INTERNATIONAL STANDARD

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Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST)

Plastiques — Matières thermoplastiques — Détermination de la température de ramollissement Vicat (VST)

Reference number
ISO 306:1994(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 306 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

This third edition cancels and replaces the second edition (ISO 306:1987), which has been technically revised.

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Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST)

1 Scope

1.1 This International Standard specifies four methods for the determination of the Vicat softening temperature (VST) of thermoplastic materials:

— Method A50 using a force of 10 N and a heating rate of 50 °C/h

— Method B50 using a force of 50 N and a heating rate of 50 °C/h

— Method A120 using a force of 10 N and a heating rate of 120 °C/h

— Method B120 using a force of 50 N and a heating rate of 120 °C/h

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 Normative references

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

ISO 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO 294:—[1], *Plastics — Injection moulding of test specimens of thermoplastic materials.*

ISO 2818:—[2], *Plastics — Preparation of test specimens by machining.*

ISO 3167:1993, *Plastics — Multipurpose 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 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 figure 1).

Unless the rod and frame members have the same linear thermal expansion coefficient, the differential

1) To be published. (Revision of ISO 294:1975)

2) To be published. (Revision of ISO 2818:1980)

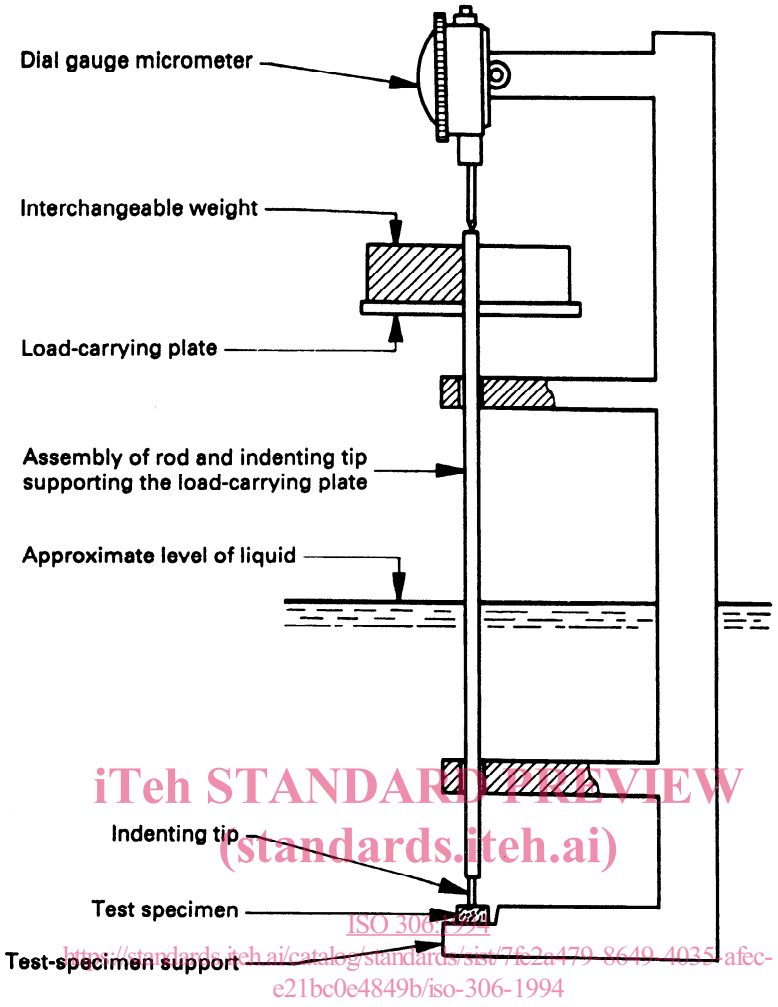

Figure 1 — Example of apparatus with a heating bath for the determination of the VST

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.[3] 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 $mm^2 \pm 0{,}015$ 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, shall be recorded (see 4.4).

NOTES

1 In certain types of apparatus, the force of the dial gauge spring is directed upwards and is subtracted from the load; in other forms, this force acts downwards 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.

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 N ± 0,2 N for methods A50 and A120 and

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

50 N ± 1 N for methods B50 and B120. 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 equipment, consisting of a heating bath (4.5.1) containing a liquid or an oven (4.5.2) with forced circulation of air or nitrogen.

