



Standard Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position¹

This standard is issued under the fixed designation D 648; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the 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 ($1/8$ 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 the standard. The values given in parentheses are for information only.

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 2—The test method described as a Method B of this test method, and test methods Ae and Be of ISO 75-1 and ISO 75-2, 1993, are technically equivalent.

2. Referenced Documents

2.1 ASTM Standards:

- D 618 Practice for Conditioning Plastics for Testing²
- D 883 Terminology Relating to Plastics²
- D 1898 Practice for Sampling of Plastics³
- D 1999 Guide for Selection of Specimens and Test Param-

eters from ISO/IEC Standards³

D 5947 Test Methods for Physical Dimensions of Solid Plastics Specimens⁴

E 1 Specification for ASTM Thermometers⁵

E 77 Test Method for Inspection and Verification of Thermometers⁵

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁶

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques⁵

E 608 Specification for Metal-Sheathed Base-Metal Thermocouples⁵

E 644 Test Methods for Testing Industrial Resistance Thermometers⁵

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁶

E 879 Specification for Thermistor Sensors for Clinical Laboratory Temperature Measurement⁵

E 1137 Specification for Industrial Platinum Resistance Thermometers⁵

2.2 ISO Standards:⁷

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

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

2.3 NIST Document:⁸

NBS Special Publication 250-22

3. Terminology

3.1 *General*—The definitions of plastics used in this test method are in accordance with Terminology D 883 unless otherwise indicated.

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

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ Discontinued; see 1997 *Annual Book of ASTM Standards*, Vol 08.01.

⁴ *Annual Book of ASTM Standards*, Vol 08.03.

⁵ *Annual Book of ASTM Standards*, Vol 14.03.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

⁷ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁸ Mangum, B. W., "Platinum Resistance Thermometer Calibration," *NBS Special Publication 250-22*, 1987. Available from National Institute of Standards and Technology, Gaithersburg, MD.

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 3). 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 3—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 may 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.

6. Interferences

6.1 The results of the test may depend 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 may depend 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 may be 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 may 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:

