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

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

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received. www.iso.org/patents

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

This fifth edition cancels and replaces the fourth edition (ISO 306:2004), which has been technically revised. The main changes are the following additions:

- new apparatus, namely heating equipment consisting of a fluidized bed;
- precision data based on round robin testing performed in 2009;
- comparison data for tests with liquid-filled and fluidized bed.

Introduction

This revision introduces heating equipment, consisting of a fluidized bed, as a new apparatus; this is as an alternative to liquid-filled heating baths and direct-contact heating units. Fluidized beds can reach higher temperatures than traditional liquid-filled heating baths; therefore, they represent a suitable way to measure the Vicat softening temperature (VST) of thermoplastic materials having improved thermo-mechanical properties.

It was also felt necessary to add

- precision data based on round robin testing performed in 2009, and
- comparison data for tests with liquid-filled and fluidized bed.

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

1 Scope

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 K/h;
- method B50 using a force of 50 N and a heating rate of 50 K/h;
- method A120 using a force of 10 N and a heating rate of 120 K/h;
- method B120 using a force of 50 N and a heating rate of 120 K/h.

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 documents, in whole or in part, are normatively referenced in this document and are indispensable to its application. 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 472, *Plastics — Vocabulary*

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

ISO 20753, *Plastics — Test specimens*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply.

3.1

penetration

distance over which the indenting tip has to penetrate into the specimen under test

Note 1 to entry: It is expressed in millimetres (mm).

**3.2
load**

force applied to test specimen by means of the indenting tip

Note 1 to entry: It is expressed in Newtons (N).

**3.3
Vicat softening temperature
VST**

temperature at which a flat-ended needle penetrates the specimen to a depth of 1 mm under a specified load using a selected uniform rate of temperature rise

Note 1 to entry: It is expressed in degrees Celsius (°C).

4 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.

5 Apparatus

5.1 Heating equipment, consisting of one of the following (5.1.1, 5.1.2 or 5.1.3) that will accept a minimum of two test frame assemblies and a cooling device (5.1.4).

The heating equipment shall be provided with means of temperature control, enabling the temperature to be raised at a uniform rate of (50 ± 5) K/h or (120 ± 10) K/h.

The heating rate shall be verified

- either by checking and recording automatically over the whole temperature range, or
- by manually checking and recording the temperature change at 6-min intervals over the temperature range being verified.

The requirement for the heating rate shall be considered satisfied if, over every 6-min interval during the test, the temperature change is $(5 \pm 0,5)$ °C or (12 ± 1) °C, respectively. For multiposition heating equipment, 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.

5.1.1 Liquid-filled heating bath, containing a liquid in which the test specimen can be immersed to a depth of at least 35 mm. Liquid paraffin, transformer oil, glycerol and silicone oil are suitable liquid heat-transfer media, but other liquids may be used. 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. Do not heat the liquid filled heating bath in excess of the flash point specified by the heat transfer media manufacturer.

5.1.2 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.

5.1.3 Fluidized bed, containing a powder bed (e.g. aluminium oxide powder), in which the test specimen can be immersed to a depth of at least 35 mm. This type of apparatus uses a micrometric aluminium oxide powder, which when mixed with a suitable flow of heated air, creates a liquid-like heating medium. The maximum working temperatures (and measurable VSTs) are therefore much higher than those attainable

with liquids according to 5.1.1. An efficient stirring mechanism shall be provided, in order to achieve a temperature homogeneity in the specimen area analogous to the case of a liquid-filled heating bath.

5.1.4 Cooling device, as an optional means to reduce the temperature of the heating device; it may be used to reduce the time between tests.

5.2 Test frame assemblies (see Figures 1 and 2), consisting of the following.

5.2.1 Rod and frame, provided with a support plate or other suitable load-application device, held in a rigid metal frame. The rod shall be able to move freely, with minimum friction, in a vertical direction. The rod shall be designed to accept weights that will apply the test load. The base of the frame supports the test specimen under the indenting tip at the end of the rod (see Figures 1 and 2). It is recommended that the rod and the frame(s) be constructed of low thermal expansion material.

