INTERNATIONAL STANDARD

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Plastics — Phenolic resins — Determination of the pseudo-adiabatic temperature rise of liquid resols when cured under acid conditions

Plastiques — Résines phénoliques — Détermination de l'élévation de température pseudoadiabatique des résols liquides thermodurcis en conditions acides

Reference number
ISO 9771:1995(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 9771 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

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Plastics — Phenolic resins — Determination of the pseudo-adiabatic temperature rise of liquid resols when cured under acid conditions

1 Scope

This International Standard specifies a method for determining the exothermic reactivity of thermosetting liquid phenolic resins when mixed with an acid hardener under specified conditions. The results of the determination are used as a means of assessing the behaviour of the resin during processing.

2 Principle

A phenolic resin is mixed with an acid hardener and allowed to harden. The highest temperature reached is measured, as well as the time taken to reach this temperature.

3 Reagent

3.1 Suitable acid hardener, depending on the composition of the resin, e.g. *p*-phenol sulfonic acid, technical grade, 65 % $(m/m) \pm 1$ % (m/m) in water.

4 Apparatus (see figure 1)

4.1 Reaction vessel: paper cup impregnated with polyethylene, with the following dimensions:

— diameter at base: about 60 mm

— diameter at rim: about 70 mm

— height: about 60 mm

4.2 Hollow foam block, made of phenolic-resin or polyurethane-resin foam with an apparent density of 30 kg/m^3 to 50 kg/m^3 and with a cavity to accommodate the reaction vessel (4.1).

The cavity shall be arranged so that the surface of the reaction mixture lies about 30 mm below the top edge of the foam block and so that the reaction vessel sits firmly in the foam material.

The thickness of the thermal-insulation layer between the reaction vessel and its surroundings shall be at least 60 mm.

NOTE 1 It is good practice to change the foam block periodically since wear of the block can affect the test result.

4.3 Non-metallic cover plate, for the foam block (4.2).

4.4 Thermocouple, with its hot junction immersed in glycol or other suitable liquid at the bottom of a test tube (4.7).

4.5 Temperature recorder.

4.6 Pipette, nominal capacity 10 ml.

4.7 Test tube, 60 mm × ⌀ 16 mm, containing 2 ml of glycol or other suitable liquid.

4.8 Stirring device: mechanical or hand stirrer.

4.9 Balance, accurate to 0,2 g.

Dimensions in millimetres

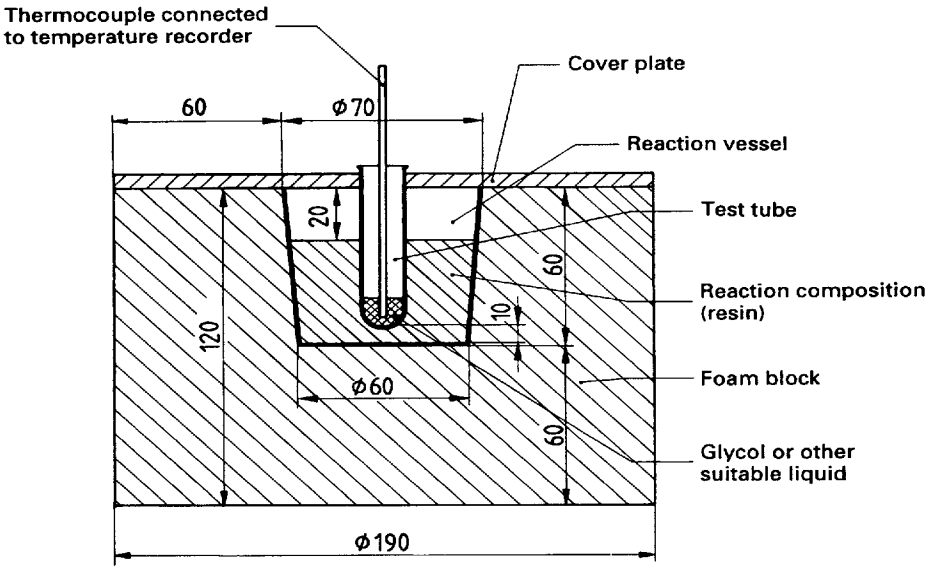

Figure 1 — Schematic diagram of apparatus

5 Procedure

Weigh 100 g ± 0,5 g of the resin sample, kept at a temperature of 23 °C ± 0,2 °C, into the reaction vessel (4.1). Condition the reaction vessel containing the test portion at 23 °C ± 0,2 °C, and place it in the foam block (4.2). Add 10 ml of hardener, also kept at 23 °C ± 0,2 °C, in one portion, using the pipette (4.6). Immediately after adding the hardener, start the temperature recorder (4.5) and mix resin and hardener thoroughly by stirring for 35 s. Other mixing times may be used if necessary, but they shall be mentioned in the test report. After mixing thoroughly, remove the stirrer and replace it by the test tube (4.7) containing the thermocouple (4.4). Lower the test tube with the thermocouple through the cover plate (4.3) so that it dips into the reaction mixture with the bottom of the test tube located in the middle of the foam block, 1 cm above the bottom of the beaker.

If a starting temperature of 23 °C does not produce a distinct temperature peak, use a higher temperature for both the resin and the hardener.

If the resin is too reactive, use a smaller amount of catalyst.

By means of the temperature recorder, record

a) the maximum temperature reached;

b) the time to reach the maximum temperature.

Carry out the procedure twice.

6 Expression of results

6.1 Calculation

Calculate the mean of the measurements of maximum temperature, in degrees Celsius, and the mean of the measurements of the time, in minutes, taken to reach each maximum temperature, and report these means as the results.

If variations greater than 5 % occur, repeat the test.

6.2 Precision (test error)

Maximum temperature:

Repeatability: ±3 %
Reproducibility: ±5 %

Time to reach maximum temperature:

Repeatability: ±5 %
Reproducibility: ±5 %

7 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the resin tested;

c) the starting temperature, in degrees Celsius;

d) the maximum temperature reached, in degrees Celsius;

e) the time, in minutes, taken to reach the maximum temperature;

f) the mixing time used.

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Descriptors: plastics, thermosetting resins, phenoplasts, tests, chemical tests, determination, chemical reactivity, temperature measurements.

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