



Standard Test Method for Distortion Temperature in Three-Point Bending by Thermomechanical Analysis¹

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 (Method A) and 1.82 MPa (Method B) (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—This test method is intended to provide results similar to those of Test Method D 648 but are performed on a thermomechanical analyzer using a smaller test specimen. Equivalence of results to those obtained by Test Method D 648 has been demonstrated on a limited number of materials. Until the user demonstrates equivalence, the results of this test method shall be considered to be independent and unrelated to those of Test Method D 648.

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

1.3 *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.*

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

2. Referenced Documents

2.1 ASTM Standards:²

¹ This test method is under the jurisdiction of Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.

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

D 648 Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position

E 473 Terminology Relating to Thermal Analysis and Rheology

E 1142 Terminology Relating to Thermophysical Properties

E 1363 Test Method for Temperature Calibration of Thermomechanical Analyzers

E 2113 Test Method for Length Change Calibration of Thermomechanical Analyzers

E 2206 Test Method for Force Calibration Of Thermomechanical Analyzers

3. Terminology

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

3.1.1 *distortion temperature* [$^{\circ}\text{C}$], 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 [$\text{Pa} = \text{N}/\text{m}^2$], 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 $<20 \mu\text{m m}^{-1} \cdot ^\circ\text{C}^{-1}$ 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 $<20 \mu\text{m m}^{-1} \cdot ^\circ\text{C}^{-1}$, to support the test specimen in a three-point bending mode (see Fig. 1).

6.1.3 *Rigid Knife Edge Compression Probe*, of inert, low expansivity material $<20 \mu\text{m m}^{-1} \cdot ^\circ\text{C}^{-1}$ 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 $\pm 0.1 \mu\text{m}$.

6.1.5 *Programmable Weight or Force Transducer*, to generate a constant force ($\pm 2.5 \%$) 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 $\pm 0.1^\circ\text{C}$.

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 $2 \pm 0.1^\circ\text{C}/\text{min}$.

6.1.8 *Means of Providing a Specimen Environment*, of inert gas at a purge rate of $50 \text{ mL}/\text{min} \pm 5 \%$.

NOTE 2—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 *Data Collection Device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for a thermomechanical analyzer are dimension ion change, temperature and time.

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 $\pm 0.001 \text{ mm}$.

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 3—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 4—The specimen shall be long enough to allow overhanging on each end of at least 10 % of the support span, that is, $l \geq 1.2 L$.

NOTE 5—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 $2 \pm 0.1^\circ\text{C}/\text{min}$.

9.2 Calibrate the deflection (length change) display of the apparatus according to the instrument manufacturer's instructions (see Test Method E 2113).

9.3 Calibrate the mechanism for applying force to the test specimen according to the instrument manufacturer's instructions (see Test Method E 2206).

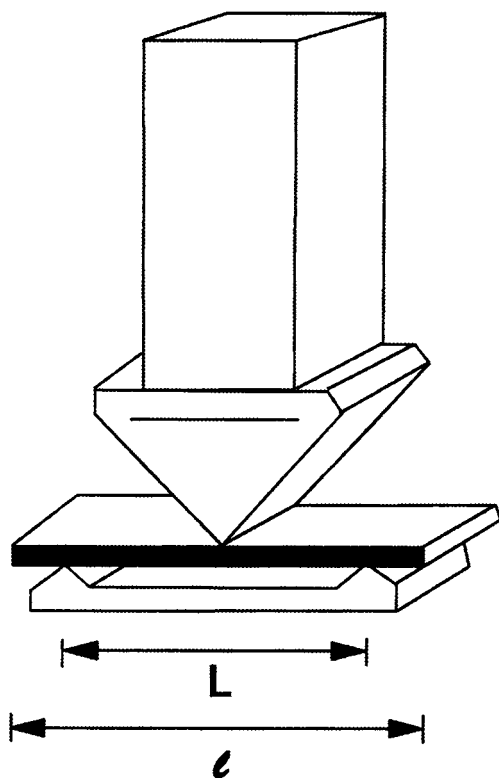


FIG. 1 Flexure Support Geometry

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 (Method A) or 1.82 MPa MPa (Method B) (66 or 264 psi) to correspond with the values used in Test Method D 648.

NOTE 6—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 7—This value is typically 2.0 mm/m (0.20 %) to correspond with the values used in Test Method D 648.

