



Designation: D1525 – 17

# Standard Test Method for Vicat Softening Temperature of Plastics<sup>1</sup>

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 ( $\epsilon$ ) 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 or 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.*

<sup>1</sup> 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).

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## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

D618 Practice for Conditioning Plastics for Testing

D883 Terminology Relating to Plastics

D1898 Practice for Sampling of Plastics (Withdrawn 1998)<sup>3</sup>

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:<sup>4</sup>

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<sup>2</sup> circular cross section will penetrate a thermoplastic specimen to a depth of 1 mm under

<sup>2</sup> 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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

<sup>4</sup> 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

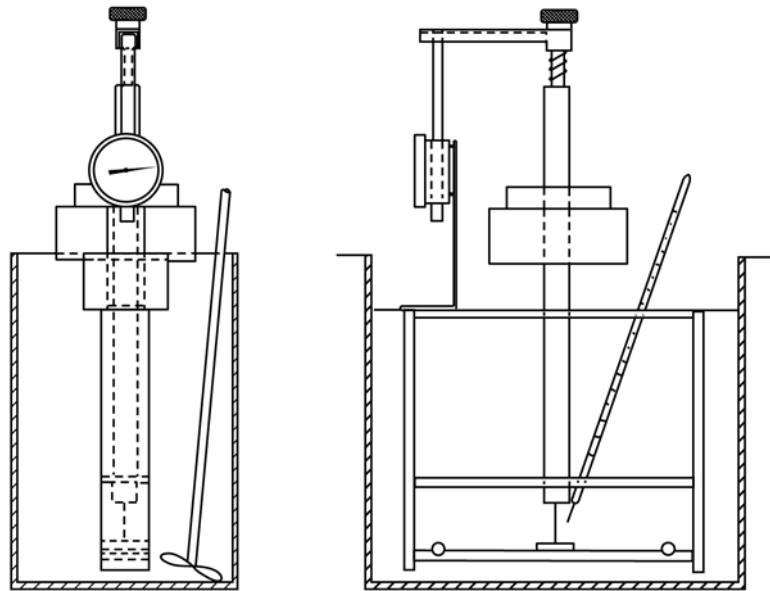


FIG. 1 Apparatus for Softening Temperature Determination (Immersion Bath)

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:

Loading 1 -  $10 \pm 0.2$  N

Loading 2 -  $50 \pm 1.0$  N

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

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

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

The temperature at which the needle has penetrated to a depth of  $1 \pm 0.01$  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.

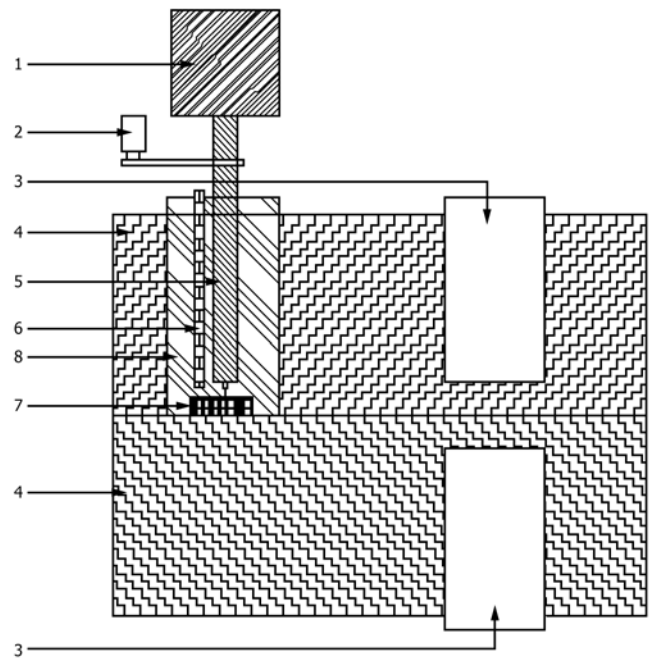
5.2 This test method is useful in the areas of quality control, development, and characterization of plastic materials.

#### 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



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)

specimens to be submerged at least 35 mm below the surface of the heat-transfer medium.

6.1.1.1 *Heat-Transfer Medium*—Shall be a liquid or fluidized powder.<sup>5</sup> Several liquids, such as silicone oils, glycerine, ethylene glycol, and mineral oil have been used successfully for various plastics.<sup>6</sup> 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.<sup>7</sup>

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  $1 \pm 0.01$  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  $10 \pm 0.2$  N (Loading 1) or  $50 \pm 1.0$  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  $150^\circ\text{C}$  or  $-5$  to  $300^\circ\text{C}$ , respectively, depending on the test range. The thermocouple or resistance thermometer and related electronics shall be accurate to at least  $\pm 0.5^\circ\text{C}$ . 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  $\pm 0.5^\circ\text{C}$ . 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  $150^\circ\text{C}$  or  $-5$  to  $300^\circ\text{C}$ , 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  $1.000 \pm 0.015$  mm<sup>2</sup> (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.

<sup>5</sup> Aluminum Oxide has been found satisfactory and safe for short-term heat cycles up to  $500^\circ\text{C}$ .

