INTERNATIONAL STANDARD

ISO 21627-3

Second edition
2009-09-15

Plastics — Epoxy resins — Determination of chlorine content —

Part 3
Total chlorine

Plastiques — Résines époxydes — Détermination de la teneur en chlore

Partie 3: Chlore total

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

This second edition cancels and replaces the first edition (ISO 21627-3:2002), 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.

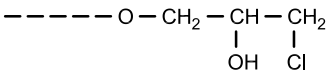
— Part 3: Total chlorine, consisting mainly of all saponifiable organic chlorine, e.g. 1,2-chlorohydrin, 1,3-chlorohydrin and 1-chloro-2-glycidylether (chloromethyl derivative) which are the result of incomplete dehydrohalogenation, along with inorganic chlorine present in the test portion of epoxy resin.

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.

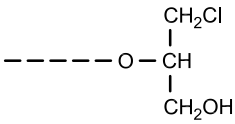
For analytical methods for impurities other than those shown below, see ISO 4615.

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

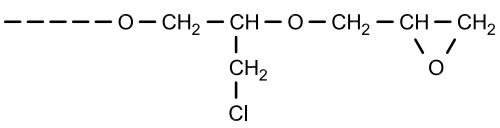
Inorganic chlorine (or ionic chlorine)



1,2-Chlorohydrin



1,3-Chlorohydrin



1-Chloro-2-glycidylether
(chloromethyl derivative)

Plastics — Epoxy resins — Determination of chlorine content —

Part 3: Total 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 the total amount of chlorine in epoxy resins.

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

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*

3 Terms and definitions

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

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 3696:1987, or better.

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 aqueous standard solution.

5.5.1 Preparation

Dissolve 17 g of silver nitrate in water and make up 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 this 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.

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

5.5.3 Calculation of concentration

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

$$c_3 = \frac{0{,}005 \times m}{58{,}5 \times (V - V_0)}$$

where

c_3 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).

5.5.4 Storage

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

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

5.6.1 Preparation

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

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 this 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.

5.6.3 Calculation of concentration

Calculate the concentration from the equation in 5.5.3, rounding the result to three significant figures.

5.6.4 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 equipped with a silver electrode and a silver chloride or mercury sulfate electrode 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.

6.6 Reflux condenser.

6.7 Graduated glass cylinder, of capacity 50 ml.

6.8 Pipette, of capacity 5 ml.

6.9 Porcelain crucible.

6.10 Beaker, of capacity 200 ml.

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

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 using a magnetic stirrer (6.11).

7.3 Add 25 ml of 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.