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 8—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 $2 \pm 0.1^\circ\text{C}/\text{min}$ 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 9—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 10—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 13.1 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 10.11 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:

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

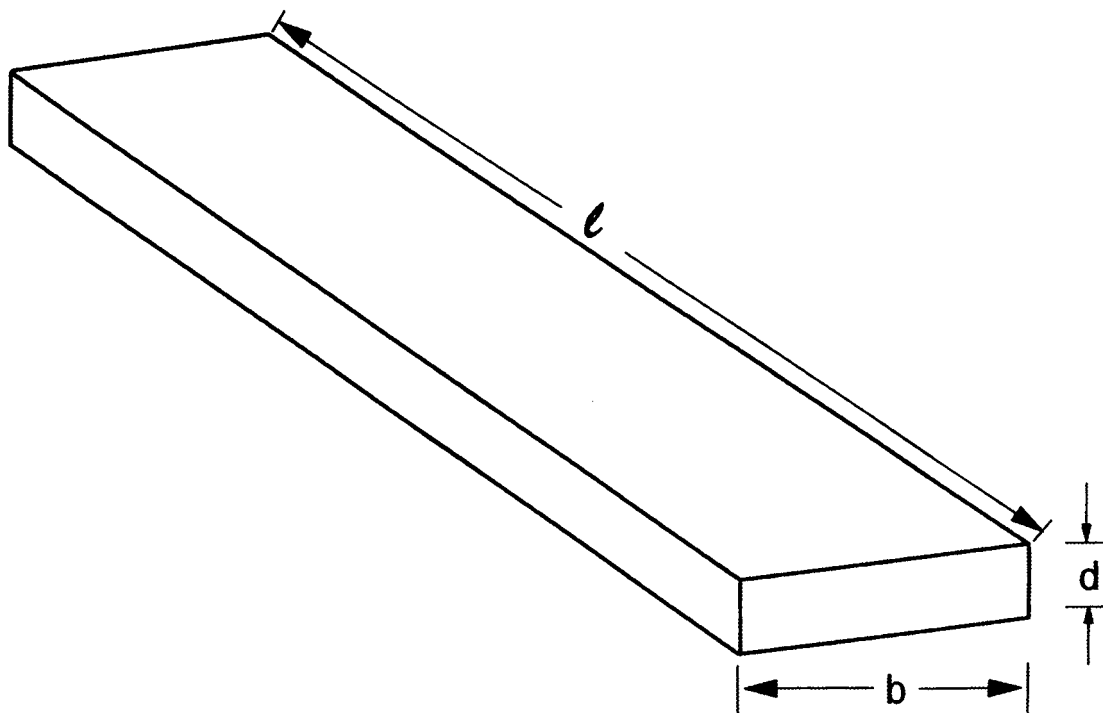


FIG. 2 Test Specimen Geometry

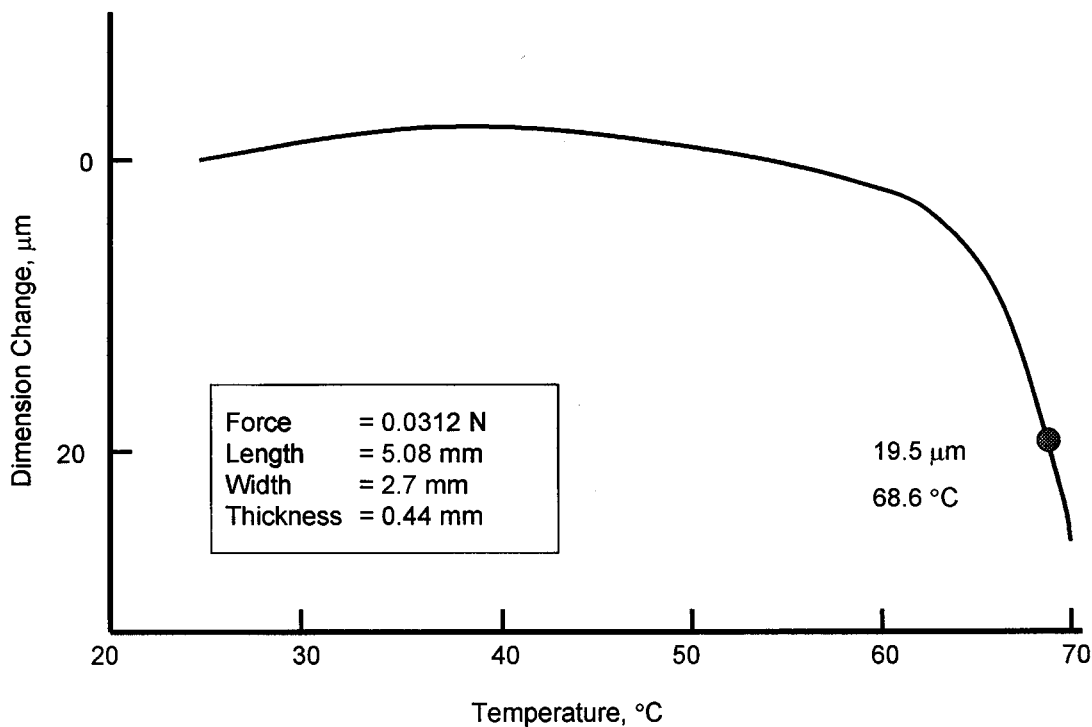


FIG. 3 Thermal Curve for Poly(vinyl chloride)

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:

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

11.2 Calculate deformation value as follows:

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

where:

r = strain, mm/m.

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

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

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 An interlaboratory test was conducted in 2003 to develop the precision and bias statement for this test method. Eight laboratories, using apparatus from two manufacturers and two stress levels, characterized a poly(vinyl chloride) sample in quntuplicate. Six laboratories produced results at a stress level of 0.455 MPa (Method A) and two laboratories at 1.82 MPa (Method B).³

13.2 Precision:

13.2.1 Within laboratory variability may be described using the repeatability value (r) obtained by multiplying the repeatability standard deviation by 2.8. The repeatability values estimates the 95 % confidence limit. That is, two results from the same laboratory should be considered suspect (at the 95 % confidence level) if they differ by more than the repeatability value.

13.2.1.1 The within laboratory repeatability standard deviation from results obtained at 0.455 MPa stress level (Method A) was found to be 0.92 °C with 20 degrees of experimental freedom.

³ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: E37-1032.

13.2.1.2 The within laboratory repeatability standard deviation for results obtained at 1.82 MPa stress level (method B) was found to be 0.84 °C with 4 degrees of experimental freedom.

