INTERNATIONAL STANDARD

ISO 12013-1

First edition
2012-10-15

Paints and varnishes — Determination of curing characteristics using a free damped oscillation method —

Part 1: Start temperature of the curing reaction

Peintures et vernis — Détermination des caractéristiques de polymérisation par une méthode utilisant un pendule amorti —

Partie 1: Température de début de réaction de polymérisation

Reference number
ISO 12013-1:2012(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12013-1 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

ISO 12013 consists of the following parts, under the general title *Paints and varnishes — Determination of curing characteristics using a free damped oscillation method*:

— *Part 1: Start temperature of the curing reaction*

— *Part 2: Glass transition temperature*

Introduction

A freshly applied coating dries and/or cures depending on its physical and chemical characteristics. There are “wet characteristics” of various substrates and the evaporation of the solvent in the drying/or curing process. The structure of the coating changes according to the drying or curing process of the coating. The start temperature of reaction is very important. It is essential to consider the evaporation of the solvent in the measurement of the start temperature of reaction.

a) While a solvent evaporates, the curing of the coating begins. The evaporation of the solvent depends on:

   1) the evaporation behaviour of the solvents;

   2) the behaviour of the solvents in the coating film during each drying stage. This is affected by the boiling point.

b) The evaporation characteristic of the solvents depends on the solubility and the thermal conductivity of the substrate.

Paints and varnishes — Determination of curing characteristics using a free damped oscillation method —

Part 1:
Start temperature of the curing reaction

1 Scope

This part of ISO 12013 specifies a free damped oscillation method for determining the start temperature of the curing reaction of coatings.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1513, *Paints and varnishes — Examination and preparation of test samples*

ISO 1514, *Paints and varnishes — Standard panels for testing*

ISO 2808, *Paints and varnishes — Determination of film thickness*

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following apply.

3.1
period
absolute value of the time between two oscillations in the same direction of the free damping oscillation

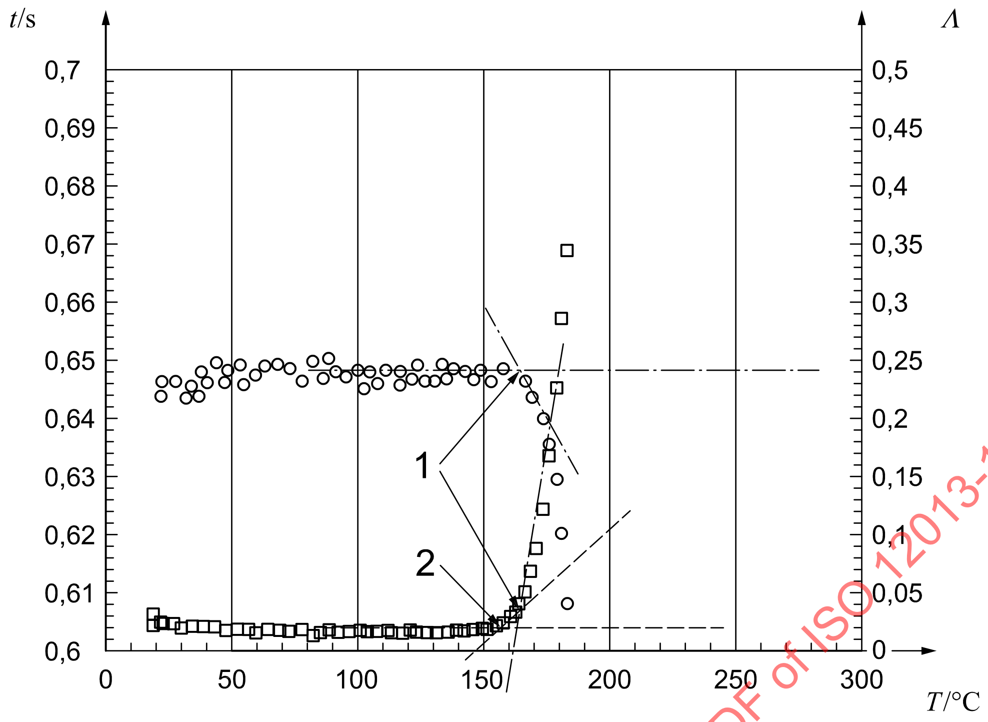
3.2
logarithmic damping ratio
Λ
logarithm of the ratio between consecutive amplitudes of a free damped oscillation

3.3
crosslinking temperature
T_{cl}
temperature at which the logarithmic damping ratio starts to increase rapidly and the period of oscillation starts to decrease rapidly

NOTE Figure 1 shows a study diagram from which the crosslinking temperature can be deduced (labelled 1).

