



BSI Standards Publication

**Paints and varnishes —
Determination of curing
characteristics using a free
damped oscillation method**
Part 2: Glass transition temperature

National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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

Part 2:

Glass transition temperature

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

Partie 2: Température de transition vitreuse





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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 12013-2 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General tests 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

When a rigid-body pendulum with a cylindrical edge is placed in contact with a cured coating, the free damped oscillation is measured as, due to heating, the viscoelasticity of the paint film changes. The attenuation of the oscillating rigid pendulum on the coating depends on the viscoelasticity of the paint. When the viscoelasticity of the paint changes, the logarithmic damping ratio of the rigid-body pendulum changes. Various paint film properties can be read from a plot of the logarithmic damping ratio versus temperature (see Annex A).

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

Part 2: Glass transition temperature

1 Scope

This part of ISO 12013 specifies a free damped oscillation method for determining the glass transition temperature of dried and cured films of paints and varnishes.

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

glass transition

reversible change in an amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one

[ISO 472:1999^[1]]

3.2

glass transition temperature

T_g

approximate midpoint of the temperature range over which the glass transition takes place

[ISO 472:1999^[1]]

NOTE 1 The rotational degrees of freedom of a polymer are excited at the glass transition temperature.

NOTE 2 The glass transition temperature varies significantly, depending upon the adhesion to the substrate of the cured coating film, as well as the test method and conditions selected to measure it.

3.3
glass transition temperature as determined by the pendulum method
 T_{gp}
glass transition temperature of dried and cured paints and varnishes determined using a free damped oscillation method

NOTE The damping ratio is determined from the amplitude of oscillation of the pendulum and plotted against the temperature. The peak of the curve thus obtained is taken as the glass transition temperature.

3.4
period
absolute value of the time between two oscillations in the same direction of the free damped oscillation

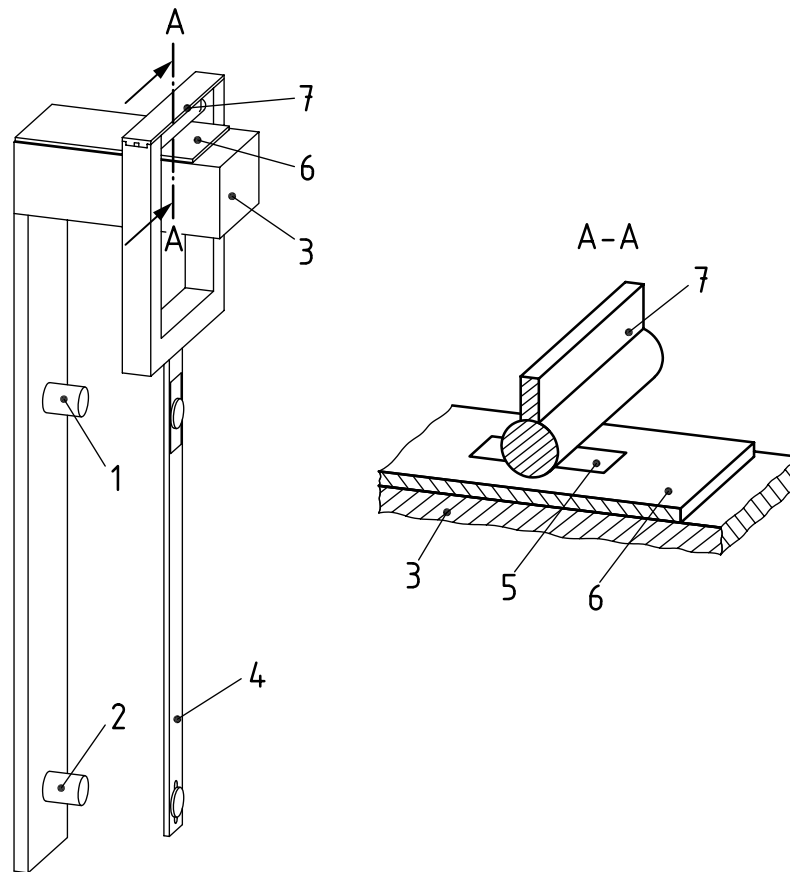
3.5
logarithmic damping ratio
 Δ
logarithm of the ratio between consecutive amplitudes of a free damped oscillation

