



Designation: D648 – 18

# Standard Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position<sup>1</sup>

This standard is issued under the fixed designation D648; 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 the determination of the temperature at which an arbitrary deformation occurs when specimens are subjected to an arbitrary set of testing conditions.

1.2 This test method applies to molded and sheet materials available in thicknesses of 3 mm ( $\frac{1}{8}$  in.) or greater and which are rigid or semirigid at normal temperature.

NOTE 1—Sheet stock less than 3 mm (0.125 in.) but more than 1 mm (0.040 in.) in thickness may be tested by use of a composite sample having a minimum thickness of 3 mm. The laminae must be of uniform stress distribution. One type of composite specimen has been prepared by cementing the ends of the laminae together and then smoothing the edges with sandpaper. The direction of loading shall be perpendicular to the edges of the individual laminae.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.4 Some older machines still use mercury-in-glass thermometers. (**Warning**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Safety Data Sheet (SDS) for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

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 2—The text of this standard references notes and footnotes that

provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

NOTE 3—This standard and ISO 75-1 and ISO 75-2 address the same subject matter, but differ in technical content, and results shall not be compared between the two test methods.

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:<sup>2</sup>

- D618 Practice for Conditioning Plastics for Testing
- D883 Terminology Relating to Plastics
- D4000 Classification System for Specifying Plastic Materials
- D5947 Test Methods for Physical Dimensions of Solid Plastics Specimens
- 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
- 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>3</sup>

- ISO 75-1 Plastics—Determination of Temperature of Deflection Under Load—Part 1: General Test Method

<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.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

**ISO 75-2 Plastics—Determination of Temperature of Deflection Under Load—Part 2: Plastics and Ebonite**

2.3 *NIST Document*:<sup>4</sup>

**NBS Special Publication 250-22**

### 3. Terminology

3.1 *General*—The definitions of plastics used in this test method are in accordance with Terminology **D883** unless otherwise indicated.

### 4. Summary of Test Method

4.1 A bar of rectangular cross section is tested in the edgewise position as a simple beam with the load applied at its center to give maximum fiber stresses of 0.455 MPa (66 psi) or 1.82 MPa (264 psi) (**Note 4**). The specimen is immersed under load in a heat-transfer medium provided with a means of raising the temperature at  $2 \pm 0.2^\circ\text{C}/\text{min}$ . The temperature of the medium is measured when the test bar has deflected 0.25 mm (0.010 in.). This temperature is recorded as the deflection temperature under flexural load of the test specimen.

**NOTE 4**—A round robin has been conducted that showed that there is no advantage to using higher loads when measuring deflection temperature of present-day plastics with present-day instruments.

### 5. Significance and Use

5.1 This test is particularly suited to control and development work. Data obtained by this test method shall not be used to predict the behavior of plastic materials at elevated temperatures except in applications in which the factors of time, temperature, method of loading, and fiber stress are similar to those specified in this test method. The data are not intended for use in design or predicting endurance at elevated temperatures.

5.2 For many materials, there may be a specification that requires the use of this test method, but with some procedural modifications that take precedence when adhering to the specification. Therefore, it is advisable to refer to that material specification before using this test method. Refer to Table 1 in Classification **D4000**, which lists the ASTM material standards that currently exist.

### 6. Interferences

6.1 The results of the test are dependent on the rate of heat transfer between the fluid and the specimen and the thermal conductivity of the fluid.

6.2 The results of this test are dependent on the measured width and depth of the specimen and the final deflection at which the deflection temperature is determined.

6.3 The type of mold and the molding process used to produce test specimens affects the results obtained in this test. Molding conditions shall be in accordance with the standard for that material or shall be agreed upon by the cooperating laboratories.

6.4 Results of testing are affected by the design of the test equipment. The test span (either 100 mm or 101.6 mm) will influence the resultant measurement. Instrumentation equipped with metal clips or other types of auxiliary supports designed to maintain specimens perpendicular to the applied load will affect the test results if the pressure is sufficient to restrict the downward motion of the specimen at its center.

### 7. Apparatus

7.1 The apparatus shall be constructed essentially as shown in **Fig. 1** and shall consist of the following:

7.1.1 *Specimen Supports*, metal supports, allowing the load to be applied on top of the specimen vertically and midway between the supports, which shall be separated by a distance, defined in 7.1.1.1 or 7.1.1.2. The contact edges of the supports and of the piece by which load is applied shall be rounded to a radius of  $3 \pm 0.2$  mm ( $0.118 \pm 0.008$  in.).

7.1.1.1 Method A— $101.6 \pm 0.5$  mm ( $4.0 \pm 0.02$  in.).

7.1.1.2 Method B— $100.0 \pm 0.5$  mm ( $3.937 \pm 0.020$  in.).

7.1.2 *Immersion Bath*—Choose a suitable liquid heat-transfer medium (**Note 5**) in which the specimen shall be immersed, which will not affect the specimen. It shall be well-stirred during the test and shall be provided with a means of raising the temperature at a uniform rate of  $2 \pm 0.2^\circ\text{C}/\text{min}$ . This heating rate shall be considered to be met if, over every 5-min interval during the test, the temperature of the bath shall rise  $10 \pm 1^\circ\text{C}$  at each specimen location.

**NOTE 5**—Mineral oil is considered safe from ignition to  $115^\circ\text{C}$ . Silicone oils may be heated to about  $260^\circ\text{C}$  for short periods of time. For still higher temperatures, special heat-transfer media should be used. Improved performance with longer oil life may be obtained by the use of  $\text{CO}_2$  or other inert gas to isolate the oil surface from the atmosphere.

7.1.3 *Deflection Measurement Device*, suitable for measuring specimen deflection of at least 0.25 mm (0.010 in.). It shall be readable to 0.01 mm (0.0005 in.) or better. Dial gauges or any other indicating or recording device, including electric displacement sensing apparatus, are acceptable.

7.1.4 *Weights*—A set of weights of suitable sizes so that the specimen are loaded to a fiber stress of 0.455 MPa (66 psi)  $\pm 2.5\%$  or 1.82 MPa (264 psi)  $\pm 2.5\%$ . The mass of the rod that applies the testing force shall be determined and included as part of the total load. If a dial gauge is used, the force exerted by its spring shall be determined and shall be included as part of the load (**Note 9**). Calculate the testing force and the mass that must be added to achieve the desired stress as follows:

$$F = 2Sbd^2/3L \quad (1)$$

$$F^1 = F/9.80665$$

<sup>4</sup> Mangum, B. W., "Platinum Resistance Thermometer Calibration," *NBS Special Publication 250-22*, 1987. Available from National Institute of Standards and Technology, Gaithersburg, MD.

