Standard Test Method for
Distortion Temperature in Three-Point Bending by Thermomechanical Analysis[1]

This standard is issued under the fixed designation E 2092; 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.

1. Scope

1.1 This test method describes the determination of the temperature at which the specific modulus of a test specimen is realized by deflection in three-point bending. This temperature is identified as the distortion temperature measured. The distortion temperature is that temperature at which a test specimen of defined geometry deforms to a level of strain under applied stress of 0.455 and 1.82 MPa (66 and 264 psi) equivalent to those used in Test Method D 648. The test may be performed over the range of temperature from ambient to 300°C.

NOTE 1—Results may or may not agree with those obtained by Test Method D 648.

1.2 Electronic instrumentation or automated data analysis and reduction systems or treatments equivalent to this test method may be used.

NOTE 2—Since all electronic data treatments are not equivalent, the user shall verify equivalency to this test method.

1.3 SI values are the standard.

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

1.5 There is no ISO standard equivalent to this test method.

2. Referenced Documents

2.1 *ASTM Standards:*
D 648 Test Method for Deflection Temperature of Plastics Under Flexural Load[2]
E 473 Terminology Relating to Thermal Analysis[3]
E 1142 Terminology Relating to Thermophysical Properties[3]

E 1363 Test Method for Temperature Calibration of Thermomechanical Analyzers[3]

3. Terminology

3.1 *Definitions*—Specific technical terms used in this standard are defined in Terminologies E 473 and E 1142.

3.1.1 *distortion temperature [°C], n*—the temperature at which an arbitrary strain level is obtained in three-point bending under an arbitrary load.

3.1.2 *strain, r [mm/m], n*—the dimension change in normalizing dimension due to an applied force.

3.1.3 *stress, S [Pa = N/m^2], n*—force per unit area.

4. Summary of Test Method

4.1 A test specimen of known dimensions is tested in three-point bending mode. A known stress is applied to the center of a test specimen supported near its ends, as it is heated at a constant rate from ambient temperature to the upper temperature limit for the material. The deflection of the test specimen is recorded as a function of temperature. The temperature at which a predetermined level of strain is observed in the test specimen is analyzed as the distortion temperature.

5. Significance and Use

5.1 Data obtained by this test method shall not be used to predict the behavior of materials at elevated temperatures except in applications in which the conditions of time, temperature, method of loading, and stress are similar to those specified in the test.

5.2 This standard is particularly suited for quality control and development work. The data are not intended for use in design or predicting endurance at elevated temperatures.

6. Apparatus

6.1 A thermomechanical analyzer consisting of:

6.1.1 *Rigid Specimen Holder*, of inert, low expansivity material <20 μm m^{-1}· °C^{-1} to center the specimen in the furnace and to fix the specimen to mechanical ground.

6.1.2 *Flexure Fixture*, of inert, low expansivity material <20 μm m^{-1}· °C^{-1}, to support the test specimen in a three-point bending mode (see Fig. 1).
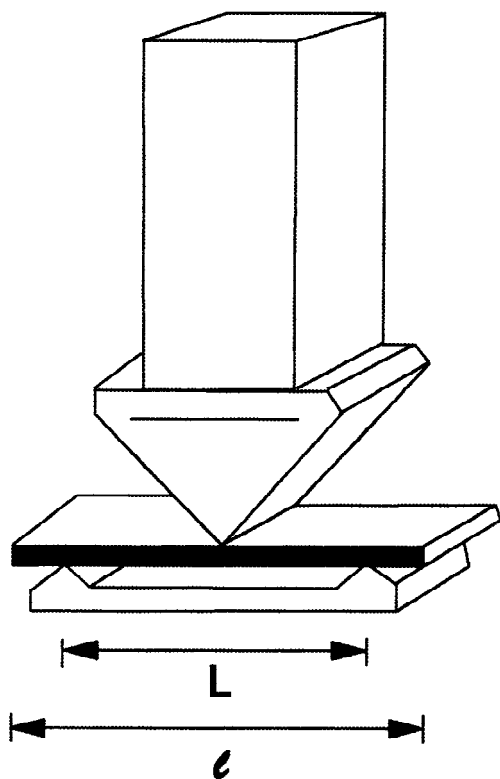