<sup>6</sup> Silicone oils having a room temperature viscosity of 100 cP have been found satisfactory and safe for short-term heat cycles up to  $260^\circ\text{C}$ .

<sup>7</sup> Borosilicate glass or Quartz has been found satisfactory for this purpose.

## 7. Sampling

7.1 Unless otherwise agreed upon between the seller and the purchaser, sample in accordance with the sections on General Sampling Procedures and Specific Sampling Procedures of Practice **D1898**. Sampling based on engineering principles, prior to packaging, shall be considered an acceptable alternative.

## 8. Test Specimen

8.1 Use at least two specimens to test each sample. The specimen shall be flat, between 3 and 6.5 mm thick, and at least 10 by 10 mm in area or 10 mm in diameter. If an individual layer of the specimen is less than 3 mm it is permissible to stack not more than three (3) layers to achieve the minimum thickness. The specimens shall be cut from sheet or molded material. The type of mold and the molding process used to produce test specimens will affect the results obtained in the test. Molding conditions shall be in accordance with the standard for the material being tested or be agreed upon between the cooperating laboratories.

**NOTE 5**—Discrepancies in test results due to variations in molding conditions often are minimized by annealing the test specimens before the test. Since different materials require different annealing conditions, annealing procedures shall be employed only if required by the material standard or if agreed upon between the cooperating laboratories.

## 9. Conditioning

9.1 If conditioning of the specimens is required, the test specimens shall be conditioned at  $23 \pm 2^\circ\text{C}$  and at  $50 \pm 10\%$  relative humidity for not less than 40 h in accordance with Procedure A of Practice **D618**, unless otherwise specified by agreement or the relevant ASTM material specification. In cases of disagreement, the tolerances shall be  $\pm 1^\circ\text{C}$  and  $\pm 5\%$  relative humidity.

**NOTE 6**—Conditioning periods less than the 40 h, as specified by Practice **D618**, may be used when it is shown that the Vicat softening temperature is not affected by the shorter conditioning time. Longer conditioning times may be required for some materials that take longer to reach temperature and humidity equilibrium. Refer to the applicable ASTM standards for those materials.

## 10. Procedure

10.1 Prepare the instrument so that the temperature of the heat-transfer medium or contact plates is between 20 and  $23^\circ\text{C}$  at the start of the test unless previous tests have shown that for a particular material under test no error is introduced by starting at a higher temperature. The immersion bath system shall be well agitated.

**NOTE 7**—Under certain conditions, it may be difficult to bring the temperature of the heat-transfer medium down to 20 to  $23^\circ\text{C}$ . In these cases, the test may be started with the bath temperature at  $30^\circ\text{C}$ . The selection of the starting temperature shall be agreed upon between the cooperating laboratories.

10.1.1 If it is not possible to reduce the temperature of the heat-transfer medium to 20– $23^\circ\text{C}$ , it is permissible to start with the bath temperature at  $30^\circ\text{C}$ . The selection of the starting temperature shall be agreed upon between the cooperating laboratories.

10.2 Place the specimen, which is at room temperature, on the specimen support so that it is approximately centered under the needle. Ensure that the needle is at least 3 mm from the edge of the specimen. Gently lower the needle rod, without the extra mass, so that the needle rests on the surface of the specimen and holds it in position.

10.3 Position the temperature measuring device so that the sensing end is located within 10 mm from where the load is applied to the surface of the specimen. Ensure that the sensing end does not touch the specimen.

10.4 Lower the assembly into the bath or close the contact plates, taking care not to jar it in any way that would damage or dislodge the specimen.

10.5 Apply the extra mass required to increase the load on the specimen to  $10 \pm 0.2\text{ N}$  (Loading 1) or  $50 \pm 1.0\text{ N}$  (Loading 2). After a 5-min waiting period, set the penetration indicator to zero.

10.6 Start the temperature rise. The rate of temperature increase shall be either  $50 \pm 5^\circ\text{C/h}$  (Rate A) or  $120 \pm 10^\circ\text{C/h}$  (Rate B) and shall be uniform throughout the test. The Rate A heating requirement shall be considered to be met if over every 12-min interval during the test, the temperature of the bath or contact plates rises  $10 \pm 1^\circ\text{C}$  at each specimen location. The Rate B heating requirement shall be considered to be met if over every 6-min interval during the test, the temperature of the bath or contact plates rises  $12 \pm 1^\circ\text{C}$  at each specimen location. The selection of the rate of rise shall be agreed upon between cooperating laboratories. See **Annex A1** for calibration of single temperature probe units.

10.7 Record the temperature of the bath or contact plates when the needle has penetrated  $1 \pm 0.01\text{ mm}$  into the test specimen. Make sure that an accurate reading of the temperature is made since the rate of penetration of the specimen will be increasing rapidly at this point.

10.8 Express the Vicat softening temperature as the arithmetic mean of the temperature of penetration of all specimens tested. If the range of penetration temperatures for the individual test specimens exceeds  $2^\circ\text{C}$ , record the individual results and repeat the test, using at least two new specimens.

**NOTE 8**—If a permanent record is desired, either read and record the penetration for each  $5^\circ\text{C}$  rise in temperature until the penetration reaches 0.4 mm, and at  $2^\circ\text{C}$  intervals thereafter, or attach a displacement transducer, having the same resolution as the gauge, to each rod and continuously record the rate of penetration by means of a multichannel recorder or similar data-acquisition device.

