Designation: D1525 – 17

Standard Test Method for Vicat Softening Temperature of Plastics[1]

This standard is issued under the fixed designation D1525; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers determination of the temperature at which a specified needle penetration occurs when specimens are subjected to specified controlled test conditions.

1.2 This test method is not recommended for ethyl cellulose, nonrigid poly(vinyl chloride), poly(vinylidene chloride), or other materials having a wide Vicat softening range.

1.3 The values stated in SI units are to be regarded as standard. No other units are included in this standard.

1.4 Due to the potential safety and environmental hazards associated with mercury-filled thermometers, the use of alternative temperature measuring devices (such as thermocouples and RTDs) is encouraged with liquid-in-glass thermometers containing mercury, the use of alternative measuring devices, such as digital thermometers using thermocouple of RTD sensors is encouraged.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health and environmental practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—This test method is equivalent to ISO 306 in all sections with the exceptions of the allowance for creep, prior to the beginning of the test and the allowance of the fluidized powder as a heat transfer medium.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

E1 Specification for ASTM Liquid-in-Glass Thermometers

E77 Test Method for Inspection and Verification of Thermometers

E608/E608M Specification for Mineral-Insulated, Metal-Sheathed Base Metal Thermocouples

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E887 Test Method for Silica in Refuse-Derived Fuel (RDF) and RDF Ash

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

2.2 *ISO Standards:*[4]

ISO 306 Plastics—Thermoplastic Material—Determination of Vicat Softening Temperature

3. Terminology

3.1 *Definitions:*

3.1.1 Definitions of plastics used in this test method are in accordance with those defined in Terminology D883, unless otherwise specified.

3.1.2 *Vicat softening temperature*—the temperature at which a flat-ended needle of 1-mm^2 circular cross section will penetrate a thermoplastic specimen to a depth of 1 mm under

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties (Section D20.30.07).

Current edition approved Aug. 1, 2017. Published August 2017. Originally approved in 1958. Last previous edition approved in 2009 as D1525 - 09. DOI: 10.1520/D1525-17.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

*A Summary of Changes section appears at the end of this standard

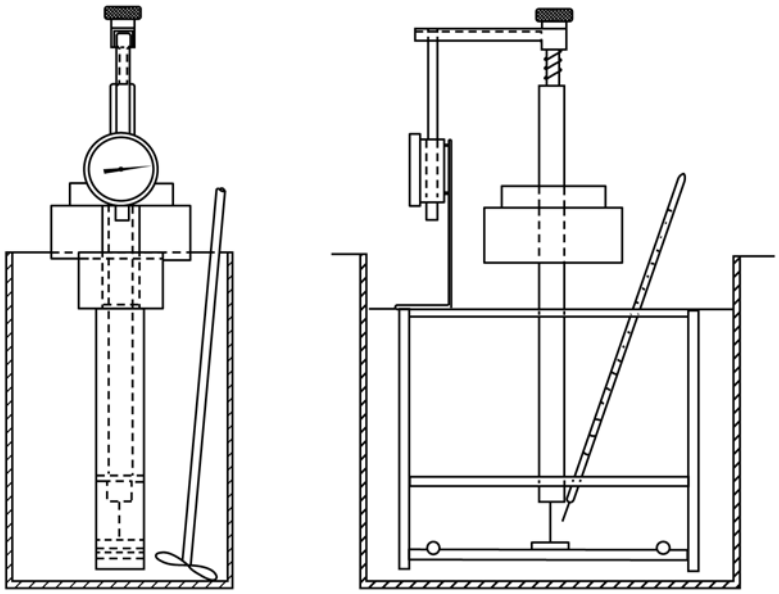


FIG. 1 Apparatus for Softening Temperature Determination (Immersion Bath)

a specified load (force) perpendicular to the test specimen using a selected uniform rate of temperature rise.

4. Summary of Test Method

4.1 A flat-ended needle loaded with a specified mass is placed in direct contact with a test specimen. The mass applied can be one of two accepted loads, as follows:

$$\text{Loading 1} - 10 \pm 0.2\,\text{N}$$

$$\text{Loading 2} - 50 \pm 1.0\,\text{N}$$

The specimen and needle are heated at either of two permissible rates, as follows:

$$\text{Rate A} - 50 \pm 5\,^{\circ}\text{C/h}$$

$$\text{Rate B} - 120 \pm 10\,^{\circ}\text{C/h}$$

The temperature at which the needle has penetrated to a depth of 1 ± 0.01 mm is recorded as the Vicat softening temperature.