3.4
start temperature of reaction
temperature at which the logarithmic damping ratio begins to increase

NOTE The start temperature of reaction is shown in Figure 1(labelled 2).



Key

t	period of oscillation	1	crosslinking temperature and network formation
Λ	logarithmic damping ratio	2	start temperature of reaction
T	temperature	O	period of oscillation
		□	logarithmic damping ratio

Figure 1 — Temperature dependence of the logarithmic damping ratio

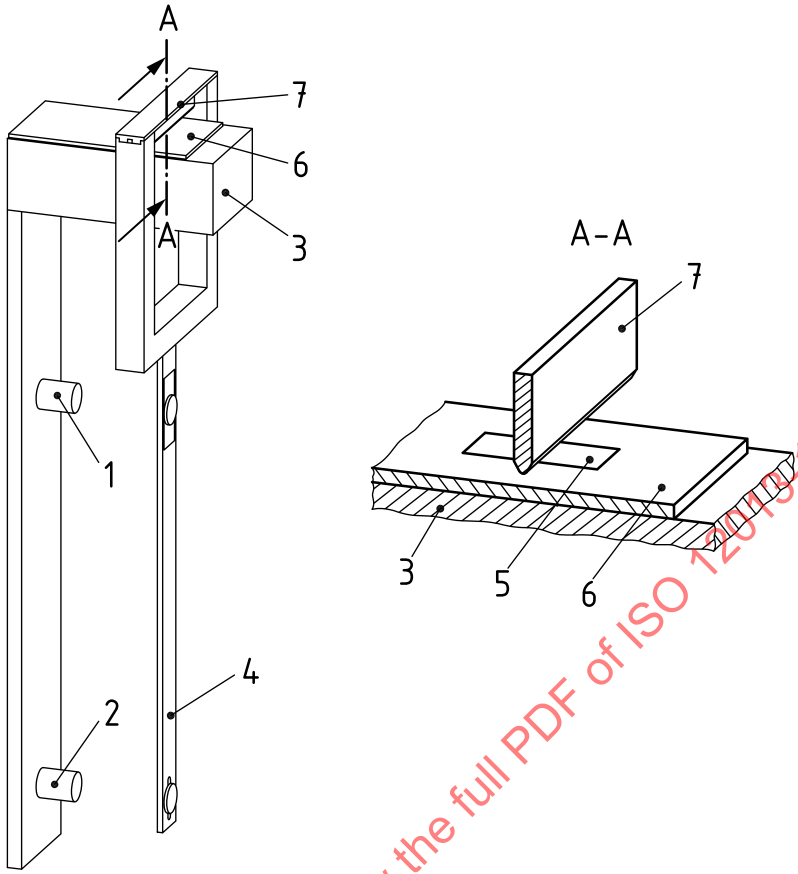
4 Principle

A coating is applied to a test panel. The coated panel is fixed in the apparatus and a rigid-body pendulum with a knife edge is lowered on to the coating. The heating rate and the amplitude of the first pendulum oscillation are set. The pendulum is released and allowed to oscillate. As the coating starts to dry or cure, the logarithmic damping ratio increases and the amplitude of oscillation of the pendulum becomes progressively smaller. The damping ratio is determined from measurements of the amplitude of the oscillation of the pendulum and plotted against the temperature. The point at which the logarithmic damping ratio begins to increase rapidly is recorded as the start temperature of reaction.

5 Pendulum-damping apparatus

5.1 General. Figure 2 shows an example of a pendulum-damping apparatus. In addition to the pendulum itself, the apparatus includes an electromagnet with which the required oscillation can be generated, a temperature-controlled test block, and a displacement sensor that detects each swing of the pendulum. The swings of the pendulum, the time that elapses from the beginning of the first oscillation and the temperature of the coating are monitored electronically.

5.2 Temperature-controlled test block, shown in Figure 3. The coated test panel is mounted on a mounting table (A), the temperature of which is regulated by means of an electric heating current passing through the block from B.

