INTERNATIONAL STANDARD

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Plastics — Unsaturated polyester resins — Measurement of gel time at 25 °C

Plastiques — Résines de polyesters non saturés — Mesurage du temps de gélification à 25 °C

Reference number
ISO 2535:1997(E)

Foreword

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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 2535 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 2535:1974), of which it constitutes a minor (editorial) revision.

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Plastics – Unsaturated polyester resins – Measurement of gel time at 25 °C

1 Scope

This International Standard specifies a method of measuring, under defined conditions, the gel time at 25 °C of unsaturated polyester resins.

This method is applicable to all resins, but it is particularly useful for cold-setting resins.

2 Normative reference

The following standard contains provision which, through reference in this text, constitute provisions of ISO 2535. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on ISO 2535 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 472:1988, *Plastics — Vocabulary*.

3 Definition

For the purposes of this International Standard, the following definition applies:

3.1 gel time: The time required for a liquid material to form a gel under specified conditions of temperature. [ISO 472]

4 Principle

A mixture of resin with specified amounts of standard accelerator and initiator is prepared at 25 °C.

This mixture is placed in a test tube of specified dimensions, which is immersed in a bath thermostatically controlled at 25 °C.

A device, which is designed to have the least possible effect on the viscometric properties of the mixture, is used to indicate the exact time that the viscosity of the mixture reaches 50 Pa·s (500 P) (the viscosity conventionally taken as corresponding to the start of the gel state). The elapsed time between the end of the addition of the initiator and accelerator and the moment when the viscosity reaches 50 Pa·s is conventionally called the "gel time at 25 °C".

NOTE 1 The type and proportion of accelerator and initiator and the temperature given in this International Standard are reference conditions.

In particular cases, however, other conditions may be agreed between the interested parties (see 7.2).

5 Reagents

5.1 Acetone, of purity ≥ 99 %.

5.2 Toluene, of purity ⩾ 99 %.

5.3 Reference accelerator: cobalt octoate solution in toluene.

Weigh into a beaker 5 g ± 0,01 g of a base solution of cobalt octoate containing 6 % of cobalt metal in an inert solvent. Transfer to a 50 ml volumetric flask fitted with a ground-glass stopper. Make up to 50 ml with toluene (5.2).

1 ml of this solution corresponds to 0,100 g of the base solution of cobalt octoate containing 6 % of cobalt metal.

5.4 Reference initiator: 50 % (*m/m*) solution of methyl ethyl ketone peroxide in dimethyl phthalate assaying 9 % of active oxygen.

Store this solution in a refrigerator and use it within 1 month of preparation or receipt.

NOTE 2 Commercial methyl ethyl ketone peroxide is a mixture of isomers in variable proportions, and two commercial products assaying the same percentage of active oxygen may give different test results (see clause 10).

WARNING — In no circumstances should the methyl ethyl ketone peroxide and cobalt octoate solutions be mixed together, as an explosive mixture is formed. Mix each separately into the polyester resin.

6 Apparatus

6.1 Borosilicate-glass test tube, having an internal diameter of 18 mm, a length of 180 mm and a wall thickness of 1,2 mm ± 0,2 mm, fitted with a stopper, to hold the test mixture.

6.2 Viscosity-measuring device, to measure the viscosity of the mixture in the tube.

The device shall be such that it has the least possible effect on the rheological properties of the mixture.

NOTE 3 A description of a suitable device (see figure 1) is given below as an example:

A glass rod, 6 mm in diameter and of sufficient length, is immersed in the mixture to a depth of 50 mm. The rod is rotated about its axis at a slow speed (1 r/min to 2 r/min) by means of a torsion wire driven by a geared electric motor.

When the torsion wire is twisted to the angle corresponding to a viscosity of about 50 Pa·s (500 P), the time is taken. An automatic device may also be used to stop the motor as well as the stopwatch and signal the end of the test.