4 Principle

Immediately after a coating material is applied to a test panel, it is dried or cured as specified. The dried or cured test panel is fixed in the apparatus and a rigid-body pendulum with a cylindrical edge (see Figure 1) is lowered on to the film. The heating rate and the amplitude of the first pendulum oscillation are set. The pendulum is released and allowed to oscillate. As the temperature is raised, the amplitude of oscillation of the pendulum becomes progressively smaller. When the temperature becomes greater than T_g , the amplitude of oscillation becomes gradually larger again. The damping ratio is determined from measurements of the amplitude of oscillation of the pendulum and plotted against the temperature. The peak of the curve thus obtained is recorded as the glass transition temperature. When the network structure of the dried or cured coatings film on the substrate is heated, it begins to change. When network structure changes, viscosity changes occur. The oscillation of the pendulum is damped by the change in viscosity. After all chemical bonds have been formed, the viscosity decreases due to a rise of the temperature. Therefore a peak is produced. The maximum height of this peak can be read off as T_{gp} .

5 Pendulum-damping apparatus

5.1 General. Figure 1 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 amplitude and period of the swings of the pendulum, the time that elapses from the beginning of the first oscillation and the temperature of the sample are monitored by computer. The logarithmic damping ratio and the temperature of the test panel are recorded.