**NOTE 9**—Some commercially available instruments record the time at which the penetration reaches a set depth. If this type of instrument is used, make a time-temperature calibration before the specimens are tested. This calibration compensates for slight variations in the heating rate. (Even though the variations may be within the specifications set forth in **10.6**, the compounded error over the range of the test can produce a substantial error in the Vicat softening temperature.)

## 11. Report

11.1 Report the following information:

- 11.1.1 Reference to this test method,
- 11.1.2 Complete identification of the material tested,

**TABLE 1 Vicat Softening Temperature Using Loading 1 and Rate B, Values Expressed in Units of °C**

Material	Average	$S_r^A$	$S_R^B$	$r^C$	$R^D$	Number of Participating Laboratories
Ethylene vinyl acetate	72.4	1.44	2.29	4.03	6.40	10
Polystyrene	97.3	0.68	2.36	1.91	6.62	10
High-density polyethylene	127.9	1.04	2.73	2.90	7.63	10
Polypropylene	152.5	1.13	2.83	3.16	7.91	10
Nylon 66	251.2	0.70	5.06	1.96	14.16	7

<sup>A</sup>  $S_r$  = within-laboratory standard deviation of the average.

<sup>B</sup>  $S_R$  = between-laboratories standard deviation of the average.

<sup>C</sup>  $r$  = within-laboratory repeatability limit = 2.8  $S_r$ .

<sup>D</sup>  $R$  = between-laboratories reproducibility limit + 2.8  $S_R$ .

**TABLE 2 Vicat Softening Temperature Using Loading 2 and Rate A, Values Expressed in Units of °C**

Material	Average	$S_r^A$	$S_R^B$	$r^C$	$R^D$
Polypropylene (PP0343)	56.2	1.07	1.86	2.99	5.22
Polypropylene (PP0114)	92.5	1.47	4.08	4.12	11.44
Impact Modified Acrylic (PMMA0230V1)	94.1	0.32	1.96	0.91	5.48
ABS	94.4	0.62	1.61	1.74	4.52
High Heat ABS (ABS0135)	100.8	0.34	1.53	0.95	4.29
Unmodified Acrylic (PMMA0141V3)	105.1	0.44	1.48	1.23	4.15
Polycarbonate (PC0136)	143.6	0.19	1.24	0.53	3.48
Polycarbonate (PC0123)	143.8	0.38	1.03	1.05	2.89

<sup>A</sup>  $S_r$  = within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of the test results from all of the participating laboratories:

$$S_r = [((S_1)^2 \dots + (S_n)^2)/n]^{1/2}$$

<sup>B</sup>  $S_R$  = between-laboratories reproducibility, expressed as standard deviation:  $S_R = [S_r^2 + S_L^2]^{1/2}$  where  $S_L$  = standard deviation of laboratory means.

<sup>C</sup>  $r$  = within-laboratory critical interval between two test results = 2.8 ×  $S_r$ .

<sup>D</sup>  $R$  = between-laboratories critical interval between two test results = 2.8 ×  $S_R$ .

**TABLE 3 VICAT Softening Temperature Using a Direct Contact Instrument (Results are repeatability data on a single instrument.)**