FIG. 1 Flexure Support Geometry

6.1.3 *Rigid Knife Edge Compression Probe*, of inert, low expansivity material <20 μm m^{-1}· °C^{-1} that contacts the specimen with an applied compression force (see Fig. 1).

6.1.4 *Deflection Sensing Element*, having a linear output over a minimum range of 5 mm to measure the displacement of the rigid compression probe (see 6.1.3) to within ± 0.1 μm.

6.1.5 *Programmable Weight or Force Transducer*, to generate a constant force (± 2.5 %) between at least 0.01 to 1.0 N, that is applied to the specimen through the rigid compression probe (see 6.1.3).

6.1.6 *Temperature Sensor*, that can be positioned reproducibly in close proximity to the specimen to measure its temperature within the range between 25 and 300°C to ± 0.1°C.

6.1.7 *Temperature Programmer and Furnace*, capable of temperature programming the test specimen from ambient to 300°C at a linear rate of at least 2 ± 0.1°C/min.

6.1.8 *Means of Providing a Specimen Environment*, of inert gas at a purge rate of 50 mL/min ± 5 %.

NOTE 3—Typically, inert purge gases that inhibit specimen oxidation are 99.9+ % pure nitrogen, helium or argon. Dry gases are recommended for all experiments unless the effect of moisture is part of the study.

6.1.9 *Recording Device*, to record and display the experimental parameters of deflection on the *Y* axis (ordinate) to a sensitivity of ± 0.1 μm and of temperature on the *X* axis (abscissa) to a sensitivity of ± 0.1°C.

6.1.10 While not required, it is convenient to have a data analysis device, that will perform and display the calculations of this standard.

6.2 Micrometer, calipers, film gage or other length-measuring device capable of measuring lengths of 0.01 to 20 mm with a precision of ± 0.001 mm.

7. Hazards

7.1 Toxic or corrosive effluents, or both, may be released when heating some materials and could be harmful to personnel and to apparatus.

7.2 Because the specimen size is small, care must be taken to ensure that each specimen is homogeneous and representative of the sample as a whole.

7.3 The test specimen shall be isotropic. See 8.3.

8. Sampling

8.1 The specimens may be cut from sheets, plates, or molded shapes, or may be molded to the desired finished dimensions.

8.2 The specimens used in this test method are ordinarily in the form of rectangular beams with aspect ratios of 1: 3: 10 for thickness or specimen depth (*d*), width (*b*) and length (*l*), depending upon the modulus of the sample and length of the support span (*L*).

NOTE 4—Other specimen and support dimensions may be used but care must be taken that the support length to specimen thickness ratio (*L/d*) be greater than 10.

NOTE 5—The specimen shall be long enough to allow overhanging on each end of at least 10 % of the support span, that is, 1 ≥ 1.2 *L*.

NOTE 6—The overhang shall be sufficient to prevent the specimen from slipping from the supports.

8.3 This test method assumes that the material is isotropic. Should the specimen be anisotropic, such as in reinforced composites, the direction of the reinforcing agent shall be reported relative to the specimen dimensions.

8.4 Since duplicate determinations are required, at least 2 specimens shall be prepared from each sample.

9. Calibration

9.1 Calibrate the temperature display of the apparatus according to Test Method E 1363 using a heating rate of 2 ± 0.1°C/min.

9.2 Calibrate the deflection display of the apparatus according to the instrument manufacturer's instructions.

9.3 Calibrate the mechanism for applying force to the test specimen according to the instrument manufacturer's instructions.

