SLOVENSKI STANDARD
SIST ISO 2535:1996

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Polimerni materiali - Nenasičene poliestrske smole - Merjenje časa želiranja pri 25° C

Plastics -- Unsaturated polyester resins -- Measurement of gel time at 25 degrees C

Plastiques -- Résines de polyesters non saturés -- Mesurage de la durée de gélification à 25 degrés C

Ta slovenski standard je istoveten z: **ISO 2535:1974**

ICS:

83.080.10 Duromeri Thermosetting materials

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INTERNATIONAL STANDARD ISO 2535

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

Matières plastiques — Résines de polyesters non saturés — Mesurage de la durée de gélification à 25 °C

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2535 was drawn up by Technical Committee ISO/TC 61, *Plastics*, and circulated to the Member Bodies in September 1971.

It has been approved by the Member Bodies of the following countries :

Australia	India	South Africa, Rep. of
Belgium	Israel	Spain
Brazil	Japan	Sweden
Czechoslovakia	Netherlands	Thailand
Egypt, Arab Rep. of	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Portugal	U.S.S.R.
Hungary	Romania	U.S.A.

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Canada
Italy
Switzerland

Printed in Switzerland

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

1 SCOPE AND FIELD OF APPLICATION

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 PRINCIPLE

Preparation at 25 °C of a mixture of resin with specified amounts of standard accelerator and initiator.

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 conventional viscosity corresponding to 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 "Gel time at 25 °C".

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

In particular cases, other conditions may be agreed between the interested parties (see clause 7).

3 REAGENTS

3.1 Acetone.

3.2 Reference accelerator : cobalt octoate solution in toluene.

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

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

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

This solution should be stored in a refrigerator and used within 1 month of preparation or receipt.

NOTES

1 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 8.)

2 **In no circumstance 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.**

4 APPARATUS

4.1 Test tube, glass, minimum length 120 mm, internal diameter 18 mm, fitted with a stopper, to be used to contain the test mixture.

4.2 Device, to measure the viscosity of the mixture in the tube.

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

NOTE – The description of a suitable device (see the figure) 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 on its axis at a slow speed (1 to 2 rev/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 stop the motor as well as the chronometer and signal the end of the test.

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

4.4 Beaker, capacity 100 ml.

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

4.6 Balance, accurate to within 0,1 g.

4.7 Spatula, stainless steel.

4.8 Chronometer, accurate to 1 s.

5 PROCEDURE

Clean the test tube (4.1) with acetone (3.1), dry and stopper it, and then immerse it to a depth of about 80 mm in the bath (4.3) thermostatically controlled at 25 °C.

Weigh 50 ± 0,1 g of resin into the beaker (4.4), place the latter in the bath at 25 °C and wait a sufficient time for the beaker and its contents to reach that temperature.

Using one of the pipettes (4.5), add 0,50 ml of cobalt octoate solution (3.2) to the resin and mix with the spatula (4.7).

Using the other pipette, add 0,70 ml of the methyl ethyl ketone peroxide solution (3.3) to the mixture, start the chronometer (4.8) 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 measuring device (4.2) is in position. Avoid wetting the sides of the tube above this level.

Replace the test tube in the thermostatic bath (4.3) in such a way that the level of the mixture in the tube is below the level of the bath.

Place the viscosity measuring device (4.2) in position.

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

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

NOTE — If the device described in the note to 4.2 and illustrated in the figure 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.

Replace the test tube in the bath as indicated. Fix the tube in a 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 in the axis of the test tube. Start the motor. When the viscosity of the mixture rises to the predetermined value, stop the motor and chronometer and continue as described.

Carry out a second test under the same conditions. The same test tube may be used if it is carefully cleaned with acetone, but it is preferable to use another test tube.

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

6 EXPRESSION OF RESULTS

Calculate the arithmetic mean of the two results obtained, and round off to the nearest 0,1 min.

The gel time at 25 °C expressed to the nearest 0,1 min is this mean value.

7 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 uses, it can sometimes prove useful

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

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

— to use other accelerator and initiator systems.

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

8 TEST REPORT

The test report shall include the following particulars :

a) the identification of the sample;

b) the gel time at 25 °C;

c) the commercial origin of the methyl ethyl ketone peroxide used (see note 1 of 3.3);

d) any variation from the reference conditions, particularly the use of an accelerator and/or initiator, different in type and/or proportion, the use of a temperature other than 25 °C, etc.

