Designation: E 831 – 03

Standard Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis[1]

This standard is issued under the fixed designation E 831; 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 covers determination of linear thermal expansion of solid materials using thermomechanical analysis techniques. Related information can be found in Refs. **(1-12)**[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 recommended lower limit of coefficient of linear thermal expansion measured with this test method is 5 µm/(m·°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 Expansion of Electrical Insulating Materials[4]

E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer[5]

E 473 Terminology Relating to Thermal Analysis[5]

E 1142 Terminology Relating to Thermophysical Properties[5]

E 1363 Test Method for Temperature Calibration of Thermomechanical Analyzers[5]

E 2113 Test Method for Length Change Calibration of Thermomechanical Analyzers[5]

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

[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

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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.

[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.

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 analytical or thermodilatometric capability for this test method includes:

6.1.1 *Rigid Specimen Holder*, of inert, low expansivity material (≤ 0.5 µm/(m • K °C)) 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 (≤0.5 µm/(m • K °C)) 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 ± 50 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 m*N* (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°C/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°C/min constant to within ± 0.1°C/min or at an isothermal temperature constant to ± 0.5°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 ± 0.5°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*, 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 ± 25 µm.

7. Test Specimens

7.1 Specimens shall be between 2 and 10 mm in length and have flat and parallel ends to within ± 25 µm. Lateral dimensions shall not exceed 10 mm. Other lengths may be used, but shall be noted in the report.

NOTE 3—This level of flatness and parallelness may be difficult to attain with some materials. Specimens that do not meet these requirements may be used, but will 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 Calibrate the length change signal using Test Method E 2113 at the same heating rate as that to be used for 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 no sample or a specimen of the material of construction of the probe is run. (see 10.1).

9. Procedure

9.1 Measure the initial specimen length in the direction of the expansion test to ± 25 µm 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 ensure that it is in contact with the specimen. Depending on the compressibility of the specimen and the temperature range to be investigated, a force of between 1 and 100 mN (0.1 to 10 g) is adequate. The actual incremental force, mass, or stress above that required to make contact with zero force shall be noted in the report.

9.5 Select appropriate ordinate and abcissa range sensitivity settings on the recorder.

NOTE 6—Normally, the expansion increases with the increase in temperature as shown in the schematic diagram of Fig. 1. An abrupt change in slope of the expansion curve indicates a transition of the material from one state to another.

9.6 Heat the specimen at a constant heating rate of 5°C/min over the desired temperature range and record the changes in specimen length and temperature to all available decimal places. Other heating rates may be used but shall be noted in the report.

NOTE 7—For best results, specimen temperature gradients should be small. High heating rates, large specimen sizes, and low specimen thermal diffusivity may lead to large specimen temperature gradients. The effects of specimen temperature gradients may be compensated for by correction found through the use of suitable reference materials whose size and