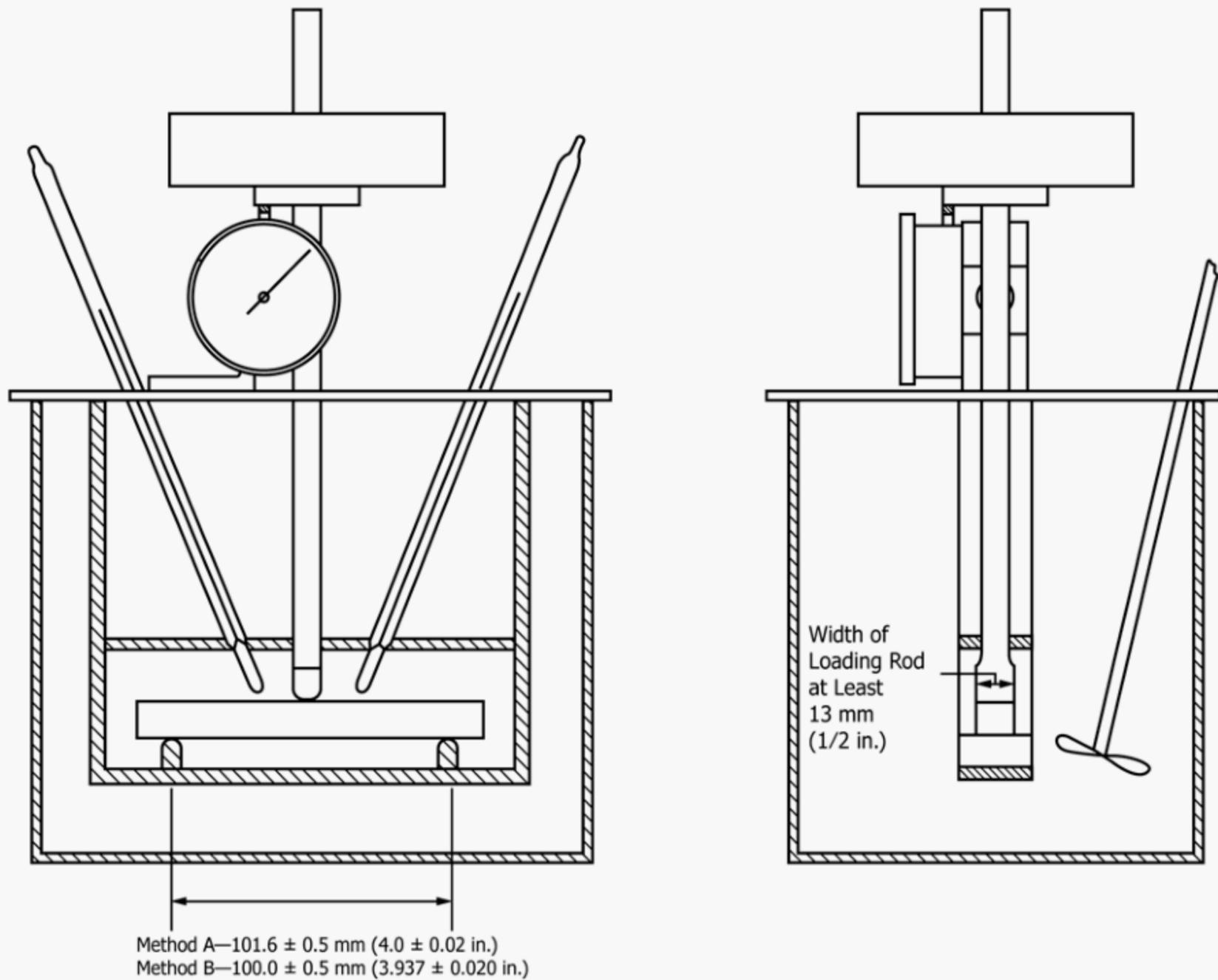


FIG. 1 Apparatus for Deflection Temperature Test

$$m_w = (F - F_s) / 9.80665 - m_r$$

where:

- $F$  = load, N,
- $F^l$  = load, kgf,
- $S$  = fiber stress in the specimen (0.455 MPa or 1.82 MPa),
- $b$  = width of specimen, mm,
- $d$  = depth of specimen, mm,
- $L$  = distance between supports, (101.6 mm—Method A, or 100 mm—Method B), see 7.1.1.1 and 7.1.1.2.
- $m_w$  = added mass, kg,
- $F_s$  = force exerted by any spring-loaded component involved, N; this is a positive value if the thrust of the spring is towards the test specimen (downwards), or a negative value if the thrust of the spring is opposing the descent of the rod, or zero if no such component is involved, and
- $m_r$  = mass of the rod that applies the testing force to the specimen, kg.

NOTE 6—In some designs of this apparatus, the spring force of the dial gauge is directed upward (opposite the direction of specimen loading), which reduces the net force applied to the specimen. In other designs, the spring force of the dial gauge acts downward (in the direction of specimen loading), which increases the net force applied to the specimen. The mass applied to the loading rod must be adjusted accordingly (increased for upward dial force and decreased for downward dial force) to compensate. Since the force exerted by the spring in certain dial gauges varies

considerably over the stroke, this force should be measured in that part of the stroke that is to be used. Suggested procedures to determine the total load required to correct for the force of the dial gauge spring are given in Appendix X1 and Appendix X2. Other procedures may be used if equivalent results are obtained. Appendix X3 provides a method of determining the spring force, uniformity of the force in the gauge's test measurement range, and whether the gauge is contaminated and sticking.

#### 7.1.5 Temperature Measurement System

7.1.5.1 *Digital Indicating System*—Consisting of a thermocouple, resistance thermometer (RTD), and so forth, as the sensor, together 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.

7.1.5.2 *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 shall be calibrated for the depth of immersion in accordance with Test Method E77.

NOTE 7—Consult Specification **E2251** for suitable alternatives to mercury-in-glass thermometers.

7.2 *Micrometers* shall meet the requirements of Test Methods **D5947** and be calibrated in accordance with that test method.

## 8. Sampling

8.1 Sample in a statistically acceptable manner. When samples are taken from a production lot or process, the process shall be in a state of statistical control.

## 9. Test Specimen

9.1 At least two test specimens of the same nominal width shall be used to test each sample at each fiber stress. The length of the specimen shall at a minimum be the distance between supports + 12.7 mm (0.500 in.). The depth of the specimen must be 12.7 mm ± 0.5 mm (0.500 ± 0.020 in.). The width of the specimen shall be any width from 3 mm to 13 mm (0.118 in. to 0.512 in.).

NOTE 8—Tolerances on the depth and width dimensions should be of the order of ±0.13 mm (0.005 in.) along the length of the specimen to minimize variations.

NOTE 9—The test results obtained on specimens approaching 13 mm in width may be 2 to 4°C above those obtained from 4 mm or narrower test specimens because of poor heat transfer through the specimen.

9.2 The specimens shall have smooth flat surfaces free from saw cuts, excessive sink marks, or flash.

9.3 Molding conditions shall be in accordance with the specification for that material or shall be agreed upon by the cooperating laboratories. Discrepancies in test results due to variations in molding conditions are often 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 by the cooperating laboratories.

