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Plastics — Phenolic resins — Determination of the gel time at a given temperature using automatic apparatus

*Plastiques — Résines phénoliques — Détermination de la durée de gélification
à une température donnée sur appareils automatiques*

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Foreword

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International Organization for Standardization
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Plastics — Phenolic resins — Determination of the gel time at a given temperature using automatic apparatus

1 Scope

This International Standard specifies two methods for determining the gel time of phenolic resins at a given temperature, using automatic apparatus under specified conditions. The test temperatures are 100 °C, 130 °C and 150 °C.

The two methods specified, which utilize different kinds of automatic apparatus, are:

- a) method A (clause 3), which employs a glass rod driven to carry out a rotary (spinning) motion;
- b) method B (clause 4), which employs a plunger driven to carry out a reciprocating vertical motion.

These methods apply to the following types of phenolic resin:

- liquid resols (methods A and B);
- novolak solutions containing hexamethylenetetramine (HMTA); the boiling point of the solvent must be higher than the test temperature (method A);
- solid resols with low melting points (method B).

Powdered resins with a short gel time, such as mixtures of novolak and HMTA, do not lend themselves to this type of determination. Such resins crosslink as soon as fusion starts, and become too viscous in the test tube to give meaningful results. Resins containing large amounts of a solvent with a low boiling point are especially unsuitable for this type of determination. With such resins, a boiling and distillation process takes place as the resin hardens.

It might be of interest to determine the time taken for the resin to gel in the presence of a catalyst. The catalyst shall be added in well defined proportions. The type of catalyst used and its treatment shall be stated in the test report.

2 Preparation of test portions

2.1 Test liquid resols as produced (method A or B).

2.2 Test solid resols as produced (method B).

2.3 Dissolve powdered resins (such as novolak + HMTA) in the proportions 1 : 1 (*m/m*) in ethylene glycol monoethyl ether

at a temperature below 60 °C. If necessary, additional solvent may be added to dissolve the resin completely (method A).

NOTE — Ethylene glycol monoethyl ether may plastify the resin and give a longer gel time.

2.4 Test solutions of novolak in high-boiling solvents containing HMTA as produced. If the solution does not contain HMTA, add 10 % of HMTA to the solution, based on the novolak content (method A).

2.5 Dissolve solid novolaks in ethylene glycol monoethyl ether and add HMTA in the following proportions by mass (method A):

10 parts of novolak;

1 part of HMTA;

11 parts of ethylene glycol monoethyl ether.

3 Method A: Apparatus with rotary stirrer

3.1 Principle

In the course of polymerization, phenolic resins pass through a critical point which is manifested by an abrupt increase in the viscosity.

The automatic apparatus used measures the time required to reach this point.

A glass rod, placed in a test tube containing the substance to be examined, is driven to carry out a rotary movement of limited torque by an electric motor.

The gel point is reached when the viscosity of the sample increases abruptly and stops the rotation of the glass rod. This causes the closure of an electric circuit, producing an audible signal. The time indicated by the timer is called the gel time at the particular temperature (method A).

3.2 Reagents

3.2.1 Solvent: Ethylene glycol monoethyl ether. (When measuring at 150 °C, ethylene glycol may be used instead.)

3.2.2 Hardener: Finely ground hexamethylenetetramine (HMTA).

3.3 Apparatus

3.3.1 Automatic apparatus for measuring gel time (see figure 1).

3.3.2 Thermostatically controlled bath, kept at the temperature specified for the determination, with for example silicone oil as the heat-transfer medium.

3.3.3 Test tube, with a mass of $10 \text{ g} \pm 1 \text{ g}$ and the following dimensions: internal diameter $16 \text{ mm} \pm 0,2 \text{ mm}$, length $160 \text{ mm} \pm 4 \text{ mm}$.

3.3.4 Balance, accurate to 0,1 g.

3.3.5 Glass rod, $6 \text{ mm} \pm 0,5 \text{ mm}$ in diameter and about 240 mm long.

3.3.6 Laboratory mortar or grinder.

3.4 Procedure

Weigh, to the nearest 0,1 g, 5 g or 10 g of the liquid resin or resin solution into the test tube (3.3.3).

