INTERNATIONAL STANDARD

ISO 21627-3

First edition
2002-08-15

Plastics — Epoxy resins — Determination of chlorine content —

Part 3: Total chlorine

Plastiques — Résines époxydes — Détermination de la teneur en chlore — Partie 3: Chlore total

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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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 3.

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 part of ISO 21627 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 21627-3 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This first edition cancels and replaces ISO 13651:1996, of which it constitutes a technical revision.

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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*

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Introduction

In producing epoxy resins based on epichlorohydrin, impurities containing chlorine may be formed. These are shown below. Since these impurities could lower 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.

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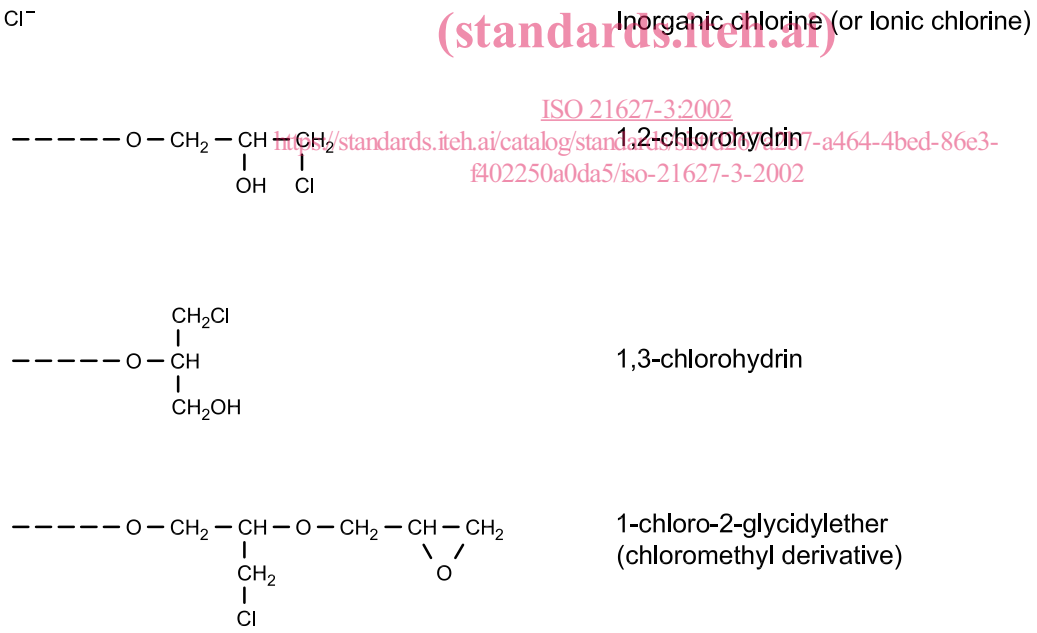

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Typical impurity types of inorganic and organic chlorine.

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Plastics — Epoxy resins — Determination of chlorine content —

Part 3: Total chlorine

WARNING — Persons using this part of ISO 21627 should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, 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 national regulatory conditions.

1 Scope

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

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

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2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 21627. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 21627 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Term and definition

For the purposes of this part of ISO 21627, the following term and definition apply.

3.1
total chlorine
amount of chlorine measurable by this method

NOTE It consists mainly of all saponifiable organic chlorine, e.g. 1,2-chlorohydrin, 1,3-chlorohydrin and 1-chloro-2-glycidylether which are the result of incomplete dehydrohalogenation along with inorganic chlorine present in the test portion of epoxy resin.

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 chlorine content is then determined by potentiometric titration of the solution with silver nitrate solution.

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5 Reagents

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

5.1 Diethylene glycol monobutyl ether.

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

Dissolve 56 g of potassium hydroxide in 1,2-propanediol, make up to 1 l 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 g of silver nitrate in water and dilute to 1 l.

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 l to give a 0,1 mol/l solution. Pipette 5 ml of the sodium chloride solution into a 200 ml beaker 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.

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Carry out a blank test in the same way, but without the addition of sodium chloride.

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5.5.3 Calculation of concentration

Calculate the concentration, to three significant figures using the following equation, rounding the result to four decimal places.

$$c_3 = \frac{0{,}005 \times m}{58{,}45 \times (V_1 - V_0)}$$

where

c_3 is the concentration of the silver nitrate solution used in the titration, expressed in moles per litre (mol/l);

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

V_1 is the volume of silver nitrate solution required for the titration, expressed in millilitres (ml);

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

5.5.4 Storage

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

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

5.6.1 Preparation

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

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5.6.2 Standardization

Weigh, to the nearest 0,1 mg, 584 mg of sodium chloride previously dried at 500 °C to 600 °C, dissolve it in water and make up to 1 l to give a 0,01 mol/l solution. Pipette 5 ml of the sodium chloride solution into a beaker (6.10) 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 manner, omitting the sodium chloride.

Calculate the concentration, from the equation in 5.5.3, rounding the result to four decimal places.

6 Apparatus

Usual laboratory apparatus, plus the following:

6.1 Potentiometric titration apparatus, comprising a suitable potentiometer equipped with a silver electrode and a silver chloride or mercury sulfate electrode, a magnetic stirrer and a titration stand.

6.2 Analytical balance, accurate to 0,1 mg.

6.3 Volumetric flask, of capacity 1 l.

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

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

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6.6 Reflux condenser.

6.7 Graduated glass cylinder, of capacity 50 ml.

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6.8 Pipette, of capacity 5 ml.

6.9 Porcelain crucible.

6.10 Beaker, of capacity 200 ml.

7 Procedure

7.1 Into a 200 ml conical flask (6.5), 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 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 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 (6.4) for 10 min while stirring.

7.4 Leave to cool, then pour 5 ml of acetone (5.4) down the reflux condenser (6.6).

7.5 Transfer the solution from the flask to a 200 ml beaker (6.10). 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 (see 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 chlorine content is less than 1 % or 0,1 mol/l silver nitrate solution (5.5) if the expected total chlorine content is greater than 1 %.

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