The heating equipment shall be provided with a means of control so that the temperature can be raised at a uniform rate of either 50 °C/h ± 5 °C/h or 120 °C/h ± 10 °C/h, as required. The requirement for the heating rate shall be considered satisfied if, over every 6 min interval during the test, the temperature change is 5 °C ± 0,5 °C or 12 °C ± 1 °C, respectively.

The apparatus may be arranged to shut off the heat automatically and sound an alarm when the specified indentation has been reached (see 7.5).

4.5.1 Heating bath, containing a liquid in which the test specimen can be immersed to a depth of at least 35 mm. An efficient stirrer shall be provided. It shall be established that the liquid chosen is stable at the temperature used and does not affect the material under test, for example by swelling or cracking.

When a heating bath is used, the temperature of the liquid, measured close to the test specimen, shall be taken as the VST (see 7.5).

NOTE 3 Liquid paraffin, transformer oil, glycerol and silicone oil are suitable liquid heat-transfer media, but other liquids may be used.

4.5.2 Oven, with forced air or nitrogen circulation of about 60 times per minute, with a volume of not less than 10 litres for each apparatus and in which the air or nitrogen flow is directed perpendicular to the upper surface of the test specimen at a speed of 1,5 m/s to 2 m/s.

The result of the test will depend on the rate of transfer of heat from the circulating air or nitrogen to the surface of the test specimen. Because of the relatively small test specimen and the fact that the lower surface is in contact with the test-specimen support, the air or nitrogen temperature shall not be taken as the VST.

Take the temperature indicated by a sensor in the rod close to the indenting tip, or in the test-specimen support, as the VST.

For an initial calibration, verify by experiment that the temperature indicated by the sensor is within ± 1 °C of the temperature that is indicated by an additional sensor embedded within a blank test specimen.

NOTE 4 Commercially available ovens are often furnished with means for suitable air or nitrogen circulation. If not, the necessary heat-transfer rate may be ensured by fitting stream plates which direct the circulating air or nitrogen perpendicular to the upper surface of the test specimen.

4.6 Temperature-measuring instrument.

4.6.1 For a heating bath

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 at the depth of immersion required by 7.2.

4.6.2 For an oven with air or nitrogen circulation

Suitable temperature-measuring instrument of appropriate range and accurate to within 0,5 °C. The sensor (thermocouple or Pt 100) shall be positioned in the rod close to the indenting tip or in the test-specimen support (see 4.5.2).

5 Test specimens

5.1 At least two test specimens shall be used to test each sample. The test specimens shall be between 3 mm 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 the 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 mm to 6,5 mm thick, in accordance with the 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 mm 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 direct contact to give a total thickness between 3 mm 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 give the same test result.

5.4 The test results obtained may depend on the moulding conditions used in the preparation of the test specimens, although such a dependence is not common. When testing materials for which the results do depend on the moulding conditions, special annealing or preconditioning procedures may be used before testing provided 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 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 Place the assembly in the heating equipment (4.5). The temperature of the heating equipment shall be 20 °C 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. When a heating bath (4.5.1) is used, the bulb of the thermometer or the sensitive part of the temperature-measuring instrument (4.6.1) 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 a sufficient weight to the load-carrying plate (4.4) so that the total thrust on the test specimen is 10 N ± 0,2 N for methods A50 and A120 and 50 N ± 1 N for methods B50 and B120. 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 heating equipment at a uniform rate of 50 °C/h ± 5 °C/h or, alternatively, 120 °C/h ± 10 °C/h; when a heating bath is used stir the liquid well during the test. For referee tests, a rate of 50 °C/h shall be used.

NOTE 5 For some materials at the higher rate (120 °C/h), Vicat softening temperatures up to 10 °C higher can be observed.

7.5 Note the temperature of the bath (see 4.6.1) or the built-in sensor (see 4.6.2) at which the indenting tip has penetrated into the test specimen by 1 mm ± 0,01 mm beyond its starting position defined in 7.3, and record it as the 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 °C, record the individual results [see clause 9, h)] and repeat the test once using a further set of at least two specimens (see 5.1).

8 Precision

The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added at the following revision.

9 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 (A50, A120, B50 or B120);

d) the thickness and the number of layers of composite test specimens (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 heat-transfer medium used;

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 unusual characteristics of the test specimen noted during the test or after removal from the apparatus.

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