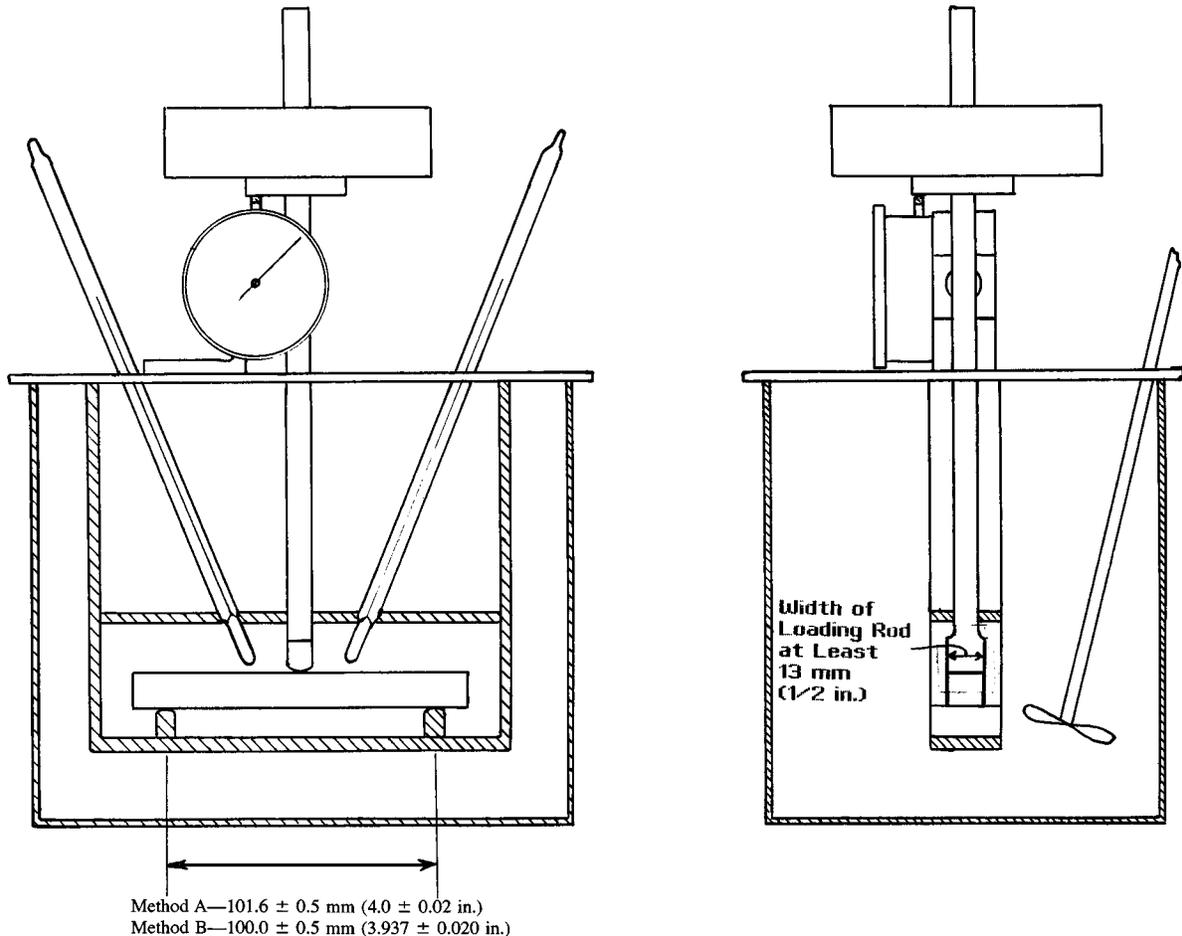


FIG. 1 Apparatus for Deflection Temperature Test

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 ± 0.2 mm (0.118 ± 0.008 in.).

7.1.1.1 Method A— 101.6 ± 0.5 mm (4.0 ± 0.02 in.).

7.1.1.2 Method B— 100.0 ± 0.5 mm (3.937 ± 0.020 in.).

NOTE 4—A test should be made on each apparatus using a test bar made of a material having a low coefficient of expansion.⁹ The temperature range to be used should be covered and a correction factor determined for each temperature. If this factor is 0.013 mm (0.0005 in.) or greater, its algebraic sign should be noted and the factor should be applied to each test by adding it algebraically to the reading of apparent deflection of the test specimen.

7.1.2 *Immersion Bath*—A suitable liquid heat-transfer medium (Note 5) in which the specimen shall be immersed. 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—A liquid heat-transfer medium shall be chosen which will not affect the specimen. Mineral oil is considered safe from ignition to 115°C . Silicone oils may be heated to about 260°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 CO_2 or other inert gas to isolate the oil surface from the atmosphere.

NOTE 6—A circulating air oven may be used if it can be shown that equivalent results are obtained.

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. The device may be a dial gage or any other indicating or recording device including electric displacement sensing apparatus.

7.1.4 *Weights*—A set of weights of suitable sizes so that the specimen can be 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 gage is used, the force exerted by its spring shall be determined and shall be included as part of the load (Note 8). Calculate the testing force and the mass that must be added to achieve the desired stress as follows:

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

$$F^l = F/9.80665$$

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

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 7—In some designs of this apparatus, the spring force of the dial gage 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 gage 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 gages 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 gage 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 gage's test measurement range, and whether the gage is contaminated and sticking.

7.1.5 *Temperature Measurement System*—Consisting of a thermocouple, thermometer, resistance thermometer, thermistor, etc., as the sensor, together with its associated conditioning and readout instrumentation to cover a suitable range. The thermometer shall be one of the following, or its equivalent, as prescribed in Specification E 1: Thermometer 1°C or 2°C , having ranges from -20 to 150°C or -5 to 300°C respectively, whichever temperature range is most suitable. Mercury in glass thermometers shall be calibrated for the depth of immersion in accordance with Test Method E 77. Thermocouples shall comply with the requirements of Specification E 608. Thermocouples shall be calibrated in accordance with Test Method E 220. Resistance thermometers shall comply with the requirements of Test Methods E 644 and Specification E 1137. Thermistors shall comply with the requirements of Specification E 879 and be calibrated in accordance with NIST Special Publication 250-22.

7.2 *Micrometers* shall meet the requirements of Test Methods D 5947 and be calibrated in accordance with that test method.

8. Sampling

8.1 Unless otherwise specified, sampling shall be in accordance with the sampling procedure prescribed in Practice D 1898. Adequate statistical sampling shall be considered an acceptable alternative.