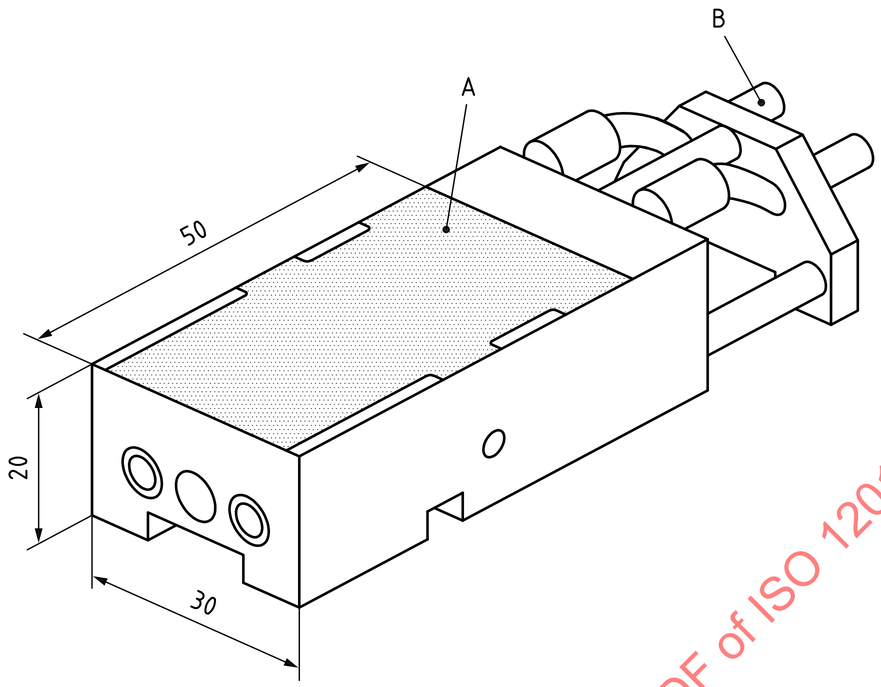


Key

1 displacement sensor
2 electromagnet
3 temperature-controlled test block
4 rigid-body pendulum
5 coating
6 test panel
7 knife edge

Figure 2 — Pendulum-damping apparatus

Dimensions in millimetres



Key

A mounting table for test panel

B connection for electric-power supply

Figure 3 — Temperature-controlled test block

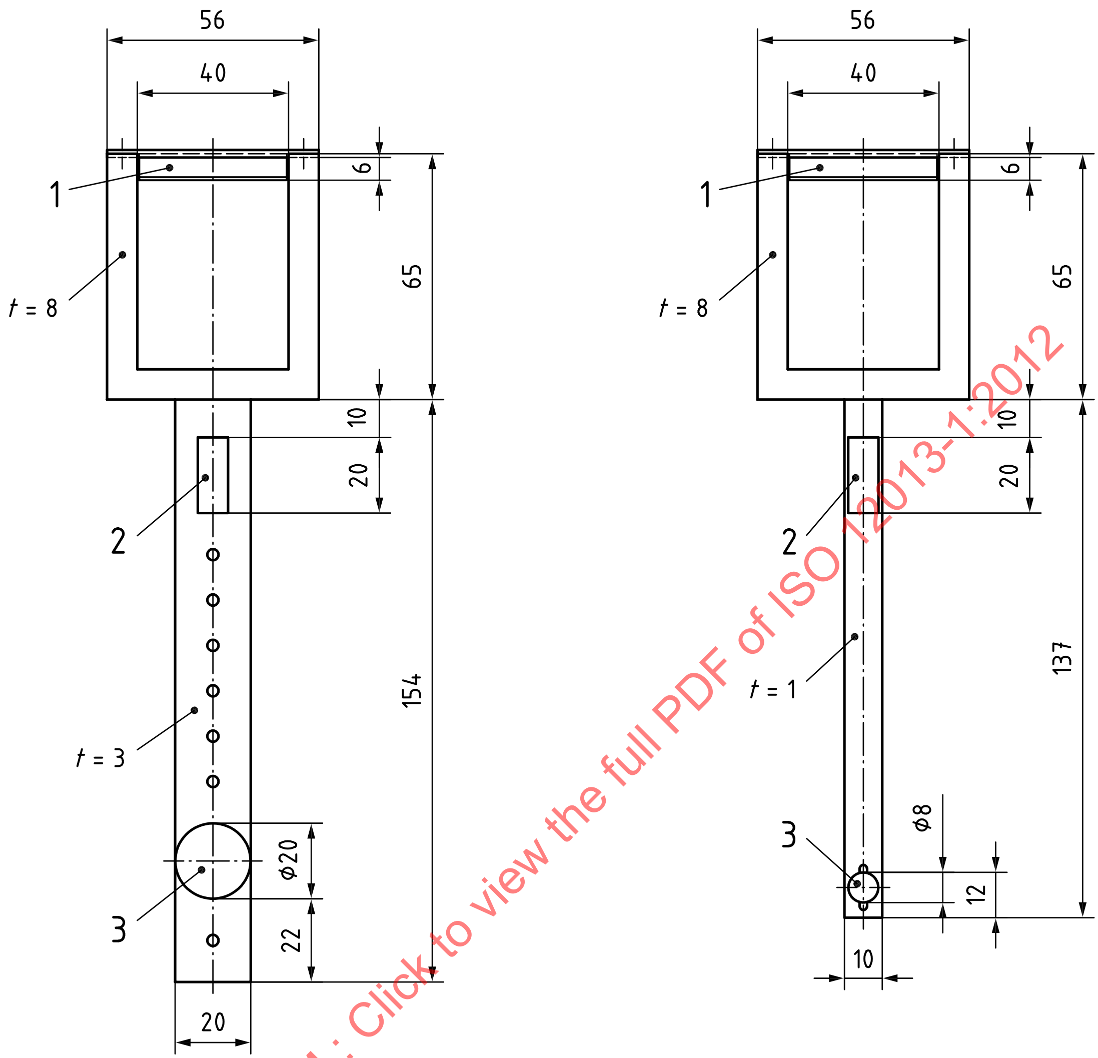
5.3 Rigid-body pendulum, of which Figure 4 shows the dimensions of two different types. These pendulums are designed so that their oscillation is generated by an electromagnet. The period and amplitude of oscillation are recorded automatically by a computer.