6.3 Bath, thermostatically controlled at 25 °C ± 0,5 °C and protected from light.

6.4 Beaker, capacity 100 ml.

6.5 Two graduated pipettes, capacity 1 ml, graduated in 0,01 ml and **clearly marked to distinguish one from the other**.

6.6 Balance, accurate to within 0,1 g.

6.7 Spatula, stainless steel.

6.8 Stopwatch, accurate to 1 s.

7 Procedure

7.1 Determination

Clean the test tube (6.1) with acetone (5.1), dry and stopper it, and then immerse it to a depth of about 80 mm in the bath (6.3) thermostatically controlled at 25 °C.

Weigh 50 g ± 0,1 g of resin into the beaker (6.4), place the latter in the bath at 25 °C and wait a sufficient time for the beaker and its contents to reach that temperature.

Dimensions in millimetres

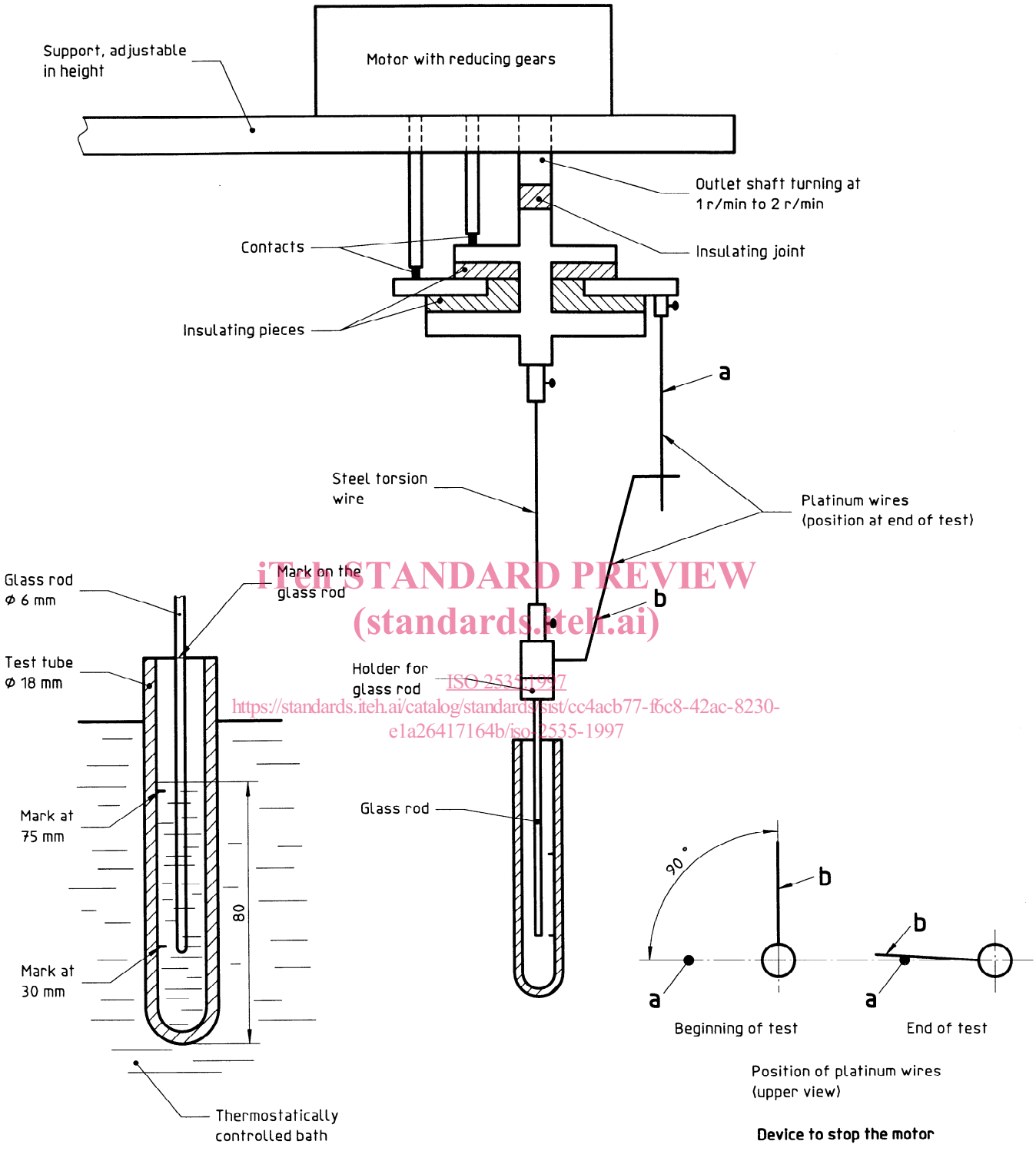

Figure 1 — Example of a suitable apparatus for the measurement of gel time using a rotating glass rod

Using one of the pipettes (6.5), add 0,50 ml of cobalt octoate solution (5.3) to the resin and mix with the spatula (6.7).

Using the other pipette, add 0,70 ml of methyl ethyl ketone peroxide solution (5.4) to the mixture, start the stopwatch (6.8) and mix with the spatula for 30 s.

Remove the test tube from the bath, unstopper it and transfer to it sufficient quantity of the mixture to ensure that the tube is filled to a depth of about 80 mm when the viscosity-measuring device (6.2) is in position. Avoid wetting the sides of the tube above this level.

Immerse the test tube in the thermostatic bath at 25 °C, so that the level of the surface of the resin in the tube is 2 cm below that of the liquid in the bath.

Place the viscosity-measuring device in position.

Stop the stopwatch when the viscosity reaches about 50 Pa·s (500 P). Record the period indicated by the stopwatch to the nearest 0,1 min.

Remove the viscosity-measuring device from the test tube, clean the assembly thoroughly with acetone, and dry.

Carry out a second determination under the same conditions with another test tube.

If the two results differ by more than 10 %, carry out more determinations until two consecutive results do not differ by more than 10 %.

If the device described in note 3 in 6.2 and illustrated in figure 1 is used, proceed as follows:

After cleaning the test tube, mark the outside of the tube at 30 mm and 75 mm from the bottom with a grease crayon, stopper it and place it in the bath.

