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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Plastics — Thermoplastic materials — Determination of Vicat softening temperature

Plastiques — Matières thermoplastiques — Détermination de la température de ramollissement Vicat

Reference number
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Foreword

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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 306 was prepared by Technical Committee ISO/TC 61, *Plastics.*

This second edition cancels and replaces the first edition (ISO 306 : 1974), of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Plastics — Thermoplastic materials — Determination of Vicat softening temperature

1 Scope and field of application

1.1 This International Standard specifies two methods for the determination of the Vicat softening temperature of thermoplastic materials:

— Method A using a load of 10 N

— Method B using a load of 50 N

1.2 The methods specified are applicable only to thermoplastics, for which they give a measure of the temperature at which the thermoplastics start to soften rapidly.

2 References

ISO 291, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 293, *Plastics — Recommended practice for compression moulding test specimens of thermoplastic materials.*

ISO 294, *Plastics — Injection moulding test specimens of thermoplastic materials.*

ISO 2818, *Plastics — Preparation of test specimens by machining.*

ISO 3167, *Plastics — Preparation and use of multi-purpose test specimens.*

3 Principle

Determination of the temperature at which a standard indenter penetrates 1 mm into the surface of a plastic test specimen under one of the loads given in 1.1 when the temperature is raised at a uniform rate.

The temperature at 1 mm penetration is quoted as the Vicat softening temperature (VST) in degrees Celsius.

4 Apparatus

The apparatus consists essentially of:

4.1 Rod, provided with a **load-carrying plate** (4.4), held in a **rigid metal frame** so that it can move freely in the vertical direction, the base of the frame serving to support the test specimen under the indenting tip at the end of the rod (see the figure).

Unless the rod and frame members have the same linear expansion coefficient, the differential change in the length of these parts introduces an error in the readings of the apparent deformation of the test specimen. A blank test shall be made on each apparatus using a test specimen made of rigid material having a low coefficient of expansion.[1] The temperature ranges to be used shall be covered and a correction term determined for each temperature. If the correction term is 0,02 mm or greater, its algebraic sign shall be noted and the term applied to each test by adding it algebraically to the reading of apparent penetration. It is recommended that the apparatus be constructed of low thermal expansion alloy.

4.2 Indenting tip, preferably of hardened steel, 3 mm long, of circular cross-section, and area 1,000 ± 0,015 mm^2, fixed at the bottom of the rod (4.1). The lower surface of the indenting tip shall be plane and perpendicular to the axis of the rod and free from burrs.

4.3 Calibrated micrometer dial gauge (or other suitable measuring instrument), to measure to ± 0,01 mm the penetration of the indenting tip into the test specimen. The thrust of the dial gauge, which contributes to the thrust on the test specimen, must be known (see 4.4).

NOTES

1 In certain forms of execution of the apparatus, the force of the dial gauge spring is directed upward and is subtracted from the load; in other forms, this force acts downward and is added to the load.

2 Since the force exerted by the spring in certain dial gauges varies considerably over the stroke, this force is measured in that part of the stroke which is to be used.

1) Invar and borosilicate glass have been found suitable for this purpose.

4.4 Load-carrying plate, fitted to the rod (4.1), and **suitable weights** added centrally so that the total thrust applied to the test specimen can be made up to 10 ± 0,2 N for method A and 50 ± 1 N for method B. The combined downward thrust due to the rod, indenting tip, load-carrying plate and the force of the dial gauge spring shall not exceed 1 N.

4.5 Heating bath, containing a suitable liquid in which the apparatus is placed so that the specimen is at least 35 mm below the surface of the liquid. An efficient stirrer shall be provided. The heating bath shall be equipped with a means of control so that the temperature can be raised at a uniform rate either of 50 ± 5 K/h or 120 ± 10 K/h, as required.

The heating rate shall be considered to be obtained if, over every 6 min interval during the test, the temperature change is within 5 ± 0,5 K or 12 ± 1 K.

NOTES

1 Liquid paraffin, transformer oil, glycerol and silicone oils may be suitable liquid heat-transfer media, but other liquids may be used. In all cases, it has to be established that the liquid chosen is stable at the temperature used and does not affect the material under test, for example by swelling, softening or cracking.