9. Test Specimen

9.1 At least two test specimens shall be used to test each sample at each fiber stress. The specimen shall be 127 mm (5 in.) in length, 13 mm ($\frac{1}{2}$ in.) in depth by any width from 3 mm ($\frac{1}{8}$ in.) to 13 mm ($\frac{1}{2}$ in.). Tolerances on dimensions (for highly reproducible work) should be of the order of ± 0.13 mm (0.005 in.) over the length of the specimen.

⁹ Invar or borosilicate glass have been found suitable for this purpose.

NOTE 8—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 may be 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. The apparatus may be 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 thermometers to the point specified in their calibration, or 76 mm (3 in.) in the case of the ASTM thermometers referred to in 7.1.5.

NOTE 9—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 at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618 unless otherwise specified in the material standard or contract between interested parties. In cases of disagreement, the tolerances shall be $\pm 1^\circ\text{C}$ (1.8°F) and $\pm 2\%$ relative humidity.

NOTE 10—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 several 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 may 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 11—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 thermometer bulb or 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 12—Verification of the load should be made on all new equipment, after replacement of dial gages, 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 13—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 14—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 D 648 Method A or D 648 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.

14. Precision and Bias

14.1 *Precision*—An interlaboratory test program¹⁰ 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 Table 1. The critical difference limits are the limits beyond which observed differences should be considered suspect.

14.2 In 1995 a second round-robin¹¹ study was conducted. Table 2 is based on this round robin conducted in accordance with Practice E 691, involving 3 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 2 individual determinations. Each laboratory obtained 4 test results for each material.

NOTE 15—**Caution:** 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 should not be applied to acceptance or rejection of material, as these data apply only

¹⁰ Supporting data are available from ASTM Headquarters. Request RR: D20-1098.

¹¹ Supporting data are available from ASTM Headquarters. Request RR: D20-1202.

TABLE 1 Statistical Information^A

Polymer	Average ^B Value	Standard Deviation	Critical ^C 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

^AAll values are given in °C.

^BAverage of pairs.

^CBetween values of a pair.

TABLE 2 Precision, Deflection Temperature

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

^A 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:

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

^B S_R = between-laboratories reproducibility, expressed as standard deviation:

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

^C r = within-laboratory critical interval between two test results = $2.8 \times S_r$

^D R = between-laboratories critical interval between two test results = $2.8 \times S_R$.

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 should apply the principles outlined in Practice E 691 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 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,¹¹ 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

TABLE 3 Deflection Temperature (Average) Obtained on Test Equipment With Span Values of 100 and 101.6 mm (3.937 and 4.0 in.), °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

ANNEX

(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 temperature meter and probe traceable to NIST, 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 NIST 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 units internal temperature display to the nearest 0.1°C. Make any necessary adjustments to the unit's temperature controller to bring the bath to ±0.1°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 NIST 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 NIST 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 ±0.1°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 NIST 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.

A1.3.3 Evaluate the data from each of the three points in the bath at both low and high temperature. If any point is greater than ±0.5°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. The unit may be electronically modified or the use of glass thermometers (as outlined in 7.1.5) may be placed at each station and manually read and recorded at the moment of specimen deflection.

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 NIST 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.

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 (½ in.) above the level of the specimen supports).

X1.2.9 Adjust the dial face on the dial gage 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 gage, lower the rod to the bridge, and then release it very gently. When the balance reaches equilibrium, the desired dial gage movement should be 0.89 ± 0.05 mm (0.035 ± 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 ± 0.05 mm is repeatedly obtained at balance.

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

X1.2.12 Adjust weight of loading rod, or spring force in dial gage, 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 gage) must be clean and free of any surface imperfections, etc., to achieve precision in calibration and also in normal test use.