10. Procedure

10.1 Measure the test length (*L*) of the test specimen as the distance between the two support points of the flexure support geometry to three significant figures (see Fig. 1).

10.2 Measure the width (*b*) and thickness (*d*) of the test specimen to three significant figures (see Fig. 2).

10.3 Select the stress (*S*) to be applied to the test specimen. This value is typically 0.455 or 1.82 MPa (66 or 264 psi) to correspond with the values used in Test Method D 648.

NOTE 7—Other values may be used but shall be reported.

10.4 Select the strain (*r*) to be used to identify the heat distortion temperature.

NOTE 8—This value is typically 2.0 mm/m (0.20 %) to correspond with the values used in Test Method D 648.
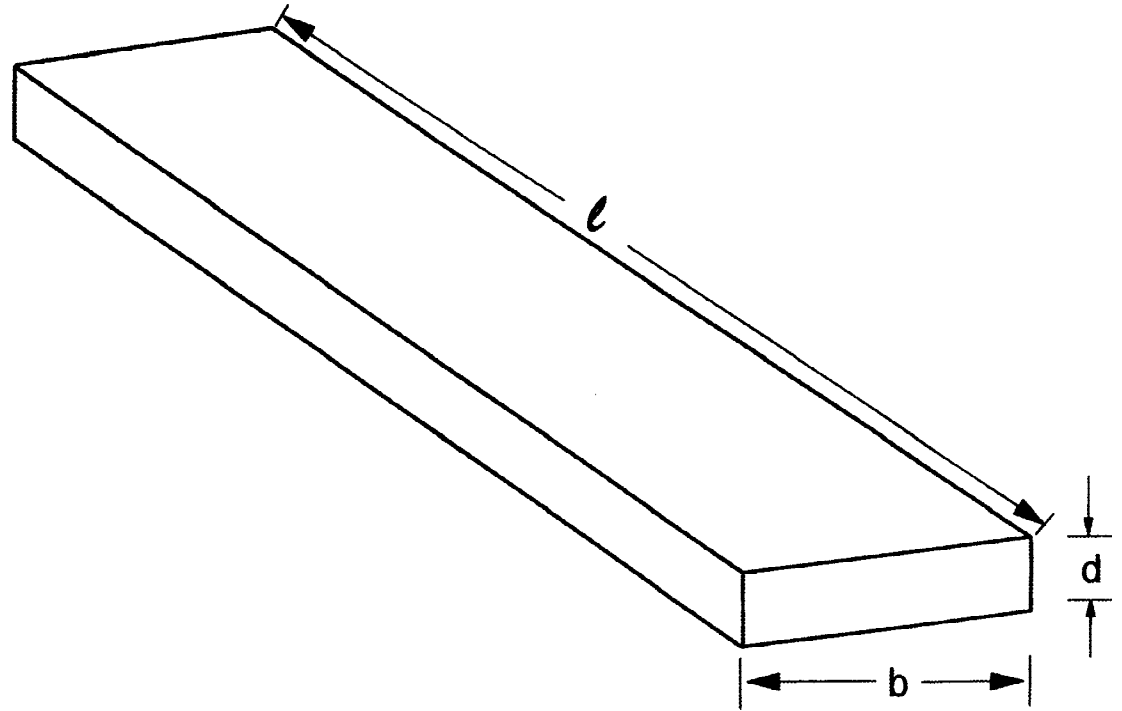
FIG. 2 Test Specimen Geometry

10.5 Using Eq 1, calculate the force (F) to be applied to the test specimen to three significant figures.

10.6 Using Eq 3, calculate the deflection (D) to be used as the experimental endpoint to three significant figures.

10.7 Center the specimen on the supports, with the long axis of the specimen perpendicular to the loading nose and supports (see Fig. 1).

NOTE 9—The typical rectangular test beam is tested flatwise on the support span, with the applied force through its thinnest dimension.

