INTERNATIONAL STANDARD

ISO 306

Fourth edition
2004-07-15

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

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

Reference number
ISO 306:2004(E)

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Published in Switzerland

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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 306 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

This fourth edition cancels and replaces the third edition (ISO 306:1994), which has been technically revised to address new equipment designs in which the specimen is not heated in a liquid bath but by direct contact with, for instance, a hot metal block. The oven used as one of the possible items of heating equipment in ISO 306:1994 is no longer included.

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

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

ISO 294-1, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens*

ISO 294-2, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 2: Small tensile bars*

ISO 294-3, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates*

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

ISO 3167, *Plastics — Multipurpose test specimens*

3 Principle

The temperature at which a standard indenting tip with a flat point penetrates 1 mm into the surface of a plastic test specimen is determined. The indenting tip exerts a specified force perpendicular to the test specimen, while the specimen is heated at a specified and uniform rate.

The temperature, in degrees Celsius, of the specimen, measured as close as possible to the indented area at 1 mm penetration, is quoted as the VST.

4 Apparatus

The apparatus consists essentially of the following:

4.1 Rod, provided with a **weight-carrying plate or other suitable load-applying device** (see 4.4), held in a **rigid metal frame** in a **liquid-filled bath or direct-contact heating unit** so that it can move freely in the vertical direction. In either case, the base of the frame supports the test specimen under the indenting tip at the end of the rod (see Figures 1 and 2).

Unless the rod has the same linear thermal expansion coefficient as the rigid metal frame, the differential change in the length of these parts introduces an error in the indentation readings. A blank test shall therefore be carried out on each rod and frame assembly, using a test specimen made of rigid material having a known, low coefficient of expansion.[1)] The blank test shall cover the temperature range typical of the type of material to be tested. A correction factor shall be determined for at least each 10 °C change in temperature, for each rod and frame assembly. If the correction factor is 0,02 mm or greater near the VST for that material, its algebraic sign shall be noted and the factor applied to each test result by adding it algebraically to the apparent indentation reading. It is recommended that the apparatus be constructed of low thermal expansion material.

4.2 Indenting tip, preferably of hardened steel, 1,5 mm to 3 mm long, of circular cross-section, and of area 1,000 $mm^2 \pm 0{,}015\ mm^2$ (corresponding to an indenting-tip diameter of 1,128 mm ± 0,008 mm), fixed at the bottom of the rod (4.1). The surface of the indenting tip in contact with the specimen 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).

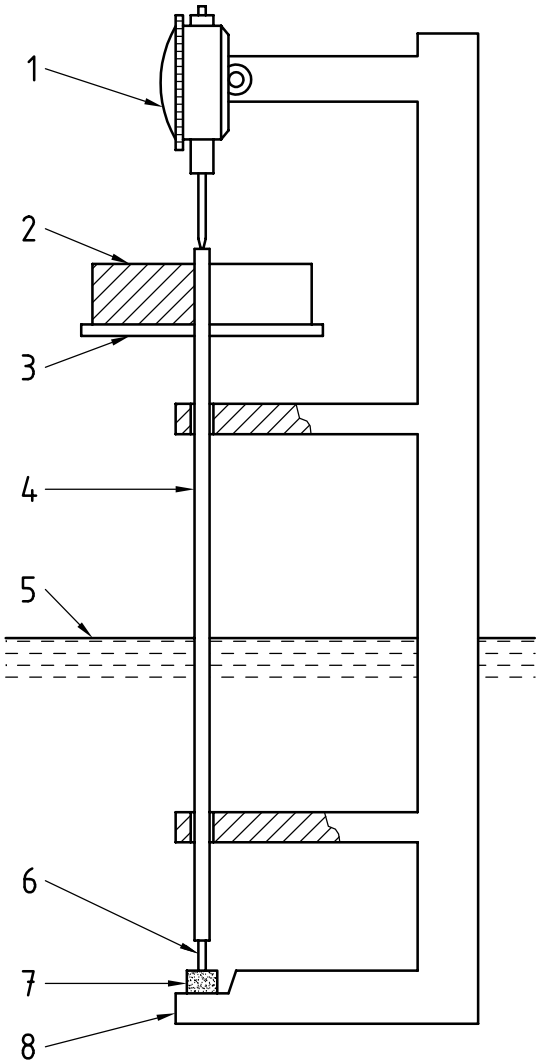
NOTE 1 In certain types of apparatus, the force of the dial gauge spring is directed upwards and is subtracted from the load; in other types, this force acts downwards and is added to the load.