5.2.2 Indenting tip, preferably of hardened steel, 1,5 mm to 3 mm long, of circular cross-section and of area $(1,000 \pm 0,015) \text{ mm}^2$ (corresponding to an indenting-tip diameter of $(1,128 \pm 0,008) \text{ mm}$), fixed at the bottom of the rod (5.2.1). The surface of the indenting tip in contact with the specimen shall be flat and perpendicular to the axis of the rod, and free from burrs.

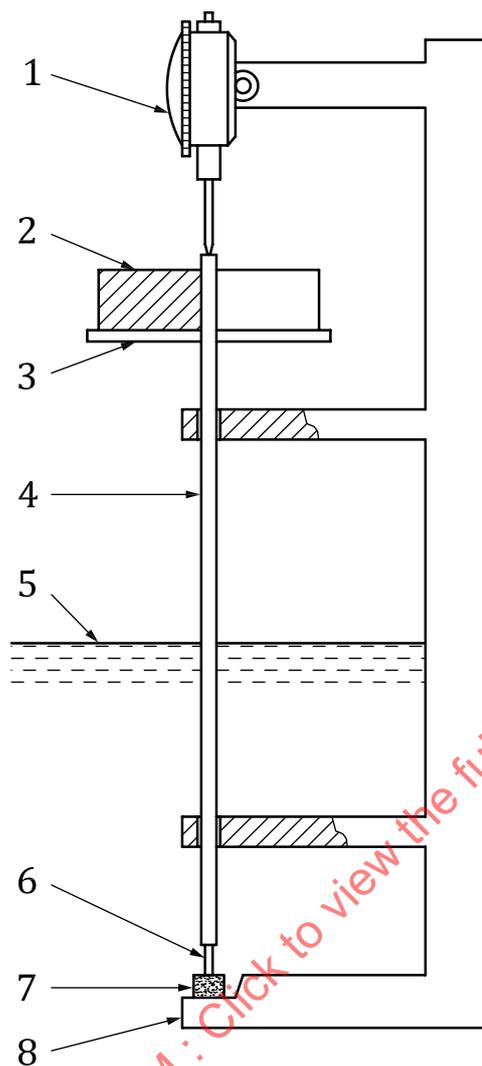
5.2.3 Weights, applied to the rod (5.2.1) centrally, so that the total load applied to the test specimen is $(10 \pm 0,2) \text{ N}$ for methods A50 and A120 and $(50 \pm 1) \text{ N}$ for methods B50 and B120.

5.2.4 Penetration-measuring device, calibrated micrometer dial gauge, LVDT (linear variable differential transformer) or other suitable measuring instrument to measure the penetration of the indenting tip into the test specimen to an accuracy of $\pm 0,01 \text{ mm}$.

5.2.5 Temperature-measuring device.

5.2.5.1 For a liquid-filled bath and a fluidized bed, use a suitable temperature-measuring instrument of appropriate range and accurate to within $\pm 0,5 \text{ K}$. Thermometers shall be calibrated at the depth of immersion required by 5.1.1 and 5.1.3. The temperature-measuring device shall be positioned as close as possible to both the indenting tip and the specimen, but avoiding direct contact between the sensor and specimen.