NOTE 11—Twenty degrees of freedom is the minimum acceptable for a sound interlaboratory test. Insufficient laboratories were available to provide results at the 1.82 MPa stress level. Thus the information provided here for this load level is for informational purposes only.

13.2.2 A Between laboratory variability may be described using the reproducibility value (R) obtained by multiplying the reproducibility standard deviation by 2.8. The reproducibility value estimates the 95 % confidence limit. That is, results obtained in two different laboratories should be considered suspect (at the 95 % confidence level) if they differ by more than the reproducibility value.

13.2.2.1 The between laboratory reproducibility standard deviation for results obtained at 0.455 MPa stress (Method A) was found to be 3.9 °C.

13.2.2.2 The between laboratory range for results obtained at 1.82 MPa (Method B) was found to be 3.9 °C for results from 2 laboratories (see Note 10).

13.3 Bias:

13.3.1 Bias is the difference between the mean value obtained and an accepted reference value for the same material.

13.3.2 The mean value for poly(vinyl chloride) characterized at 0.455 MPa (Method A) was found to be 68.6 °C with 20 degrees of experimental freedom. The value reported (by the

test sample manufacturer) is 70 °C as measured by D 648. Therefore the bias is $(68.7 - 70) ^\circ\text{C} = 1.3 ^\circ\text{C}$.

13.3.3 The mean value for poly(vinyl chloride) characterized at 1.82 MPa (Method A) was found to be 69.0°C with 4 degrees of experimental freedom. The value reported (by the test sample manufacturer) is 67 °C as measured by Test Method D 648.

13.3.4 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.

13.3.5 A comparison was made between this test method and the DTUL measurements of Test Method D 648 using a series of 4 polyvinyl chloride samples⁵. The results derived from TMA data are 1.3°C lower than were the DTUL values.

14. Keywords

14.1 deflection; deflection temperature under load; heat distortion temperature; strain; stress; temperature; thermomechanical analysis (TMA)

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.

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