INTERNATIONAL STANDARD

ISO 21627-2

Second edition
2009-09-15

Plastics — Epoxy resins — Determination of chlorine content —

Part 2: Easily saponifiable chlorine

Plastiques — Résines époxydes — Détermination de la teneur en chlore —

Partie 2: Chlore facilement saponifiable

Reference number
ISO 21627-2:2009(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 21627-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 21627-2:2002), which has been technically revised.

ISO 21627 consists of the following parts, under the general title *Plastics — Epoxy resins — Determination of chlorine content*:

— *Part 1: Inorganic chlorine*

— *Part 2: Easily saponifiable chlorine*

— *Part 3: Total chlorine*

Introduction

In producing epoxy resins based on epichlorohydrin, impurities containing chlorine may be formed. These are shown below. Since these impurities could impair the final properties of the cured resins, it is necessary to control their formation. Their chemical activities differ significantly, so different analytical procedures are needed for their analysis.

ISO 21627 specifies methods for the determination of these organic and inorganic chlorides which occur as impurities in epoxy resins derived from epichlorohydrin:

— Part 1: Inorganic chlorine (also called ionic chlorine).

— Part 2: Easily saponifiable chlorine, consisting mainly of chlorine which is present as 1,2-chlorohydrin as the result of incomplete dehydrohalogenation.

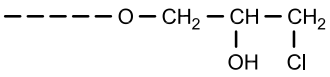
— Part 3: Total chlorine, consisting mainly of all saponifiable organic chlorine, e.g. 1,2-chlorohydrin, 1,3-chlorohydrin and 1-chloro-2-glycidylether (chloromethyl derivative) which are the result of incomplete dehydrohalogenation, along with inorganic chlorine present in the test portion of epoxy resin.

Since the purposes of Parts 1 to 3 of ISO 21627 differ, one of these methods should be selected, depending on the impurities to be measured.

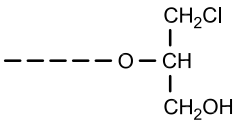
For analytical methods for impurities other than those shown below, see ISO 4615.

Typical types of inorganic and organic chlorine impurity are shown below:

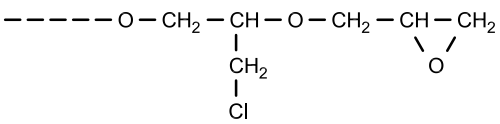
Inorganic chlorine (or ionic chlorine)



1,2-Chlorohydrin



1,3-Chlorohydrin



1-Chloro-2-glycidylether
(chloromethyl derivative)

Plastics — Epoxy resins — Determination of chlorine content —

Part 2: Easily saponifiable chlorine

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

1 Scope

This part of ISO 21627 specifies a method for the determination of easily saponifiable chlorine in epoxy resins.

The easily saponifiable chlorine content is the quantity of easily saponifiable chlorine in a given quantity of epoxy resin.

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

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 21627-1, *Plastics — Epoxy resins — Determination of chlorine content — Part 1: Inorganic chlorine*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
easily saponifiable chlorine
amount of chlorine saponifiable by this test method, consisting mainly of chlorine present as 1,2-chlorohydrin as a result of incomplete dehydrohalogenation

4 Principle

Epoxy resins, except glycidyl esters, are reacted with NaOH solution at room temperature in 2-butoxyethanol.

Glycidyl esters are reacted with NaOH solution at 50 °C in methanol.

The mixture is acidified and the concentration of chloride ions resulting from the saponification is determined by potentiometric titration with standardized silver nitrate solution. A correction is made for the inorganic chlorine content of the sample, determined by the method specified in ISO 21627-1.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and water of grade 3 purity, as defined in ISO 3696:1987, or better.

5.1 Glacial acetic acid.

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. The threshold limit value is a volume fraction of 5×10^{-5}.

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 achieve complete dissolution. Cool and make up to 1 l with the same solvent.

5.6 Acetone.

5.7 Silver nitrate solution, 0,01 mol/l.

5.7.1 Preparation

Dissolve 1,7 g of silver nitrate in water and make up to 1 l.

5.7.2 Standardization

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

Pipette 5 ml of this solution into a 200 ml beaker and add 100 ml of acetone (5.6) and 2 ml of glacial acetic acid (5.1). Then titrate potentiometrically with the silver nitrate solution prepared in 5.7.1.

Conduct a blank test in the same way, leaving out the sodium chloride.