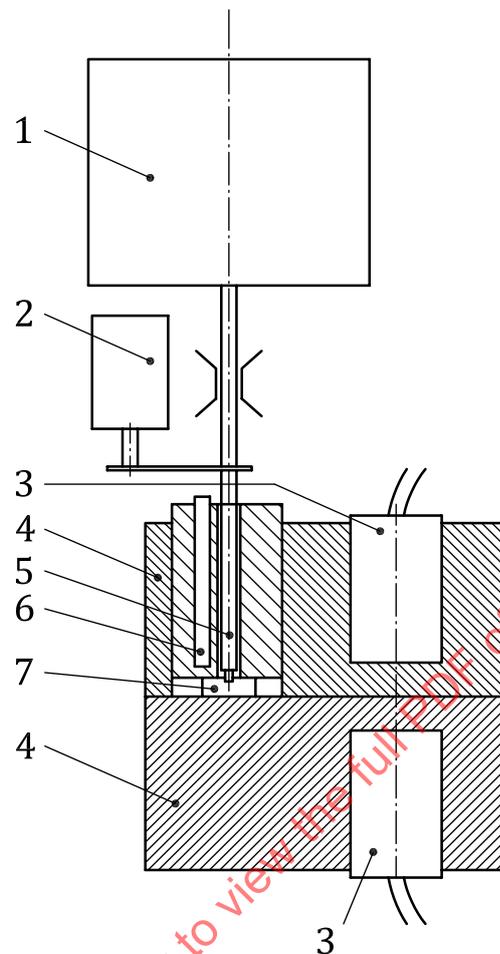
5.2.5.2 For a direct-contact heating unit, use a suitable temperature-measuring instrument of appropriate range and accurate to within $\pm 0,5 \text{ K}$. The sensor shall be positioned as close as possible to both the indenting tip and the specimen, but avoiding direct contact between the sensor and specimen.



Key

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

Figure 1 — Schematic view of one type of testing apparatus with heating equipment filled with liquid or fluidized powder bed 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 — Schematic view of testing apparatus with a direct-contact heating unit for determination of the VST

6 Test frame assembly calibration

6.1 When analogue dial gauges are used, the thrust of the dial gauge, which contributes to the thrust on the test specimen, shall be recorded. 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. 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. The combined downward thrust, determined during calibration of the apparatus, due to the rod, the indenting tip 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.

6.2 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 test shall therefore be carried out on each frame assembly, using a test specimen made of a rigid material known to have a low coefficient of expansion (e.g. quartz or borosilicate glass). This test shall cover the temperature range typical of the type of material to be tested. A correction 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.

7 Test specimens

7.1 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.

7.2 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 20753 if no material specification exists. If these are not applicable, other procedures may be used as agreed between the interested parties.

7.3 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 (specified in 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 of 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.

7.4 The test results obtained can 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.

8 Conditioning

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

9 Procedure

9.1 If using a liquid-filled heating bath (5.1.1) or a Fluidized bed (5.1.3), mount the test specimen horizontally under the indenting tip (5.2.2) of the unloaded rod (5.2.1), perpendicular to the indenting tip. If using a direct-contact heating unit (5.1.2), 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.

9.2 If using a liquid-filled heating bath or a fluidized bed, place the rod/frame assembly in the heating equipment. 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 liquid-filled heating bath or a fluidized bed is used, the bulb of the thermometer or the sensor of the temperature-measuring instrument (see [5.2.5.1](#)) 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 [5.2.5.2](#).

9.3 With the indenting tip still in position, add a sufficient weight to the support plate (or load the indenting tip in another suitable way), so that the total thrust on the test specimen will be $(10 \pm 0,2)$ N for methods A50 and A120 and (50 ± 1) N for methods B50 and B120. After 5 min with the load applied, note the reading of the indentation-measuring instrument) (see [5.2.4](#)) or set the instrument to zero.

9.4 Increase the temperature at a uniform rate of (50 ± 5) K/h or (120 ± 10) K/h. When a liquid-filled heating bath or a fluidized bed is used, stir the heating medium well during the test. For referee tests, a rate of 50 K/h shall be used.

NOTE For some materials tested at the higher heating rate (120 K/h), Vicat softening temperatures can be observed which are up to 10 °C higher than those obtained when testing at 50 K/h.

9.5 Note the temperature of the heating medium (see [5.2.5.1](#)) or the heating block (see [5.2.5.2](#)) when the indenting tip has penetrated into the test specimen by $(1 \pm 0,01)$ mm from its starting position as defined in [9.3](#), and record it as the VST of the test specimen.