10.8 Set initial experimental conditions by placing the test specimen into flexure support, loading the compression probe onto the center of the test specimen in the three-point bending mode with the force calculated in 10.5. Set the deflection-axis signal to be zero at ambient temperature.

10.9 Program the temperature from ambient temperature at $2 \pm 0.1°C/min$ until the deflection, $D \pm 10\%$, determined in 10.6, is obtained while recording specimen deflection and temperature. Once the deflection value is achieved, terminate the temperature program and remove the load from the test specimen. Cool the apparatus to ambient temperature.

NOTE 10—Should the approximate distortion temperature of the material be known, the temperature program might be started at an elevated temperature that shall be at least 50°C below the anticipated distortion temperature.

10.10 Perform a baseline determination similar to section 10.9 except that the test specimen is the inverted flexure fixture.

NOTE 11—This step measures the deflection of the rigid specimen holder, flexure fixture and rigid knife edge compression probe.

10.11 For ease of interpretation, display the thermal curves from sections 10.9 and 10.10 with deflection displayed on the

Y-axis and temperature on the X-axis as illustrated in Fig. 3, both axes set to the same scale sensitivity.

10.12 Using the same Y-axis scale sensitivity, subtract the baseline curve from 10.10 from the test specimen curve from 10.9.

10.13 The distortion temperature is taken as the temperature at which the test specimen achieves a distortion of the value of D from the initial condition in the baseline subtracted curve of 10.12.

10.14 Repeat the experiment on a second, different test specimen. Report the result as the mean value of duplicate determinations.

11. Calculation

11.1 Calculate force value as follows:

$$F = (2\,S\,b\,d^2)/(3\,L) \qquad (1)$$

where:
F = force, N,
S = stress, MPa,
b = sample width, mm,
d = sample thickness, mm, and
L = length of the flexure fixture support span, mm.

11.1.1 As an example, if:

S = 0.455 MPa
b = 2.7 mm
d = 0.44 mm
L = 5.08 mm

then:

$$F = \frac{(2 \times 0.455\ \text{MPa} \times 2.7\ \text{mm} \times 0.44\ \text{mm} \times 0.44\ \text{mm})}{(3 \times 5.08\ \text{mm})} = 0.0312\ \text{N}$$
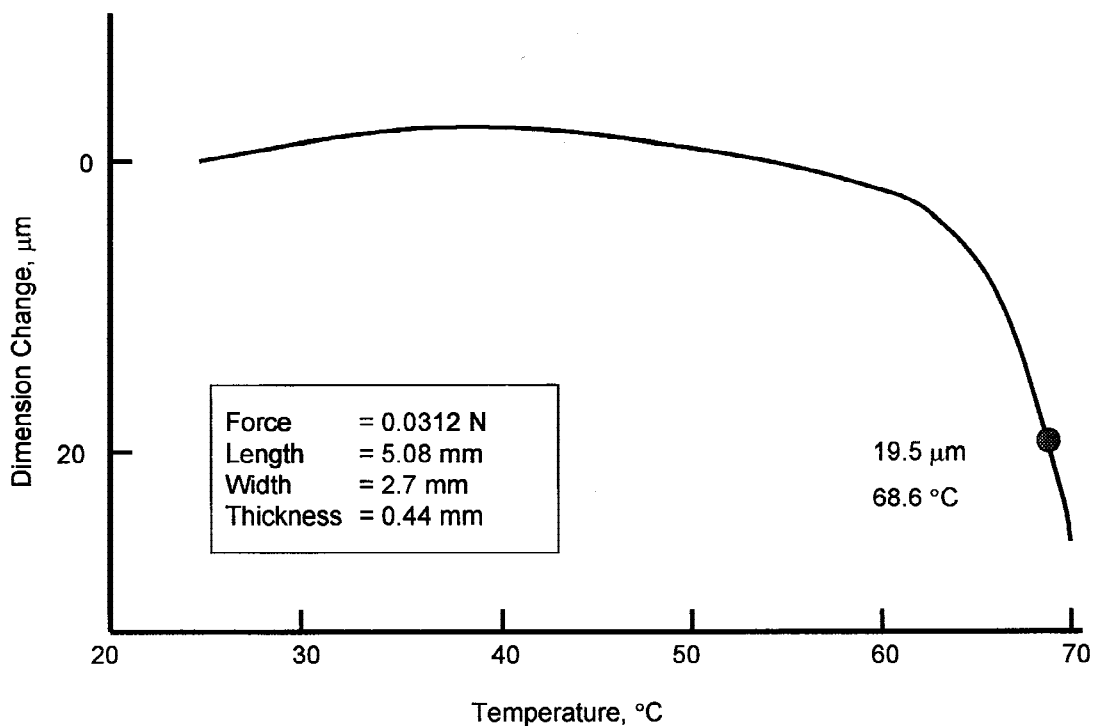
$$\qquad (2)$$