NOTE 2 Since the force exerted by the spring in certain dial gauges varies considerably over the stroke, this force is measured at the position where the indenting tip has penetrated 1 mm into the specimen.

4.4 Weight-carrying plate, fitted to the rod (4.1), and **suitable weights** added centrally so that the total load applied to the test specimen can be made up to 10 N ± 0,2 N for methods A50 and A120 and 50 N ± 1 N for methods B50 and B120. The combined downward thrust, determined during calibration of the apparatus, due to the rod, the indenting tip, the weight-carrying plate and the upward or downward force exerted by the dial gauge spring in the measurement range used during the test, shall not exceed 1 N.

Other suitable devices for applying the load can be used provided the requirements specified above are met.

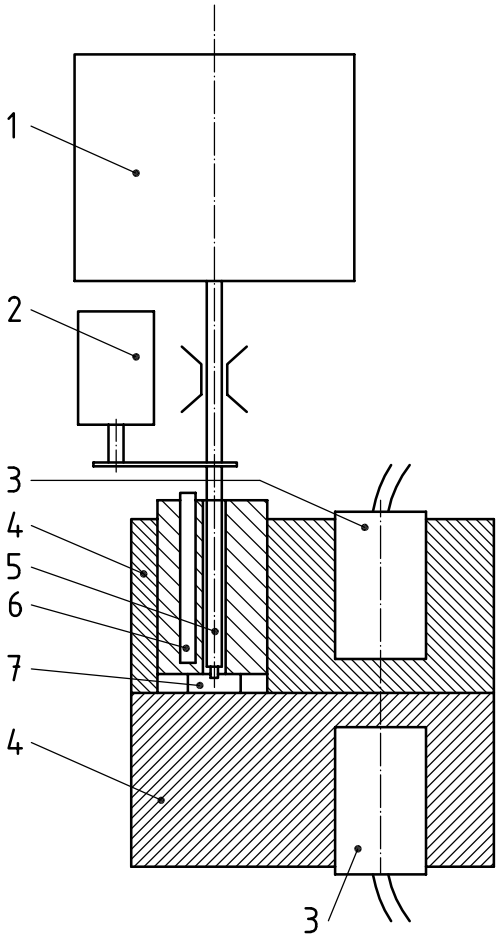
1) Invar and borosilicate glass have been found suitable for this purpose.



Key

1 micrometer dial gauge
2 replaceable weight
3 weight-carrying plate
4 rod with indenting tip
5 approximate level of liquid
6 indenting tip
7 test specimen
8 test-specimen support

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



Key

1 weight
2 displacement-measurement device
3 heater
4 heating block
5 rod with indenting tip
6 temperature-measurement unit
7 test specimen

Figure 2 — Example of apparatus with a direct-contact heating unit for determination of the VST

4.5 Heating equipment, consisting of a heating bath (4.5.1) containing a liquid or of a direct-contact heating unit (4.5.2). The heating equipment shall be provided with temperature-control means enabling the temperature to be raised at a uniform rate of 50 °C/h ± 5 °C/h or 120 °C/h ± 10 °C/h

The heating rate shall be verified

— either by checking the automatically read temperature;

— or by checking manually the temperature change at intervals of, at the most, 6 min during the whole of the test.

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. For multi-position baths, the heating rate shall be verified at each test station.

The apparatus may be designed 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.

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

4.5.2 Direct-contact heating unit, containing heaters and blocks which, through conductive heating, raise the temperature of the specimen at a controlled rate until the VST is reached.

4.6 Temperature-measuring instrument

4.6.1 For a heating bath

Use a mercury-in-glass thermometer of the partial-immersion type, or another 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. For mechanical and thermal reasons, the temperature-measuring instrument shall not make direct contact with the specimen.