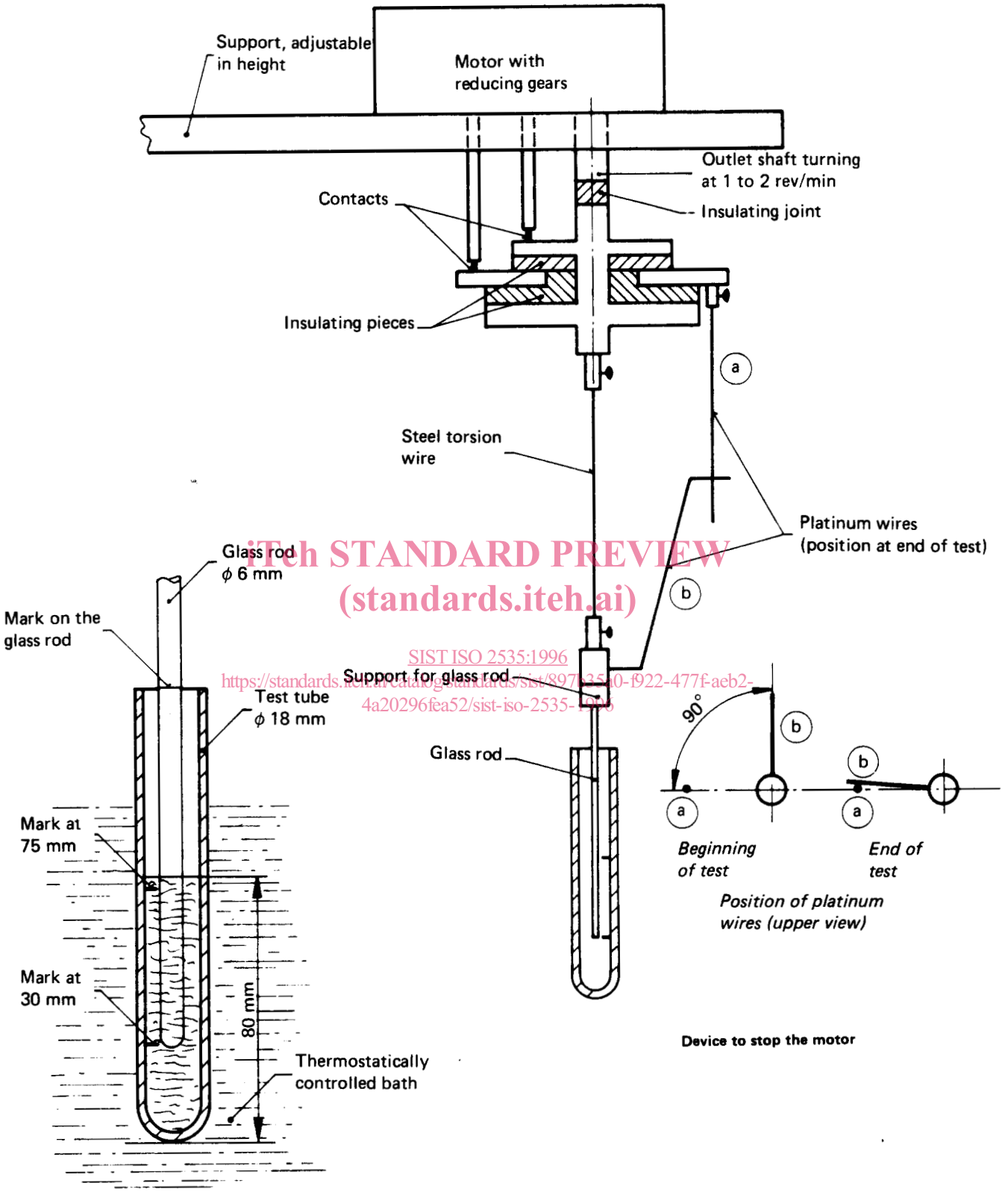

FIGURE — **Example of a suitable apparatus for the measurement of gel time using a rotating glass rod**

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

ERRATUM

Outside front cover

Replace the first element of the French sub-title by :

"Plastiques".