FIG. 3 Thermal Curve for Poly(vinyl chloride)

11.2 Calculate deformation value as follows:

$$D = (r L^2)/(6 d) \qquad (3)$$

where:

r = strain, mm/m.

11.2.1 As an example, if $r = 2.0$ mm/m, then:

$$D = \frac{(2.0 \text{ mm/m} \times 5.08 \text{ mm} \times 5.08 \text{ mm})}{(6 \times 0.44 \text{ mm})} = 19.6 \text{ μm} \qquad (4)$$

12. Report

12.1 Report the following information:

12.1.1 Complete identification and description of the material tested including source, manufacturer code, and any thermal or mechanical pretreatment,

12.1.2 Description of the instrument used, including model number and location of the temperature sensor,

12.1.3 Details of the procedure used to calculate the heat distortion temperature including strain and the resultant force, stress and resultant strain, as well as specimen dimensions including flexure distance,

12.1.4 Heating rate, °C/min, and temperature range,

12.1.5 A copy of all original records that are presented,

12.1.6 The mean value of the distortion temperature in three-point bending, °C, and

12.1.7 The specific dated version of this test method used.

13. Precision and Bias

13.1 *Precision*:

13.1.1 Limited information from one laboratory on eight materials indicates that the repeatability relative standard deviation is ± 1.6 % for materials ranging from 100 to 180°C.[4]

13.1.2 An interlaboratory test program is being planned for 2003–2004 to determine the precision of this standard. Anyone wishing to participate in this test program should contact ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

13.2 *Bias*:

13.2.1 A comparison was made between this test method and the deflection temperature under load (DTUL) measurements of Test Method D 648 for a series of polymeric materials.[4] A linear correlation was obtained with a linear correlation coefficient $r^2 = 0.99$. A bias of 1.7°C was observed.

13.2.2 A comparison was made between this test method and the DTUL measurements of Test Method D 648 using a series of 4 polyvinyl chloride samples.[5] The results derived from TMA data are 1.3°C lower than were the DTUL values.

13.2.3 At least one of the materials to be used in the interlaboratory test program described in 13.1.1 is intended to be tested by Test Method D 648. In this way a bias statement

[4] Riga, A.T., and Collins, E.A., "Material Characterization by Thermochemical Analysis: Industrial Applications," *Materials Characterization by Thermomechanical Analysis*, ASTM STP 1136, ASTM, 1991, pp. 71-83.

[5] Yanai, H.S., Freund, W.J., and Carter, O.L., "Determination of the Deflection Temperature Under Load, Vicat Softening Temperature, and Clash-Berg T_f of Plastics by a New Method," *Thermochim. Acta 4*, 1972, pp. 199-202.

between the deflection temperature under load and the distortion temperature in three-point bending may be achieved.

chanical analysis (TMA)

14. Keywords

14.1 deflection; deflection temperature under load; heat distortion temperature; strain; stress; temperature; thermome-