Material	Thickness	Method	1	2	3	4	5	6	min	max	Average	st. dev.	
(PC)	4.0 mm	B2	141.1	140.2	141.3	140.5	141.7	140.7	140.2	141.7	140.9	0.6	
	4.0 mm	B2	141.2	140.1	141.3	140.5	142.0	140.6	140.1	142.0	141.0	0.7	
	4.0 mm	B2	141.2	140.5	141.4	140.7	141.9	140.3	140.3	141.9	141.0	0.6	
	4.0 mm	B2	141.3	140.5	141.4	140.6	141.8	140.8	140.5	141.8	141.1	0.5	
	4.0 mm	B2	141.5	140.5	141.6	140.8	140.9	140.9	140.5	141.6	141.1	0.5	
			min	141.1	140.1	141.3	140.5	141.7	140.3	140.1	141.7	140.8	0.6
			max	141.3	140.5	141.6	140.8	142.0	140.9	140.5	142.0	141.2	0.6
			average	141.2	140.4	141.4	140.6	141.9	140.7	140.4	141.9	141.0	0.6
			st. dev.	0.1	0.2	0.1	0.1	0.1	0.2	0.1	0.2	0.1	0.1
		4.0 mm	A2	140.3	139.9	140.3	140.2	140.6	140.0	139.9	140.6	140.2	0.2
		4.0 mm	A2	140.5	139.8	140.4	140.1	140.8	140.0	139.8	140.8	140.3	0.4
			min	140.3	139.8	140.3	140.0	140.6	140.0	139.8	140.6	140.2	0.3
		max	140.5	140.2	140.4	140.2	140.8	140.0	140.0	140.8	140.4	0.3	
		average	140.4	140.0	140.4	140.1	140.7	140.0	140.0	140.7	140.3	0.3	
		st. dev.	0.1	0.2	0.1	0.1	0.1	0.0	0.0	0.2	0.1	0.1	
(PS)	4.0 mm	A2	88.7	88.4	88.5	88.3	88.9	88.0	88.0	88.9	88.5	0.3	
	4.0 mm	A2	88.5	88.3	88.5	88.1	88.6	88.0	88.0	88.6	88.3	0.2	
	4.0 mm	A2	88.5	88.4	88.5	88.1	88.7	87.8	87.8	88.7	88.3	0.3	
			min	88.5	88.3	88.5	88.1	88.6	87.8	87.8	88.6	88.3	0.3
			max	88.7	88.4	88.5	88.3	88.9	88.0	88.0	88.9	88.5	0.3
			average	88.6	88.4	88.5	88.2	88.7	87.9	87.9	88.7	88.4	0.3
			st. dev.	0.1	0.1	0.0	0.1	0.2	0.1	0.0	0.2	0.1	0.1
(HDPE)	3.1 mm	A1		123.3	125.5	123.2	124.5	123.4	123.2	125.5	124.0	1.0	
	3.1 mm	A1	124.0	124.0	125.8	124.4	125.2	123.8	123.8	125.8	124.5	0.8	
	3.1 mm	A1		123.2	125.4	124.0	124.9	124.1	123.2	125.4	124.3	0.9	
	3.1 mm	A1	123.5	122.9	125.3	124.0	124.8	123.5	122.9	125.3	124.0	0.9	
	3.1 mm	A1	123.5	123.1	124.8	124.0	125.0	123.7	123.1	125.0	124.0	0.7	
			min	123.5	122.9	124.8	123.2	124.5	123.4	122.9	124.8	123.7	0.8
			max	124.0	124.0	125.8	124.4	125.2	124.1	124.0	125.8	124.6	0.7
			average	123.7	123.3	125.4	123.9	124.9	123.7	123.3	125.4	124.1	0.8
			st. dev.	0.3	0.4	0.4	0.4	0.3	0.3	0.3	0.4	0.3	0.1

11.1.3 Method of preparing test specimens, including conditioning and annealing methods used,

11.1.4 Initial starting temperature,

11.1.5 Rate of temperature rise, Rate A (50°C/h) or Rate B (120°C/h),

11.1.6 Total load applied to the specimen, Loading 1 (10 ± 0.2 N) or Loading 2 (50 ± 1.0 N),

11.1.7 Thickness of the specimen and the number of layers of the material that were used,

11.1.8 Type of instrument used: Immersion bath and heat-transfer medium used or contact plates,

11.1.9 Vicat softening temperature, expressed as the arithmetic mean of the Vicat softening temperatures of the individual specimens, and

11.1.10 Any observations relating to the test.

## 12. Precision and Bias<sup>8</sup>

12.1 *Precision*—Tables 1 and 2 have been developed in accordance with Practice E691. Table 1, for the case using Loading 1 (10 ± 0.2 N) and Heating Rate B (120 ± 10°C/h) is based on round-robin tests conducted in 1982 involving five materials and differing numbers of laboratories as noted in the table. Each laboratory obtained three test results for each material. Table 2, for the case using Loading 2 (50.0 ± 1.0 N) and Heating Rate A (50 ± 5°C/h) is based on round-robin tests conducted in 1994 involving 8 materials and six laboratories. Each laboratory obtained two test results for each material. In both cases, for each material, all of the individual specimens from all material samples were prepared by one source. Each test result was the average of two individual determinations. (**Warning**—The following explanations of *r* and *R* (see 12.1.1 – 12.1.1.3) are intended only to present a meaningful way of

considering the approximate precision of this test method. The data given in Tables 1 and 2 shall not be applied rigorously to the acceptance or rejection of material, as those data are specific to the round-robin test and are not representative of other lots, conditions, materials, or laboratories. Users of this test method need to apply the principles outlined in Practice E691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 12.1.1 – 12.1.1.3 would then be valid for such data.)

12.1.1 *Concept of r and R*—If  $S_r$  and  $S_R$  have been calculated from a large enough body of data, and for test results that were averages from testing two specimens, the following applies:

12.1.1.1 *Repeatability, r*—The value that the absolute difference between two individual test results under repeatability conditions are expected to occur with a probability of approximately 0.95 (95 %).

12.1.1.2 *Reproducibility, R*—The value that the absolute difference between two test results obtained under reproducibility conditions are expected to occur with a probability of approximately 0.95 (95 %).

12.1.1.3 Conducting equivalence testing on numerical data from two sources shall be conducted in accordance with E2935 or any known method for judging the equivalence of two means.

NOTE 10—Example a t-test.

12.2 *Bias*—There are no recognized standards by which to estimate the bias of this test method.

NOTE 11—Reinforced materials have been shown to sometimes break at the point of penetration at temperatures above 24°C, which can affect the repeatability of the test results.

## 13. Keywords

13.1 plastics; thermoplastics; Vicat softening temperature

<sup>8</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:D20-1194.

## ANNEXES

### (Mandatory Information)

#### A1. CALIBRATION OF SINGLE-(CENTRALIZED) TEMPERATURE PROBE UNITS

A1.1 If the unit in operation has only one temperature probe in the bath, and this probe is monitored to record the softening temperature of the specimen at all the stations in the unit, then the following calibration and checks must be undertaken to ensure comparable results with units that have a temperature probe at each station.