## 10. Preparation of Apparatus

10.1 The apparatus shall be arranged so that the deflection of the specimen at midspan is measured by the deflection measurement device described in 7.1.3. It is acceptable if the apparatus is arranged to shut off the heat automatically and sound an alarm or record the temperature when the specific deflection has been reached. Sufficient heat transfer liquid shall be used to cover the sensing end of the temperature measuring device to the point specified in their calibration.

NOTE 10—It is desirable to have a means to cool the bath in order to reduce the time required to lower the temperature of the bath after the test has been completed. This may be accomplished by using a cooling coil installed in the bath, or an external heat transfer system that passes the hot oil through it. If the rate of temperature rise of the oil is adversely affected by the presence of residual coolant in the coils, the coolant should be purged prior to starting the next test.

## 11. Conditioning

11.1 *Conditioning*—Condition the test specimens in accordance with Procedure A of Practice **D618** unless otherwise specified by contract or the relevant ASTM material specification. Conditioning time is specified as a minimum. Temperature and humidity tolerances shall be in accordance with

Section 7 of Practice **D618** unless specified differently by contract or material specification.

NOTE 11—Shorter conditioning periods may be used when it is shown that they do not affect the results of this test. Longer conditioning times may be required for some materials that continue to change with time.

## 12. Procedure

12.1 Measure the width and depth of each specimen with a suitable micrometer (as described in 7.2) at three points along the span. Average these respective readings to obtain the nominal width and depth value for the specimen. These values are used to determine the amount of applied force necessary to produce the specified fiber stress in each specimen (see 7.1.4).

12.2 Position the test specimens edgewise in the apparatus and ensure that they are properly aligned on the supports so that the direction of the testing force is perpendicular to the direction of the molding flow. If the specimen support unit has metal clips or auxiliary supports on it to hold the specimen perpendicular to the load and to prevent the specimen from being displaced by the circulating oil, only one surface of the clip or auxiliary support shall touch the specimen at any one time. The presence of any clip or auxiliary support shall not impede the deflection of the specimen or place additional force on the specimen that will result in more load having to be applied to achieve deflection.

NOTE 12—Holding of the specimens upright on the specimen supports by the use of clips or auxiliary supports that apply pressure to the specimen have been shown to alter the deflection temperature when testing at the 0.45 MPa stress level.

12.3 The sensitive part of the temperature measuring device shall be positioned as close as possible to the test specimen (within 10 mm) without touching it. The stirring of the liquid-heat transfer medium shall be sufficient to ensure that temperature of the medium is within 1.0°C at any point within 10 mm of the specimen. If stirring is not sufficient to meet the 1.0°C requirement, then the temperature measuring device shall be placed at the same level as the specimen and within 10 mm of the point at which the specimen is loaded.

12.4 Ascertain that the temperature of the bath is suitable. The bath temperature shall be at ambient temperature at the start of the test unless previous tests have shown that, for the particular material under test, no error is introduced by starting at a higher temperature.

12.5 Carefully apply the loaded rod to the specimen and lower the assembly into the bath.

12.6 Adjust the load so that the desired stress of 0.455 MPa (66 psi) or 1.82 MPa (264 psi) is obtained.

NOTE 13—Verification of the load should be made on all new equipment, after replacement of dial gauges, or following any other change that could affect the loading. Verification of the load should also be performed periodically to ensure that the equipment is within calibration (see **Appendix X1**, **Appendix X2**, and **Appendix X3**). Depending on the type of deflection measurement device used, it may be necessary to adjust the device such that it records the deflection in the displacement range of the device where the test is to be made.

12.7 Five minutes after applying the load, adjust the deflection measurement device to zero or record its starting position. Heat the liquid heat-transfer medium at a rate of  $2.0 \pm 0.2^\circ\text{C}/\text{min}$ .

NOTE 14—The 5-min waiting period is provided to compensate partially for the creep exhibited by some materials at room temperature when subjected to the specified nominal surface stress. That part of the creep that occurs in the initial 5 min is usually a significant fraction of that which occurs in the first 30 min.

12.8 Record the temperature of the liquid heat-transfer medium at which the specimen has deflected the specified amount at the specified fiber stress.

NOTE 15—Continuous reading of the deflection versus temperature even beyond the standard deflection might be useful in special situations.

**13. Report**

- 13.1 Report the following information:
  - 13.1.1 Full identification of the material tested,
  - 13.1.2 Method of test specimen preparation,
  - 13.1.3 Conditioning procedure,
  - 13.1.4 Test method, reported as D648 Method A or D648 Method B,
  - 13.1.5 The width and depth of the specimen, measured to 0.025 mm,
  - 13.1.6 The standard deflection, the deflection temperature, and the resultant maximum fiber stress for each specimen,
  - 13.1.7 The immersion medium, the temperature at the start of the test, and the actual heating rate,
  - 13.1.8 Average deflection temperature,
  - 13.1.9 Any nontypical characteristics of the specimen noted during the test or after removal from the apparatus, (such as twisting, nonuniform bending, discoloration, swelling), and
  - 13.1.10 Type of apparatus: automated or manual.

**TABLE 1 Statistical Information<sup>A</sup>**

Polymer	Average <sup>B</sup> Value	Standard Deviation	Critical <sup>C</sup> Difference, Within Laboratories	Critical Difference, Between Laboratories
Polyethylene, 0.455 MPa	85.3	4.8	6.0	9.4
Polycarbonate, 0.455 MPa	142.0	2.0	2.3	3.9
Methyl methacrylate, 1.82 MPa	97.6	2.9	4.0	5.7
Polysulfone, 1.82 MPa	173.8	2.8	2.3	5.5

<sup>A</sup>All values are given in  $^\circ\text{C}$ .  
<sup>B</sup>Average of pairs.  
<sup>C</sup>Between values of a pair.

**14. Precision and Bias**

14.1 *Precision*—An interlaboratory test program<sup>5</sup> was carried out with seven laboratories participating and utilizing both manual and automated instruments. Four polymers were included in the program. Statistical information is summarized in

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1098.

**TABLE 2 Precision, Deflection Temperature**

Material	Units Expressed in $^\circ\text{C}$				
	Average	$S_r^A$	$S_R^B$	$r^C$	$R^D$
ABS, 1.8 MPa	81.6	1.15	1.67	3.21	4.68
PP natural, 0.45 MPa	83.8	3.11	4.71	8.70	13.20
PP filled, 0.45 MPa	114.7	2.16	4.62	6.06	12.92

<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:

$$S_r = [((S_1)^2 + (S_2)^2 + \dots + (S_n)^2)/n]^{1/2}$$

<sup>B</sup> $S_R$  = between-laboratories reproducibility, expressed as standard deviation:

$$S_R = [S_r^2 + S_L^2]^{1/2}, \text{ where } S_L = \text{standard deviation of laboratory means.}$$

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

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

**TABLE 3 Deflection Temperature (Average) Obtained on Test Equipment With Span Values of 100 and 101.6 mm (3.937 and 4.0 in.),  $^\circ\text{C}$**

Material	100-mm (3.937-in.) Span	101.6-mm. (4.0-in.) Span
ABS, 1.8 MPa	81.9	81.0
PP natural, 0.45 MPa	85.2	80.9
PP filled, 0.45 MPa	116.6	112.0
Nylon, 1.8 MPa	156.1	153.8

**Table 1.** The critical difference limits are the limits beyond which observed differences are to be considered suspect.

