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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
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

This third edition cancels and replaces the second edition (ISO 8974:1997), which has been technically revised.

Annexes A and B of this International Standard are for information only.

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 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series*

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.

6.8 Volumetric flask, capacity 250 ml, 500 ml or 1 000 ml (see ISO 1042).

6.9 Beaker, capacity 50 ml or 100 ml.

6.10 Conical flasks with ground-glass necks, capacity 100 ml.

7 Procedure

7.1 General

This International Standard describes two methods for the determination of the residual phenol content by gas chromatography:

- Method A: a weighed amount of internal standard is added to each test portion (this method is the reference method).
- Method B: a stock solution of internal standard is used (this method is useful for routine analysis).

7.2 Operating conditions

7.2.1 Injector

Set the temperature at a fixed value, between 180 °C and 250 °C, compatible with the temperature recommended for normal use of the precolumn/insert. The injector is usually set at about 50 °C above the boiling point of the phenol.

For examples, see annex A.

7.2.2 Column

The aim is to ensure optimum resolution of the volatile constituents.

The column can be operated isothermally or using a temperature programme.

For examples, see annex A.

7.2.3 Detector

Set the temperature at a fixed value, between 230 °C and 300 °C, compatible with the temperature recommended for normal use of the detector.

For examples, see annex A.

7.3 Calibration

7.3.1 General

Follow this procedure for both method A and method B.

7.3.2 Preparation of calibration solutions

Into a series of at least six conical flasks (6.10) weigh, to the nearest 0,1 mg, suitable quantities of the phenol and the internal standard such that the mass ratio is between 3:1 and 1:3.

Dilute with solvent (5.4) to a concentration suitable for the column being used, taking account of the following:

- for a packed column, the phenol content shall be of the order of 10 mg/ml;

- for a megabore column with a diameter of 0,5 mm, the phenol content shall be of the order of 5 mg/ml when using a split injection technique or 0,5 mg/ml without a splitter;
- for a capillary column with a diameter of 0,2 mm, the phenol content shall be of the order of 1 mg/ml when using a split injection technique or 0,5 mg/ml without a splitter.

7.3.3 **Preparation of the calibration curve** (see annex B for an example)

Using the microsyringe (6.2), inject the solutions prepared in 7.3.2. Use at least six standard solutions for the initial calibration. Average the results of at least two injections for each solution.

Record the peaks and measure the peak areas.

Plot the peak area ratio against the mass ratio. The values should lie on a straight line passing through the origin. If required, the degree of fit and the absence of a significant intercept can be checked using a weighted least-squares regression technique.

Average the ratios of peak area ratio/mass ratio. Take the inverse of the result as the response factor. The response factor will allow subsequent conversion of peak area ratios to mass ratios.

The calibration should preferably be checked periodically by injection of two or three standard solutions. Calculate the response factor as described above and plot it against time. The calculated average response factor should agree with the original value to within ± 2 %. A wider variation will require full recalibration of the apparatus. As already noted, it is advisable to do this periodically anyway, and certainly if any change is made in the column/detector system or the recorder.

7.4 Determination

7.4.1 Method A

7.4.1.1 Preparation of test solution

Weigh a test portion of resin, to the nearest 0,1 mg, into a conical flask (6.10). Add a quantity of internal standard (5.3) such that the mass ratio lies between 3:1 and 1:3. The ideal ratio is about 1:1. Add solvent (5.4) in order to obtain a phenol concentration as indicated in 7.3.2.

Table 1 — Examples of suitable test solution compositions

Phenol content of sample % by mass	**Mass of test portion** g	**Mass of internal standard** mg
$\leqslant 1$	2 to 5	25
> 1 but $\leqslant 10$	0,5 to 5	50
> 10	0,1 to 0,5	50

7.4.1.2 Injection

Using the microsyringe (6.2), inject the volume required for the column being used.

Record the peaks and measure the peak areas. Average the results of at least two injections of each solution. Make sure, as previously indicated, that no constituent of the resin has the same elution time as the phenol or the internal standard.