A1.2 This procedure must be performed annually as a minimum to ensure proper temperature distribution and accuracy of probe and display.

A1.3 Calibration will require the use of temperature meter and probe traceable to NMI (National Metrology Institute), with accuracy and display resolution of 0.1°C or better, a stopwatch, and any tools needed to open and adjust the unit.

A1.3.1 Low-temperature calibration of the unit is accomplished by placing the NMI-traceable probe within 10 mm of specimen height, in the bath at three different points in the bath. The three points will be at the center and left and right ends of the bath. Start with the station closest to the centralized probe,

while the unit is programmed to maintain a constant temperature between 20 and 50°C, with all stirrers operating. Allow the bath to stabilize for a minimum of 5 min. Read and record the readout of the calibrated probe and the unit's internal temperature display to the nearest 0.1°C. Make any necessary adjustments to the unit's temperature controller to bring the bath to  $\pm 0.1^\circ\text{C}$  of the bath set point, allowing a stabilization time of a minimum of 5 min between adjustment(s) and readings. Once the calibrated probe indicates the bath is at the set point, make adjustments to the centralized probe's display as necessary.

A1.3.1.1 Move the NMI-traceable probe to the other two points maintaining the probe within 10 mm of specimen height. Read and record the temperatures at these points, after allowing the probe to stabilize a minimum of 5 min.

A1.3.2 High-temperature calibration will be accomplished by programming the unit to maintain an elevated temperature near, but not exceeding, the highest temperature allowed by the heat transfer media. All covers and stations must be in place and stirrer motors operating. Place the NMI probe within 10 mm of specimen height at the station closest to the centralized probe, and allow the bath to stabilize for a minimum of 5 min. Read and record the readout of the calibrated probe and the unit internal temperature display to the nearest 0.1°C. Make any necessary adjustments to the unit's temperature controller to bring the bath to  $\pm 0.1^\circ\text{C}$  of the bath set point, allowing a stabilization time of a minimum of 5 min between adjustment(s) and readings. Once the calibrated probe indicates the bath is at the set point make adjustments to the centralized probe's display as necessary.

A1.3.2.1 Move the NMI-traceable probe to the other two points maintaining the probe within 10 mm of specimen height. Read and record the temperatures at these points, after allowing the probe to stabilize for a minimum of 5 min.

A1.3.3 Evaluate the data from each of the three points in the bath at both low and high temperature. If any point is greater than  $\pm 0.5^\circ\text{C}$  from the set point, have the unit serviced or repaired to correct this error. If it is not possible to correct the bath uniformity to less than  $0.5^\circ\text{C}$ , then a thermal sensing device must be placed at each station and used to record the temperature of the bath at the time of deflection while running tests. The unit can be electronically modified or the use of glass thermometers (as outlined in 6.1.6) can be placed at each station and manually read and recorded at the moment of specimen deflection.

A1.3.4 If the steps given in A1.3.1 – A1.3.2.1 have been taken and successfully completed, cool the bath down to a normal start temperature and allow the bath to stabilize. Place the NMI probe at the point in the bath that the preceding gathered data shows the greatest error. Start a test at 120°C/h or 50°C/h. Read and record the temperature of both the unit's display and the readout of the NMI probe. An offset of 10 to 15 s between the two readings is acceptable as long as this interval is maintained throughout this test. Start the stopwatch when the first temperature is recorded. Read and record the temperature of the unit's display and the NMI probe, maintaining any delay interval, if used, every 5 min for 1 h.

A1.3.5 Evaluate the data acquired during the test given in A1.3.4. Ensure that the temperature of the bath is rising at the correct rate as outlined in 10.6, at both the centralized probe and the other selected test point. If either is outside the limits for the rate of rise, the unit must be serviced and rechecked before further use. If a unit fails to pass this calibration test the unit must be serviced or replaced. Placing a temperature sensing device at each station will not correct the problem observed in A1.3.4, as the unit's rate of rise is outside the tolerances of this test method.

## A2. CALIBRATION OF MULTI TEMPERATURE SENSOR INSTRUMENTS

A2.1 This procedure is to be used in addition to manufacturer's requirements and procedures to calibrate a VICAT instrument that has multiple temperature sensors in the medium to control the temperature of the medium, or record the deflection temperature, or both. If the unit under test has only a single temperature sensor please refer to [Annex A1](#).

A2.2 This procedure shall be performed at a frequency that conforms to the end user's quality system requirements.

A2.3 All test equipment, that is, temperature meters, temperature sensors, gauge blocks, stopwatches, etc., used to perform this procedure must be calibrated and traceable through a National Metrology Institute (NMI) to the International System of Units (SI). Temperature measuring equipment must have a resolution of 0.1°C or better, gauge blocks used to calibrate the deflection must be accurate to 0.001 mm or better, stopwatches must be accurate to 0.1 s or better.

A2.4 Temperature calibration shall be done in accordance with the manufacturer's procedures and the following guidelines:

A2.4.1 The temperature shall be calibrated at a minimum of two points. One being at or near<sup>9</sup> the start temperature of the test and the other at or above the maximum temperature used by the end user. Care must be taken not to exceed the maximum safe temperature of the heat transfer media.