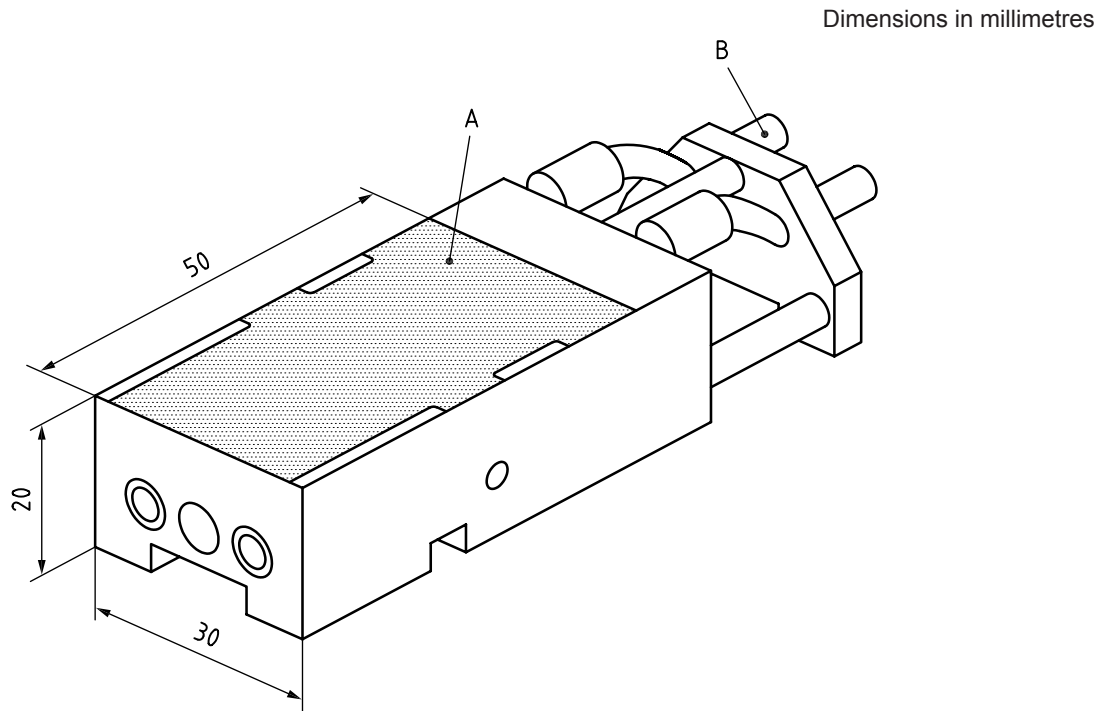


Key

- | | | | |
|---|---|---|---------------------------------------|
| 1 | displacement sensor | 5 | dried or cured film on the test panel |
| 2 | electromagnet | 6 | test panel |
| 3 | temperature-controlled test block | 7 | cylindrical edge |
| 4 | rigid-body pendulum with cylindrical edge | | |

Figure 1 — Pendulum-damping apparatus

5.2 Temperature-controlled test block, shown in Figure 2. The dried or cured 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

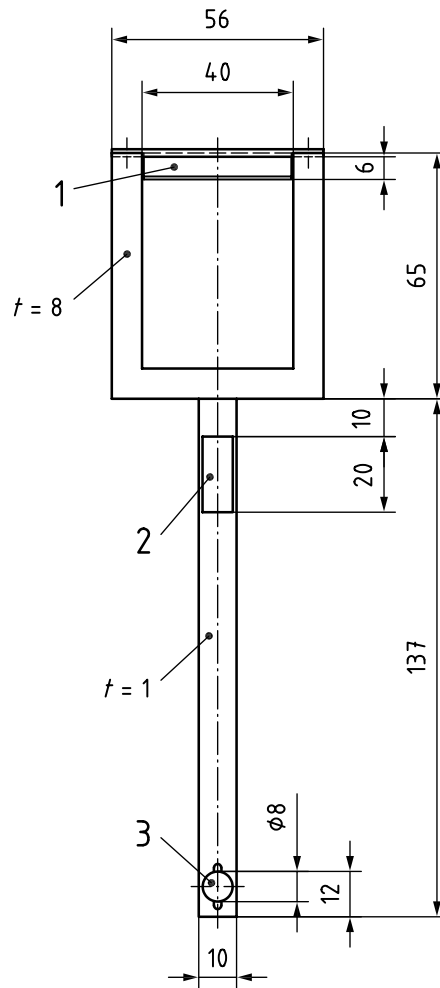
A mounting table for test panel

B connection for electric-power supply

Figure 2 — Temperature-controlled test block

5.3 Rigid-body pendulum, of which Figure 3 shows the dimensions. The pendulum is designed so that its oscillation is generated by an electromagnet. The period and amplitude of oscillation are recorded automatically by a computer.

Dimensions in millimetres



Key

- 1 cylindrical edge 2 displacement sensor 3 electromagnet

Figure 3 — Rigid-body pendulum, steel, mass 45 g

5.4 Cylindrical edge, attached to the rigid-body pendulum and placed in contact with the dried or cured film of coating material on the test panel at the beginning of the test. The shape and dimensions of the cylindrical edge are shown in Figure 4.

Dimensions in millimetres

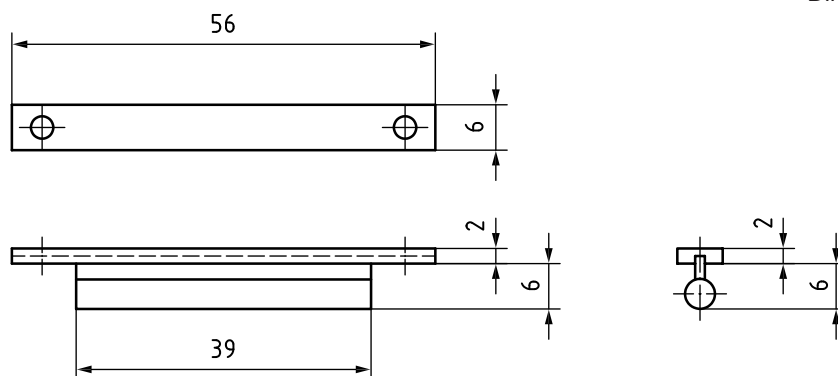


Figure 4 — Cylindrical edge, brass, mass 4 g

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.

