INTERNATIONAL STANDARD

ISO 4583

Second edition
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Plastics — Epoxy resins and related materials — Determination of easily saponifiable chlorine

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

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 4583 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 4583:1978), which has been technically revised.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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

1 Scope

This International Standard specifies a method for the determination of easily saponifiable chlorine in epoxy resins (see 7.1) and glycidyl esters (see 7.2).

The values obtained are indicative of the concentration of easily saponifiable chlorine of chlorohydrin groups in the compounds.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 11376:1997, *Plastics — Epoxy resins and glycidyl esters — Determination of inorganic chlorine.*

3 Definition

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

easily saponifiable chlorine: The amount of chlorine saponifiable by this test method. It consists mainly of chlorine which is present as 1,2-chlorohydrin as the result of incomplete dehydrohalogenation.

The easily saponifiable chlorine content is the quantity of easily saponifiable chlorine in a given quantity of epoxy resin and/or glycidyl ester.

4 Principle

A test portion is reacted with sodium hydroxide solution for 2 h:

at room temperature in 2-butoxyethanol for epoxy resins;

at 50 °C in methanol for glycidyl esters.

A shorter saponification time of 30 min is also possible (see 7.1.1 and 7.2.1).

The mixture is acidified and the chloride ion concentration resulting from the saponification determined by potentiometric titration with standard volumetric silver nitrate solution. A correction is made for the inorganic chlorine content of the sample, determined by the method specified in ISO 11376.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water of grade 3 purity as defined in ISO 3696.

5.1 Acetic acid, glacial (ρ = 1,05 g/ml).

5.2 2-Butoxyethanol (ethylene glycol monobutyl ether), stored in a brown bottle in the dark.

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.

5.3 2-Butanone (methyl ethyl ketone).

5.4 Methanol.

WARNING — Methanol is toxic. Avoid inhalation of vapour. Prevent contact with skin and eyes. Work under a fume hood or in a well ventilated area.

5.5 Sodium hydroxide, 120 g/l solution

in 2-butoxyethanol (for epoxy resins);

in methanol for glycidyl esters.

Dissolve 120 g of sodium hydroxide in 75 ml of water plus sufficient 2-butoxyethanol (5.2) or methanol (5.4) to ensure complete dissolution. Cool and make up to 1 litre with the same solvent.

5.6 Sodium chloride.

5.7 Acetone.

5.8 Silver nitrate, 0,01 mol/l standard volumetric solution.

5.8.1 Preparation

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

5.8.2 Standardization

Weigh, to the nearest 0,1 mg, 0,584 g of sodium chloride (5.6), previously dried at 500 °C to 600 °C, and dissolve in 1 litre of water.

Pipette 5 ml of the sodium chloride solution into a 200 ml beaker (6.4). Add 100 ml of acetone (5.7) and 2 ml of acetic acid (5.1). Then titrate the solution potentiometrically with silver nitrate solution (5.8).

Carry out a blank test on the solvent (5.7) in the same way.

5.8.3 Calculation of the concentration

The concentration c of the silver nitrate solution, expressed in mol/l, is given by the equation

$$c = \frac{5 \times m}{58{,}45 \times (V - V_0)}$$

where

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

V is the volume, in ml, of the silver nitrate solution (5.8) used in the titration;

V_0 is the volume, in ml, of the silver nitrate solution (5.8) used in the blank determination.

Round the result to four significant figures.

5.8.4 Storage

Store the solution in the dark.

6 Apparatus

Ordinary laboratory apparatus, together with the following:

6.1 Potentiometric titration apparatus: a suitable potentiometer equipped with a glass/silver chloride electrode, a titration stand and a 10 ml microburette.

6.2 Magnetic stirrer, with a polytetrafluoroethylene-coated bar.

6.3 Analytical balance, accurate to 0,1 mg.

6.4 Beaker, of capacity 200 ml.

6.5 Volumetric flask, of capacity 1 litre.

6.6 Pipettes, of capacity 2 ml, 5 ml and 25 ml.

6.7 Graduated glass cylinder, of capacity 100 ml.

6.8 Water bath, capable of being maintained at 50 °C.

6.9 Electric furnace, capable of being heated to 500 °C to 600 °C.

7 Procedure

7.1 Epoxy resins

7.1.1 Weigh, to the nearest 0,1 mg, a test portion containing not more than 1,78 mg of easily saponifiable chlorine into the beaker (6.4). Pipette 25 ml of 2-butoxyethanol (5.2) into the beaker and dissolve the test portion, using the magnetic stirrer (6.2) and by heating if necessary. Cool the solution to room temperature and pipette 25 ml of sodium hydroxide solution in 2-butoxyethanol (see 5.5) into the beaker. Mix well, cover the beaker, and allow the reaction mixture to stand at room temperature for 2 h.

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

7.1.2 Add 100 ml of 2-butanone (5.3) and 25 ml of acetic acid (5.1) to 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.

7.1.3 Place the electrodes (see 6.1) in the sample solution and titrate the solution potentiometrically with silver nitrate solution (5.8).

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

7.1.4 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.

7.1.5 If it is found that less than 1 ml of silver nitrate solution is 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 mol/l potassium chloride solution added to the solution (and also the blank test solution) prior to the titration. Titrate immediately after addition of the potassium chloride solution.

7.1.6 Determine the inorganic chlorine content of the sample in accordance with the method specified in ISO 11376.

7.2 Glycidyl esters

7.2.1 Weigh, to the nearest 0,1 mg, a test portion containing not more than 1,78 mg of easily saponifiable chlorine into the beaker (6.4). Pipette 25 ml of methanolic sodium hydroxide solution (see 5.5) into the beaker and dissolve the test portion, using the magnetic stirrer (6.2). Cover the beaker and allow the reaction mixture to stand in the water bath (6.8) at 50 °C for 2 h.

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

7.2.2 Proceed in accordance with 7.1.2 to 7.1.6.

8 Expression of results

The easily saponifiable chlorine content W of the sample, expressed in mg/kg (parts per million by mass), is given by the equation

$$W = \frac{35{,}5 \times c \times (V_1 - V_2) \times 1\,000}{m_0} - I$$

where

m_0 is the mass, in g, of the test portion (see 7.1.1 or 7.2.1);

V_1 is the volume, in ml, of silver nitrate solution (5.8) used in the titration of the test portion;

V_2 is the volume, in ml, of silver nitrate solution (5.8) used in the blank test;

c is the concentration, in mol/l, of the silver nitrate solution;

I is the inorganic chlorine content, expressed in mg/kg (parts per million by mass) (see 7.1.6).

Round the result to three significant figures.

9 Precision

The precision of this test method is not known because inter-laboratory data are not available. Inter-laboratory data are being obtained and a precision statement will be added at the following revision.

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for complete identification of the material tested;

c) the inorganic chlorine content, determined in accordance with ISO 11376;

d) the saponification time, if shorter than 2 h;

e) the test result;

f) the date and location of the test;

g) any deviation, by agreement or otherwise, from the procedure specified, and any incident which may have influenced the result.

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Descriptors: plastics, resins, epoxy resins, chemical analysis, determination of content, saponifiable matter, chlorine.

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