INTERNATIONAL STANDARD

ISO 8974

First edition
1988-12-01

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Plastics — Phenolic resins — Determination of residual phenol content by gas chromatography

Plastiques — Résines phénoliques — Dosage du phénol résiduel par chromatographie en phase gazeuse

Reference number
ISO 8974 : 1988 (E)

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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8974 was prepared by Technical Committee ISO/TC 61, *Plastics*. It complies with the provisions of ISO 2718 : 1974, *Standard layout for a method of chemical analysis by gas chromatography*.

Printed in Switzerland

Plastics — Phenolic resins — Determination of residual phenol content by gas chromatography

1 Scope

This International Standard specifies a gas chromatographic method for the measurement of the residual phenol content in phenolic resins.

For the determination of traces of phenol [< 0,5 % (*m*/*m*)], this International Standard must be modified.

For alkaline resins, the method is not usable. The phenol is not totally elutable, because it is in the phenolate state.

IMPORTANT NOTE — The following apparatus and conditions are recommended. However, it is possible to use other apparatus and conditions (for example other detectors and capillary columns) if it has been verified that they give the same results with a precision of the same order.

2 Principle

Dissolution of a test specimen in a suitable solvent and determination of the phenol content by gas chromatography.

3 Materials

3.1 Carrier gas: nitrogen.

3.2 Detector gas: hydrogen.

3.3 Internal standard: *m*-cresol, phenol-free, or anisole, phenol-free.

NOTE — In cases of possible interference, for example presence of cresol in the resin, it is advisable to use anisole as the internal standard.

4 Apparatus

4.1 Chromatograph

Any laboratory chromatograph fitted with a flame ionization detector is suitable (see IMPORTANT NOTE in clause 1).

4.2 Injection device

Microsyringe, to inject approximately 1 µl of the solution prepared in 5.3.1.

4.3 Column

4.3.1 Tube

Type: stainless steel

Length: 2 m

Internal diameter: 2,2 mm

4.3.2 Packing

4.3.2.1 Support: Chromosorb W/AW/DMCS [1], grain size: 150 to 180 µm (80 to 100 mesh).

4.3.2.2 Stationary phase: Carbowax 20 M [2], 10 g per 100 g of dry base material for water-free products. A non-polar stationary phase should be preferred for products containing water or solutions in water, for example Silicon rubber OV 1701 [2].

4.3.3 Conditioning of the column

15 h at 150 °C

4.4 Detector

Flame ionization

1) Chromosorb W/AW/DMCS is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

2) Carbowax 20 M and Silicon rubber OV 1701 are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

4.5 Recorder

Potentiometric

5 Procedure

5.1 Operating conditions

5.1.1 Injector

Temperature: 200 °C

5.1.2 Column

5.1.2.1 Temperature: 150 °C

5.1.2.2 Flow rate: 30 ml/min

5.1.3 Detector

Hydrogen: 30 ml/min

Air: 250 ml/min

5.1.4 Recorder: 1 mV full scale

5.2 Calibration

Determination of the relative correction factor, expressed as the mass of phenol relative to *m*-cresol or anisole.

This correction factor shall be determined using a standard mixture in proportions similar to the solutions to be analysed. It will be valid for all concentrations when operating within the dynamic range of linearity of the detector.

$$F\,(2/1) = \frac{C_2}{C_1} \times \frac{A_1}{A_2}$$

where

$F\,(2/1)$ is the relative correction factor, expressed as the mass of phenol relative to that of the *m*-cresol or anisole;

C_2 and C_1 are the respective concentrations by mass of phenol (2) and *m*-cresol or anisole (1);

A_1 and A_2 are the respective areas of the *m*-cresol or anisole (1) and the phenol (2) peaks.

5.3 Determination

5.3.1 Preparation of the test solution

The solvent used for dissolving the sample should preferably be acetone. In certain cases, methanol, toluene or a 50 % (V/V) toluene/acetone mixture may be used without prejudicing the chromatographic separation.

Unless the resin is found to contain material, particularly *m*-cresol, which interferes with the use of *m*-cresol as an internal reference (in which case anisole free from phenol shall be used as the internal reference), use as the internal reference *m*-cresol which has been shown to be free from phenol.

NOTE — Depending on the concentration, dilution may be necessary. For example, for a concentration between 0,5 and 5 % (m/m), weigh 3 g (m_0) of resin, then 0,25 g (m_1) of *m*-cresol or anisole, into 10,00 ml of acetone.

5.3.2 Introduction of the test portion

Inject, using the microsyringe, approximately 1 µl of the solution prepared in 5.3.1.

5.3.3 Recording

Make the recording. Measure the peak area with a planimeter or an integrator.

6 Expression of results

6.1 Method of calculation

The residual phenol content, expressed as a percentage by mass, as phenol, is given by the formula

$$\frac{m_1}{m_0} \times F\,(2/1) \times \frac{A_2}{A_1} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the internal reference (*m*-cresol or anisole);

$F\,(2/1)$, A_1 and A_2 have the same meanings as in 5.2.

6.2 Precision (test error)

6.2.1 Residual phenol ⩽ 2 % (m/m)

Repeatability 0,1 % (m/m)

Reproducibility 0,2 % (m/m)

6.2.2 Residual phenol > 2 % (m/m)

Repeatability 5 % (relative)

Reproducibility 10 % (relative)

7 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) complete identification of the resin tested;

c) the test conditions, if different from those specified;

d) the result of the test, as specified in clause 6.

UDC 678.632 : 543.544.25 : 547.56

Descriptors : plastics, resins, phenoplasts, chemical analysis, determination of content, chemical residues, phenol, gas chromatography.

Price based on 2 pages