4.6.2 For a direct-contact heating unit

Use a suitable temperature-measuring instrument of appropriate range and accurate to within 0,5 °C. The sensor shall be positioned as close as possible to both the indenting tip and the specimen, but avoiding direct contact between the sensor and specimen.

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 as agreed upon by the interested parties.

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 294-1, ISO 294-2, ISO 294-3 or ISO 3167 if no material specification exists. If these are not applicable, other procedures maybe used as agreed between the interested parties.

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

Condition in accordance with ISO 291 or with the appropriate material specification.

7 Procedure

7.1 If using a heating bath (4.5.1), mount the test specimen horizontally under the indenting tip (4.2) of the unloaded rod (4.1), perpendicular to the indenting tip. If using a direct-contact heating unit (4.5.2), place the test specimen horizontally and perpendicular to the direction of travel of the indenting tip, without placing the indenting tip on the specimen.

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 If using a heating bath, place the rod/frame assembly in the bath. If using a direct-contact heating unit, position the specimen between the two blocks and lower the indenting tip on to the specimen. The temperature of the heating equipment shall be a maximum of 25 °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 is used, the bulb of the thermometer or the sensor of the temperature-measuring instrument (see 4.6.1) shall be at the same level as, and as close as possible to, the test specimen. If using a direct-contact heating unit, the sensor shall be positioned in the heating block, as close as possible to the specimen as specified in 4.6.2.

7.3 With the indenting tip still in position, add a sufficient weight to the weight-carrying plate (4.4) (or load the indenting tip in another suitable way), so that the total thrust on the test specimen will be 10 N ± 0,2 N for methods A50 and A120 and 50 N ± 1 N for methods B50 and B120. After 5 min with the load applied, note the reading of the indentation-measuring instrument) (see 4.3) or set the instrument to zero.

7.4 Increase the temperature at a uniform rate of 50 °C/h ± 5 °C/h or 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 For some materials tested at the higher heating rate (120 °C/h), Vicat softening temperatures can be observed which are up to 10 °C higher than those obtained when testing at 50 °C/h.

7.5 Note the temperature of the bath (see 4.6.1) or the heating block (see 4.6.2) when the indenting tip has penetrated into the test specimen by 1 mm ± 0,01 mm from its starting position as 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, unless the range of individual results exceeds 2 °C. If the range is greater than 2 °C, record the individual results [see Clause 9, item h)] and repeat the test a second time using an additional set of at least two specimens (see 5.1). In the event of repeat testing, report the individual values from both the first and second tests. Report the VST to three significant figures.

8 Repeatability

See Annex B.

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 method used to heat the specimens;

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

h) the mean Vicat softening temperature (VST) of the material, in degrees Celsius, unless the range of the first set of results exceeds 2 °C in which case all the individual results shall be reported;

i) any unusual characteristics of the test specimens noted during the test or after removal from the apparatus;

j) the date of the test.

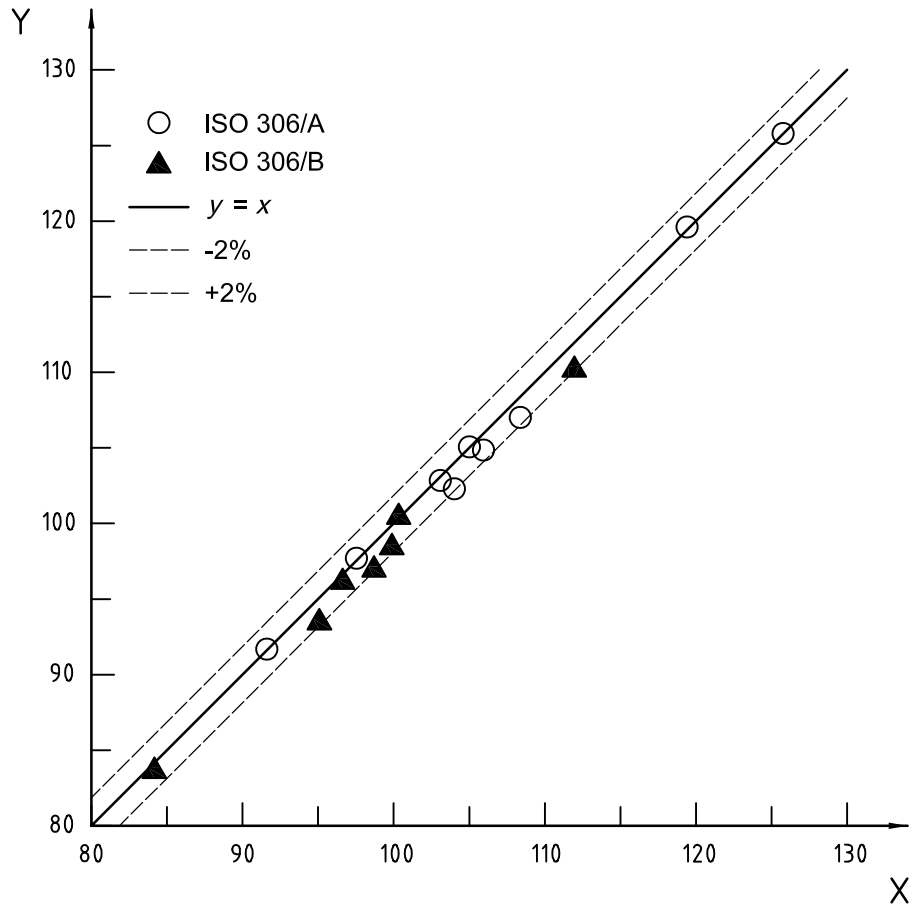
Annex A
(informative)