A2.4.2 If moving the reference temperature sensor(s) from location to location in the medium, a minimum of 5 min must be allowed between moving the temperature sensor and reading the temperature values.

A2.4.3 Test stations and covers shall be in their normal test position when possible and all agitators operating during the calibration.

<sup>9</sup> Near is defined in this Annex as  $\pm 5^\circ\text{C}$ .

A2.4.4 Reference temperature sensor(s) sensitive part shall be placed as close as possible to the Unit Under Test (UUT) sensor(s), and  $\leq 10$  mm from the specimens.

A2.4.5 Adjustment of the UUT shall be made so the display(s) of the UUT is  $\pm 0.1^\circ\text{C}$  of the values indicated by the reference temperature sensor(s).

A2.5 Once the static temperature calibration has been completed, cool the instrument down to a normal start temperature and allow the medium temperature to stabilize. Program the UUT to ramp up the medium temperature at a rate of  $120^\circ\text{C/h}$  or  $50^\circ\text{C/h}$  as dictated by the use of the UUT. If the UUT is used at both ramp rates separate tests must be conducted at each ramp rate. Read and record the temperature at each station at intervals not to exceed those stated in section 10.6 of this test method, until the UUT reaches the high temperature calibration point. These temperatures shall be read and recorded by software control or data acquisition from the UUT using the internal temperature sensors after they have been calibrated by the above steps or by the use of external traceable temperature measurement equipment. Perform multiple ramps if necessary to verify each station.

A2.5.1 Evaluate the data acquired during the preceding test(s) to ensure that the temperature rate of rise at each station is within the tolerances outlined in Section 4 of this test method. It is allowable for the first 10 min of the ramp to be outside of the prescribed tolerances as many instruments use a PID control for the heating, and it is normal for the controller to tune itself to the correct power and interval requirements to perform the required ramp rate. If any station is found to be outside the prescribed tolerances beyond the first 10 min, that station shall not be used for testing until repairs or adjustments are made to bring the station back into tolerance.

A2.6 A test must be made on each station using a test specimen made of a material having a low coefficient of

expansion<sup>10</sup> to determine the thermal expansion of the station, load rod and associated parts. The calibrated temperature range of the UUT shall be covered and a compensation value determined at a minimum of each  $20^\circ\text{C}$  rate of rise. If the UUT is used at both ramp rates of  $120^\circ\text{C/h}$  and  $50^\circ\text{C/h}$  then a compensation value must be determined independently for each ramp rate. If this compensation value is greater than 0.02 mm (0.0008 in.) its algebraic sign shall be noted and the compensation value shall be applied to each test by adding it algebraically to the reading of apparent deflection of the test specimen. It is permissible to perform this test in conjunction with the rate of rise test as outlined in A2.5.

A2.7 The deflection indicators and critical mechanical dimensions, that is, needle point(s), must also be calibrated/verified using traceable calibration tools. The manufacturer's requirement and procedures will provide details on how to perform the actual tasks; the following are intended to provide the user with tolerances and other necessary guidelines.

A2.7.1 The deflection indicators must be calibrated to a tolerance of  $\pm 0.01$  mm of the reference.

A2.7.2 The critical mechanical dimensions must meet the requirements outlined in 6.1.4 and 6.1.7 of this test method.

A2.7.3 The weights must be verified and conform to the specification outlined in 6.1.5 of this test method.

A2.7.4 When determining the weight of the load rod(s) and deflection indicator any spring force acting on the specimen must be accounted for. If the design of the apparatus uses a spring force that acts downward (as part of the load) or upwards (reducing the applied load), this force must be added or subtracted as necessary so that the actual load applied to the specimens is determined.

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<sup>10</sup> Borosilicate (quartz) has been found suitable for this purpose.

## APPENDIXES

### (Nonmandatory Information)

#### X1. PROCEDURE FOR DETERMINATION OF CORRECT SPECIMEN LOADING UTILIZING EQUILIBRIUM WEIGHING OF THE LOADING ROD

##### X1.1 Apparatus:

X1.1.1 The apparatus is constructed essentially as shown in Fig. X1.1 and consists of the following:

X1.1.1.1 *Laboratory Scale*, having a resolution of at least 0.1 g.

X1.1.1.2 *Platform Assembly*, for supporting the test unit above the scale.

X1.1.1.3 *Bridge Platform*, for supporting the loading rod on the scale.

##### X1.2 Procedure:

X1.2.1 Calculate the load required to apply the desired force on the specimen using the equation given in 6.1.5.

X1.2.2 Level the mounting assembly on top of the tester. Shim or clamp if necessary for firm seating.

X1.2.3 Level the scale.

X1.2.4 Position the test frame on the cross bar above the scale.