9.6 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 K. If the range is greater than 2 K, record the individual results [see [Clause 11](#), list item h)] and repeat the test a second time using an additional set of at least two specimens (see [6.1](#)). In the event of repeat testing, report the individual values from both the first and second tests. Report the VST to three significant figures.

10 Precision

For precision, see [Annex C](#).

11 Test report

The test report shall include the following information:

- a) a reference to this International Standard, i.e. ISO 306;
- 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 type of heating equipment;
- 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 K, in which case, all the individual results shall be reported;

- i) any unusual characteristics of the test specimen noted during the test or after removal from the apparatus.
- j) the date of the test.

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Annex A (informative)

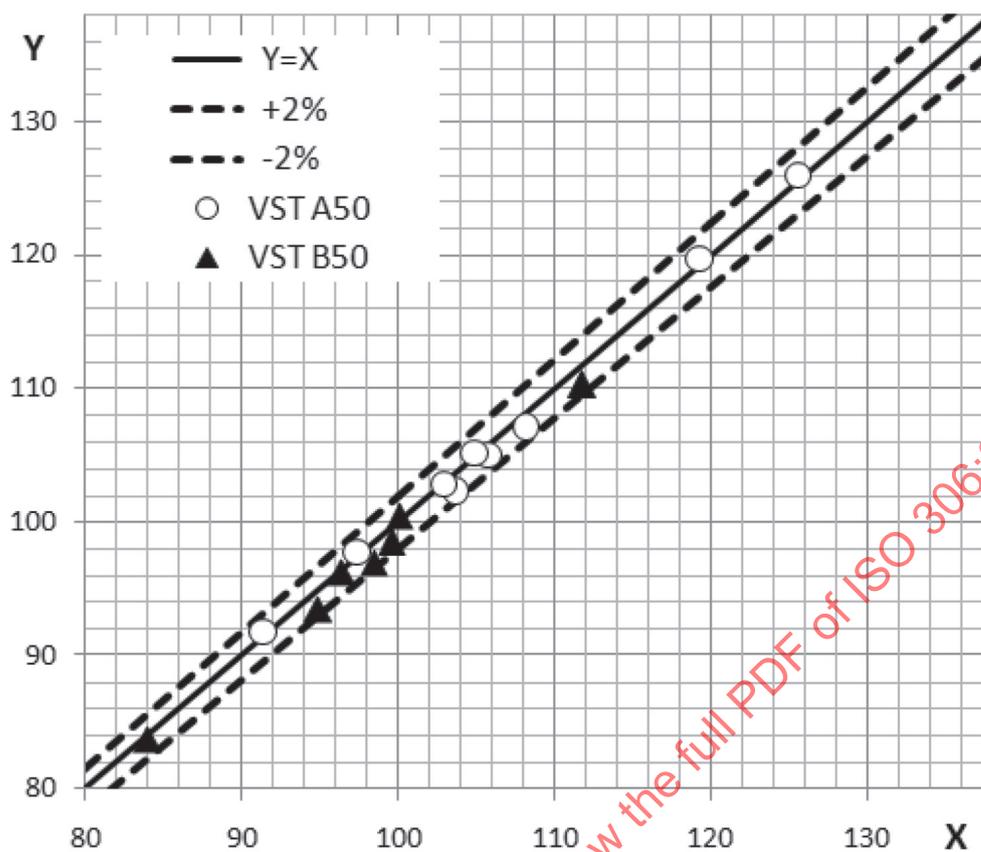
Comparison of VST results obtained with liquid-filled heating bath and direct-contact heating unit

A study was conducted to determine the VST of 10 materials measured using a liquid-filled 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 ± 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 may be considered to give identical values.

NOTE These data was obtained by round robin testing in 2009 (see [Annex C](#)).