14.2 In 1995 a second round-robin<sup>6</sup> study was conducted. **Table 2** is based on this round robin conducted in accordance with Practice **E691**, involving three materials tested by 15 laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each test result was the average of two individual determinations. Each laboratory obtained four test results for each material. (**Warning**—The following explanation for  $r$  and  $R$  (**14.3 – 14.3.3**) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in **Table 2** shall not be applied to acceptance or rejection of material, as these data apply only to materials tested in the round robin and are unlikely to be rigorously representative of the other lots, formulations, conditions, material, or laboratories. Users of this test method shall apply the principles outlined in Practice **E691** to generate data specific to their materials and laboratory (or between specific laboratories). The principles of **14.3 – 14.3.3** would then be valid for such data.)

14.3 *Concept of  $r$  and  $R$  in Table 2*—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 for each test result, then:

14.3.1 *Repeatability*— $r$  is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the

<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1202.

same day in the same laboratory. Two test results shall be judged not equivalent if they differ by more than the  $r$  value for the material.

14.3.2 *Reproducibility*— $R$  is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories, not necessarily on the same day. Two test results shall be judged not equivalent if they differ by more than the  $R$  value for that material.

14.3.3 Any judgment in accordance with 14.3.1 or 14.3.2 would have an approximate 95 % (0.95) probability of being correct.

14.4 There are no recognized standards by which to estimate bias of this test method.

NOTE 16—Based on the round-robin test data,<sup>6</sup> a bias may exist between data obtained on test equipment with a span between supports of 101.6 mm (4.0 in.) (Method A) and 100 mm (3.937 in.) (Method B), with results being of 1.0-4.5°C higher for the equipment with a span width between supports of 100 mm, and the value of the difference is material dependent (see Table 3).

## 15. Keywords

15.1 deflection temperature; flexural load; flexure

## ANNEXES

### (Mandatory Information)

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

A1.1 If the unit in operation is of the type that has only one temperature probe in the bath, and this probe is monitored to record the deflection 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 a calibrated reference temperature meter and probe traceable to the International System of Units (SI) through NIST or another 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 reference probe within 10 mm of specimen, 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. While the unit is programmed to maintain a constant temperature between 20 and 50°C, with all stirrers operating, place the traceable probe in the bath at one of the required points in the bath, allow the bath and reference probe to stabilize for a minimum of 5 minutes. Read and record the readout of the calibrated probe and the units internal temperature display to the nearest 0.1°C. Move the 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 minutes.

A1.3.1.1 With the reference probe at the location closest to the unit's centralized temperature sensor 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 minutes 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 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 traceable probe within 10 mm of specimen height at one of the required points in the bath. Allow the bath and reference probe to stabilize for a minimum of 5 minutes. Read and record the readout of the calibrated probe and the unit internal temperature display to the nearest 0.1°C. Move the 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 minutes. 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 minutes 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 With the reference probe at the location closest to the unit's centralized temperature sensor 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 minutes 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.

A1.3.3 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°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.

A1.3.4 If the preceding steps have been taken and successfully completed, cool the bath down to a normal start temperature and allow the bath to stabilize. Place the reference probe at the point in the bath that the preceding gathered data shows the greatest error. Start a test at 120°C/h. Read and record the temperature of both the unit's display and the readout of the NIST 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 NIST probe, maintaining any delay interval, if used, every 5 min for 1 h.

A1.3.5 Evaluate the data acquired during the preceding test. Ensure that the temperature of the bath is rising at the correct rate as outlined in 7.1.2, 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 INSTRUMENTS

A2.1 This procedure is to be used in addition to manufacturers requirements and procedures to calibrate an HDT (DTUL) instrument that has multiple temperature sensors in the bath to control the temperature of the bath, 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 to the International System of Units (SI) through NIST or another National Metrology Institute. 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 second 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>7</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 bath, a minimum of five minutes 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.

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 to a normal start temperature and allow the bath temperature to stabilize. Program the UUT to increase the bath temperature at a rate of 2°C/min (120°C/h). Read and record the temperature at each station at intervals of five minutes 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 to ensure that the temperature rate of rise at each station is within the tolerances outlined in 7.1.2. It is allowable for the first ten minutes 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 ten minutes, 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>8</sup> to determine the thermal expansion of the station,

<sup>7</sup> Near is defined as  $\pm 5^\circ\text{C}$ .

<sup>8</sup> Invar or borosilicate glass has been found suitable for this purpose.

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°C rate of rise. If this compensation value is greater than 0.013 mm (0.0005 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, support radius) 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 [7.1.1](#).

A2.7.3 The weights must be verified and conform to the specification outlined in [7.1.4](#). Note that it is permissible that the smaller weights ( $\leq 4$  grams) individually do not meet the  $\pm 2.5$  % requirements, but when used in conjunction with other larger weights the total applied mass must conform to the requirements.

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. 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.

## 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 shall be constructed essentially as shown in [Fig. X1.1](#) and shall consist of the following:

X1.1.1.1 *Single-Pan or Equal-Arm Laboratory Balance*, having a sensitivity of at least 0.1 g.

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

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

##### X1.2. Procedure

X1.2.1 Calculate the load required to give the desired fiber stress in accordance with [Eq 1](#).

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 balance.

X1.2.4 Start oil bath stirrer on tester and heat oil to 75 to 100°C and continue operating during calibration.

X1.2.5 Determine tare weight of the bridge.

X1.2.6 Position the test unit on the cross bar above the balance pan.

X1.2.7 Lubricate the rod and guide hole surfaces with light oil.

X1.2.8 Lift the loading rod and put the bridge in place on the balance pan so that it will support the loading rod (bridge height dimension is such that it supports the rod 13 mm ( $\frac{1}{2}$  in.) above the level of the specimen supports).

X1.2.9 Adjust the dial face on the dial gauge so that the needle points to zero (with no depression of the spindle).

X1.2.10 With the deflector arm in position over the dial gauge, lower the rod to the bridge, and then release it very gently. When the balance reaches equilibrium, the desired dial gauge movement should be  $0.89 \pm 0.05$  mm ( $0.035 \pm 0.002$  in.) (0.64 mm (0.025 in.) as in zero point, plus 0.25 mm (0.010 in.) for deflection of the test bar in the normal test). Read just the deflector arm position until  $0.89 \pm 0.05$  mm is repeatedly obtained at balance.