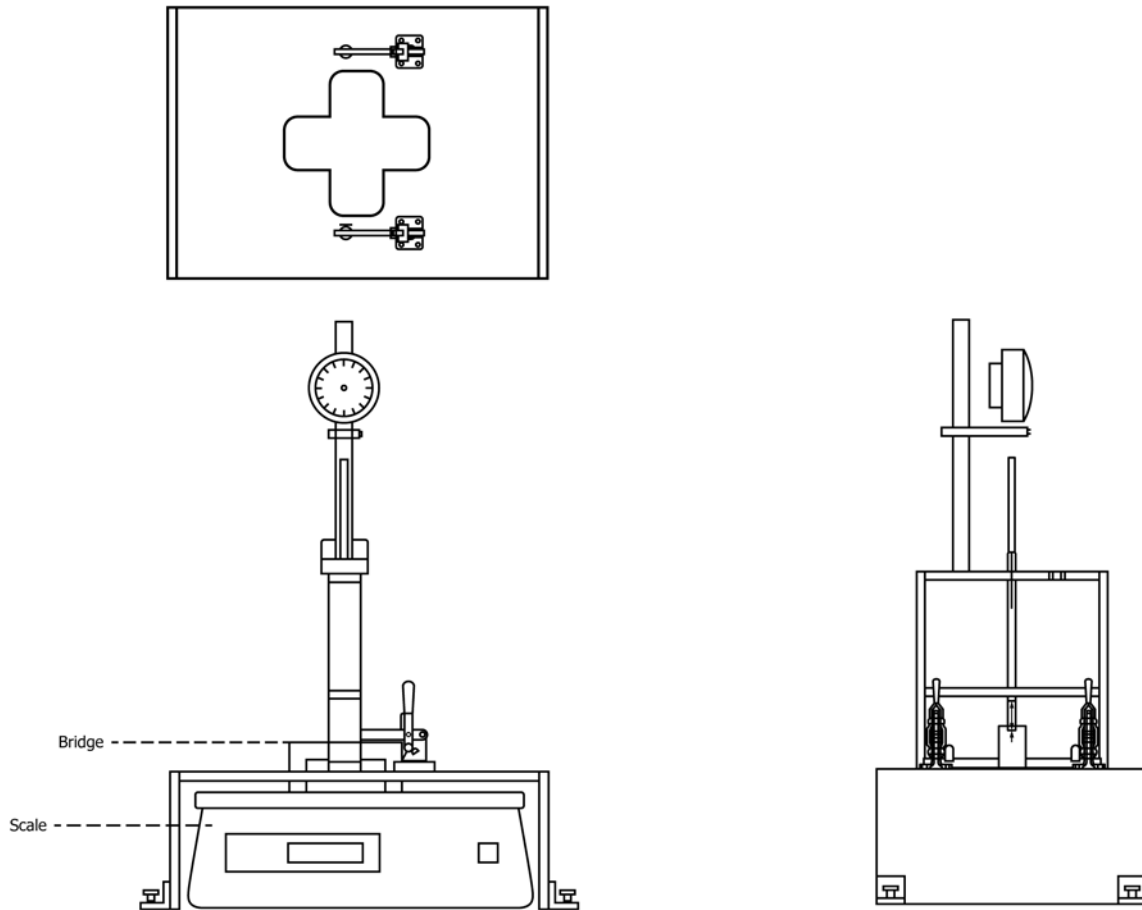


FIG. X1.1 Calibration Apparatus Using a Laboratory Scale

X1.2.5 Lubricate the rod and guide hole surfaces with a light oil.

X1.2.6 Lift the loading rod and put the bridge in place on the scale pan so that it will support the loading rod. The bridge height dimension is such that it supports the rod approximately 2 mm (or the thickness of the test specimen normally used) above the base of the test frame.

X1.2.7 Determine the mass of the bridge.

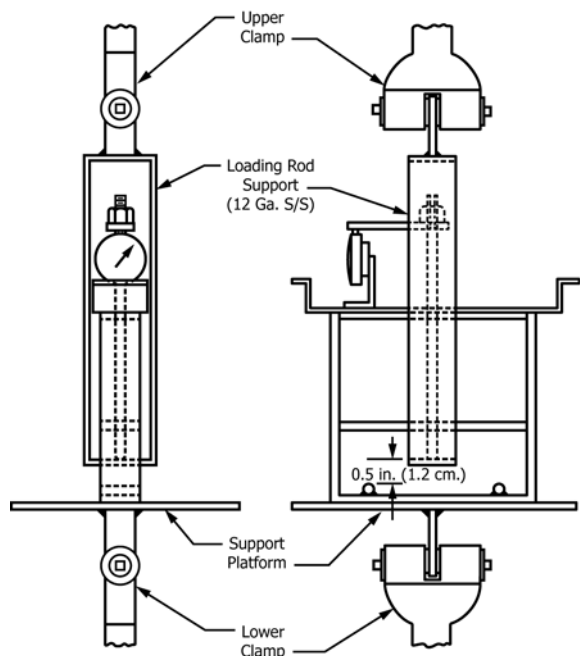
X1.2.8 With the deflector arm in position over the dial gauge, lower the rod to the bridge and release it very gently. In

this position, the dial gauge should be adjusted so that it is approximately in the middle of its travel, with at least 1.0 mm of travel left to allow for penetration of the specimen during a normal test.

X1.2.9 Record the force in grams. This amount, when converted to newtons, should equal  $(F - F_s)$  as calculated in 6.1.5.

NOTE X1.1—The test units (rod, guide surface, and dial gauge) shall be clean and free of any surface imperfections, etc., to achieve precision in calibration and normal test use.

