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

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

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Foreword

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

1 Scope and field of application

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 — For phenolic resins, the term "non-volatile matter" is based upon arbitrary test conditions (see 4.2, note 1). 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 is applicable.

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, tinplate 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 — 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, and with a perforated metal shelf placed at one-third of the height of the oven, and capable of being controlled at the test temperature (see 4.2).

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), controlled 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 ± 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 of resin therein (see the note in 3.1).

4.2 Place the dish at the centre of the shelf in the oven, heated to the preferred test temperature of 135 ± 1 °C. An alternative test temperature (see note 1) may be used and shall be recorded in the test report. Recommended alternative temperatures are 120 ± 1 °C and 150 ± 1 °C (see note 2 and annex).

NOTES

1 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.

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

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 of the resin.

4.4 *Make a duplicate determination simultaneously.*

NOTE — As the temperature is not the same in all parts of the oven at all times, the two dishes should be placed 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

$$\text{NV}_{\theta\,°\text{C},\,1\,\text{h}} = \frac{m_2 - m_0}{m_1} \times 100$$

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 test results 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;

b) complete identification of the resin tested;

c) type of dish used;

d) test temperature;

e) if necessary, the solvent used (test at 150 °C; see the annex);

f) individual test results and the arithmetic mean.

Annex

Special procedures for testing at 150 °C

(This annex forms an integral part of the Standard.)

WARNING — If no explosion-proof class of oven is used, the determination of non-volatile matter performed at 150 ± 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

A.1.1 Distilled toluene.

A.1.2 Isopropanol.

A.1.3 Methyl ethyl ketone.

A.2 Procedure

Predry a dish (3.1) in the oven (3.3), controlled 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 ± 0,05 g and record the mass (m_1), to the nearest 1 mg, of the test portion of the resin in the dish.

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

NOTE — Methyl ethyl ketone cannot be used with resols.

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

When the resin has dissolved and is distributed evenly over the base of the dish, place the dish in the oven, controlled at 150 ± 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 of the resin. Continue as in 4.4.

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