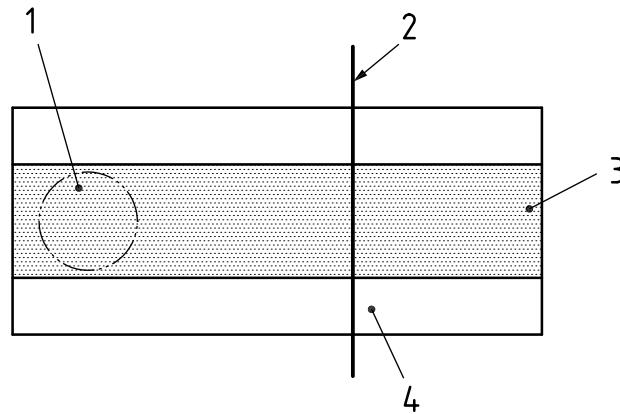
7 Test panels

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, coat it with the product under test and dry or cure the coating as specified by the manufacturer. The test coating shall be smooth and free from surface irregularities.



Key

- | | | | |
|---|--------------------------------|---|---------------------------------|
| 1 | position of temperature sensor | 3 | dried or cured coating material |
| 2 | position of cylindrical edge | 4 | test panel (substrate) |

Figure 5 — Test arrangement of temperature sensor, dried or cured coating material and cylindrical edge

7.3 Thickness of coating

After completion of the test, determine the dry-film thickness, in micrometres, of the 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 material 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.

- Carry out the determination at a temperature of $(23 \pm 2) ^\circ\text{C}$ and a relative humidity of $(50 \pm 5) \%$.
- Set the temperature at which the measurements are to begin to approximately $30 ^\circ\text{C}$ and the heating rate to $10 ^\circ\text{C}/\text{min}$.

- c) Set the temperature at which the measurements are to finish to more than 30 °C above the expected glass transition temperature.
- d) Measure the logarithmic damping ratio Δ 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.

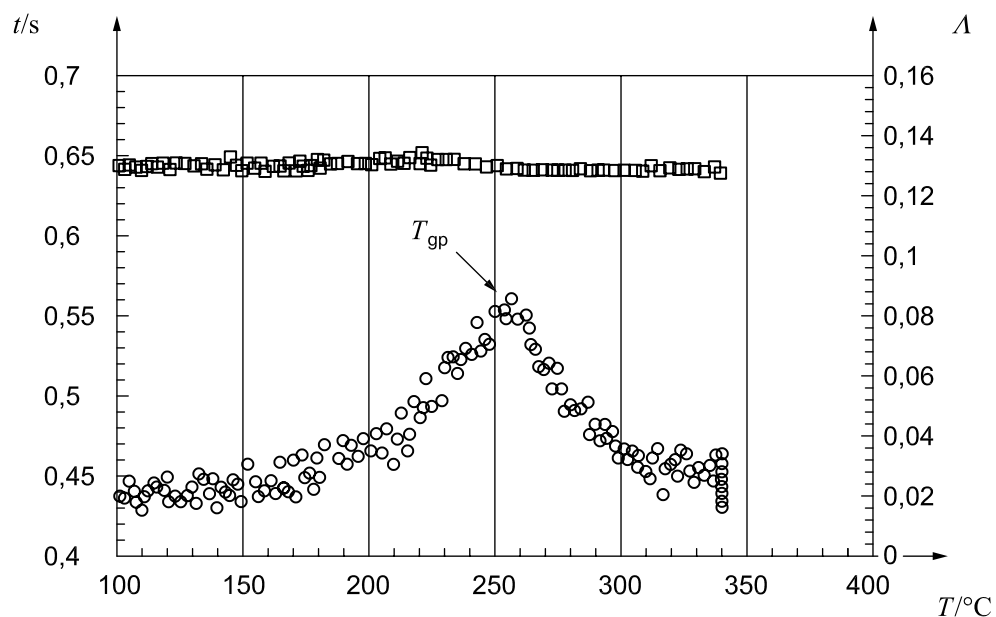
Place the test panel carrying the dried or cured coating material on the test block (5.2). Mount the temperature sensor on to the dried or cured coating (see Figure 5) and record the temperature.