5. Significance and Use

5.1 Data obtained by this test method is used to compare the heat-softening qualities of thermoplastic materials.

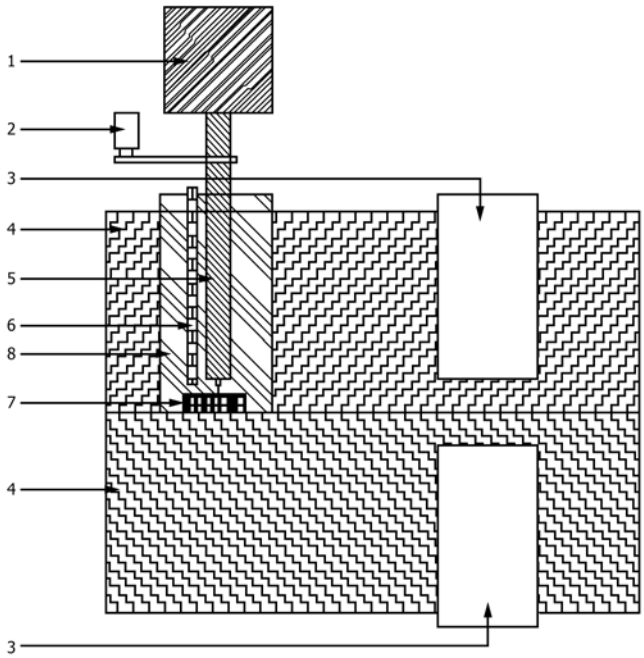
5.2 This test method is useful in the areas of quality control, development, and characterization of plastic materials.

6. Apparatus

6.1 The instrument shall have a heating system consisting of a heated bath (see Fig. 1) or a contact heating system (see Fig. 2):

The equipment shall be constructed essentially as shown in Fig. 2 and shall consist of the following:

6.1.1 *Immersion Bath*—An immersion bath containing the heat-transfer medium shall be equipped with an agitation system, temperature-measuring device, and heater. The heater shall have automatic control of the selected bath temperature-rise rate (see 4.1). The bath shall be constructed to allow the specimens to be submerged at least 35 mm below the surface of the heat-transfer medium.



Key
1 Weight
2 Displacement-measurement device
3 Heater(s) (Note that multiple heaters are placed symmetrically throughout the instrument and not just in the one location shown.)
4 Heating block (contains lower contact plate)
5 Load rod with needle
6 Temperature-measuring device
7 Test specimen
8 Contact plate

FIG. 2 Apparatus for Softening Temperature Determination (Direct Contact)

6.1.1.1 *Heat-Transfer Medium*—Shall be a liquid or fluidized powder.[5] Several liquids, such as silicone oils, glycerine, ethylene glycol, and mineral oil have been used successfully for various plastics.[6] The medium used shall be free from contaminants and shall have no short-time effect at elevated temperatures on the material being tested, liquid mediums shall be of low viscosity at room temperature.

NOTE 2—The results of the test can depend on the thermal diffusivity of the heat-transfer medium.

6.1.2 *Contact Heating Unit*—Shall consist of heater and heater blocks which, through conductive heating, raises the temperature of the specimen at the required controlled rate until the VICAT Softening Temperature is reached.

NOTE 3—It is desirable to have a method of cooling or heating the bath in order to reduce the time required to change the temperature of the bath between tests more quickly. This may be accomplished by using a cooling coil installed in the bath or an external heat-transfer system. If the temperature rise rate is adversely affected by the presence of residual coolant in the cooling coils, the coolant should be purged prior to beginning the test.