Table A.1 — Results of comparative study (heating rate 50 K/h), VST [°C]

| Test material | Type of material | VST using liquid-filled 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 | — | 125,9 | — |
| PE Sample 1 | Polyethylene | 91,4 | — | 91,7 | — |
| PE Sample 2 | Polyethylene | 97,4 | — | 97,7 | — |
| Terluran GP-22 | ABS | 105,8 | 99,6 | 105,0 | 98,5 |
| Terluran GP-35 | ABS | 103,7 | 96,4 | 102,3 | 96,2 |
| Terluran HI-10 | ABS | 104,9 | 98,5 | 105,1 | 97,0 |
| Terluran EGP-7 | ABS | 108,2 | 100,1 | 107,1 | 100,5 |
| Terluran HH-12 | ABS | 119,3 | 111,8 | 119,7 | 110,3 |
| Terluran 967K | ABS | 103,0 | 94,9 | 102,8 | 93,5 |
| PS 143E | Polystyrene | — | 84,0 | — | 83,7 |



Key

X VST using liquid-filled heating bath

Y VST using direct-contact heater

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)

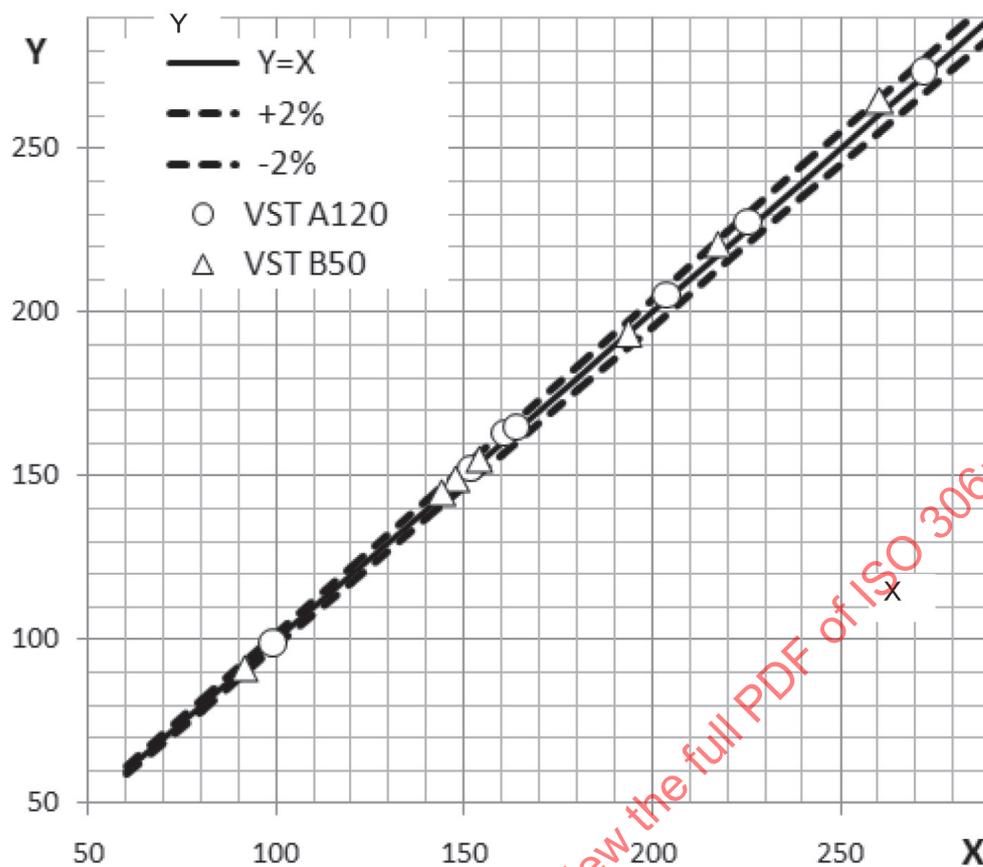
Comparison of VST results obtained with liquid-filled heating bath and fluidized bed