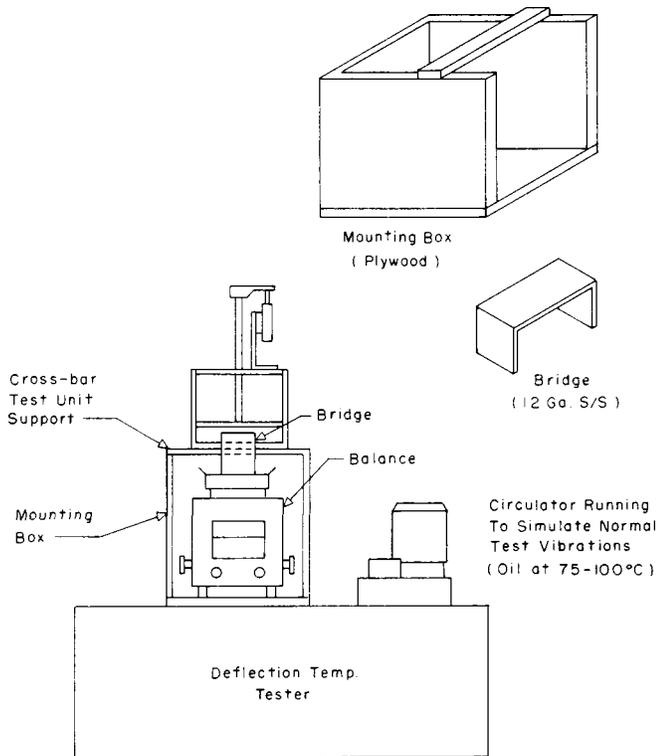


FIG. X1.1 Calibration Apparatus Using Platform Balance

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.

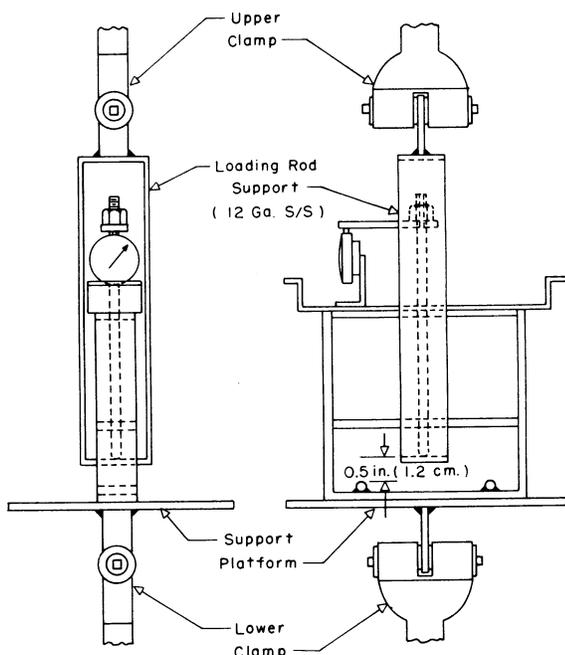


FIG. X2.1 Calibration Apparatus Using a Tensile Machine

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.

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

X2.2.5 Adjust the dial gage 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 gage.

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 gage shows movement, activate the chart drive at the rate of 1 in./min.

X2.2.7 Record the force, in grams, at 0.89 ± 0.05 -mm (0.035 ± 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.

X3.2 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.

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.

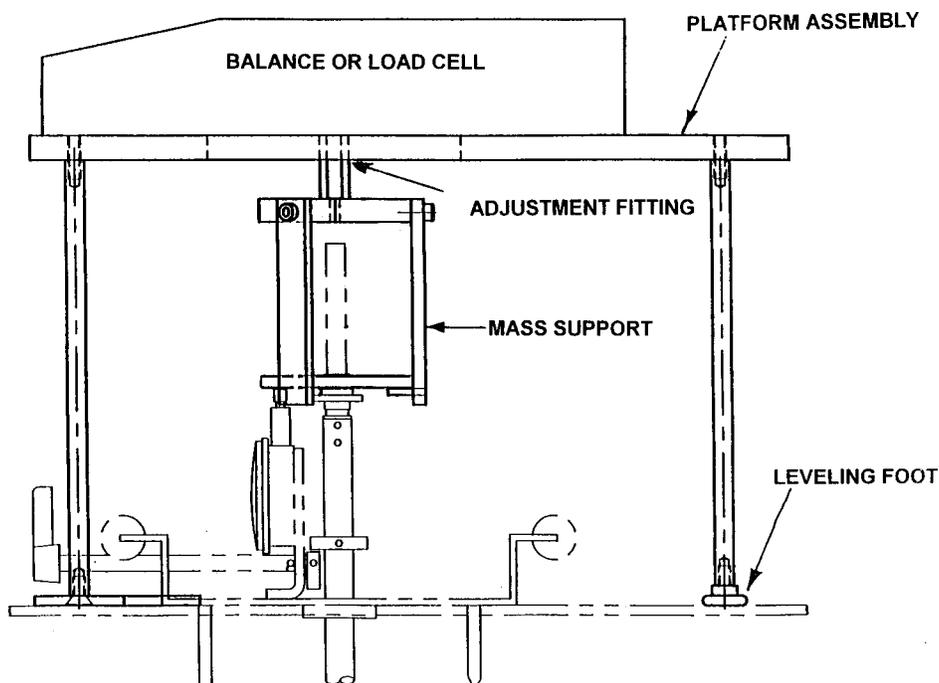


FIG. X3.1 Apparatus for Determination of Correct Specimen Loading

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 gages 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 GAGE 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 gage blocks that, when stacked together, are comparable in height to a typical test

specimen. At least one of the gage blocks should be a 1.00-mm block. If a 1.0-mm gage block is not available, a 0.040-in. (1.016-mm) gage block can be substituted.