NOTE Guidance on the selection of the pendulum is given in Annex A.

5.4 Knife edge, attached to the pendulum and placed in contact with the coated test panel at the beginning of the test. The shape and dimensions of the knife edge are shown in Figure 5.

NOTE Guidance on the selection of the knife edge is given in Annex A.

Dimensions in millimetres



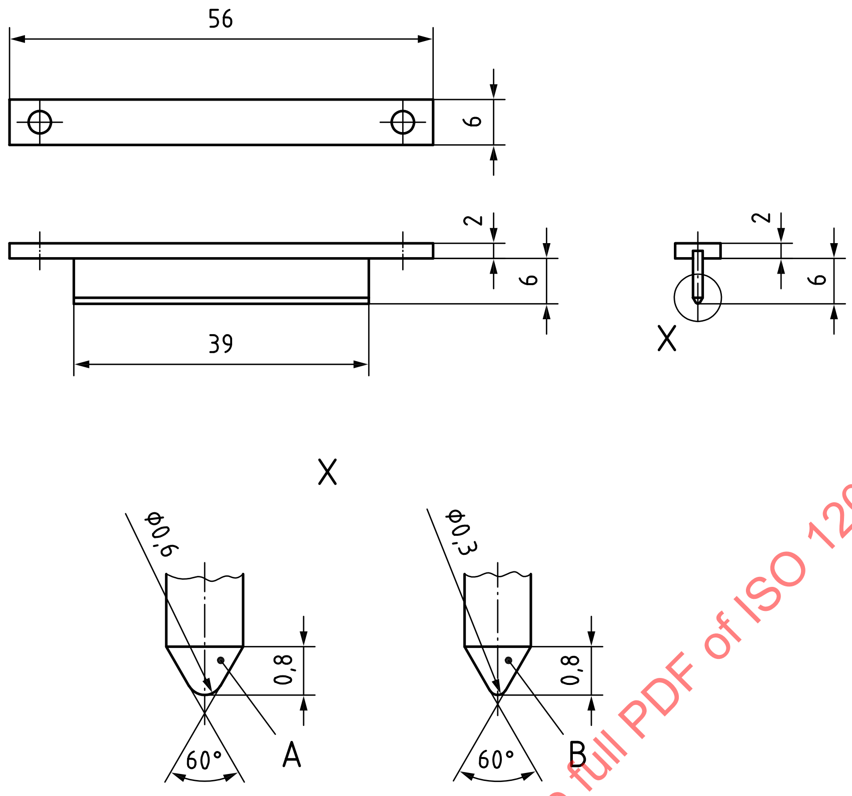
a) **Type 1, steel, mass 51 g** **b)** **Type 2, steel, mass 45 g**

Key

1 knife edge
2 displacement sensor
3 electromagnet

Figure 4 — Rigid-body pendulum

Dimensions in millimetres



Key

A knife edge A, material steel, mass 6 g, see Annex A

B knife edge B, material steel, mass 6 g, see Annex A

Figure 5 — Dimension of knife edge

6 Sampling

Take a representative sample of the product to be tested (or of each product in the case of a multicoat system), as specified in ISO 15528.

Examine and prepare each sample for testing as specified in ISO 1513.