Insert the test tube into the thermostatically controlled bath (3.3.2) (see figure 1) maintained at a temperature of $100 \text{ }^\circ\text{C} \pm 0,5 \text{ }^\circ\text{C}$ or $130 \text{ }^\circ\text{C} \pm 0,5 \text{ }^\circ\text{C}$ or $150 \text{ }^\circ\text{C} \pm 0,5 \text{ }^\circ\text{C}$, as agreed between the parties concerned. Adjust the glass rod (3.3.5) so that its tip is 6 mm from the bottom of the test tube containing the resin or solution. The level of the liquid in the tube shall be 10 mm below the level of the liquid in the thermostatically controlled bath (see figure 1).

Switch on the automatic apparatus as soon as the test tube is inserted in the bath, then set the button of the bell or buzzer to "ON"; the timer will stop automatically when the resin sets, thereby actuating the bell or buzzer.

The speed of rotation of the glass rod stirrer shall be mentioned in the test report.

When the resin has set, remove the tube from the bath and switch off the bell or buzzer. Note the time elapsed. Remove the glass rod from the tube and clean it with a cloth soaked with methanol or acetone.

Take two measurements with different test portions; if there is a difference of more than 5 % between the results obtained, repeat the test.

3.5 Test report

The test report shall contain the following information :

- a) a reference to this International Standard;
- b) the method used (method A);
- c) all details necessary for the complete identification of the sample of resin tested and all details of its pretreatment;
- d) the mass of the test portion (5 g or 10 g);
- e) the arithmetic mean of the individual values measured for the gel time, expressed in minutes to the nearest 0,1 min;
- f) the temperature at which the test was performed;
- g) the speed of rotation used.

4 Method B: Apparatus with vertically reciprocating stirrer

4.1 Principle

A plunger placed in a test tube containing the substance to be examined is driven to carry out a reciprocating motion. The gel time is reached when the viscosity of the specimen is such that the tube is lifted with the plunger, thus stopping a timer.

4.2 Apparatus

4.2.1 Automatic apparatus for measuring gel time (see figure 2). The frequency of reciprocation of the plunger shall be 10 s^{-1} or as otherwise agreed between the parties concerned. The frequency shall be mentioned in the test report.

4.2.2 Thermostatically controlled bath, maximum temperature $200 \text{ }^\circ\text{C}$, tolerance $\pm 0,5 \text{ }^\circ\text{C}$ for the temperature chosen for the test (see 4.3). The density of the bath liquid, e.g. silicone oil, shall be $1 \text{ g/cm}^3 \pm 0,1 \text{ g/cm}^3$.

4.2.3 Test tube, with a mass of $10 \text{ g} \pm 1 \text{ g}$ and the following dimensions: inside diameter $16 \text{ mm} \pm 0,2 \text{ mm}$, length $160 \text{ mm} \pm 4 \text{ mm}$.

