Plast – Fenolharts – Bestämning av restfenolhalt med gaskromatografi
(ISO 8974:2002)

Plastics – Phenolic resins – Determination of residual phenol content by gas chromatography
(ISO 8974:2002)


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Plastics - Phenolic resins - Determination of residual phenol content by gas chromatography (ISO 8974:2002)

Plastiques - Résines phénoliques - Dosage du phénol résiduel par chromatographie en phase gazeuse (ISO 8974:2002)


This European Standard was approved by CEN on 2 April 2002.

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Foreword

This document (EN ISO 8974:2002) has been prepared by Technical Committee ISO/TC 61 “Plastics” in collaboration with 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of an identical text or by endorsement, at the latest by November 2002, and conflicting national standards shall be withdrawn at the latest by November 2002.

This document supersedes EN ISO 8974:1999.

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, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 8974:2002 has been approved by CEN as a European Standard without any modifications.

NOTE Normative references to International Standards are listed in annex ZA (normative).
Plastics — Phenolic resins — Determination of residual phenol content by gas chromatography

1 Scope

This International Standard specifies two methods, using gas chromatography, of measuring the residual phenol content of phenolic resins.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard 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 1042:1998, Laboratory glassware — One-mark volumetric flasks
ISO 10082:1999, Plastics — Phenolic resins — Classification and test methods

3 Principle

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

4 Terms and definitions

For the purposes of this International Standard, the terms and definitions contained in ISO 10082 and the following terms and definitions apply.

4.1 internal standard
reference material added in known quantity to the test solution to determine the response factor for the phenol

4.2 mass ratio
ratio of the mass of the phenol to the mass of an internal standard in the test solution

4.3 peak area ratio
ratio of the phenol peak area to the internal-standard peak area

NOTE In the chromatogram, the phenol peak area is the area contained within the phenol peak and the baseline. The internal-standard peak area is the area contained within the internal-standard peak and the baseline.
4.4 response factor
response for the phenol compared to the response for the internal standard:

\[ K = \frac{\text{Mass ratio}}{\text{Peak area ratio}} \]

4.5 retention time
time between injection of the solute and the vertex of the supposed symmetrical peak

5 Reagents and materials

During the analysis, use only reagents of recognized analytical grade (as listed in ISO 6353-2) except where otherwise stated.

5.1 Carrier gas: hydrogen, nitrogen or helium, chromatographic grade.

NOTE For safety reasons, the use of helium or nitrogen is recommended.

5.2 Detector gas: hydrogen and compressed air.

5.3 Internal standard: any product which is not usually a constituent of phenolic resins and which meets the following requirements:

— is chemically pure;
— has good long-term stability;
— is chemically inert to phenolic resins under the test conditions;
— does not interfere with any of the constituents of the sample;
— gives a linear response within the range of concentrations to be measured;
— preferably has a retention time close to that of the phenol and gives a Gaussian peak.

Examples of reagents which meet the above-mentioned requirements are:

— 1-octanol (especially recommended);
— 1-undecanol.

5.4 Solvent: any product which will completely dissolve the internal standard and the test portion of phenolic resin to give a stable solution, and which meets the following requirements:

— is inert (does not react with the resin or the internal standard under the test conditions);
— is compatible with the constituents of the chromatographic column;
— has a retention time different from those of the phenol and the internal standard.
Examples of solvents which meet the above-mentioned requirements are:

— acetone (recommended);
— methanol;
— ethanol.

6  Apparatus

6.1 Chromatograph: any laboratory chromatograph fitted with a flame ionization detector.

NOTE  Examples of apparatus and the operating conditions recommended are shown below. It is possible to use different procedures providing it can be verified that they would give the same results.

6.2 Microsyringe, capable of injecting 0,5 µl to 1 µl.

6.3 Precolumn/injection-chamber insert: any device capable of trapping all the non-volatile constituents present in the test portion. Suitable are:

— an empty quartz tube or one filled with a stationary phase or glass wool (depending on the type of column);
— a Vigreux column type quartz tube.

For examples, see annex A.

6.4 Column

6.4.1 Column characteristics

Any column with characteristics (length, diameter, stationary phase) which will ensure resolution of the volatile constituents (phenol, internal standard, solvent) is suitable.

WARNING — As in any chromatographic analysis, one constituent of the product being analysed may have the same elution time as the phenol. Some technical procedures can solve the problem, for example by using a column with a different polarity or by using a modified temperature programme.

For examples, see annex A.

NOTE  Capillary columns with a resolution higher than that of packed columns allow the determination of residual phenol contents of less than 0,5 % by mass.

6.4.2 Conditioning of the column

Condition the column in accordance with the manufacturer's instructions.

6.5 Recorder: various types may be used:

— potentiometric recorder;
— integrator;
— computerized data-acquisition system.

6.6 Balance, accurate to 0,1 mg.

6.7 Pipette, capacity 10 ml.