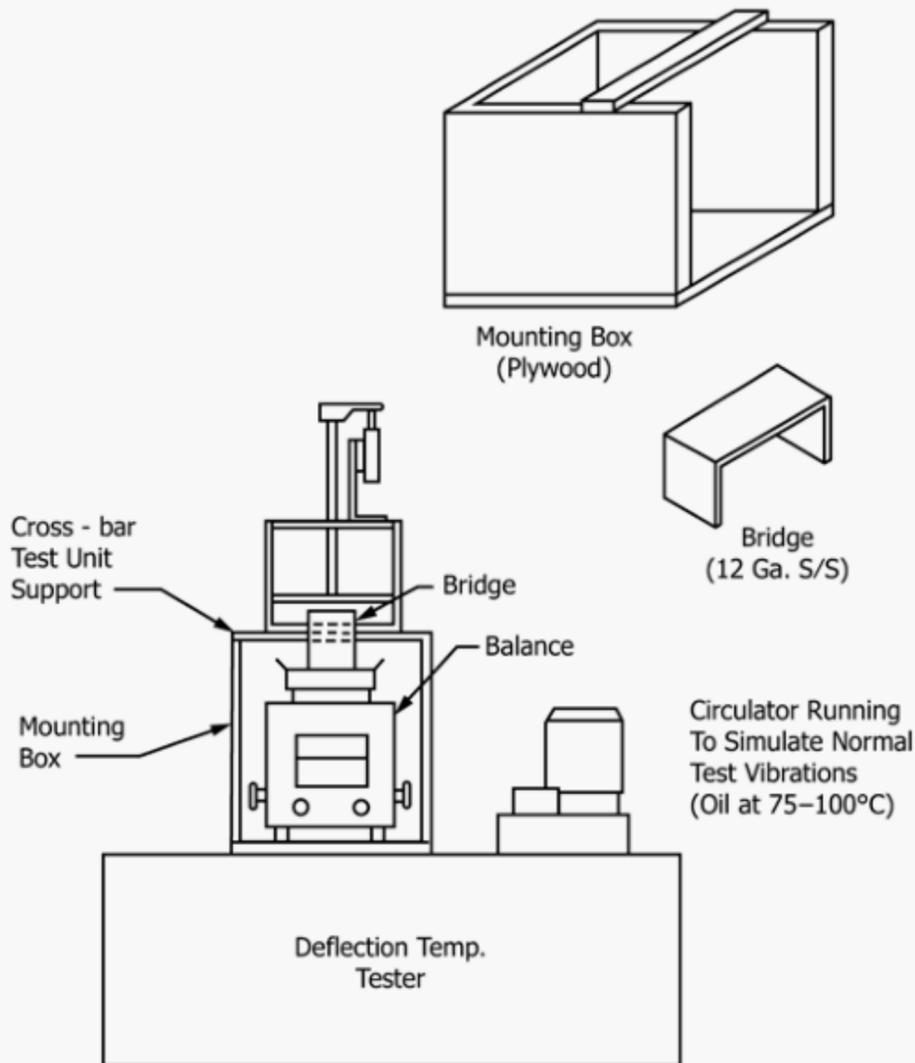


FIG. X1.1 Calibration Apparatus Using Platform Balance

X1.2.11 Record the force, in grams, at the  $0.89 \pm 0.05$ -mm ( $0.035 \pm 0.002$ -in.) equilibrium deflection.

X1.2.12 Adjust weight of loading rod, or spring force in dial gauge, to provide the loading required for a desired stress at 0.89-mm (0.035-in.) deflection in accordance with Eq 1.

NOTE X1.1—The test units (rods, guide surfaces, and dial gauge) must be clean and free of any surface imperfections, and so forth, to achieve precision in calibration and also in normal test use.

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

### X2.1. Apparatus

X2.1.1 The apparatus shall be constructed essentially as shown in Fig. X2.1 and shall consist 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 grip separation. The testing machine used should be capable of measuring loads of at least 2000 g. The rate of separation of the jaws shall be capable of adjustment to 0.51 mm (0.02 in.)/min.

X2.1.1.2 *Platform*, square, approximately 203 by 203 mm (8 by 8 in.) 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

X2.2.1 Mount the support platform in the lower crosshead clamps.

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

X2.2.3 Secure the deflection temperature test unit on the support platform and adjust the loading rod support so that the tip of the loading rod is 12.7 mm ( $\frac{1}{2}$  in.) from the top of the specimen supports.

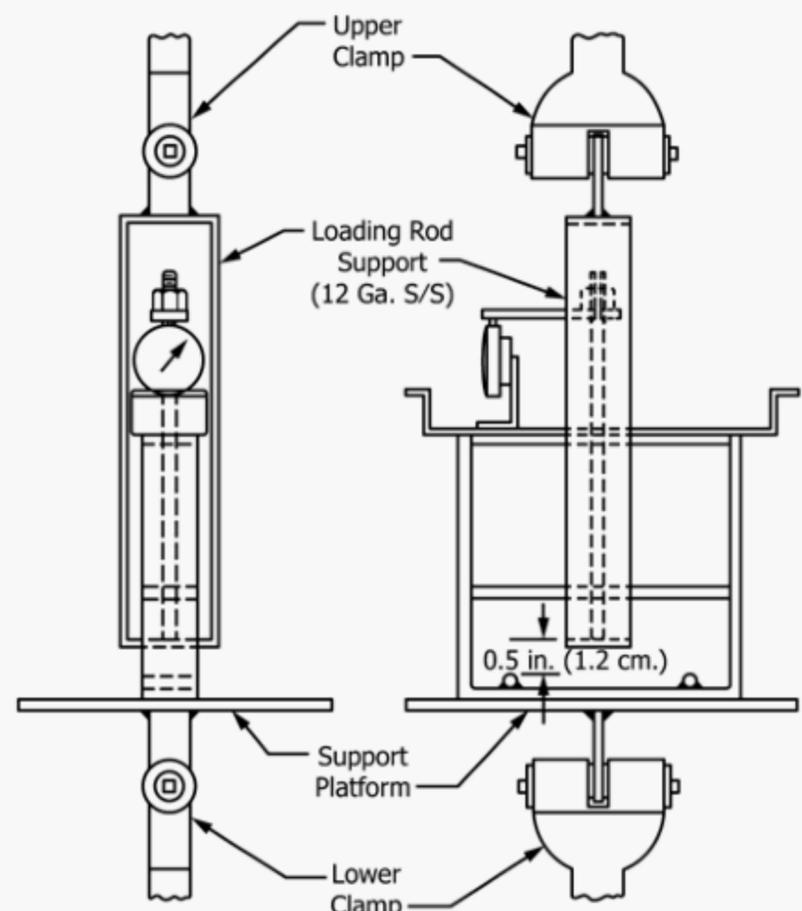


FIG. X2.1 Calibration Apparatus Using a Tensile Machine

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 (0.02 in.)/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 1 in./min.

X2.2.7 Record the force, in grams, at  $0.89 \pm 0.05$ -mm ( $0.035 \pm 0.002$ -in.) deflection.

X2.2.8 Adjust the weight of the loading rod required to give the desired maximum fiber stress in accordance with Eq 1.