If no suitable liquid can be found for use as a heat-transfer medium, some different heating arrangement for which air may be found to be a suitable heat-transfer medium will be necessary.

2 The results of the test may depend on the thermal conductivity of the heat-transfer medium.

4.6 Mercury-in-glass thermometer, of the partial-immersion type or **other suitable temperature-measuring instrument** of appropriate range and accurate to within 0,5 °C. Mercury-in-glass thermometers shall be calibrated for the depth of immersion required by 7.2.

5 Test specimens

5.1 At least two specimens shall be used to test each sample. The test specimens shall be between 3 and 6,5 mm thick and at least 10 mm square or of 10 mm diameter. Their surfaces shall be flat and parallel and free from flash. They shall be made in accordance with specifications, if any, for the material under test. In the absence of such specifications, any suitable procedure may be used for the preparation of test specimens.

5.2 If the samples submitted for test are in the form of moulding materials (for example, powder or granulated materials), these shall be moulded into specimens 3 to 6,5 mm thick, in accordance with specifications relating to the material under test, or in accordance with ISO 293, ISO 294 or ISO 3167 if no material specification exists. If these are not applicable, any other reproducible procedure may be followed that modifies the properties of the material as little as possible.

5.3 For sheet materials, the thickness of the test specimens shall be equal to the thickness of the sheet, except as follows:

a) if the thickness exceeds 6,5 mm, the test specimens shall be reduced in thickness to 3 to 6,5 mm by machining one surface (see ISO 2818), the other surface being left intact; the test surface shall be the intact one;

b) if the thickness of the sheet is less than 3 mm, not more than three pieces shall be stacked together in good contact to give a total thickness between 3 and 6,5 mm and the thickness of the upper (measured) piece shall be at least 1,5 mm. Stacking of pieces of lesser thickness does not always result in the same value of the measured quantity.

5.4 The test results obtained may depend on the moulding conditions used in the preparation of the specimens, although such a dependence is not common. When testing materials for which the results do depend on moulding conditions, special annealing or preconditioning procedures may be used before testing if they are agreed to by the interested parties.

6 Conditioning

Unless otherwise required by the specification for the material being tested, the specimens shall be conditioned and tested in accordance with ISO 291.

7 Procedure

7.1 Mount the test specimen horizontally under the indenting tip (4.2) of the unloaded rod (4.1). The indenting tip shall at no point be nearer than 3 mm to the edge of the test specimen. The surface of the test specimen in contact with the base of the apparatus shall be flat.

7.2 Immerse the assembly in the heating bath (4.5). The temperature of the bath should be 20 to 23 °C at the start of each test, unless previous tests have shown that, for the material under test, no error is caused by starting at another temperature. The bulb of the thermometer or sensitive part of the temperature-measuring instrument (4.6) shall be at the same level as, and as close as possible to, the test specimen.

7.3 After 5 min, with the indenting tip still in position, add the weight to the load-carrying plate (4.4) so that the total thrust on the test specimen is 10 ± 0,2 N for method A and 50 ± 1 N for method B. Then note the reading of the micrometer dial gauge (or other indentation-measuring instrument) (4.3) or set the instrument to zero.

7.4 Increase the temperature of the bath at a uniform rate of 50 ± 5 K/h or, alternatively, of 120 ± 10 K/h; stir the liquid well during the test. For referee tests, a rate of 50 K/h shall be used.

NOTE — For some materials, at the higher rate (120 K/h) Vicat softening temperatures up to 4 K higher can be observed.

7.5 Note the temperature of the bath at which the indenting tip has penetrated into the test specimen by 1 ± 0,01 mm beyond its starting position defined in 7.3 and record it as the Vicat softening temperature (VST) of the test specimen.

7.6 Express the VST of the material under test as the arithmetic mean of the VSTs of the specimens tested. If the range of individual results exceeds 2 K, record the individual results [see clause 8, h)] and repeat the test once using a further set of at least two specimens (see 5.1).

8 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) full identification of the material tested;

c) the method employed (A or B) and the rate of temperature increase (50 or 120 K/h), noted as A50, A120, B50, or B120;

d) the thickness and number of layers of composite test specimen (i.e. specimens consisting of more than one layer) if these are used;

e) the method of preparation of the test specimens used;

f) the immersion medium;

g) the conditioning and annealing procedures used, if any;

h) the Vicat softening temperature (VST) of the material, in degrees Celsius; if the individual results after two measurements differ by more than the limit given in 7.6, all individual results shall be reported;

i) any peculiar characteristics of the test specimen noted during the test or after removal from the apparatus.

