



SLOVENSKI STANDARD

SIST EN ISO 9396:2001

01-junij-2001

Nadomešča:
SIST EN ISO 9396:1999

Polimerni materiali - Fenolne smole - Ugotavljanje časa želiranja pri določeni temperaturi z avtomatskim aparatom (ISO 9396:1997)

Plastics - Phenolic resins - Determination of the gel time at a given temperature using automatic apparatus (ISO 9396:1997)

Kunststoffe - Phenolharze - Bestimmung der Gelierzeit mit einem Automaten unter spezifischen Bedingungen (ISO 9396:1997)

Plastiques - Résines phénoliques - Détermination de la durée de gélification a une température donnée sur appareils automatiques (ISO 9396:1997)

Ta slovenski standard je istoveten z: EN ISO 9396:2000

ICS:

83.080.10 Duomeri Thermosetting materials

SIST EN ISO 9396:2001 en

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 9396

September 2000

ICS 83.080

Supersedes EN ISO 9396:1995

English version

Plastics - Phenolic resins - Determination of the gel time at a given temperature using automatic apparatus (ISO 9396:1997)

Plastiques - Résines phénoliques - Détermination de la durée de gélification à une température donnée sur appareils automatiques (ISO 9396:1997)

Kunststoffe - Phenolharze - Bestimmung der Gelierzeit mit einem Automaten unter spezifischen Bedingungen (ISO 9396:1997)

This European Standard was approved by CEN on 9 August 2000.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Foreword

The text of the International Standard from Technical Committee ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

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 March 2001, and conflicting national standards shall be withdrawn at the latest by March 2001.

This European Standard supersedes EN ISO 9396:1995.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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Endorsement notice

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The text of the International Standard ISO 9396:1997 has been approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

**ISO
9396**

Second edition
1997-07-15

Plastics — Phenolic resins — Determination of the gel time of resols under specific conditions using automatic apparatus

*Plastiques — Résines phénoliques — Détermination du temps
de gélification des résols dans des conditions spécifiques sur appareils
automatiques*
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Reference number
ISO 9396:1997(E)

ISO 9396:1997(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 9396 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 9396:1989), in which method A (which used a rotary stirrer) has been deleted and a precision clause added.

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Printed in Switzerland

Plastics — Phenolic resins — Determination of the gel time of resols under specific conditions using automatic apparatus

1. Scope

This International Standard specifies a method 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 method specified employs a plunger driven to carry out a reciprocating vertical motion.

This method applies to the following types of phenolic resin:

- liquid resols
- solid resols with low melting points

Powdered resins with a short gel time, such as mixtures of novolak and hexamethylenetetramine, 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.

2.2 Test solid resols as produced.

3. 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. Apparatus

- 4.1 **Automatic apparatus for measuring gel time** (see figure 1): The cycle time of the plunger shall be 10 s or as otherwise agreed between the parties concerned. The frequency shall be mentioned in the test report.
- 4.2 **Thermostatically controlled bath**, maximum temperature 200 °C, tolerance $\pm 0,5$ °C for the temperature chosen for the test (see 5). The density of the bath liquid, e.g. silicone oil, shall be $1 \text{ g/cm}^3 \pm 0,1 \text{ g/cm}^3$.
- 4.3 **Plunger**: metal wire with helix, length approx. 23 cm, diameter approx. 1 mm; helix at the end of the wire, height approx. 5 mm, diameter 5 mm to 6 mm.
- 4.4 **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 $160 \text{ mm} \pm 4 \text{ mm}$.
- 4.5 **Metal ring**, weighing 10 g or 20 g, to hold down the test tube (4.4) in the bath (4.2).
- 4.6 **Balance**, accurate to 0,1 g.

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5. 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 (4.4). Place the metal ring (4.5) round the top of the tube to hold it down in the bath (4.2) 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 (4.1) 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.

6. Precision (test error)

6.1 Repeatability (r)

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method, may be expected to lie with a 95 % probability is

2-8 % (depending on resin type)

6.2 Reproducibility (R)

The value below which the absolute difference between two test results, each the mean of duplicates, obtained on identical materials by operators in different laboratories using the standardized test method, may be expected to lie with a 95 % probability is

20 %

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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 sample of resin tested;
- c) the arithmetic mean of the individual values measured for the gel time, expressed in minutes to the nearest 0,1 min.
- d) the temperature at which the test was performed;
- e) the frequency of the plunger motion.