

INTERNATIONAL STANDARD

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AMENDMENT 1
1998-05-01

Coating powders —

Part 6:

Determination of gel time of thermosetting
coating powders at a given temperature

AMENDMENT 1

Poudres pour revêtement —

*Partie 6: Détermination du temps de gélification à une température donnée
de poudres thermodurcissables*

AMENDEMENT 1

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Foreword

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Amendment 1 to ISO 8130-6:1992 was prepared by ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

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Clause 8

In paragraph 5, line 4, after “start the timer (6.4)”, add “and commence stirring the material.”

New clause 9

Add the following clause:

“9 Precision

9.1 Repeatability, r

The value below which the absolute difference between two single test results 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 5 %.

9.2 Reproducibility, R

Due to the nature of the material, reproducibility cannot be determined.”

Renumber the current clause 9 (Test report) as clause 10.

ICS 87.040

Descriptors: coatings, powdery materials, thermosetting materials, paints, gelation, tests, determination, setting time.

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International Standard ISO 8130-6 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Sub-Committee SC 9, *General test methods for paints and varnishes*.

ISO 8130 consists of the following parts, under the general title *Coating powders*:

- *Part 1: Determination of particle size distribution by sieving*
- *Part 2: Determination of density by gas comparison pyknometer (referee method)*
- *Part 3: Determination of density by liquid displacement pyknometer*
- *Part 4: Calculation of lower explosion limit*
- *Part 5: Determination of flow properties of a powder/air mixture*
- *Part 6: Determination of gel time of thermosetting coating powders at a given temperature*
- *Part 7: Determination of loss of mass on stoving*
- *Part 8: Assessment of the storage stability of thermosetting powders*

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— *Part 9: Sampling*

Coating powders —

Part 6:

Determination of gel time of thermosetting coating powders at a given temperature

1 Scope

This part of ISO 8130 specifies a method for the determination of the time for a thermosetting coating powder to gel at a specified temperature, normally 180 °C.

NOTE 1 The determination of the gel time is a very simple method for the characterization and quality control of coating powders. However, the gel time determined by this method is not directly related to the time for a coating powder to cure in practical applications.

The method is not applicable to coating powders with ultra-short gel times (less than 15 s).

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 8130. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8130 are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 8130-9:1992, *Coating powders — Part 9: Sampling*.

3 Definition

For the purposes of this part of ISO 8130, the following definition applies.

3.1 gel time: The time taken for a specified volume of coating powder to become non-deformable, under specified conditions, after melting.

4 Principle

A test portion of a specified volume of coating powder is heated to a specified temperature in a depression in a heating block. The time at which threads can no longer be pulled from the molten product is determined.

5 Materials

5.1 Test substances of known melting point for checking the temperature of the heating block (6.1).

NOTE 2 For a test temperature of 180 °C, D-camphor is a suitable material.

5.2 Release agent, such as an aerosol dispersion of polytetrafluoroethylene.

6 Apparatus

6.1 Heating block, consisting of an electrically heated steel block of sufficient mass to maintain temperature stability, i.e. to ensure that the temperature selected within the range of 130 °C to 230 °C does not vary by more than ± 1 °C. The temperature shall be controlled by means of a thermoregulator.

The block shall have a spherical, polished depression, with a diameter of $(16 \pm 0,1)$ mm and a radius of curvature of $(10 \pm 0,1)$ mm in the centre of the top surface to contain the test portion.

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A hole of sufficient diameter shall be situated to take a temperature-measuring device (6.2) near the centre of one of the sides of the heating block, just below the top surface, extending horizontally to near the centre of the block and ending within 2 mm of the central depression.

NOTES

3 An example of a suitable heating block is shown in figure 1. The dimensions indicated are given for illustrative purposes only.

4 Thermal insulation of the heating block is desirable. For the heating block shown in figure 1, a heating device rated at 500 W has been found suitable.

6.2 Temperature-measuring device, of sufficient range, reading to 1 °C.

6.3 Measuring spoon, of capacity $(0,25 \pm 0,01)$ ml, for transferring the test portion to the depression in the heating block.

6.4 Timer, accurate to 1 s.

6.5 Stirrer, of very low heat capacity and of suitable size.

NOTE 5 For the heating block shown in figure 1, a stirring needle with a round pin-head, diameter 1 mm, and

attached to an insulating handle may be used. A small wooden stirrer has also been found to be satisfactory.

6.6 Scraper, made of a material softer than that of the heating block, for removing the test material from the heating block without scratching its surface.

7 Sampling

Take a representative sample of the product to be tested, as described in ISO 8130-9.