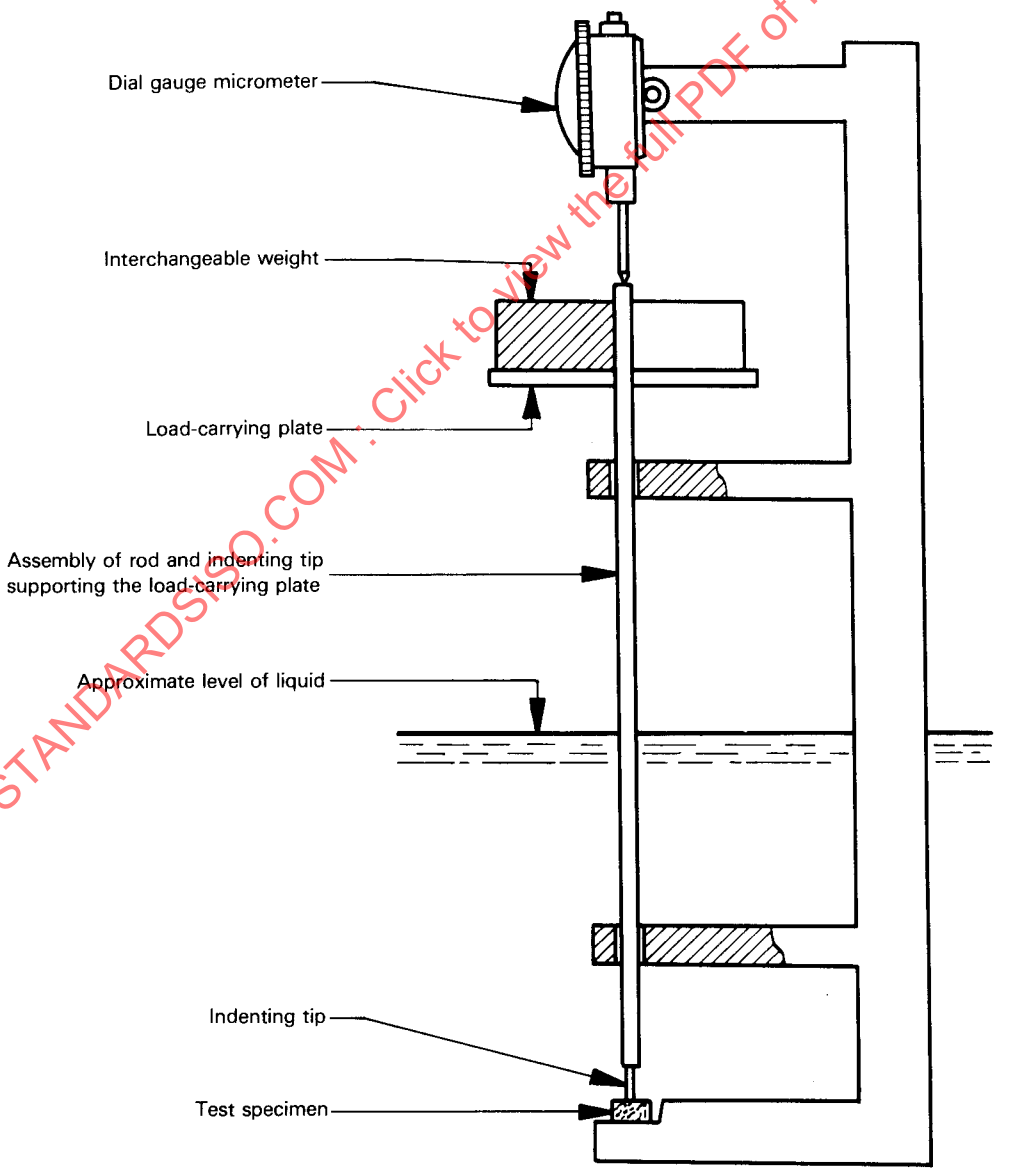

Figure — Example of apparatus for the determination of the Vicat softening temperature