

Plastics — Epoxy resins — Determination of chlorine content —

Part 3: Total chlorine

The European Standard EN ISO 21627-3:2003 has the status of a
British Standard

ICS 83.080.10

National foreword

This British Standard is the official English language version of EN ISO 21627-3:2003. It is identical with ISO 21627-3:2002. It supersedes BS ISO 13651:1996 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/42, Fibre reinforced thermosetting plastics and prepregs, which has the responsibility to:

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- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
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Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, the EN ISO foreword page, the ISO title page, pages ii to v, a blank page, pages 1 to 5, the Annex ZA page, an inside back cover and a back cover.

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English version

**Plastics - Epoxy resins - Determination of chlorine content
Part 3: Total chlorine (ISO 21627-3:2002)**

Plastiques - Résines époxydes - Détermination de la teneur
en chlore - Partie 3: Chlore total (ISO 21627-3:2002)

Kunststoffe - Epoxidharze - Bestimmung des Chlorgehaltes
- Teil 3: Gesamtgehalt an Chlor (ISO 21627-3:2002)

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Management Centre: rue de Stassart, 36 B-1050 Brussels

EN ISO 21627-3:2003**Foreword**

The text of ISO 21627-3:2002 has been prepared by Technical Committee ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 21627-3:2003 by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2004, and conflicting national standards shall be withdrawn at the latest by May 2004.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of ISO 21627-3:2002 has been approved by CEN as EN ISO 21627-3:2003 without any modifications.

NOTE Normative references to International Standards are listed in Annex ZA (normative).

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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Contents

	Page
1 Scope	1
2 Normative references	1
3 Term and definition	1
4 Principle	1
5 Reagents	2
6 Apparatus	3
7 Procedure	3
8 Expression of results	4
9 Precision	4
10 Test report	4
Bibliography.....	5

EN ISO 21627-3:2003

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.

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*

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.

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 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.

EN ISO 21627-3:2003**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.

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, 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.

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.

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.

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 %.

EN ISO 21627-3:2003

7.8 Carry out a blank test in the same way.

8 Expression of results

Calculate the total chlorine content, using the following equation, rounding the result to three places of decimals:

$$w_3(\text{Cl}^-) = \frac{35,45 \times c_3 \times (V_1 - V_0) \times 1\,000}{m}$$

where

$w_3(\text{Cl}^-)$ is the total chlorine content of the sample, expressed in milligrams per kilogram (mg/kg);

c_3 is the concentration of the silver nitrate solution, calculated in accordance with 5.5.3, expressed in moles per litre (mol/l);

V_1 is the volume of silver nitrate solution (5.5 or 5.6) used for titration of the test portion, expressed in millilitres (ml);

V_0 is the volume of silver nitrate solution (5.5 or 5.6) used for the blank test, expressed in millilitres (ml);

m is the sample mass of the test portion, expressed in grams (g).

9 Precision

The accuracy data were determined from an experiment organized in 1994 involving 10 laboratories and 3 levels and analysed in accordance with ISO 5725-2. Data from 10 laboratories contained outliers. The outliers were not included in the calculation of the repeatability standard deviation and the reproducibility standard deviation. A description of the materials used in the accuracy experiment shall be added, especially when the trueness or precision depend on the materials.

Type of epoxy resin	Repeatability s_r	Reproducibility s_R	Average total chlorine content
BPA	32	46	1 497
ECN	28	37	1 071
BPA is bisphenol A type liquid epoxy resin; ECN is o-cresol novolac type solid epoxy resin; s_r is the repeatability (within-laboratory) standard deviation; s_R is the reproducibility (between laboratory) standard deviation.			

10 Test report

The test report shall include the following information:

- reference to this part of ISO 21627, i.e. ISO 21627-3;
- all details necessary for identification of the sample;
- results of the test;
- date of the test;
- any other necessary information.

Bibliography

- [1] ISO 4615:1979, *Plastics — Unsaturated polyesters and epoxide resins — Determination of total chlorine content*
- [2] ISO 21627-1, *Plastics — Epoxy resins — Determination of chlorine content — Part 1: Inorganic chlorine*
- [3] ISO 21627-2, *Plastics — Epoxy resins — Determination of chlorine content — Part 2: Easily saponifiable chlorine*

Annex ZA (normative)

Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

NOTE Where an International Publication has been modified by common modifications, indicated by (mod.), the relevant EN/HD applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	<u>EN</u>	<u>Year</u>
ISO 3696	1987	Water for analytical laboratory use - Specification and test methods	EN ISO 3696	1995

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