### X3. PROCEDURE FOR DETERMINATION OF CORRECT SPECIMEN LOADING BY WEIGHING THE APPLIED LOAD IN SITU

#### X3.1. Scope

X3.1.1 This procedure covers an alternate technique for measuring the net force that is applied to a deflection temperature specimen at midspan.

X3.1.2 The net force is measured with the specimen support unit and loading assembly in place, that is, immersed in the heat-transfer medium.

X3.1.3 This technique permits the user to account for discrepancies in the actual load applied to the specimen as a result of spring forces, friction, buoyancy, etc.

X3.1.4 *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.*

#### X3.2. Apparatus

X3.2.1 The apparatus shall be constructed essentially as shown in Fig. X3.1 and shall consist of the following:

X3.2.1.1 *Electronic Weighing System with Load Cell* (for example, digital scale or tensile testing machine), single-pan balance, or equal-arm laboratory balance, with a minimum capacity of 2000 g and a sensitivity of 0.1 g.

X3.2.1.2 *Platform Assembly*, for supporting the scale or balance above the deflection temperature bath unit.

X3.2.1.3 *Mass Support Unit*, to hold the loading rod and mass in position while the force measurement is determined.

X3.2.1.4 *Adjustment Fitting*, for connection of the mass support to the load cell or balance. This fitting should facilitate adjusting the test fixture so that the loading force can be measured at the desired position.

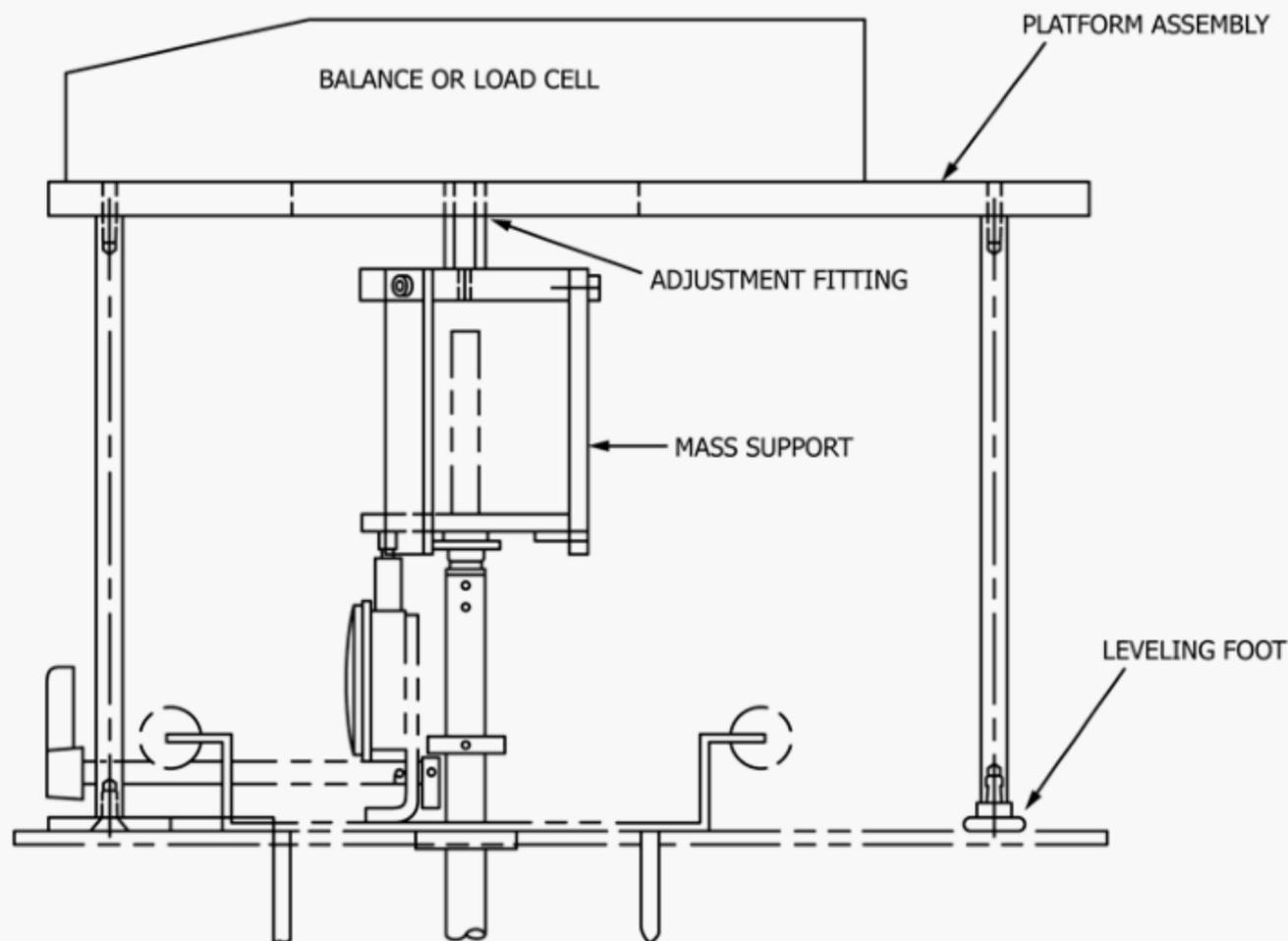


FIG. X3.1 Apparatus for Determination of Correct Specimen Loading

### X3.3. Procedure

X3.3.1 Determine the loading required to give the desired fiber stress in accordance with [Eq 1](#).

X3.3.2 Place the necessary mass on the loading rod.

X3.3.3 Lower the specimen support unit and loading assembly into the bath.

X3.3.4 Start the circulator, provided that the vibration produced by the circulator motor does not affect the weighing system adversely.

**NOTE X3.1**—Some vibration from the circulator may be dampened by using rubber feet on the platform assembly, or by designing the platform assembly so that it spans the bath unit rather than rest on top of it.

X3.3.5 If a scale or balance is used, position the platform assembly on top of the deflection temperature bath unit and level it. Place the scale or balance on top of the platform assembly and verify that it is level.

X3.3.6 Attach the adjustment fitting to the bottom of the load cell or balance.

X3.3.7 Attach the mass support to the bottom of the adjustment fitting.

X3.3.8 If a load cell is used, allow it to warm up before making the measurements. Tare out the weight due to the mass support and adjustment fitting.

X3.3.9 Position the mass support so that it bears the weight of the loading rod and mass.

X3.3.10 Verify that the load cell or balance, adjustment fitting, mass support, and loading rod are uniaxially aligned. It is very important to ensure that the test setup does not introduce any off-center loading into the system that will result in incorrect force measurements.

X3.3.11 Use the adjustment fitting to position the loading assembly so that it corresponds to the zero deflection position. Zero the deflection measurement device of the machine, if necessary. Dial gauges should be adjusted in accordance with [Appendix X5](#).

X3.3.12 Record the indicated load at the zero deflection position to the nearest 0.1 g.

