INTERNATIONAL STANDARD

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Plastics — Epoxy resins and glycidyl esters — Determination of inorganic chlorine

Plastiques — Résines époxydes et esters glycidiques — Dosage du chlore inorganique

Reference number
ISO 11376:1997(E)

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 11376 was prepared by Technical Committee ISO/TC 61, *Plastics,* Subcommittee SC 12, *Thermosetting materials.*

This International Standard cancels and replaces ISO 4573:1978, of which it constitutes a technical revision.

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Plastics — Epoxy resins and glycidyl esters — Determination of inorganic chlorine

1 Scope

This International Standard specifies a direct potentiometric method for the determination of inorganic chlorine in epoxy resins and glycidyl esters, called also "inorganic chlorine" or "ionic chlorine".

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard 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.*

3 Definition

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

3.1 inorganic chlorine; ionic chlorine (Cl^-): The inorganic chlorine content, expressed in milligrams per kilogram, of an epoxy resin or glycidyl ester.

4 Principle

A test portion is dissolved in a suitable solvent and the inorganic chlorine determined by potentiometric titration with standard volumetric silver nitrate solution.

5 Reagents

During the analysis, use only reagents of recognized analytical grade, and only grade 3 water conforming to ISO 3696.

5.1 Acetone.

In some cases, acetone does not dissolve the resin. In such cases, use butanone (methyl ethyl ketone) or THF (tetrahydrofuran) or another suitable solvent, and record the solvent used in the test report.

5.2 2-Propanol.

5.3 Glacial acetic acid.

5.4 Solution of silver nitrate in 2-propanol, 0,002 mol/l.

5.4.1 Preparation

Dissolve 17,0 g of silver nitrate in water and dilute to 1 l (0,1 mol/l). Take 20 ml of this aqueous 0,1 mol/l silver nitrate solution in a 1 l graduated flask, and dilute to 1 l with 2-propanol.

5.4.2 Standardization

Weigh, to the nearest 0,1 mg, 115 mg to 120 mg of sodium chloride, previously dried at 500 °C to 600 °C, and dissolve in 1 l of water.

Take 5 ml of this solution in a 200 ml beaker, and add 100 ml of acetone (5.1) and 2 ml of glacial acetic acid (5.3). Then titrate potentiometrically with the silver nitrate solution prepared in 5.4.1.

Conduct a blank test in the same way.

5.4.3 Calculation of concentration

Calculate the concentration c, expressed in moles per litre, of the silver nitrate solution to three significant figures using the following equation:

$$c = \frac{0{,}005m}{58{,}45(V - V_0)}$$

where

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

V is the volume, in millilitres, of the silver nitrate solution used in the titration;

V_0 is the volume, in millilitres, of the silver nitrate solution used in the blank test.

6 Apparatus

Usual laboratory apparatus, plus the following:

6.1 Potentiometric titration apparatus, comprising a suitable potentiometer equipped with a glass-silver electrode system and titration stand, magnetic stirrer and 10 ml microburette.

6.2 Analytical balance, accurate to 0,1 mg.

6.3 Beaker, of capacity 200 ml.

6.4 Graduated glass cylinder, of capacity 100 ml.

6.5 Pipettes, of capacities 1 ml and 2 ml.

7 Procedure

7.1 Weigh, to the nearest 0,1 mg, 10 g of a sample into a 200 ml beaker (6.3). Add 100 ml of acetone (5.1) and dissolve the test portion at room temperature, using a magnetic stirrer (see 6.1).

7.2 Add 2 ml of water and 1 ml of glacial acetic acid (5.3).

7.3 Place the beaker on the titration stand (see 6.1) and adjust its position so that the electrodes are about half immersed. Fill the microburette with 0,002 mol/l silver nitrate solution (5.4), and place the burette in position on the titration stand so that the tip extends approximately 10 mm below the surface of the liquid in the beaker. Adjust the speed of the stirrer to give vigorous stirring without splattering. Record the initial burette and meter (cell potential) readings.

7.4 Add small amounts of silver nitrate solution and, after waiting until a constant potential is reached, record the burette and meter readings. In regions between points of inflection, where the potential change is small for each increment of silver nitrate solution, add volumes of up to 0,1 ml.

When the rate of change of cell potential becomes higher than 5 mV per 0,02 ml, reduce the increments of silver nitrate solution to less than 0,02 ml.

7.5 Continue the titration until the rate of change of cell potential becomes lower than 2 mV per 0,02 ml of silver nitrate solution again. Remove the titrated solution, rinse the electrodes well with water, wipe with a dry cloth and burnish lightly with fine emery cloth. Between titrations, keep the electrodes immersed in water.

7.6 Plot the cumulative volumes of added silver nitrate solution against the cell potential. Take as the end point the middle of the steepest part of the curve (the point of inflection). Read from the plot, to the nearest 0,01 ml, the volume of silver nitrate solution required to reach the end point.

7.7 Conduct a blank test at the same time as the determination, following the same procedure.

8 Expression of results

Calculate the inorganic chlorine content $w(Cl^-)$ of the sample, expressed in milligrams per kilogram, using the following equation:

$$w(Cl^-) = \frac{(V_1 - V_2)\, c \times 35{,}5 \times 1\,000}{m_0}$$

where

V_1 is the volume, in millilitres, of silver nitrate solution (5.4) used in the titration of the test portion;

V_2 is the volume, in millilitres, of silver nitrate solution (5.4) used in the blank test;

c is the concentration of the silver nitrate solution (5.4) calculated in accordance with 5.4.3;

m_0 is the mass, in grams, of the test portion.

Round the result to the first decimal place.

9 Precision

Inorganic chlorine content	Repeatability		Reproducibility	
mg/kg	s_r	r	s_R	R
Less than 1	0,05	0,1	0,13	0,4
From 1 to 3	0,14	0,4	0,27	0,8
From 3 to 5	0,25	0,7	0,55	1,5

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for identification of the sample;

c) the solvent used, if different from that indicated in 5.1 (acetone);

d) the test results;

e) the date of the test;

f) any other relevant information.

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