



SLOVENSKI STANDARD SIST EN ISO 9396:1999

01-maj-1999

Določa način določanja časa nastajanja gela pri določeni temperaturi s pomočjo avtomatske naprave (ISO 9396:1989).

Plastics - Phenolic resins - Determination of the gel time at a given temperature using automatic apparatus (ISO 9396:1989)

Kunststoffe - Phenolharze - Bestimmung der Gelierzeit mit einem Automaten bei vorgegebener Temperatur (ISO 9396:1989)

Plastiques - Résines phénoliques - Détermination de la durée de gélification à une température donnée sur appareils automatiques (ISO 9396:1989)

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Ta slovenski standard je istoveten z: EN ISO 9396:1995

ICS:

83.080.10 Duromeri Thermosetting materials

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EUROPEAN STANDARD

EN ISO 9396

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July 1995

ICS 83.080,10

Descriptors: plastics, phenoplasts, resins, tests, determination, gelation

English version

Plastics - Phenolic resins - Determination of the
gel time at a given temperature using automatic
apparatus (ISO 9396:1989)

Plastiques - Résines phénoliques -
Détermination de la durée de gélification à une
température donnée sur appareils automatiques
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Temperatur (ISO 9396:1989)

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SIST EN 9396:1999
REPUBLICA SLOVENIJA
MINISTRSTVO ZA ZNANOST IN TEHNOLOGIJO
Urad RS za standardizacijo in meroslovje
LJUBLJANA

SIST... EN ISO 9396.....

PREVZET PO METODI RAZGLASITVE

-85- 1995

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 35 B-1050 Brussels

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Ref. No. EN ISO 9396:1995 E

Foreword

The text of the International Standard from ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) has been taken over as a European Standard by the Technical Committee CEN/TC 249 "Plastics".

This European Standard shall be given the status of a National Standard, either by publication of an identical text or by endorsement, at the latest by January 1996, and conflicting national standards shall be withdrawn at the latest by January 1996.

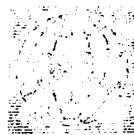
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Endorsement notice

The text of the International Standard ISO 9396:1989 has been approved by CEN as a European Standard without any modification.

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SIST EN ISO 9396:1999

61

INTERNATIONAL STANDARD

**ISO
9396**

First edition
1989-12-15

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

iTeh STANDARD PREVIEW
*Plastiques — Résines phénoliques — Détermination de la durée de gélification
à une température donnée sur appareils automatiques*
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Reference number
ISO 9396 : 1989 (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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9396 was prepared by Technical Committee ISO/TC 61, *Plastics*.

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

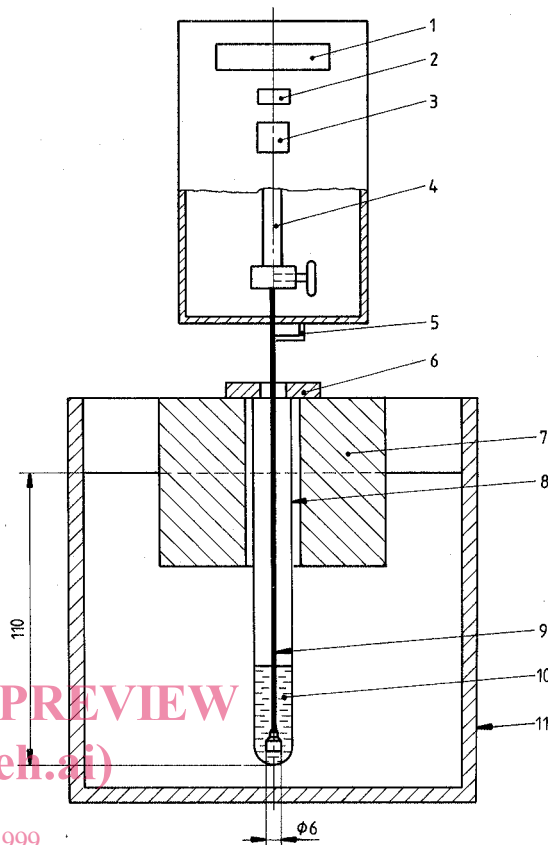
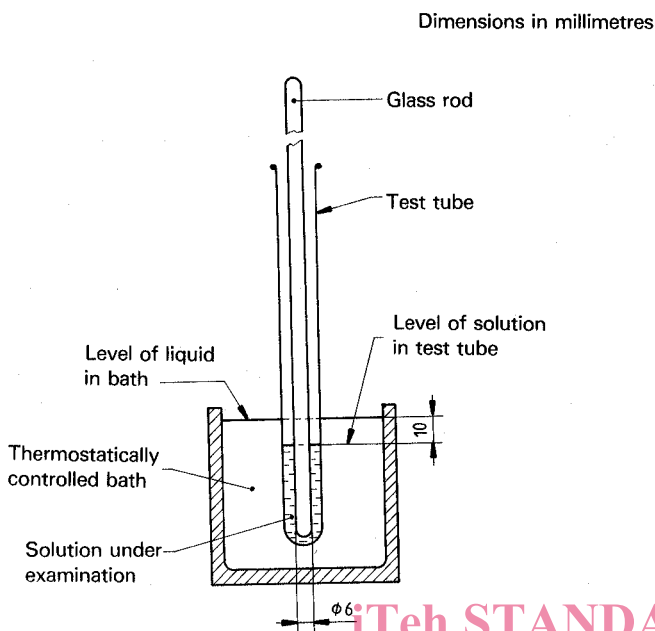


Figure 1 — Rotating-stirrer apparatus [SIST EN ISO 9396:1999](https://standards.iteh.ai/catalog/standards/sist/27031b62-a6ac-4465-a6d2-2658b88afca6/sist-en-iso-9396-1999)
for determining gel time <https://standards.iteh.ai/catalog/standards/sist/27031b62-a6ac-4465-a6d2-2658b88afca6/sist-en-iso-9396-1999>

- 1 Electric timer
- 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