TC 61

# INTERNATIONAL STANDARD ISO 4573

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## Plastics — Epoxide resins and glycidyl esters — Determination of inorganic chlorine

*Plastiques — Résines d'époxydes et esters glycidiques — Dosage du chlore inorganique*

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## FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4573 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in December 1976.



It has been approved by the member bodies of the following countries :



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No member body expressed disapproval of the document.



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# Plastics — Epoxide resins and glycidyl esters — Determination of inorganic chlorine

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a direct potentiometric method for the determination of inorganic substances in epoxide resins and glycidyl esters, called also "inorganic chlorine" or "ionic chlorine".

## 2 REFERENCE

ISO 3696, *Water for laboratory use — Specifications.* [1)]



## 3 PRINCIPLE



Dissolution of a test portion in a suitable solvent. Determination of the chloride ion by potentiometric titration with a standard volumetric silver nitrate solution.

## 4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only grade 1 water conforming to ISO 3696.

**4.1 Acetic acid,** glacial, $\rho$ 1,05 g/ml.

**4.2 Butanone** (methyl ethyl ketone).

NOTE — In some cases, butanone does not dissolve the resin; another, more suitable solvent should then be used, and mentioned in the test report.

**4.3 Hydrochloric acid,** 0,1 N solution.

**4.4 Potassium chloride,** 0,01 N solution.

**4.5 Silver nitrate,** 0,01 N standard volumetric solution.

**4.5.1** *Preparation*

Dissolve 1,70 g of silver nitrate in water and dilute to 1 litre.

**4.5.2** *Standardization*

Weigh, to the nearest 0,1 mg, 20 to 25 mg of sodium chloride, previously dried at 120 °C. Transfer to a 300 ml conical flask, and dissolve in 50 ml of water. Titrate with the silver nitrate solution (4.5.1) in accordance with 7.3.

**4.5.3** *Calculation of concentration*

The concentration $T$, expressed as normality, is given by the formula

$$T = \frac{m}{58{,}45\ V}$$

where

$m$ is the mass, in milligrams, of sodium chloride used;

$V$ is the volume, in millilitres, of the silver nitrate solution (4.5.1) used in the titration.

## 5 APPARATUS

Usual laboratory apparatus, and

**5.1 Microburette,** 10 ml capacity, graduated in 0,02 ml; length of delivery tube approximately 120 mm.

**5.2 Magnetic stirrer.**

**5.3 pH-millivoltmeter,** with glass and silver electrodes and titration stand.

1) At present at the stage of draft.

## 6 PREPARATION OF ELECTRODES

Before each titration, wash the glass electrode (see 5.3) with the solvent (4.2), rinse with water, soak the electrode for at least 10 min in the hydrochloric acid (4.3), and rinse again with water and solvent. Polish the silver electrode before each titration with fine abrasive cloth (for example, grain coarseness P 120*) until a clean polished surface is obtained, and rinse with water.

NOTES

1 Observance of these instructions for preparing the electrodes is very important, as otherwise the electrodes will not respond properly during the titration. However, in practice when frequent measurements on the same type of product are made, it is only necessary to prepare the electrodes daily.

2 Other types of electrodes and other methods of preparing the electrodes can be used provided that there is agreement between participants.

## 7 PROCEDURE

**7.1** Weigh, to the nearest 1 mg, a test portion containing not more than 1,42 mg of inorganic chlorine in a 250 ml beaker. Pipette 100 ml of the solvent (4.2) into the beaker and dissolve the test portion at room temperature, using the magnetic stirrer (5.2).

**7.2** Add, by means of pipettes, 1 ml of the potassium chloride solution (4.4) and 25 ml of the acetic acid (4.1) to the mixture. Titrate immediately in accordance with 7.3.

**7.3** Place the electrodes and the microburette (5.1) containing the silver nitrate solution (4.5) in position, immersing the tip of the microburette about 10 mm in the liquid. Stir throughout the determination. Add the silver nitrate solution carefully and wait until a constant potential has been established.

Record the microburette and meter readings and plot them rapidly as a graph. When the potential change is small for each increment of silver nitrate solution added, add volumes as large as 0,1 ml. When the rate of change of potential becomes greater than 10 mV per 0,1 ml, use 0,05 ml increments. Stop the titration about 0,3 ml after the deflection point.

**7.4** Carry out a blank test at the same time as the determination, following the same procedure but omitting the test portion.

## 8 EXPRESSION OF RESULTS

**8.1** Note the equivalence point from the graph prepared as described in 7.3.

**8.2** The "inorganic chlorine" content $C$ of the sample, expressed in milligrams per kilogram (parts per million by mass) is given by the formula :

$$C = \frac{(V_1 - V_0) \times 35{,}5 \times T \times 1\,000}{m_0}$$

where

$V_0$ is the volume, in millilitres, of the silver nitrate solution (4.5) used in the blank test (see 7.4);

$V_1$ is the volume, in millilitres, of the silver nitrate solution (4.5) used in the titration of the test portion (see 7.3);

$T$ is the normality of the silver nitrate solution (4.5) calculated in accordance with 4.5.3;

$m_0$ is the mass, in grams, of the test portion (see 7.1).



## 9 TEST REPORT

The test report shall include the following particulars :

a) reference to this International Standard;

b) the solvent used, if it is different from that indicated in 4.2 (butanone);

c) the complete identification of the material tested;

d) the type of electrode and the method of preparation used, if different from that specified in clause 6;

e) the test result and the method of expression used.

* Designation from EFPA Classification (European Federation of Producers of Abrasives).