



Standard Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis¹

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1. Scope

1.1 This test method covers determination of linear thermal expansion of solid materials using thermomechanical analysis techniques. Related information can be found in Refs. (1-2)².

1.2 This test method is applicable to solid materials that exhibit sufficient rigidity over the test temperature range such that the sensing probe does not produce indentation of the specimen.

1.3 The lower limit of coefficient of linear thermal expansion measured with this test method is 5 $\mu\text{m}/(\text{m}\cdot^\circ\text{C})$. The test method may be used at lower (or negative) expansion levels with decreased accuracy and precision (see Section 11).

1.4 This test method is applicable to the temperature range from -120 to 600°C . The temperature range may be extended depending upon the instrumentation and calibration materials used.

1.5 Computer or electronic based instruments, techniques, or data treatment equivalent to this test method may also be used.

NOTE 1—Users of this test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user to determine the necessary equivalency prior to use.

1.6 SI values are the standard.

1.7 This test method is related to ISO 11359-2 but is significantly different in technical detail.

1.8 *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 determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 696 Test Method for Coefficient of Linear Thermal Expansion of Plastics Between -30°C and 30°C ³

D 3386 Test Method for Coefficient of Linear Thermal

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² The boldface numbers in parentheses refer to the list of references at the end of this standard.

³ *Annual Book of ASTM Standards*, Vol 08.01.

Expansion of Electrical Insulating Materials⁴

E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer⁵

E 473 Terminology Relating to Thermal Analysis⁵

E 1142 Terminology Relating to Thermophysical Properties⁵

E 1363 Test Method for Temperature Calibration of Thermomechanical Analyzers⁵

2.2 ISO Standards:⁶

ISO 11359-2 Plastics—Thermomechanical Analysis (TMA)—Part 2: Determination of Coefficient of Linear Thermal Expansion and Glass Transition Temperature

3. Terminology

3.1 *Definitions*—Thermal analysis terms in Terminologies E 473 and E 1142 shall apply to this test method.

4. Summary of Test Method

4.1 This test method uses a thermomechanical analyzer or similar device to determine the linear thermal expansion of solid materials when subjected to a constant heating rate.

4.2 The change of the specimen length is electronically recorded as a function of temperature. The coefficient of linear thermal expansion can be calculated from these recorded data.

5. Significance and Use

5.1 Coefficients of linear thermal expansion are used, for example, for design purposes and to determine if failure by thermal stress may occur when a solid body composed of two different materials is subjected to temperature variations.

5.2 This test method is comparable to Test Method D 3386 for testing electrical insulation materials, but it covers a more general group of solid materials and it defines test conditions more specifically. This test method uses a smaller specimen than Test Methods E 228 and D 696.

6. Apparatus

6.1 *Thermomechanical Analyzers (TMA)*—The essential instrumentation required providing minimum thermomechanical

⁴ *Annual Book of ASTM Standards*, Vol 10.02.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

⁶ Available from the American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

analytical or thermodilatometric capability for this test method includes:

6.1.1 *Rigid Specimen Holder*, of inert, low expansivity material ($\leq 0.5 \mu\text{m}/(\text{m} \cdot \text{K})$) to center the specimen in the furnace and to fix the specimen to mechanical ground.

6.1.2 *Rigid Expansion Probe*, of inert, low expansivity material ($\leq 0.5 \mu\text{m}/(\text{m} \cdot \text{K})$) that contacts the specimen with an applied compressive force.

6.1.3 *Sensing Element*, linear over a minimum range of 2 mm to measure the displacement of the rigid expansion probe to within $\pm 50 \text{ nm}$ resulting from changes in length of the specimen.

6.1.4 *Weight or Force Transducer*, to generate a constant force of 1 to 100 mN (0.1 to 10 g) that is applied through the rigid expansion probe to the specimen.

6.1.5 *Furnace*, capable of providing uniform controlled heating (cooling) of a specimen to a constant temperature or at a constant rate between 2 and $10^\circ\text{C}/\text{min}$ within the applicable temperatures range of between -120 and 600°C .

6.1.6 *Temperature Controller*, capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change of 2 to $10^\circ\text{C}/\text{min}$ constant to within $\pm 0.1^\circ\text{C}/\text{min}$ or at an isothermal temperature constant to $\pm 0.5^\circ\text{C}$.