Place the cylindrical edge, mounted on the pendulum, in contact with the dried or cured coating on the test panel (see Figure 5). Start the apparatus.

During the test, the temperature of the test panel and the logarithmic damping ratio is recorded. The logarithmic damping ratio Δ is calculated from the oscillation amplitudes of a pendulum and plotted against the temperature.

9 Expression of results

The glass transition temperature determined by pendulum method, T_{gp} , of dried or cured paint on substrate is estimated as the logarithmic damping ratio, Δ , for the temperature. An example of such a plot is given in Figure 6. Record the peak of the plot as the glass transition temperature. Calculate the mean of the values obtained in the two individual determinations and round this mean value to the nearest whole number.



Key

- t period of oscillation, in seconds
- Δ logarithmic damping ratio
- T temperature, in degrees celsius
- logarithmic damping ratio
- period of oscillation

Figure 6 — Example of a plot for determining the glass transition temperature T_{gp} of a dried or cured film of coating material on a substrate

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 3 °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 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 9 °C.

11 Test report

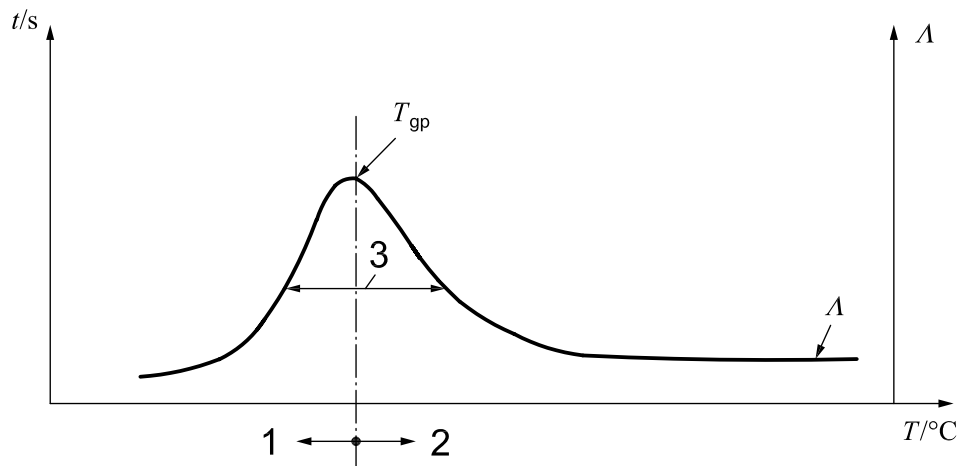
The test report shall contain at least the following information:

- 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 12013-2:2012);
- c) details of the coated test panel (substrate material, film thickness, state of surface);
- d) the test conditions used (i.e. 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.

Annex A (informative)

Viscoelastic properties

The plot of logarithmic damping ratio against temperature obtained by the method specified in this part of ISO 12013 (see Figure A.1) not only indicates the glass transition temperature, but also gives information on the viscoelastic behaviour of the dried or cured coating, such as the change in hardness with temperature (related to the reciprocal of the logarithmic damping ratio) at temperatures up to T_{gp} and the degree of crosslinking.



Key

t	period of oscillation	1	soft
Λ	logarithmic damping ratio	2	hard (crosslinking structure)
T	temperature	3	degree of crosslinking

Figure A.1 — Reading physical data of cured paint

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- [1] ISO 472:1999, *Plastics — Vocabulary*
- [2] SCHOFF C.K. Automotive coating defects — Part 2: Weathering processes and their effects on coating properties. *JCT — CoatingsTech* 2004, 1(3), pp. 22–26

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