X4.5 Place the stacked gage blocks in the test frame where the specimen is normally positioned. Lower the loading rod onto the gage 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 gage 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 gage block. Record the reading on the indicator. The reading should be equal to 1.00 ± 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 gage blocks.

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

X5. PROCEDURE FOR DETERMINATION OF SPRING FORCE AND CONDITION OF GAGE

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 gage 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 gage on the bottom fixed or movable member of the test machine. Position the dial gage 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 gage. Position the anvil so that it is just touching the stem of the dial gage and less than a 1 g of force is observed on the chart recorder.

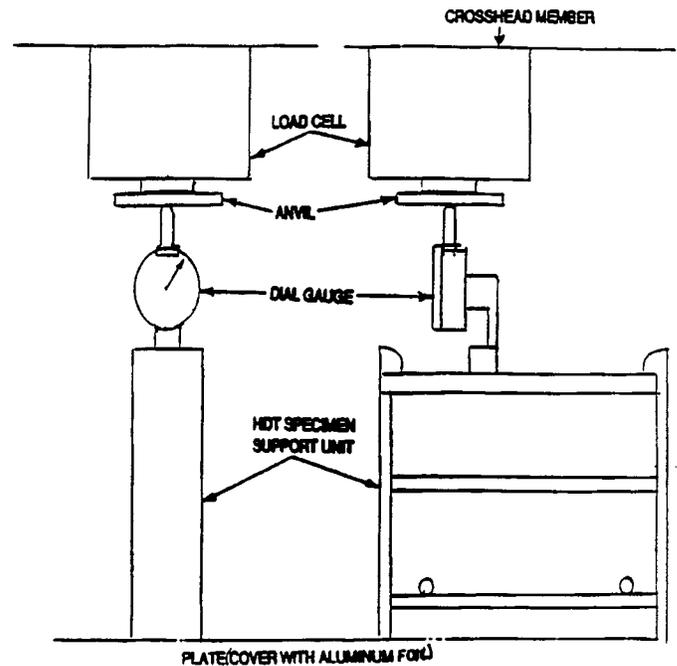


FIG. X5.1 Calibration Apparatus for Determining Spring Force

X5.2.6 Start the crosshead moving to deflect the stem of the dial gage. The load on the chart will increase as the spring in

the dial gage 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 gage 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 gage is working properly, the curve should be similar to the one in Fig. X5.2. If the gage 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 gage 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 gage should be replaced or reworked to correct the erratic behavior.

