INTERNATIONAL STANDARD

ISO 13651

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

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

Reference number
ISO 13651:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 13651 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

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Plastics – Epoxy resins and related materials – Determination of total saponifiable chlorine

1 Scope

This International Standard specifies a method for the determination of the total saponifiable chlorine contained in epoxy resins and related materials.

The amount of chlorine measured by the method, referred to as total saponifiable chlorine, includes saponifiable organic chlorine and inorganic chlorine.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitutes 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 method.*

3 Definition

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

3.1 total saponifiable chlorine content: The ratio of the mass of all saponifiable organic and inorganic chlorine in a test portion of epoxy resin or related material to the mass of the test portion.

NOTE 1 "Inorganic chlorine content" is defined in ISO 11376:—[1], *Plastics — Epoxy resins and glycidyl esters — Determination of inorganic chlorine.*

4 Principle

A test portion is dissolved in diethylene glycol monobutyl ether and the solution saponified with an alcoholic solution of potassium hydroxide by heating under reflux. The total saponifiable chlorine content is then determined by potentiometric titration of the solution with silver nitrate solution.

5 Reagents

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

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1) To be published.

5.1 Diethylene glycol monobutyl ether.

5.2 1 mol/l solution of potassium hydroxide in 1,2-propanediol.

Dissolve 56 g of potassium hydroxide in 1,2-propanediol, make up to 1 litre with 1,2-propanediol and mix.

5.3 Glacial acetic acid.

5.4 Acetone.

5.5 Silver nitrate, 0,1 mol/l standard aqueous solution.

5.5.1 Preparation

Dissolve 17,0 g of silver nitrate in water and dilute to 1 litre.

5.5.2 Standardization

Weigh, to the nearest 0,1 mg, 5,845 g of sodium chloride previously dried at 500 °C to 600 °C, dissolve it in water and make up to 1 litre to give a 0,1 mol/l solution. Pipette 5 ml of the sodium chloride solution into a 200 ml beaker (6.12) and add 100 ml of acetone (5.4) and 2 ml of glacial acetic acid (5.3). Then titrate the solution potentiometrically with the silver nitrate solution prepared in 5.5.1.

Carry out a blank test in the same way.

5.5.3 Calculation of concentration

Calculate the concentration c, in moles per litre, from the following equation, rounding the result to four places of decimals:

$$c = \frac{5 \times m}{58{,}45 \times (V_1 - V_0)}$$

where

m is the mass, in grams, of sodium chloride taken;

V_1 is the volume, in millilitres, of silver nitrate solution required for the titration;

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

5.6 Silver nitrate, 0,01 mol/l standard aqueous solution.

5.6.1 Preparation

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

5.6.2 Standardization

Weigh, to the nearest 0,1 mg, 0,584 g of sodium chloride previously dried at 500 °C to 600 °C, dissolve it in water and make up to 1 litre to give a 0,01 mol/l solution. Pipette 5 ml of the sodium chloride solution into a beaker (6.12) and add 100 ml of acetone (5.4) and 2 ml of glacial acetic acid (5.3). Then titrate the solution potentiometrically with the silver nitrate solution prepared in 5.6.1.

Carry out a blank test in the same way.

Calculate the concentration c, in moles per litre, from the equation in 5.5.3, rounding the result to four places of decimals.

6 Apparatus

Usual laboratory apparatus, plus the following:

6.1 Potentiometric titration apparatus, including a suitable potentiometer equipped with a silver electrode and a silver chloride or mercury sulfate electrode, plus a titration stand.

6.2 Analytical balance, accurate to 0,1 mg.

6.3 Magnetic stirrer (with polytetrafluoroethylene-coated bar).

6.4 Measuring flask, of capacity 1 litre.

6.5 Hotplate or **oil bath**, capable of being heated to above 200 °C.

6.6 Conical flask, of capacity 200 ml, with a ground-glass stopper.

6.7 Reflux condenser.

6.8 Graduated glass cylinder, of capacity 50 ml.

6.9 Pipette, of capacity 5 ml.

6.10 Porcelain crucible.

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

6.12 Beaker, of capacity 200 ml.

7 Procedure

7.1 Into a 200 ml conical flask (6.6), weigh to the nearest 0,1 mg:

— a test portion of a size such that it contains 0,5 mg to 1,5 mg of chlorine, when the expected total saponifiable chlorine content is less than 1 %;

or

— a test portion of a size such that it contains 5 mg to 15 mg of chlorine, when the expected total saponifiable chlorine content is greater than 1 %.

7.2 Add 25 ml of diethylene glycol monobutyl ether (5.1) and dissolve the test portion.

7.3 Add 25 ml of 1 mol/l potassium hydroxide solution in 1,2-propanediol (5.2). Reflux the solution on a hotplate or in an oil bath (6.5) for 10 min while stirring.

7.4 Leave to cool, then pour 5 ml of acetone (5.4) down the reflux condenser (6.7).

7.5 Transfer the solution from the flask to a 200 ml beaker (6.12). Wash the inside of the flask into the beaker three times, using a total of 50 ml of glacial acetic acid (5.3).

7.6 Immerse the electrodes (6.1) in the solution. Adjust the stirrer speed to give vigorous stirring without splattering.

7.7 Carry out the potentiometric titration with

— 0,01 mol/l silver nitrate solution (5.6) if the expected total saponifiable chlorine content is less than 1 %;

or

— 0,1 mol/l silver nitrate solution (5.5) if the expected total saponifiable chlorine content is greater than 1 %.

7.8 Carry out a blank test in the same way.

8 Expression of results

Calculate the total saponifiable chlorine content $w(\mathrm{Cl})$ of the sample, expressed in milligrams per kilogram (parts per million by mass), from the following equation, rounding the result to three places of decimals:

$$w(\mathrm{Cl}) = \frac{35{,}45 \cdot c \cdot (V_1 - V_0) \cdot 1\,000}{m}$$

where

c is the concentration, in moles per litre, of the silver nitrate solution used (5.5 or 5.6);

m is the mass, in grams, of the test portion;

V_1 is the volume, in millilitres, of silver nitrate solution (5.5 or 5.6) used for the titration of the test portion;

V_0 is the volume, in millilitres, of silver nitrate solution (5.5 or 5.6) used for the blank test.

9 Precision

The precision of this test method was determined in accordance with ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* (now withdrawn), following round-robin testing organized in Japan (1992-1993).

The repeatability and reproducibility calculated from the round-robin testing are as follows:

Type of epoxy resin	Repeatability		Reproducibility		Average total saponifiable chlorine content
	s_r	r	s_R	R	
BPA	32	90	46	129	1 497
ECN	28	78	37	104	1 071

BPA is bisphenol A type liquid epoxy resin;

ECN is *o*-cresol novolac type solid epoxy resin;

s_r is the repeatability (within-laboratory) standard deviation;

r is the repeatability (absolute value);

s_R is the reproducibility (between laboratory) standard deviation;

R is the reproducibility (absolute value).

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 results of the test;

d) the date of the test;

e) any other necessary information.

ICS 83.080.10

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

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