A comparison study was conducted measuring the VST (method B50 and method A120) by means of a liquid-filled heating bath apparatus containing silicone oil (for seven materials) and a fluidized bed apparatus with aluminium oxide powder (for 11 materials). The results obtained and the number of laboratories involved are shown in [Table B.1](#) and [Figure B.1](#). These results show that all values fall within a scatter band of ± 2 %. The slope of the regression curve is 1,0157 (linear regression with a correlation coefficient of 0,999 7). Hence, for practical purposes the two techniques may be considered to give identical values over the common temperature range.

NOTE These data were obtained by round robin testing in 2009 (see [Annex C](#)).

Table B.1 — Results of comparative study, VST [°C]

| — | | Test method B50 | | Test method A120 | |
|-------------------------------|------------------------|--|------------------------------|--|------------------------------|
| | | Heating rate 50 K/h 50 N load | | Heating rate 120 K/h 10 N load | |
| Material | Type of material | VST using liquid-filled heating bath | VST using flu- idized bed | VST using liquid-filled heating bath | VST using flu- idized bed |
| PS | Polystyrene | 91,6 | 91,5 | 99,2 | 98,9 |
| POM 1 | Polyoxymethylene | 147,8 | 149,1 | 160,7 | 162,9 |
| PC | Polycarbonate | 144,1 | 144,8 | 151,7 | 152,3 |
| POM 2 | Polyoxymethylene | 153,9 | 155,3 | 163,9 | 164,6 |
| PPE | Polyphenylene ether | 193,4 | 193,4 | 203,9 | 205,3 |
| PES | Polyetersulfone | 217,4 | 220,8 | 225,5 | 227,4 |
| PPS | Polyphenylene sulfide | 260,4 | 264,6 | 272,3 | 273,5 |
| LCP 1 | Liquid-crystal polymer | — | 231,9 | — | 302,3 |
| LCP 2 | Liquid-crystal polymer | — | 221,8 | — | 303,2 |
| PEEK | Polyetheretherketone | — | 330,3 | — | 340,0 |
| LCP 4 | Liquid-crystal polymer | — | 269,6 | — | 361,4 |
| Number of laboratories | | 6 | 2 | 7 | 2 |
| Number of materials | | 7 | 11 | 7 | 11 |



Key

X VST using liquid-filled heating bath [°C]

Y VST using fluidized bed [°C]

Linear regression

$$y = -1,508\ 64 + 1,015\ 74x$$

$$R^2 = 0,999\ 69$$

Figure B.1 — Plot of data presented in Table B.1 — Methods B50 and A120

Annex C (informative)

Repeatability and precision

C.1 Precision

Round robin testing involving 11 materials and seven laboratories was conducted in 2009 in accordance with ISO 5725-2 to determine the precision of the method specified in the previous edition of this International Standard.

C.2 Test conditions

Specimens made of 11 different materials were sent to seven laboratories. Test specimens for each material were injection-moulded by one laboratory which offered the sample.

The other test conditions were the following:

- test method: the previous edition of this International Standard;
- heating equipment: liquid-filled, fluidized bed;
- specimens tested; six (three specimens, twice);
- methods: A120 (10N, 120 K/h) and B50 (50N, 50 K/h).

Not every laboratory tested every material using every type of equipment. For liquid-filled heating bath, seven laboratories tested seven materials. For fluidized bed, two laboratories tested 11 materials. Some materials could not be tested with a liquid-filled heating bath because their VST was too high for the silicone oil.

C.3 Precision data

The results are shown in [Tables C.1](#), [C.2](#), [C.3](#) and [C.4](#).

In [Tables C.1](#) to [C.4](#), the statistical properties used are:

s_r = within laboratory standard deviation;

s_R = between laboratory standard deviation;

r = 95 % repeatability limit = 2,8 s_r ;

R = 95 % reproducibility limit = 2,8 s_R .