X3.3.13 Use the adjustment fitting to lower the loading assembly to the final deflection position, typically 0.25 mm.

X3.3.14 Record the indicated load at the final deflection point to the nearest 0.1 g.

**NOTE X3.2**—These force measurements may be made with the bath at any convenient temperature. The effect of temperature on the buoyancy force over the usable range of the machine is generally negligible for commonly used silicone fluids and loading assembly designs. The decrease in the oil density is offset by the increased volume of oil dispersed. If desired, the user may perform this load verification procedure at two different temperatures to confirm the condition.

X3.3.15 Based on these measurements, adjust the mass so that the applied force corresponds to the calculated force of [X3.3.1](#).

X3.3.16 The difference between the force measurement at the zero deflection position (0.00 mm) and the force measurement at the final deflection position (typically 0.25 mm) should be within the  $\pm 2.5\%$  tolerance as specified in [7.1.4](#).

**NOTE X3.3**—If the force differential is excessive over the deflection measuring range, the user should attempt to identify the component responsible for the deviation, implement the necessary corrections, and repeat this procedure to ensure that the proper adjustments have been made. It may be possible to adjust the machine so that the calculated load is achieved at an intermediate position (for example, 0.12 mm), thereby permitting the load at the zero deflection position (0.00 mm) and the final deflection position (typically 0.25 mm) to fall within the allowable tolerance.

## X4. PROCEDURE FOR VERIFYING THE CALIBRATION OF PENETRATION MEASURING DEVICES USING GAUGE BLOCKS

X4.1 This procedure is intended to provide a method of verifying the calibration of penetration measuring devices typically found on DTUL measuring instruments. It is not a calibration method. If the user finds that the measuring device on one or more of the test frames is out of calibration, the manufacturer of the instrument, or a qualified calibration service company should be consulted to have the problem corrected. This procedure may be used for dial indicator, LVDT, and encoder-type penetration measurement devices.

X4.2 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 machine, the frame may 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. Verification should be made using the minimum load that may be encountered during testing.

X4.3 Thoroughly clean the loading nose and the anvils where the specimen is normally positioned.

X4.4 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. If a 1.00-mm gauge block is not available, a 1.016-mm (0.040-in.) gauge block can be substituted.

X4.5 Place the stacked gauge 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 loading nose rests in the middle of the block. Add the required weight to the rod to apply force to the block, simulating test conditions. Zero the indicator or record the reading on the display.

**NOTE X4.1**—Care must be taken to avoid damaging the gauge blocks when using heavy loads.

X4.6 Lift the loading rod and carefully remove the 1.00-mm block from beneath the rod without changing the position of

the remaining block. Lower the rod onto the remaining gauge block. Record the reading on the indicator. The reading should be equal to  $1.00 \pm 0.02$  mm.

X4.7 Repeat the procedure at least twice to ensure repeatability. Intermediate reading can be verified in a similar manner

by using different gauge blocks.

X4.8 Repeat the procedure on all of the instrument's test frames.

## X5. PROCEDURE FOR DETERMINATION OF SPRING FORCE AND CONDITION OF GAUGE

### X5.1. Apparatus

X5.1.1 The apparatus should be setup essentially as shown in Fig. X5.1 and should consist of the following:

X5.1.1.1 *Testing Machine*—A testing machine of the constant-rate-of-crosshead-movement type, equipped with devices for recording the load and movement of the crosshead.

X5.1.1.2 *Load Measurement Device*—The load measurement device shall be accurate to 0.5 g.

X5.1.1.3 *Event Detector (Optional)*—The event detector is used to mark specific points along the graph to indicate various deflections of the dial gauge stem.

### X5.2. Procedure

X5.2.1 Set up the testing machine as shown in Fig. X5.1.

X5.2.2 Calibrate and zero the tensile test machine's force and position displays.

X5.2.3 Position the support unit and dial gauge on the bottom fixed or movable member of the test machine. Position the dial gauge stem directly beneath the center of the load cell anvil.

X5.2.4 Set the crosshead speed of the testing machine to approximately 0.3 mm/min. Set the chart speed to approximately 60 mm/min.

X5.2.5 Zero the dial gauge. Position the anvil so that it is just touching the stem of the dial gauge and less than a 1 g of force is observed on the chart recorder.

X5.2.6 Start the crosshead moving to deflect the stem of the dial gauge. The load on the chart will increase as the spring in the dial gauge is stretched. At each 0.05 mm of deflection use the event marked or manually mark a position along the load-deflection curve.

NOTE X5.1—If the dial gauge has a needle contact pointer to provide an electrical signal to the controller, ensure that this pointer does not come into contact with the moving pointer during the test. Contact will result in a significant increase in load, and a false reading of the spring tension.

X5.2.7 Examples of the load-deflection curves are shown in Figs. X5.2 and X5.3. If the gauge is working properly, the curve should be similar to the one in Fig. X5.2. If the gauge is sticking or has other problems, it will show the behavior shown in Fig. X5.3.

X5.2.8 From the load-deflection curve determine the average spring force in the displacement range of the dial gauge where the test measurements are determined. Determine the lowest and highest loads from the curve for the displacement range in which the test will be conducted. If the difference between the low and high values is greater than 5 % of the total mass calculated from Eq 1, then the gauge should be replaced or reworked to correct the erratic behavior.

