INTERNATIONAL STANDARD

ISO 8974

Second edition
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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 8974:1997( )

Foreword

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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.

Annex A forms an integral part of this International Standard.

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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.

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.

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.

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)

Detector temperature: 300 °C ± 10 °C

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.

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,

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.

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.

6 Expression of results

6.1 Method of calculation

The residual phenol content, expressed (as phenol) as a percentage by mass, 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, anisole or octanol);

$F(2/1)$, A_1 and A_2 are as defined in 5.2.

6.2 Precision

6.2.1 Repeatability (*r*)

The value below which the difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method may be expected to lie with a 95 % probability is 5 % (relative).

6.2.2 Reproducibility (*R*)

The value below which the difference between two test results, each the mean of duplicates, obtained on identical materials by operators in different laboratories using the standardized test method may be expected to lie with a 95% probability is 10 % (relative).

7 Test report

The test report shall include the following information:

a) a reference to this International Standard;
b) all details necessary for complete identification of the resin tested;
c) the test conditions, if different from those specified;
d) the individual results, as specified in clause 6, and their average;
e) the date of the test.

Annex A
(normative)

Modified method for alkaline resins

Neutralize alkaline resins when preparing the test solution, and then carry out the determination as with acid resins.

Example:

Weigh, to the nearest 1 mg, 2,5 g of alkaline resin into a 50 ml glass flask. Add 30 ml of methanol, dissolve the test portion with stirring and adjust the pH to 6,5 to 7,0 with 15 % sulfuric acid, using a pH-meter.

Weigh a quantity of internal standard depending on the expected phenol content into the flask (see 5.3.1).

Continue the procedure as described in 5.3.2.

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