6.1.3 *Specimen Support*—A suitable stand or support for the specimen to be placed in the bath or be part of the contact heating structure. The vertical members that attach the specimen support to the upper plate shall be made of a material having the same coefficient of expansion as that used for the rod through which the load is applied in order that the penetration-measuring device reading caused by differential expansion over the intended temperature range does not exceed 0.02 mm when the specimen is replaced by a piece of heat-resistant material.[7]

6.1.4 *Penetration-Measuring Device*—The device used for measuring the penetration of the specimen shall be capable of measuring a penetration depth of at least 1 ± 0.01 mm. The measuring device shall be an analog or digital dial gauge or similar device, including an electronic-displacement sensing apparatus.

6.1.5 *Masses*—A set of masses of suitable sizes shall be supplied so that the net force on the needle point shall be equal to 10 ± 0.2 N (Loading 1) or 50 ± 1.0 N (Loading 2) when the apparatus is assembled. The net force shall consist of the weight of the needle rod assembly, the force attributed to action of the penetration-measuring device, and the extra weight that is required to balance the system. The required mass is calculated as follows:

$$\text{Required mass, } m_w = (F - F_s)/9.80665 - m_r$$

F = total force to be applied to the specimen, N,
F_s = force exerted by any penetration-measuring device, N (this is a positive value if the thrust of the spring is towards the specimen (downward), a negative value if the thrust of the spring is opposing the descent of the rod, or zero if no such device is involved),
m_r = mass of the needle rod assembly, kg, and
m_w = extra mass applied to attain the desired force, kg.

Verification of the load shall be made on new equipment and after replacing penetration-measuring devices, or at any time to ensure that the equipment is in calibration. The calibration procedure for dial-gauge-type penetration-measuring devices is described in Appendix X1 and Appendix X2. The methods for determination of the thrust contributed by dial-gauge-type penetration-measuring devices are also given in Appendix X1 and Appendix X2.

6.1.6 *Temperature-Measuring Device*, Consisting of a sensor (for example, thermocouple, resistance thermometer (RTD), thermistor probe (E887-12), or platinum resistance thermometer (PRT). or thermometer adequate to cover the range being tested. The thermometer shall be one of the following, or its equivalent, in accordance with Specification E1: Thermometer 1C or 2C, having ranges from –20 to 150°C or –5 to 300°C, respectively, depending on the test range. The thermocouple or resistance thermometer and related electronics shall be accurate to at least ±0.5°C. Liquid-in-glass thermometers (refer to Specification E2251) shall be calibrated for the depth of immersion in accordance with Test Method E77. Thermocouples shall comply with the requirements of Specification E608/E608M. Resistance thermometers shall comply with the requirements of Specification E1137/E1137M.

6.1.6.1 *Digital Thermometer*—A digital indicating device incorporating a sensor (that is, thermocouple probe, thermistor probe, or platinum resistance thermometer (PRT)) with associated conditioning, conversion, and readout instrumentation adequate to cover the range being tested. The sensor and related electronics shall be accurate to at least ±0.5°C. Thermocouples shall comply with the requirements of Specification E608/E608M. Resistance thermometers shall comply with the requirements of Specification E1137/E1137M.

6.1.6.2 *Liquid-in-Glass Thermometer*—Older systems still in existence use a thermometer for temperature measurement at each individual test station. The thermometer shall be one of the following, or its equivalent, as prescribed in Specification E1: Thermometer 1C or 2C, having ranges from –20 to 150°C or –5 to 300°C, respectively, whichever temperature range is most suitable. Liquid-in-glass thermometers (Refer to Specification E2251a) shall be calibrated for the depth of immersion in accordance with Test Method E77.

NOTE 4—WARNING—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website- http://www.epa.gov/mercury/faq.htm - for additional information. Users should be aware that selling mercury and/or mercury containing products into your state may be prohibited by state law.

6.1.7 *Needle*—A flat-tipped, hardened steel needle with a cross-sectional area of 1.000 ± 0.015 mm^2 (diameter of 1.120 to 1.137 mm) shall be used. The tip shall be free of burrs and be perpendicular to the axis of the rod. The needle shall protrude at least 2 mm from the end of the rod.

[5] Aluminum Oxide has been found satisfactory and safe for short-term heat cycles up to 500°C.

[6] Silicone oils having a room temperature viscosity of 100 cP have been found satisfactory and safe for short-term heat cycles up to 260°C.

[7] Borosilicate glass or Quartz has been found satisfactory for this purpose.