8 Procedure

Carry out the determination in duplicate.

Carry out the test at (180 ± 1) °C unless otherwise specified or otherwise agreed between the interested parties.

Place the heating block (6.1) in an atmosphere free from draughts. Raise the temperature of the heating block to the specified temperature and allow it to stabilize for at least 10 min.

NOTE 6 A check that the surface temperature of the heating block has reached the required temperature may be made by placing on it a small portion of material (5.1) that melts at the required temperature.

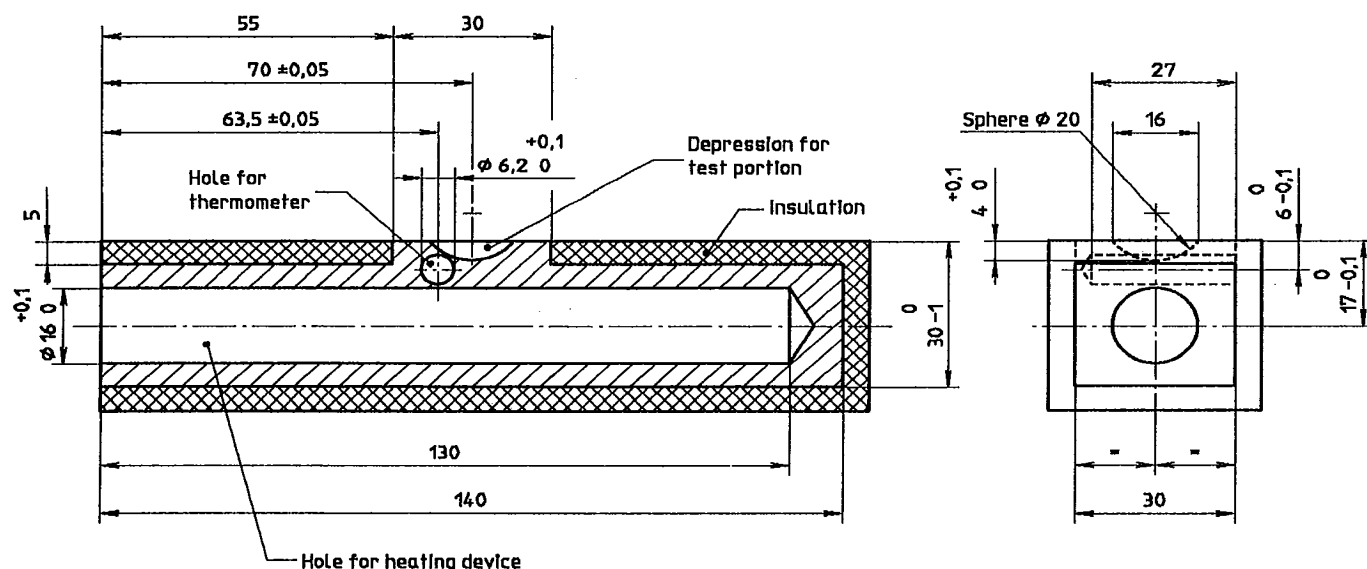


Figure 1 — Heating block (example)

If required, treat the depression and surrounding top surface of the heating block with the release agent (5.2) in accordance with the manufacturer's instructions.

By means of the measuring spoon (6.3), transfer a test portion of 0,25 ml of the product under test to the depression in the heating block and immediately after all the powder has melted start the timer (6.4). Stir the molten material in small circular movements with the stirrer (6.5). When thickening starts, whilst maintaining the overall stirring action, lift the stirring device every 2 s to 3 s to about 10 mm away from the molten material. When the threads thus formed become brittle such that they break and can no longer be drawn from the molten material, stop the timer and record the time to the nearest 1 s. This is the gel time. Immediately scrape the test material from the heating block, using the scraper (6.6) and taking care not to damage the surface.

Repeat the determination with a fresh test portion. If the two results differ by less than 5 % of the lower value, calculate and report the arithmetic mean. If the difference between the two results exceeds 5 %, carry out a third determination and calculate and report the arithmetic mean of all three results to the nearest 1 s.

If the difference between the result of the third determination and those of the other determinations is also greater than 5 %, state this and the individual results in the test report.

9 Test report

The test report shall contain at least the following information:

- a) all details necessary to identify the product tested;
- b) a reference to this part of ISO 8130 (ISO 8130-6);
- c) the test temperature;
- d) whether the heating block was treated with a release agent;
- e) the result of the test as indicated in clause 8;
- f) any deviation from the test method specified;
- g) the date of the test.

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