5.7.3 Calculation of concentration

Calculate the concentration using the following equation, rounding the result to three significant figures:

$$c_2 = \frac{0{,}005 \times m}{58{,}5 \times (V - V_0)}$$

where

c_2 is the concentration of the silver nitrate solution, expressed in moles per litre (mol/l);

m is the mass of sodium chloride used, expressed in milligrams (mg);

58,5 is the gram equivalent of sodium chloride (g/mol);

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

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

5.7.4 Storage

Store the silver nitrate solution in a brown bottle in the dark.

6 Apparatus

Usual laboratory apparatus, plus the following:

6.1 Potentiometric-titration apparatus, comprising a suitable potentiometer or autotitrator equipped with a glass-silver/silver chloride electrode system, titration stand and 10 ml microburette.

6.2 Analytical balance, accurate to 0,1 mg.

6.3 Beaker, of capacity 200 ml.

6.4 Volumetric flask, of capacity 1 l.

6.5 Pipettes, of capacities 2 ml, 5 ml and 25 ml.

6.6 Graduated glass cylinder, of capacity 100 ml.

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

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

6.9 Reflux condenser.

6.10 Magnetic stirrer, with a PTFE (polytetrafluoroethylene) coated stirring bar.

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 (6.3). Pipette 25 ml of 2-butoxyethanol (5.2) into the beaker and dissolve the test portion using the magnetic stirrer (6.10) 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.

7.1.2 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.

7.1.3 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 has dissolved.

7.1.4 Place the electrodes (see 6.1) in the test solution and titrate the solution potentiometrically with silver nitrate solution (5.7).

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

7.1.5 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.

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 exactly 1 ml, accurately measured, of 0,01 mol/l potassium chloride solution added to the solution (and also to 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 21627-1.

7.2 Glycidyl esters

7.2.1 Glycidyl esters shall be pretreated by one of the following two methods:

a) method A, using a beaker;

b) method B, using a conical flask and reflux condenser.

NOTE Method B is preferred for reasons of safety and hygiene.

7.2.2 **Method A:** Weigh, to the nearest 0,1 mg, a test portion containing not more than 1,78 mg of easily saponifiable chlorine into the beaker (6.3). Pipette 25 ml of methanolic sodium hydroxide solution (see 5.5) into the beaker and dissolve the test portion using the magnetic stirrer (6.10). Cover the beaker and allow the reaction mixture to stand in the water bath (6.7) at 50 °C for 2 h.

7.2.3 **Method B:** Weigh, to the nearest 0,1 mg, a test portion containing not more than 1,78 mg of easily saponifiable chlorine into the conical flask (6.8). Pipette 25 ml of methanolic sodium hydroxide solution (see 5.5) into the conical flask and dissolve the test portion using the magnetic stirrer (6.10). Fit the reflux condenser (6.9) on the flask and allow the reaction mixture to stand in the water bath (6.7) at 50 °C for 2 h.

7.2.4 Following pretreatment, proceed in accordance with 7.1.2 to 7.1.6.

8 Expression of results

Calculate the saponifiable chlorine content of the sample using the following equation:

$$w_2(\mathrm{Cl}^-) = \frac{35{,}5 \times c_2 \times (V_1 - V_2) \times 1000}{m_0} - c_1$$

where

$w_2(\mathrm{Cl}^-)$ is the saponifiable chlorine content of the sample, expressed in milligrams per kilogram (mg/kg);

V_1 is the volume of silver nitrate solution (5.7) used in the titration of the test portion, expressed in millilitres (ml);

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

c_2 is the concentration of the silver nitrate solution, calculated in accordance with 5.7.3 (mol/l);

35,5 is the gram equivalent of chlorine (g/mol);

c_1 is the inorganic chlorine content (see 7.1.6), expressed in milligrams per kilogram (mg/kg);

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

Round the result to three significant figures.

9 Precision

The precision of this test method is not known because interlaboratory data are not available. When precision data are obtained, a precision statement will be added at the following revision.

10 Test report

The test report shall include the following information:

a) a reference to this part of ISO 21627, i.e. ISO 21627-2;

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

c) the inorganic chlorine content, determined in accordance with ISO 21627-1;

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 details of any incident which may have influenced the result.

Bibliography

[1] ISO 4615, *Plastics — Unsaturated polyesters and epoxide resins — Determination of total chlorine content*

[2] ISO 21627-3, *Plastics — Epoxy resins — Determination of chlorine content — Part 3: Total chlorine*

ICS 83.080.10

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