4.2.4 Metal ring, weighing 10 g or 20 g, to hold down the test tube (4.2.3) in the bath (4.2.2).

4.2.5 Balance, accurate to 0,1 g.

Dimensions in millimetres

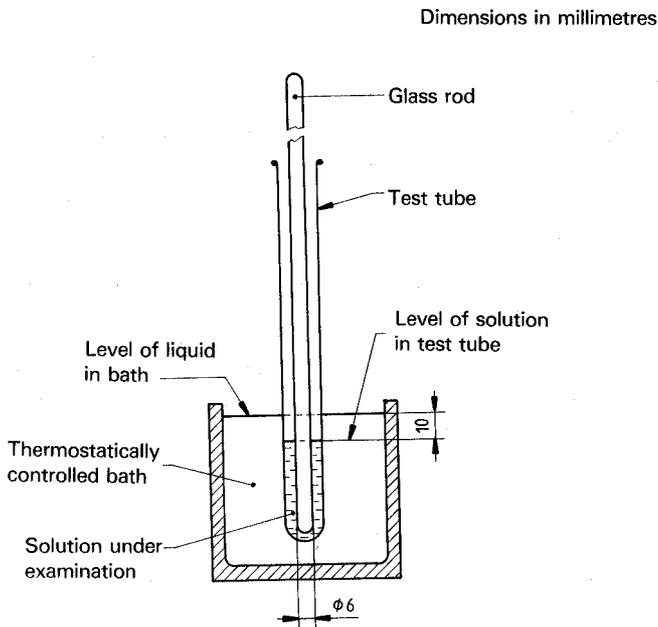
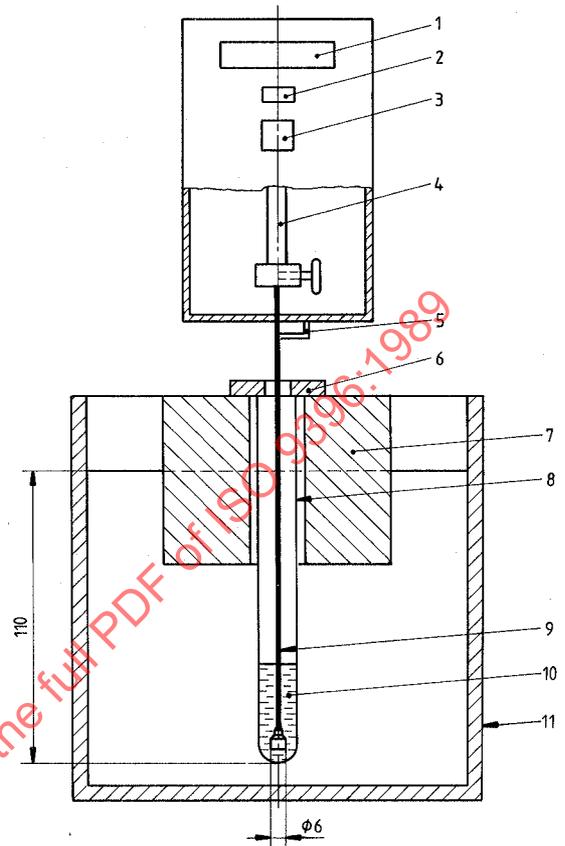


Figure 1 — Rotating-stirrer apparatus for determining gel time



- 1 Electric timer
- 2
- 3 Timer setting button
- 3 Start button for plunger and timer
- 4 Plunger drive motor
- 5 Stop lever for plunger and timer
- 6 Metal ring
- 7 Test tube holder
- 8 Test tube
- 9 Plunger (of material other than aluminium)
- 10 Phenolic resin
- 11 Thermostatically controlled bath containing a liquid suitable for use at the highest temperature employed

Figure 2 — Reciprocating-plunger apparatus for determining gel time

4.3 Procedure

Perform the test in duplicate, or on a greater number of test portions as agreed between the parties concerned.

Weigh $5 \text{ g} \pm 0,1 \text{ g}$ of resin into the test tube (4.2.3). Place the metal ring (4.2.4) round the top of the tube to hold it down in the bath (4.2.2) and to prevent it being lifted prematurely as the viscosity rises before the gel point is reached. The mass of the ring shall be

10 g for liquid resols

or

20 g for solid or powdered resols.

The test temperature shall be 100 °C, 130 °C or 150 °C, as agreed between the parties concerned.

Start up the apparatus (4.2.1) as soon as the test tube containing the resin to be tested is in its holder. In the case of liquid resins, connect up the plunger immediately. For solid resins, it will be necessary to wait until the resin has melted. During the test, the test tube shall remain immersed to a depth of 11 cm

in the liquid in the thermostatically controlled bath, with part of the test tube holder also being immersed.

The gel time is reached when the test tube follows the to-and-fro motion of the plunger, i.e. when the test tube starts to move in its holder. The apparatus will stop automatically when the gel time is reached.

4.4 Test report

The test report shall contain the following information:

- a) a reference to this International Standard;
- b) the method used (method B);
- c) all details necessary for the complete identification of the sample of resin tested;
- d) the arithmetic mean of the individual values measured for the gel time, expressed in minutes to the nearest 0,1 min.
- e) the temperature at which the test was performed;
- f) the frequency of the plunger motion.

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