**X2. PROCEDURE FOR DETERMINATION OF CORRECT SPECIMEN LOADING BY WEIGHING THE APPLIED LOAD WITH A TENSION-TESTING MACHINE**



**FIG. X2.1 Calibration Apparatus Using a Tensile Machine**

**X2.1 Apparatus:**

X2.1.1 The apparatus is constructed essentially as shown in Fig. X2.1 and consists of the following:

X2.1.1.1 *Tension-Testing Machine*, of the constant-rate-of-jaw-separation type, equipped with devices for recording the tensile load and the grip separation. The testing machine used should be capable of measuring loads of at least 2000 g. The rate of separation of the jaws should be capable of adjustment to 0.51 mm/min.

X2.1.1.2 *Platform*, square, approximately 203 by 203 mm, to be mounted on the lower crosshead of the tensile machine to support the deflection temperature test unit.

X2.1.1.3 *Loading Rod Support*—A saddle-like device to be clamped in the upper grips of the tensile machine so that it extends under the bottom tip of the loading rod.

**X2.2 Procedure:**

NOTE X2.1—This technique is applicable to dial-type test instruments only.

X2.2.1 Mount the support platform in the lower cross-head clamps.

X2.2.2 Fit the loading rod support into the upper clamps and calibrate the tensile-testing machine.

X2.2.3 Secure the Vicat softening point test unit on the support platform and adjust the loading rod support so that the tip of the loading rod is 2.54 mm from the top of the specimen support.

X2.2.4 Lubricate the rod and guide hole surfaces with light oil.

X2.2.5 Adjust the dial gauge so that it reads zero, then turn the nut on top of the loading rod clockwise until the deflector arm almost makes contact with the contact arm on top of the dial gauge.

X2.2.6 Start the lower crosshead in the up direction at the rate of 0.51 mm/min. This in effect causes the loading rod to move down as in an actual test. When the pointer on the dial gauge shows movement, activate the chart drive at the rate of 25.4 mm/min.

X2.2.7 Record the force, g, at  $1.00 \pm 0.05$ -mm penetration.

X2.2.8 Adjust the mass of the loading rod required to give the desired maximum load according to the equation given in 6.1.5.

**X3. PROCEDURE FOR VERIFYING THE CALIBRATION OF PENETRATION MEASURING DEVICES USING GAUGE BLOCKS**

X3.1 This procedure provides a method of verifying the calibration of penetration measuring devices typically found on vicat softening temperature measuring instruments. It is not a calibration method. If the user finds that the measuring device on one or more test stations is out of calibration, it is recommended the manufacturer of the instrument, or a qualified calibration service company be consulted to have the problem corrected. This procedure can be used for dial indicator, LVDT and encoder-type penetration-measuring devices.

X3.2 Remove the test frame from the bath. Wipe excess heat transfer medium from the frames and place on a sturdy, level surface. If it is not possible to remove the test frame from the bath, the frame can be positioned on top of the instrument,

providing the frame is level during the verification procedure so that the loading rod will apply its full load as it would during a test.

X3.3 Thoroughly clean the needle tip and the surface of the frame where the specimen is normally positioned.

X3.4 Select a minimum of two gauge blocks that, when stacked together, are comparable in height to a typical test specimen. At least one of the gauge blocks should be a 1.00-mm block.

NOTE X3.1—If a 1.00-mm gauge block is not available, a 0.040-in. (1.016-mm) gauge block can be substituted.

X3.5 Place the stacked blocks in the test frame where the specimen is normally positioned. Lower the loading rod onto

the gauge blocks in such a way that the penetrating needle is in the middle of the block. Add the required weight to the rod to apply force to the block (either 10 or 50 N), simulating test conditions. Zero the indicator or record the reading on the display.

NOTE X3.2—Care must be taken to avoid damaging the gauge blocks when using the 50-N force.

X3.6 Lift the loading rod and remove the 1.00-mm (or 0.040-in.) block from beneath the rod and lower the rod onto the remaining gauge block. Do not change the position of the

remaining gauge block. Record the reading on the indicator. The change in the reading should be equal to  $1.00 \pm 0.01$  mm (or 0.040 in.).

X3.7 Repeat the procedure at least twice to ensure repeatability. Intermediate readings can be verified in a similar manner by using different size gauge blocks.

X3.8 Repeat this procedure for all of the instrument's penetration measuring devices.

## SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D1525–09) that may impact the use of this standard. (August 1, 2017)

- (1) Editorial changes and removal of permissive language.
- (2) Revised last sentence in section 6.1.1.1 and moved it as a new Note(#2)—content is informational only.
- (3) Revised 6.1.6 for using temperature measuring devices—refer to D648-HDT testing
- (4) Changed Note 5—mandatory information—incorporated this as a new 10.1.1 within the body of Section 10.

- (5) Reworded for clarification of Appendixes X3.1
- (6) Added new Note 4 regarding mercury hazard associated with E1 with thermometers containing mercury.
- (7) Added thermistor probe, or platinum resistance thermometer (PRT) or equivalent to section 6.1.6.1.
- (8) Added Specification E2251 in Section 2, which is identified in 6.1.6.

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