INTERNATIONAL
STANDARD

ISO
21627-1

Second edition
2009-09-15

Plastics — Epoxy resins — Determination of chlorine content —

Part 1:
Inorganic chlorine

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

Partie 1: Chlore inorganique

Reference number
ISO 21627-1:2009(E)

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Published in Switzerland

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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 21627-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 21627-1:2002), 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.

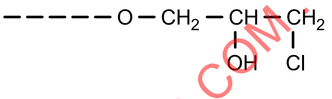
Since the purposes of Parts 1 to 3 of ISO 21627 differ, one of these methods should be selected, depending on the impurities to be measured.

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

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

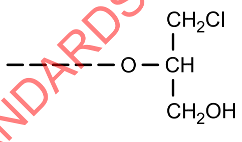
Cl^-

Inorganic chlorine (or ionic chlorine)



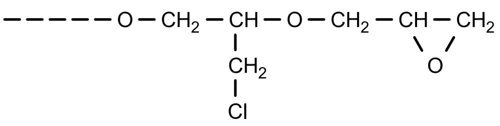
$\text{------O}-\text{CH}_2-\underset{\text{OH}}{\text{CH}}-\underset{\text{Cl}}{\text{CH}_2}$

1,2-Chlorohydrin



$\text{------O}-\underset{\text{CH}_2\text{OH}}{\overset{\text{CH}_2\text{Cl}}{\text{CH}}}$

1,3-Chlorohydrin



1-Chloro-2-glycidylether
(chloromethyl derivative)

Plastics — Epoxy resins — Determination of chlorine content —

Part 1:
Inorganic 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 direct potentiometric method for the determination of inorganic chlorine in epoxy resins, also called “ionic chlorine”.

The inorganic chlorine content is expressed in milligrams per kilogram of epoxy 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*

3 Terms and definitions

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

3.1
inorganic chlorine
ionic chlorine
amount of chlorine present in a resin as Cl^-

4 Principle

A test portion is dissolved in a suitable solvent and the inorganic chlorine determined by potentiometric titration with standardized silver nitrate solution.

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 **Acetone**.

In some cases, acetone may not dissolve the resin. In such cases, use butanone (methyl ethyl ketone) or THF (tetrahydrofuran) or another suitable solvent and record the solvent used in the test report.

5.2 **2-Propanol**.

5.3 **Glacial acetic acid**.

5.4 **Solution of silver nitrate in 2-propanol**, 0,002 mol/l.

5.4.1 Preparation

Dissolve 17 g of silver nitrate in water and make up to 1 l (0,1 mol/l). Put 20 ml of this aqueous 0,1 mol/l silver nitrate solution in a 1 l graduated flask and dilute to 1 l with 2-propanol (5.2).

5.4.2 Standardization

Weigh, to the nearest 0,1 mg, 115 mg to 120 mg of sodium chloride, previously dried at 500 °C to 600 °C, dissolve it in water and make up to 1 l. Pipette 5 ml of this solution into a 200 ml beaker and add 100 ml of acetone (5.1) and 2 ml of glacial acetic acid (5.3). Then titrate potentiometrically with the silver nitrate solution prepared in 5.4.1.

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

5.4.3 Calculation of concentration

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

$$c_1 = \frac{0{,}005 \times m}{58{,}5 \times (V - V_0)}$$

where

c_1 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).

5.4.4 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/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 Graduated glass cylinder, of capacity 100 ml.

6.5 Pipettes, of capacities 1 ml, 2 ml and 5 ml.

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

7 Procedure

7.1 Weigh, to the nearest 0,1 mg, 10 g of sample into a 200 ml beaker (6.3). Add 100 ml of acetone (5.1) and dissolve the test portion at room temperature, using a magnetic stirrer (6.6).

7.2 Add 2 ml of water and 1 ml of glacial acetic acid (5.3).

7.3 Place the beaker on the titration stand (see 6.1) and adjust its position so that the electrodes (see 6.1) are about half immersed. Fill the microburette with 0,002 mol/l silver nitrate solution (5.4), then place the burette in position on the titration stand so that the tip extends approximately 10 mm below the surface of the liquid in the beaker. Adjust the speed of the stirrer to give vigorous stirring without splashing. Record the initial burette and meter (cell potential) readings.

7.4 Add small amounts of silver nitrate solution and, after waiting until the potential reaches a constant value, record the burette and meter readings. In regions between points of inflection, where the change in potential is small for each increment of silver nitrate solution, add volumes of up to 0,1 ml. Where the rate of change of the cell potential becomes higher than 5 mV per 0,02 ml, reduce the increments of silver nitrate solution to less than 0,02 ml.

7.5 Continue the titration until the rate of change of cell potential again becomes lower than 2 mV per 0,02 ml of silver nitrate solution. Remove the titrated solution, rinse the electrodes well with water, wipe with a dry cloth and burnish lightly with fine emery cloth. Between titrations, keep the electrodes immersed in water.

7.6 Plot the cumulative volumes of added silver nitrate solution against the cell potential. Take as the end point the middle of the steepest part of the curve (the inflection point). Read from the plot, to the nearest 0,01 ml, the volume of silver nitrate solution required to reach the end point.

7.7 Conduct a blank test at the same time as the determination, following the same procedure.

8 Expression of results

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

$$w_1(\mathrm{Cl}^-) = \frac{(V_1 - V_2) \times c_1 \times 35{,}5 \times 1\,000}{m_0}$$

where

$w_1(\mathrm{Cl}^-)$ is the inorganic chlorine content of the sample, expressed in milligrams per kilogram (mg/kg);