otherwise specified) and only distilled water, or water of equivalent purity.

4.1 Acetic acid, glacial, ρ 1,05 g/ml.

4.2 2-Butoxyethanol (ethylene glycol monobutyl ether), technical grade (only needed for epoxide resins).

WARNING — 2-Butoxyethanol is toxic. Avoid inhalation of vapour. Prevent contact with skin and eyes. Work under a fume hood or in a well ventilated area. Threshold limit value is 50 ppm.

Purify this product as follows :

Add 15 g of sodium hydroxide to 1 litre of technical 2-butoxyethanol and reflux the solution for 4 h. Distil the 2-butoxyethanol at a rate of 5 ml/min. collecting the fraction boiling above 165 °C. Discard the last 100 ml fraction. Store the 2-butoxyethanol in a brown bottle under nitrogen in the dark.

4.3 Butanone (Methyl ethyl ketone).

4.4 Sodium hydroxide, 120 g/l solution in 2-butoxyethanol (for epoxide resins). For glycidyl esters and 1-chloro-2,3-epoxypropane, replace the 2-butoxyethanol by methanol.

Dissolve 120 g of sodium hydroxide in 75 ml of water, cool and dilute to 1 litre with the 2-butoxyethanol (4.2).

4.5 Hydrochloric acid, 0,4 % (m/m) solution.

Mix 9 ml of concentrated hydrochloric acid, ρ approximately 1,19 g/ml, with water and dilute to 1 litre.

4.6 Silver nitrate, 0,01 N standard volumetric solution.

4.6.1 *Preparation*

Dissolve 1,70 g of silver nitrate in water and dilute to 1 litre.

4.6.2 *Standardization*

Weigh, to the nearest 0,1 mg, 20 to 25 mg of sodium chloride, previously dried at 120 °C. Transfer to a 250 ml conical flask, and dissolve in 50 ml of water. Titrate with the silver nitrate solution (4.6.1) in accordance with 6.1.4.

4.6.3 *Calculation of the concentration*

The concentration T, expressed as normality, is given by the formula

$$T = \frac{m}{58{,}45\ V}$$

where

m is the mass, in milligrams, of sodium chloride used;

V is the volume, in millilitres, of the silver nitrate solution (4.6.1) used in the titration.

4.6.4 *Storage*

Store the solution in the dark.

5 APPARATUS

Usual laboratory apparatus, and

5.1 Microburette, of capacity 10 ml graduated in 0,02 ml, with a capillary tip which extends approximately 120 mm below the stopcock.

5.2 Magnetic stirrer.

5.3 pH-millivoltmeter, preferably of the compensating type, regular glass electrode and silver electrode.

5.4 Titration beaker, of capacity 400 ml.

6 PROCEDURE

6.1 Epoxide resins

6.1.1 Weigh a test portion containing not more than 1,78 mg of easily saponifiable chlorine in the beaker (5.4). Pipette 25 ml of the 2-butoxyethanol (4.2) into the beaker and dissolve the test portion, using the magnetic stirrer (5.2) and by heating if necessary. Cool the solution to room temperature and pipette 25 ml of the sodium hydroxide solution (4.4) into the beaker. Mix well, cover the beaker, and allow the reaction mixture to stand at room temperature for 2 h.

6.1.2 Pipette 100 ml of the butanone (4.3) and 25 ml of the acetic acid (4.1) into the mixture while stirring. Stir for a few minutes more, until all of the precipitate which is formed during the addition of the acetic acid is dissolved.

6.1.3 Before each titration, wash the glass electrode (see 5.3) with the butanone, rinse with water, soak the electrode for at least 10 min in the hydrochloric acid (4.5), and rinse again with water and butanone. Polish the silver electrode before each titration with fine abrasive cloth (for example, grain size P 120*) until a clean polished surface is obtained, and rinse with water. (See note 1.)

NOTES

1 Observance of these instructions for preparing the electrodes is very important, as otherwise the electrodes will not respond properly during titration. However, in practice when frequent measurements on the same type of product are made, it is only necessary to prepare the electrodes daily.

2 It is essential to carry out the titration as soon as possible after adding acetic acid, otherwise lower values may be obtained.

6.1.4 Place the electrodes and the microburette (5.1) containing the silver nitrate solution (4.6) in position, immersing the tip of the microburette about 10 mm in the liquid. Stir throughout the determination. Add the silver nitrate solution carefully and wait each time until a constant potential has been established.

Record the microburette and meter readings and plot them rapidly as a graph. When the potential change is small for each increment of silver nitrate solution added, add volumes as large as 0,5 ml. When the rate of change of potential becomes greater than 10 mV per 0,5 ml, use 0,1 ml increments. Stop the titration about 0,5 ml after the inflection point.

NOTE — If it is found that less than 1 ml of silver nitrate solution if required for the titration (and thus also for the blank titration), repeat the test with an accurately measured quantity of 1 ml of 0,01 N potassium chloride solution added to the solution (and also to the blank test solution) prior to titration. Titrate immediately after addition of the potassium chloride solution.

6.1.5 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.

6.1.6 Make a determination of "inorganic chlorine" content of the sample in accordance with the method specified in ISO 4573.

6.2 Glycidyl esters and 1-chloro-2,3-epoxypropane

WARNING — 1-chloro-2,3-epoxypropane is highly toxic, its toxicity being cumulative, and is flammable. Avoid inhalation of vapour. Prevent contact with skin and eyes. Work under a fume hood or in a well ventilated area. 1-chloro-2,3-epoxypropane may react explosively with concentrated sulphuric acid, concentrated alkali metal hydroxides and anhydrous metal halides, such as tin(IV) and iron(III) chlorides. Wear safety goggles and surround the apparatus with a safety shield. Threshold limit value is 5 ppm.

6.2.1 Weigh a test portion containing not more than 1,78 mg of easily saponifiable chlorine in the beaker (5.4). Pipette 25 ml of the methanolic sodium hydroxide solution (4.4) into the beaker and dissolve the test portion, using the magnetic stirrer (5.2). Cover the beaker and allow the reaction mixture to stand at 50 °C for 2 h.

6.2.2 Proceed in accordance with 6.1.2 to 6.1.6.

* Designation from EFPA Classification (European Federation of Producers of Abrasives).

7 EXPRESSION OF RESULTS

7.1 The easily saponifiable chlorine content of the sample, expressed as a percentage by mass, is given by the formula

$$\frac{35{,}5\ T\ (V_1 - V_0)}{10\ m_0} - \frac{C}{10\ 000}$$

where

V_0 is the volume, in millilitres, of the silver nitrate solution (4.6) used in the blank test (see 6.1.5);

V_1 is the volume, in millilitres, of the silver nitrate solution (4.6) used in the titration of the test portion (see 6.1.4);

T is the normality of the silver nitrate solution (4.6) calculated in accordance with 4.6.3;

m_0 is the mass, in grams, of the test portion (see 6.1.1 or 6.2.1);

C is the "inorganic chlorine" content, expressed in milligrams per kilogram (parts per million by mass) (see 6.1.6).

7.2 If required, the purity of 1-chloro-2,3-epoxypropane samples, expressed as a percentage by mass, is given by the formula

$$\frac{92{,}5\ T\ (V_1 - V_0)}{10\ m_0} - \frac{92{,}5\ C}{35{,}5 \times 10^4}$$

where the symbols have the same meaning as in 7.1.

8 TEST REPORT

The test report shall include the following particulars :

a) the reference to this International Standard;

b) the complete identification of the material tested;

c) the "inorganic chlorine" content determined in accordance with ISO 4573;

d) the test result and the method of expression used.