

Plastics — Epoxy resins — Determination of chlorine content —

Part 1: Inorganic chlorine

The European Standard EN ISO 21627-1:2003 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 21627-1:2003. It is identical with ISO 21627-1:2002. It supersedes BS EN ISO 11376:2001 which is withdrawn.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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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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Plastics - Epoxy resins - Determination of chlorine content
Part 1: Inorganic chlorine (ISO 21627-1:2002)

Plastiques - Résines époxydes - Détermination de la teneur
en chlore - Partie 1: Chlore inorganique (ISO 21627-
1:2002)

Kunststoffe - Epoxidharze - Bestimmung des Chlorgehaltes
- Teil 1: Anorganisches Chlor (ISO 21627-1:2002)

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EN ISO 21627-1:2003

Foreword

The text of ISO 21627-1:2002 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 21627-1:2003 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of an identical text or by endorsement, at the latest by May 2004, and conflicting national standards shall be withdrawn at the latest by May 2004.

This document supersedes EN ISO 11376:2001.

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 21627-1:2002 has been approved by CEN as EN ISO 21627-1:2003 without any modifications.

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

INTERNATIONAL
STANDARD

ISO
21627-1

First edition
2002-08-15

**Plastics — Epoxy resins — Determination
of chlorine content —**

Part 1:
Inorganic chlorine

*Plastiques — Résines époxydes — Détermination de la teneur en chlore —
Partie 1: Chlore inorganique*



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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	2
7 Procedure	3
8 Expression of results	3
9 Precision	4
10 Test report	4
Bibliography.....	5

EN ISO 21627-1:2003

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

This first edition cancels and replaces ISO 11376:1997 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 1:

Inorganic 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 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 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

inorganic chlorine

also called ionic chlorine, it is chlorine present into the 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.

EN ISO 21627-1:2003**5 Reagents**

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

5.1 Acetone.

NOTE 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 dilute 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, and dissolve in 1 l of water.

Pipette 5 ml of this solution into a 200 ml beaker, then 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:

$$c_1 = \frac{0,005 \times m}{58,45 \times (V - V_0)}$$

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);

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 or autotitrator equipped with a glass-silver/silver chloride electrode system, magnetic stirrer, 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.

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.

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 (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 splattering. Record the initial burette and meter (cell potential) readings.

7.4 Add small amounts of silver nitrate solution and, after waiting until a constant potential is reached, record the burette and meter readings. In regions between points of inflection, where the potential change is small for each increment of silver nitrate solution, add volumes of up to 0,1 ml.

When the rate of change of 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:

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

where

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

V_1 is the volume of silver nitrate solution (5.4) used in the titration of the test portion, expressed in millilitres (ml);

V_2 is the volume of silver nitrate solution (5.4) used in the blank test, expressed in millilitres (ml);

c_1 is the concentration of the silver nitrate solution (5.4) calculated in accordance with 5.4.3;

m_0 is the sample mass, of the test portion, expressed in grams (g).

Round the result to the first decimal place.

EN ISO 21627-1:2003**9 Precision**

The accuracy data were determined from an experiment organized and analysed in accordance with ISO 5725-2 involving 10 laboratories and 3 levels. 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.

Inorganic chlorine content mg/kg	Repeatability s_r	Reproducibility s_R
$w_1(\text{Cl}^-) < 1$	0,05	0,13
$1 \leq w_1(\text{Cl}^-) < 3$	0,14	0,27
$3 \leq w_1(\text{Cl}^-) \leq 5$	0,25	0,55

s_r is the repeatability (within-laboratory) standard deviation;
 s_R is the reproducibility (between laboratories) standard deviation.

10 Test report

The test report shall include the following information:

- reference to this part of ISO 21627, i.e. ISO 21627-1;
- all details necessary for identification of the sample;
- solvent used, if different from that indicated in 5.1 (acetone);
- test results;
- date of the test;
- any other relevant information.

Bibliography

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