SLOVENSKI STANDARD
SIST EN ISO 8974:1999

01-maj-1999

Polimerni materiali ï Fenolne smole ï Določevanje deleža nezreagiranega fenola s plinsko kromatografijo (ISO 8974:1997)

Plastics - Phenolic resins - Determination of residual phenol content by gas chromatography (ISO 8974:1997)

Kunststoffe - Phenolharze - Bestimmung des freien Phenols durch Gas-Chromatographie (ISO 8974:1997)

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Plastiques - Résines phénoliques - Dosage du phénol résiduel par chromatographie en phase gazeuse (ISO 8974:1997)

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Ta slovenski standard je istoveten z: **EN ISO 8974:1999**

ICS:

83.080.10	Duromeri	Thermosetting materials

SIST EN ISO 8974:1999 **en**

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 8974

February 1999

ICS 83.080.10

Supersedes EN ISO 8974:1995

English version

Plastics - Phenolic resins - Determination of residual phenol content by gas chromatography (ISO 8974:1997)

Plastiques - Résines phénoliques - Dosage du phénol résiduel par chromatographie en phase gazeuse (ISO 8974:1997)

Kunststoffe - Phenolharze - Bestimmung des freien Phenols durch Gas-Chromatographie (ISO 8974:1997)

This European Standard was approved by CEN on 10 February 1999.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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REPUBLIKA SLOVENIJA
MINISTRSTVO ZA ZNANOST IN TEHNOLOGIJO
Urad RS za standardizacijo in meroslovje
LJUBLJANA
SIST... EN ISO 8974 ...
PREVZET PO METODI RAZGLASITVE
-05- 1999

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Foreword

The text of the International Standard from Technical Committee ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard replaces EN ISO 8974:1995.

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 August 1999, and conflicting national standards shall be withdrawn at the latest by August 1999.

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, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

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The text of the International Standard ISO 8974:1997 has been approved by CEN as a European Standard without any modification.

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INTERNATIONAL STANDARD

ISO 8974

Second edition
1997-12-01

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

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

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Reference number
ISO 8974:1997(E)

SIST EN ISO 8974:1999

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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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International Standard ISO 8974 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 8974:1988), which has been technically revised.

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Annex A forms an integral part of this International Standard.

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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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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 of phenolic resins. Alkaline resins (i.e. those containing alkali-metal phenolates) with a pH > 7 may be determined using the modified method given in annex A.

2 Principle

A test specimen is dissolved in a suitable solvent and the phenol content is determined by gas chromatography.

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The apparatus, materials and conditions described are suitable. However, it is possible to use other apparatus and conditions (for example detectors and columns) if it has been verified that they give the same results with a precision of the same order of magnitude. In the procedure described, capillary columns and a flame ionization detector are used.

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3 Materials

3.1 **Carrier gas:** hydrogen, helium or nitrogen.

3.2 **Detector gas:** hydrogen and air.

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

NOTE - If it considered that interference is likely, caused for example by the presence of cresol in the resin, it is advisable to use anisole or octanol as the internal standard.

4 Apparatus

4.1 Chromatograph

4.1.1 **Microsyringe,** capable of injecting approximately 0,5 µl of test solution (see 5.3.1).

4.1.2 **Injection port,** with glass liner to retain non-volatile compounds.

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4.1.3 **Capillary column**

Type: Quartz capillary
Length: 25 m
Internal diameter: 0,32 mm
Stationary phase: e.g. Permabond OV-1701 (cyanopropylphenylmethylsilane)[1]. Condition for 2 h at 250 °C ± 10 °C before use.

4.1.4 **Flame ionization detector**

4.2 **Data-processing unit,** with built in printer-plotter.

5 Procedure

5.1 Operating conditions

Injection-port temperature: 200 °C ± 10 °C

Column temperature: 130 °C ± 2 °C

Carrier-gas flow conditions (for hydrogen): approx. 6 bar, split 1:30
(1 bar = 10^5 N/m^2 = 0,1 MPa)

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Detector temperature: 300 °C ± 10 °C

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Flow rates of flame gases:
hydrogen: 40 ml/min ± 1 ml/min
air: 400 ml/min ± 10 ml/min

Integrator programmed with necessary data from 5.2 and 6.1

5.2 Calibration

Determine the correction factor, expressed as the mass of phenol relative to that of the internal standard, using a standard mixture containing proportions similar to the solution to be analysed. The correction factor will be valid for all concentrations within the linearity range of the detector.

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

1) Permabond OV 1701 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.

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where

F(2/1) is the correction factor, expressed as the mass of phenol relative to that of the internal standard;

C_2 and C_1 are the concentrations by mass of phenol (2) and the internal standard (1), respectively;

A_1 and A_2 are the areas of the internal-standard (1) and phenol (2) peaks, respectively.

5.3 Determination

5.3.1 Preparation of test solution (see also annex A)

The preferred solvent for dissolving the sample is acetone. Methanol, toluene or a 50% (*V*/*V*) toluene/acetone mixture may also be used without prejudicing the chromatographic separation.

Use as the internal reference *m*-cresol which has been shown to be free from phenol 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 use anisole free from phenol, or octanol, as the internal reference,

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Example:

For a phenol concentration between 0,5 % (*m*/*m*) and 5 % (*m*/*m*), weigh, to the nearest 1 mg, 0,5 g of resin (m_0) then 0,05 g of internal standard (m_1) into 10 ml of acetone. For other phenol concentrations, see the following table.

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Phenol content % (*m*/*m*)	Mass of resin g
< 0,5	1
0,5 to 10	0,5
> 10	0,25

5.3.2 Injection of test portion

Using the microsyringe, inject approximately 0,5 µl of the test solution prepared in 5.3.1

5.3.3 Recording of results

This will be done automatically by the integrator.

5.3.4 Number of determinations

Carry out three determinations.