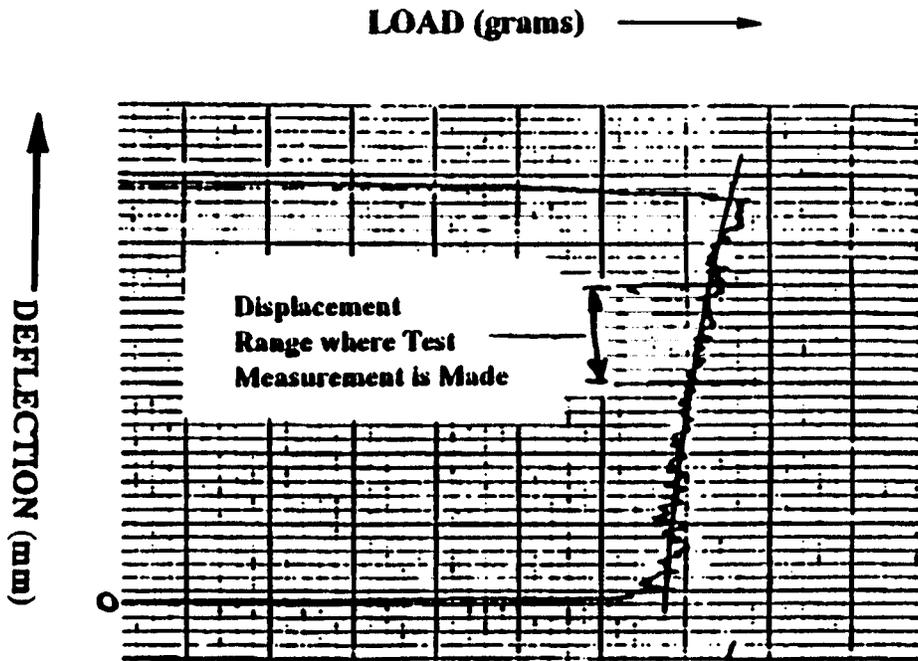


FIG. X5.2 Load Versus Deflection Curve for Gage With No Current Problems

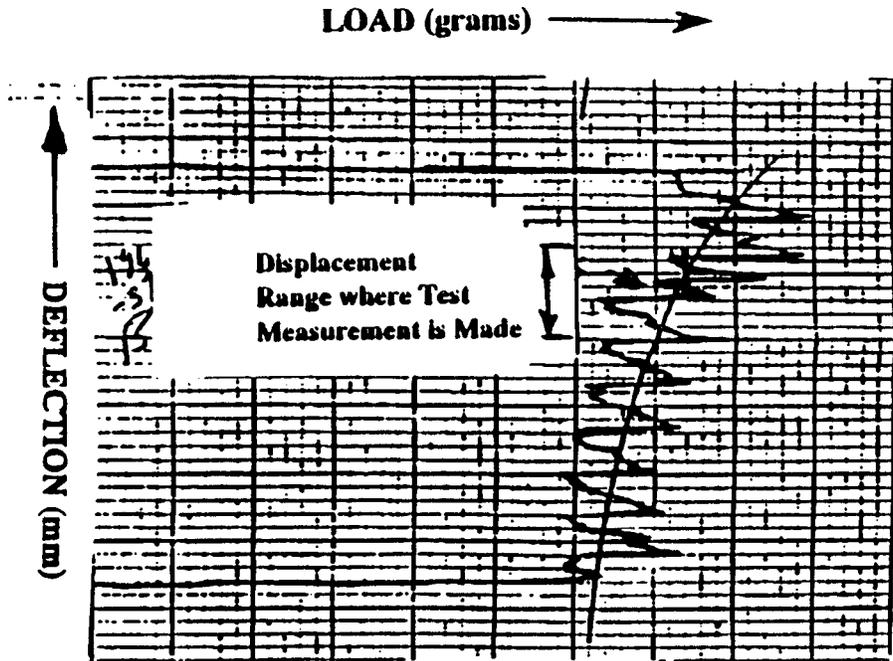


FIG. X5.3 Load Versus Deflection Curve for Gage With Problems

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 648 – 97:

(1) Addition of Annex A1 on calibration of single temperature probe units.

D 648 – 98:

(1) Modified ISO Equivalency Statement.

D 648 – 98c:

(1) Title Change.

(2) Revised 1.4.

(3) Revised Section 3, Terminology.

(4) Revised 4.1, changing units for fiber stress.

(5) Added new Section 6, Interferences.

(6) Revised 7.1.1, Apparatus, adding tolerances on the required span and on the radius of the load points.

(7) Revised Note 4.

(8) Revised 7.1.4, clarifying calculations of fiber stress.

(9) Revised 7.1.5, Temperature Measurement System, clarifying requirements for temperature measurement.

(10) Revised Section 8, Sampling.

(11) Added Note 9.

(12) Added 9.3.

(13) Revised Section 10.

(14) Added Note 10.

(15) Revised Section 11, Conditioning.

(16) Added Note 12.

(17) Revised Section 12, Procedure, to clarify testing procedure, and added Note 13.

(18) Revised Section 13, Report.

(19) Added Appendix X4 and Appendix X5.

(20) Added new Section 13, Precision and Bias.

(21) Deleted Note 12 regarding bias between manual and instrumented units.

(22) Added new Note 13, Caution.

D 6480 – 00:

(1) Added Methods A and B (see 7.1.1.1 and 7.1.1.2).

D 648 – 00a:

(1) 12.2—Added additional requirements to the end of the paragraph.

(2) Added Note 11 and renumbered subsequent notes.

D 648 – 01:

(1) Added Section 6.4.



D 648

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