Clean the glass rod with acetone and dry it. Insert the glass rod into the test tube so that its tip is 30 mm from the bottom of the tube, and make a mark on the rod with a grease crayon level with the top of the test tube.

Pour the mixture, prepared in the beaker, into the test tube up to the 75 mm mark.

Immerse the test tube in the bath as indicated. Fix the tube in the vertical position and place the glass rod in the mixture in such a way that the mark on the rod is level with the top edge of the tube. Adjust the position of the tube so that the glass rod is placed correctly along the axis of the test tube. Start the motor. When the viscosity of the mixture rises to the predetermined value, stop the motor and stopwatch and continue as described.

7.2 Notes on the procedure

The test temperature of 25 °C and the concentration of 0,1 % of cobalt octoate (6 % Co) and 1,4 % of methyl ethyl ketone peroxide (9 % active oxygen) given in this International Standard correspond to the conditions most commonly used in practice, and are the reference conditions.

Nevertheless, for certain resins and/or for certain applications, it can sometimes prove useful

— to carry out the determination at 20 °C or 30 °C;

— to use different concentrations, lower or higher, of the above reagents;

— to use other accelerator and/or initiator systems.

These different conditions will normally be agreed between the interested parties.

8 Expression of results

Calculate the arithmetic mean of the two results obtained, and round to the nearest 0,1 min. This mean value, rounded in this way, is the gel time at 25 °C.

9 Precision

The precision of this method is not known because interlaboratory data are not available. Interlaboratory data are being obtained, and a precision statement will be added at the next revision.

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the sample;

c) the gel time at 25 °C;

d) the commercial origin of the methyl ethyl ketone peroxide used (see note 2 in 5.4);

e) details of any deviation from the reference conditions, particularly the use of an accelerator and/or initiator different in type and/or in different proportions, the use of a temperature other than 25 °C, etc.;

f) the date of the test;

g) any other relevant information.

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