6.1.7 *Temperature Sensor*, that can be attached to, in contact with, or reproducibly positioned in close proximity to the specimen to indicate the specimen/furnace temperature to $\pm 0.5^\circ\text{C}$.

6.1.8 A means of sustaining an environment around the specimen of inert gas at a purge gas rate of 10 to 50 mL/min.

NOTE 2—Typically, greater than 99 % pure nitrogen, argon, or helium is used when oxidation in air is a concern. Unless effects of moisture are to be studied, use of dry purge gas is recommended and is essential for operation at subambient temperatures.

6.1.9 *Recording Device*, either digital or analog, capable of recording and displaying any fraction of the specimen dimension signal (TMA curve), including signal noise, on the Y-axis versus any fraction of the temperature signal, including noise, on the X-axis.

6.2 *Cooling Capability*, to sustain a subambient specimen temperature (if subambient measurements are to be made) or to hasten cool down of the specimen from elevated temperatures.

6.3 *Micrometer*, or other length-measuring device with a range of up to 10 mm to determine specimen dimensions to within $\pm 25 \mu\text{m}$.

7. Test Specimens

7.1 Specimens shall be between 2 and 10 mm in length and have flat and parallel ends to within $\pm 25 \mu\text{m}$. Lateral dimensions shall not exceed 10 mm. Other lengths may be used, but must be noted in the report.

NOTE 3—This level of flatness and parallelness may be difficultly obtained with some materials. Specimens that do not meet these requirements may be used, but these may result in increased imprecision.

7.2 The specimens are ordinarily measured as received. Where some heat or mechanical treatment is applied to the specimen prior to test, this should be noted in the report.

NOTE 4—Some materials, particularly composites, may require heat

treatment to condition the specimen prior to test to relieve stresses or distortions. Such heat treatment must be included in the report.

8. Calibration

8.1 Calibrate the temperature and length changes signals according to the procedures in the manufacturer's operation manual.

8.2 Calibrate the temperature signal using Test Method E 1363.

8.3 The length change measuring and recording system can be calibrated by measuring the linear expansion of a material having a known expansion when heated at the same rate as the test specimens. The observed expansion must be corrected for the difference in expansion between the specimen holder and probe obtained from a blank run in which either no sample or a specimen of the material of construction of the probe is run (see 10.1).

8.4 The linear expansions of high-purity aluminum, commonly supplied by instrument manufacturers and useful as a working standard, are tabulated in Table 1 along with those for platinum.

9. Procedure

9.1 Measure the initial specimen length in the direction of the expansion test to $\pm 25 \mu\text{m}$ at 20 to 25°C .

NOTE 5—Direct readout of zero position and specimen length using the analyzer sensing element, where available, with a sufficient range has been found to be an accurate means of length determination.

9.2 Place the specimen in the specimen holder under the probe. Place the specimen temperature sensor in contact with the specimen or as near to the specimen as possible.

9.3 Move the furnace to enclose the specimen holder. If measurements at subambient temperature are to be made, cool the specimen to at least 20°C below the lowest temperature of interest. The refrigerant used for cooling shall not come into direct contact with the specimen.

9.4 Apply an appropriate load force to the sensing probe to

TABLE 1 Expansion Coefficients^A

Temperature, °C	Aluminum Mean Coefficient Of Linear Thermal Expansion, $\mu\text{m}/(\text{m}\cdot^\circ\text{C})$ (3-7)	Platinum Mean Coefficient Of Linear Thermal Expansion, $\mu\text{m}/(\text{m}\cdot^\circ\text{C})$ (8-10)
800		
700		10.75
600		10.42
550	35.3	
500	33.2	10.15
450	31.8	
400	30.3	9.92
350	29.2	
300	27.8	9.68
250	26.8	
200	26.2	9.42
150	25.5	
100	24.5	9.17
50	23.6	9.05
0	22.6	8.85
-50	20.9	8.53
-100	18.8	8.10
-150		

^AMean coefficient of linear thermal expansion values are calculated for $\pm 50^\circ\text{C}$ from the indicated temperature except in the case of platinum where values are for $\pm 100^\circ\text{C}$ of the indicated temperature for the range of between 200 and 700°C .