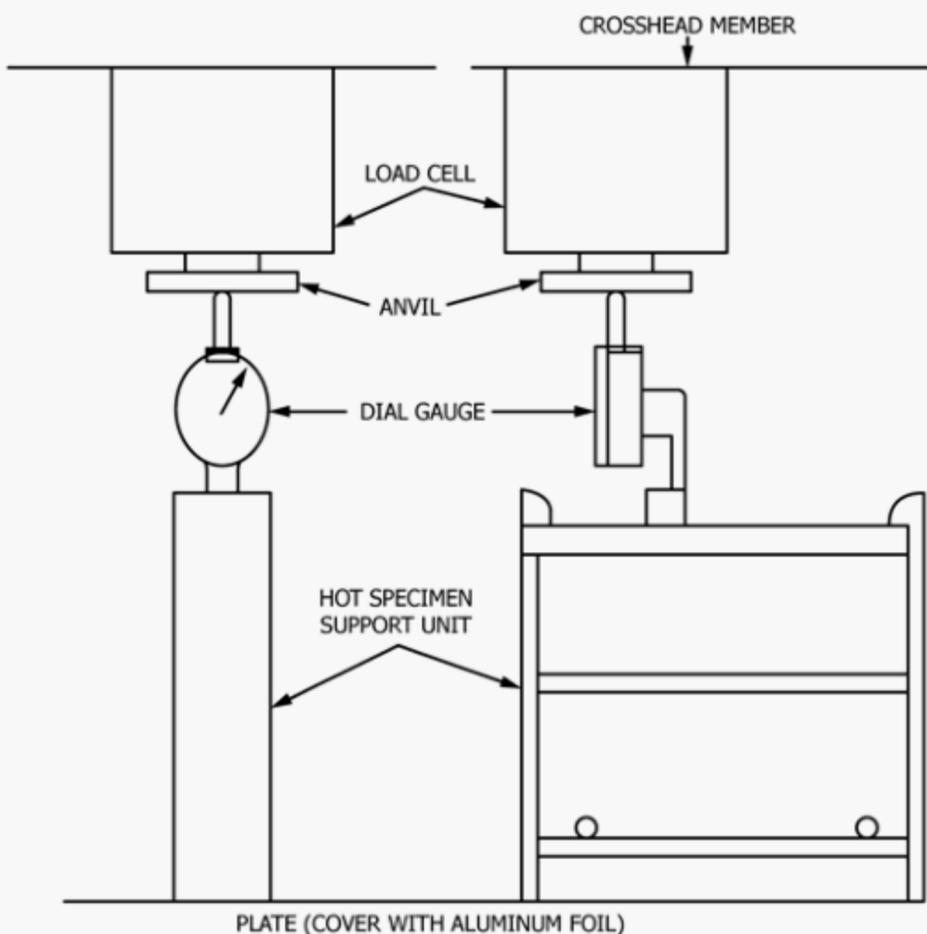


FIG. X5.1 Calibration Apparatus for Determining Spring Force

LOAD (grams) →

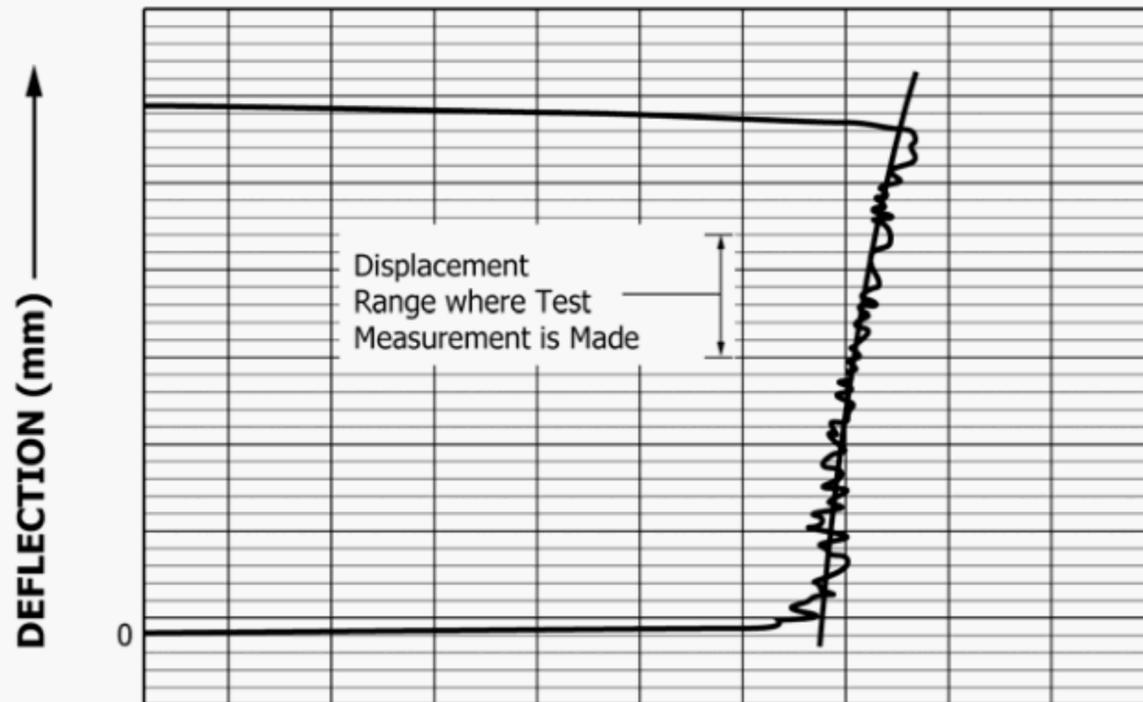


FIG. X5.2 Load Versus Deflection Curve for Gauge With No Current Problems

LOAD (grams) →

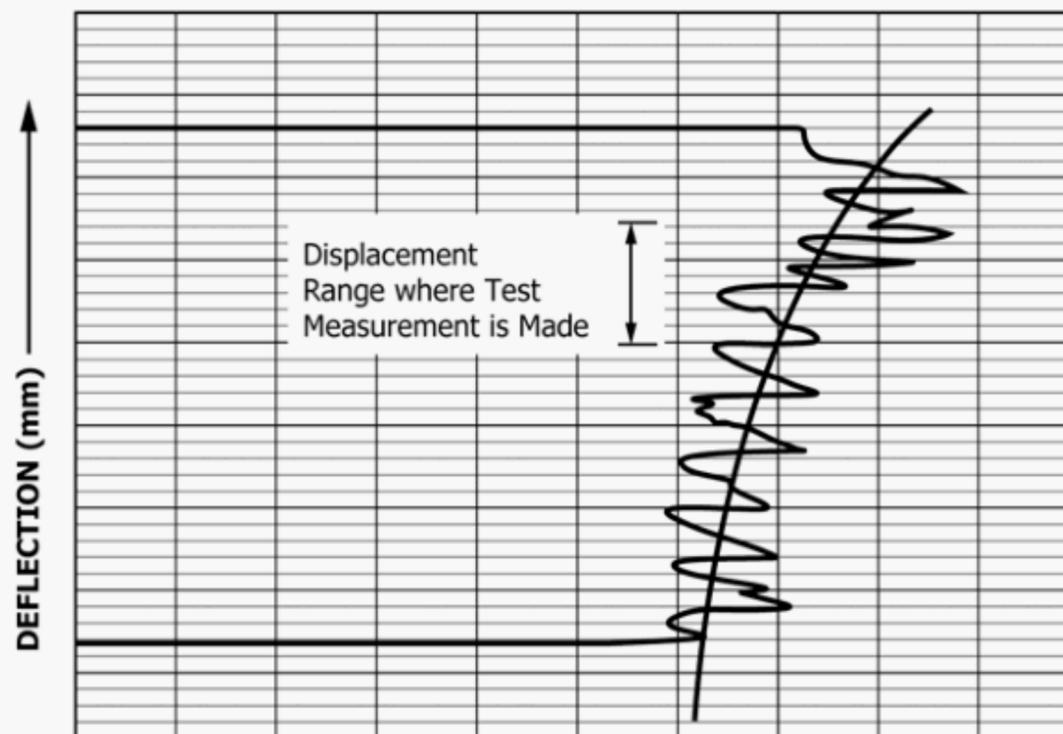


FIG. X5.3 Load Versus Deflection Curve for Gauge With Problems

### SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D648 - 16) that may impact the use of this standard. (April 1, 2018)

(1) Revised 9.1 and Note 8.

(2) Revised Annex A1.3 through A1.3.4.

(3) Revised Annex A2.3.

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