Comparison of VST results obtained with heating bath and direct-contact heating unit

A study was conducted to determine the VST of 10 materials measured using a heating bath containing silicone oil and a direct-contact heating technique in which heat was transmitted to the specimens by direct contact with metal surfaces. The results are shown in Table A.1 and Figure A.1, all values falling within a scatter band of $\pm$ 2 %. The slope of the regression curve is 1,008, suggesting that the difference in VST between the two heating techniques is less than 1 %. Hence, for practical purposes the two techniques can be considered to give identical values.

Table A.1 — Results of comparative study (heating rate 50 °C/h**)**

Test material	Type of material	VST using heating bath		VST using direct-contact heating	
		10 N load	50 N load	10 N load	50 N load
PE 4261 A	Polyethylene	125,6 °C	—	125,9 °C	—
PE Sample 1	Polyethylene	91,4 °C	—	91,7 °C	—
PE Sample 2	Polyethylene	97,4 °C	—	97,7 °C	—
Terluran GP-22	ABS	105,8 °C	99,6 °C	105,0 °C	98,5 °C
Terluran GP-35	ABS	103,7 °C	96,4 °C	102,3 °C	96,2 °C
Terluran HI-10	ABS	104,9 °C	98,5 °C	105,1 °C	97,0 °C
Terluran EGP-7	ABS	108,2 °C	100,1 °C	107,1 °C	100,5 °C
Terluran HH-112	ABS	119,3 °C	111,8 °C	119,7 °C	110,3 °C
Terluran 967K	ABS	103,0 °C	94,9 °C	102,8 °C	93,5 °C
PS 143 E	Polystyrene	—	84,0 °C	—	83,7 °C



Key

X VST using heating bath, °C

Y VST using direct-contact heater, °C

Linear regression:

$$y = -1{,}291\,23 + 1{,}007\,94x$$

$$R^2 = 0{,}994\,65$$

Figure A.1 — Plot of data presented in Table A.1

Annex B
(informative)

Repeatability

The data given in Table B.1 are based on a repeatability study, using method A120, involving one laboratory testing four materials. Three replicates were tested at two different times.

Table B.1 — Results of repeatability study

Material	Average	s_r [a]	r [b]
PC	154,50	0,71	1,98
ABS	108,40	0,14	0,40
PP	145,60	0,21	0,59
PMMA	125,10	0,07	0,20

[a] s_r = within-laboratory standard deviation

[b] $r = 2{,}83 s_r$

r is the interval representing the critical difference (95 % confidence level) between two test results for the same material obtained by the same operator using the same equipment in the same laboratory.

This explanation of r is only intended to present a meaningful way of considering the approximate repeatability of the test method. The data in Table B.1 should not be rigorously applied to acceptance or rejection of material, as they are specific to the study and may not be representative of other lots, conditions, materials or laboratories.

ICS 83.080.20

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