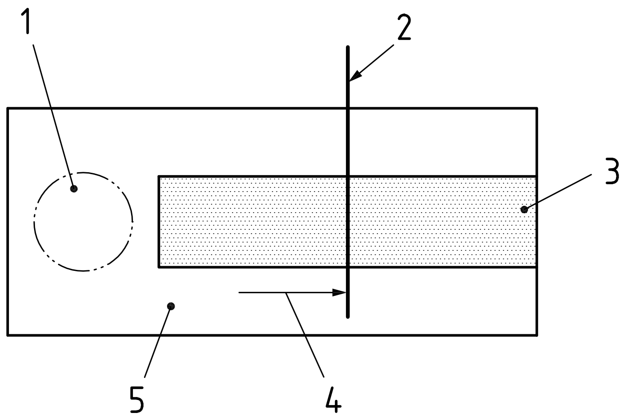
7 Test panels

7.1 Substrate

Select the substrate from those specified in ISO 1514, ensuring that the panels are planar and rigid. Metal, glass or plastic panels are recommended, with dimensions of 25 mm × 50 mm × (0,3 mm to 1,0 mm).

7.2 Preparation and coating

Prepare each test panel in accordance with ISO 1514 and then coat it with the product under test by using the draw-down method. The direction of application shall be as indicated by the arrow in Figure 6. The test coating shall be smooth and free from surface irregularities.



Key

1 position of temperature sensor
2 position of knife edge
3 coating
4 direction of application of coating
5 test panel (substrate)

Figure 6 — Test arrangement of temperature sensor, coating, and knife-edge

7.3 Thickness of coating

After completion of the test, determine the thickness, in micrometres, of the dry coating, on a part of the test panel not affected by the test, by one of the procedures specified in ISO 2808.

8 Procedure

Carry out the determination in duplicate.

The starting temperature and the temperature increase are dependent not only on the type of coating and the substrate used, but also on the test conditions. The test conditions should preferably be agreed between the interested parties. If the conditions have not been agreed, a) to d) are recommended.

a) Carry out the determination at a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) %.

b) Set the temperature at which the measurements are to begin to preferably 30 °C and the heating rate to 10 °C/min.

c) Set the temperature at which the measurements are to finish to more than 20 °C above the expected start temperature of the reaction.

d) Measure the logarithmic damping ratio Λ every 12 s. The oscillation of the rigid-body pendulum is performed at each reading interval. The time to draw a rigid-body pendulum to a magnet is 1 s to 2 s.

Immediately after the product under test has been applied to the test panel, place the coated test panel on the test block (5.2). Mount the temperature sensor on the upper surface of the test panel (see Figure 6) and record the temperature.

Place the knife edge, mounted on the pendulum, in contact with the coating on the test panel (see Figure 6). Start the apparatus.

During the test, the temperature of the test panel and the period of oscillation of the pendulum are recorded. The logarithmic damping ratio Λ is calculated from the amplitude of oscillation of the pendulum and plotted against the temperature.

9 Expression of results

The starting temperature of reaction is taken to be the temperature, in degrees Celsius, at which the logarithmic damping ratio start to increase rapidly (see Figure 1). Calculate the mean of the values obtained in the two individual determinations and round this mean value to the nearest whole number.

10 Precision

10.1 General

Precision data for this method have been obtained with a melamine–polyester resin.

10.2 Repeatability limit

The repeatability limit r is the value below which the absolute difference between two single test results, each the mean of duplicates, can be expected to lie with a 95 % probability when this method is used under repeatability conditions (i.e. test results obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method). For the particular melamine–polyester resin tested, r has been found to be 4 °C.

10.3 Reproducibility limit

The reproducibility limit R is the value below which the absolute difference between two-single test results, each the mean of duplicates, can be expected to lie with a 95 % probability when this test method is used under reproducibility conditions (i.e. test results obtained on identical material by operators in different laboratories using the standardized test method). For the particular melamine–polyester resin tested, R has been found to be 20 °C.

11 Test report

The test report shall contain at least the following information:

a) all details necessary for complete identification of the product tested (manufacturer, trade name, batch number, etc.);

b) a reference to this part of ISO 12013 (ISO 12013-1:2012);

c) details of the coated test panel (substrate material, film thickness, state of surface);

d) the equipment and test conditions used (i.e. the type of rigid-body pendulum and knife edge, the temperature at which the test was begun, the heating rate, and the temperature at which the test was finished);

e) the results of the test, including both the results of the individual determinations and their mean as specified in Clause 9;

f) any specific agreements between the interested parties;

g) any deviations from the procedure specified;

h) any unusual features (anomalies) observed during the test;

i) the date of the test.

