INTERNATIONAL STANDARD

ISO 21627-2

First edition
2002-08-15

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 21627-2:2002(E)

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

Printed in Switzerland

Contents

Page

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

This first edition cancels and replaces ISO 4583:1998, of which it constitutes a technical revision.

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 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).

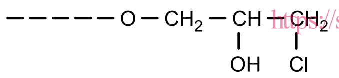
— Part 2: Easily saponifiable chlorine consisting mainly of chlorine, which is present as 1,2-chlorohydrin as the result of incomplete dehydrohalogenation.

— 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.

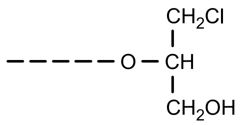
Since the purposes of parts 1 to 3 of ISO 21627 differ, one of these methods should be selected depending on the impurities to be measured.

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

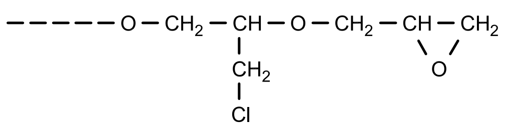
Cl^- Inorganic chlorine (or Ionic chlorine)



1,2-chlorohydrin



1,3-chlorohydrin



1-chloro-2-glycidylether
(chloromethyl derivative)

Typical impurity types of inorganic and organic chlorine.

Plastics — Epoxy resins — Determination of chlorine content —

Part 2: Easily saponifiable 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 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 of chlorohydrin groups in the compounds.

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.

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 Term and definition

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

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

4 Principle

Epoxy resins, excluding 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 chloride ion concentration 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, unless otherwise stated, use only reagents of recognized analytical grade and water of grade 3 purity or better conforming to ISO 3696:1987.

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. Threshold limit value is 5×10^{-5} volume fraction.

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 Sodium chloride.

5.7 Acetone.

5.8 Silver nitrate, 0,01 mol/l.

5.8.1 Preparation

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

5.8.2 Standardization

Weigh, to the nearest 0,1 mg, 584 mg of sodium chloride (5.6), previously dried at 500 °C to 600 °C, and dissolve in 1 l of water.

Pipette 5 ml of the sodium chloride solution into a 200 ml beaker. Add 100 ml of acetone (5.7) and 2 ml of acetic acid (5.1). Then titrate the solution potentiometrically with the silver nitrate solution prepared in 5.8.1.

Carry out a blank test on the solvent (5.7) in the same manner.

5.8.3 Calculation of the concentration

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

$$c_2 = \frac{0{,}005 \times m}{58{,}45 \times (V - V_0)}$$

where

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

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

V is the volume of silver nitrate solution (5.8) used in the titration; expressed in millilitres (ml),

V_0 is the volume of silver nitrate solution (5.8) used in the blank determination, expressed in millilitres (ml).

5.8.4 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 glass/silver chloride electrode, a magnetic stirrer, a titration stand and a 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 litre.

6.5 Pipettes, of capacity 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.

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 and by heating if necessary. Cool the solution to room temperature and pipette 25 ml of sodium hydroxyde 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 is dissolved.