

INTERNATIONAL
STANDARD

ISO
8618

Second edition
1995-12-15

**Plastics — Liquid phenolic resins —
Conventional determination of non-volatile
matter**

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*Plastiques — Résines phénoliques liquides — Détermination de l'extrait
sec conventionnel*

ISO 8618:1995

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Reference number
ISO 8618:1995(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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 8618 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 8618:1987) which has been revised to include an air circulation oven as an alternative to the static convection oven.

Annex A forms an integral part of this International Standard.

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Plastics — Liquid phenolic resins — Conventional determination of non-volatile matter

1 Scope

This International Standard specifies a method for the conventional determination of the non-volatile matter of liquid phenolic resins (resols, novolak solutions, etc.). It can be used for commercial products or for resins in their various stages of manufacture.

NOTE 1 For phenolic resins, the term "non-volatile matter" is based upon arbitrary test conditions (see 4.2, note 3). Results obtained by this method may not agree with the results obtained in industrial applications of these resins.

2 Definition

For the purpose of this International Standard, the following definition applies.

2.1 non-volatile matter: The percentage residue obtained when the volatile components of a specific mass of a liquid phenolic resin are evaporated under specified conditions of temperature and time.

3 Apparatus

3.1 Small disposable dishes made of aluminium, nickel, stainless steel, tinfoil or galvanized sheet metal, with a flat base, an inner diameter at the base of 75 mm and a height of approximately 25 mm.

NOTE 2 Dishes of different diameters may be used if the mass of the test portion m_1 , in grams, is calculated according to the following relation:

$$\frac{m_1}{A} = \frac{3}{4\,412} = 0,000\,68 \text{ g/mm}^2$$

where A is the area, in square millimetres, of the dish base.

3.2 Precision balance, accurate to 1 mg.

3.3 Static convection oven, with natural ventilation, with a perforated metal shelf placed at one-third of the height of the oven and capable of being maintained at the test temperature (see 4.2).

or

Air circulation oven, with horizontal ventilation, having an air circulation speed of 0,8 m/s to 1,2 m/s.

The type of oven used shall be stated in the test report since the results of the test depend on the type used (static convection or air circulation).

3.4 Desiccator, containing dehydrated calcium chloride or other suitable drying agent.

4 Procedure

4.1 Degrease a small dish (3.1), dry it in the oven (3.3), maintained at 135 °C (or the selected test temperature, see 4.2), and store it in the desiccator (3.4) until used.

Determine the mass (m_0) of the clean dry dish to the nearest 1 mg. Transfer 3,0 g ± 0,5 g of completely homogenized resin into the dish and record the mass (m_1), to the nearest 1 mg, of the test portion therein (see 3.1, note 2).

4.2 Place the dish at the centre of the shelf in the oven, heated to the preferred test temperature of 135 °C ± 1 °C. An alternative test temperature (see note 3) may be used and shall be recorded in the test report. Recommended alternative temperatures are 120 °C ± 1 °C and 150 °C ± 1 °C (see note 4 and annex A).

NOTES

3 The test temperature must be such that the volatile components evaporate without decomposition of the resin. When selecting the temperature, possible reactions between the individual components and polycondensation should also be considered.

4 When testing is performed at 150 °C, a slightly different procedure may be used (dissolving the test portion in a solvent). (See annex A.)

4.3 After 1 h ± 5 min at the selected test temperature, remove the dish from the oven, allow it to cool for at least 1 h in the desiccator and determine the mass (m_2), to the nearest 1 mg, of the dish plus the non-volatile matter of the test portion.

4.4 Carry out a duplicate determination simultaneously.

As the temperature is not the same in all parts of the oven at all times, place the two dishes at the same height and next to one another.

5 Expression of results

5.1 Calculate the non-volatile matter NV, as a percentage by mass, using the equation

$$NV_{\theta, 1h} = \frac{m_2 - m_0}{m_1} \times 100$$

where

m_0 is the mass, in grams, of the dish (see 4.1);

m_1 is the mass, in grams, of the test portion (see 4.1);

m_2 is the mass, in grams, of the dish plus the non-volatile matter of the test portion (see 4.3);

θ is the test temperature, in degrees Celsius (see 4.2).

5.2 Calculate the arithmetic mean (to one decimal place) of the two determinations. If the difference between the two determinations is more than 5 % in relative value, repeat the test.

6 Test report

The test report shall include the following particulars:

- a reference to this International Standard;
- all details necessary for complete identification of the resin tested;
- the type of dish used;
- the test temperature;
- the type of oven used;
- if necessary, the solvent used (test at 150 °C; see annex A);
- the results of the individual determinations and their arithmetic mean.

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Annex A (normative)

Special procedures for testing at 150 °C

WARNING — If the oven used is not an explosion-proof one, the determination of non-volatile matter at 150 °C ± 1 °C using solvent must not exceed the use of two small dishes. Fire and/or explosion could result if the number of test portions in small dishes were to be increased beyond two.

A.1 Solvents

0,05 g and record the mass (m_1), to the nearest 1 mg, of the test portion in the dish.

A.1.1 Distilled toluene.

Dissolve the resin in 5 ml of distilled toluene (A.1.1) or isopropanol (A.1.2). If the resin does not dissolve in these solvents, use methyl ethyl ketone (A.1.3).

A.1.2 Isopropanol.

A.1.3 Methyl ethyl ketone.

NOTE 5 Methyl ethyl ketone cannot be used with resols.

The dissolution process can be speeded up by heating briefly in the oven (10 s to 15 s).

A.2 Procedure

Predry a dish (3.1) in the oven (3.3), maintained at 150 °C, allow to cool to ambient temperature and store in the desiccator (3.4).

Determine the mass (m_0) of the clean dry dish to the nearest 1 mg. Transfer sufficient resin into the dish so that the expected non-volatile matter will be 0,5 g ±

When the resin has dissolved and is distributed evenly over the base of the dish, place the dish in the oven, maintained at 150 °C ± 1 °C, so that the dish is horizontal and heat it for 2 h. Remove the dish from the oven and allow it to cool at ambient temperature in the desiccator. Determine the mass (m_2), to the nearest 1 mg, of the dish plus the non-volatile matter of the test portion. Continue as in 4.4.

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