Designation: E831 – 06

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

This standard is issued under the fixed designation E831; 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 determines the apparent coefficient 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 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 900 °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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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:*[3]

D696 Test Method for Coefficient of Linear Thermal Expansion of Plastics Between -30°C and 30°C with a Vitreous Silica Dilatometer

D3386 Test Method for Coefficient of Linear Thermal Expansion of Electrical Insulating Materials[4]

E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer

E473 Terminology Relating to Thermal Analysis and Rheology

E1142 Terminology Relating to Thermophysical Properties

E1363 Test Method for Temperature Calibration of Thermomechanical Analyzers

E2113 Test Method for Length Change Calibration of Thermomechanical Analyzers

2.2 *ISO Standards:*[5]

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 E473 and E1142 shall apply to this test method.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *apparent coefficient of linear thermal expansion, ($\alpha_m$)*—the change in length, relative to the specimen length at ambient temperature, accompanying a unit change in temperature identified by the midpoint temperature of the temperature range of measurement

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

Current edition approved March 15, 2006. Published April 2006. Originally approved in 1981. Last previous edition approved in 2005 as E831 – 05. DOI: 10.1520/E0831-06.

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

[3] 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.

[4] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

## 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 D3386 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 and substantially different apparatus than Test Methods E228 and D696.

## 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 • °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 • °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 –150 and 1000 °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 capable of indicating 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—It has been found with some materials that this level of flatness and parallelness cannot be attained. Specimens that do not meet these requirements 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 Prepare the instrument for operation according to the procedures in the manufacturer's operation manual.

8.2 Calibrate the temperature signal using Test Method E1363.

8.3 Calibrate the length change signal using Test Method E2113 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 graphical representation.

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 conductivity 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 thermal conductivity are close to that of the test specimen.

NOTE 8—Intralaboratory testing indicates that no detectable increase in imprecision is observed for specimen sizes from 2 to 10 mm in length, for heating rates from 2 to 10 °C/min, and for thermal conductivities from 0.2 to 400 W/(m·°C) if only one parameter is changed.

9.7 Measure the measurement instrument baseline by repeating 9.3 through 9.6 using the same test parameters but without a test specimen, that is, with the probe in contact with the specimen holder. The measured $\Delta L$ for the specimen should normally be corrected for this instrument baseline, especially for low expansion specimens.

9.8 Test at least three specimens of the same material. Retest of a specimen may be used only as reference and shall not be treated as an independent test of a new specimen.

## 10. Calculation

10.1 Calculate the mean coefficient of linear thermal expansion rounded to the nearest 0.1 µm/(m·°C) for a desired temperature range as follows:

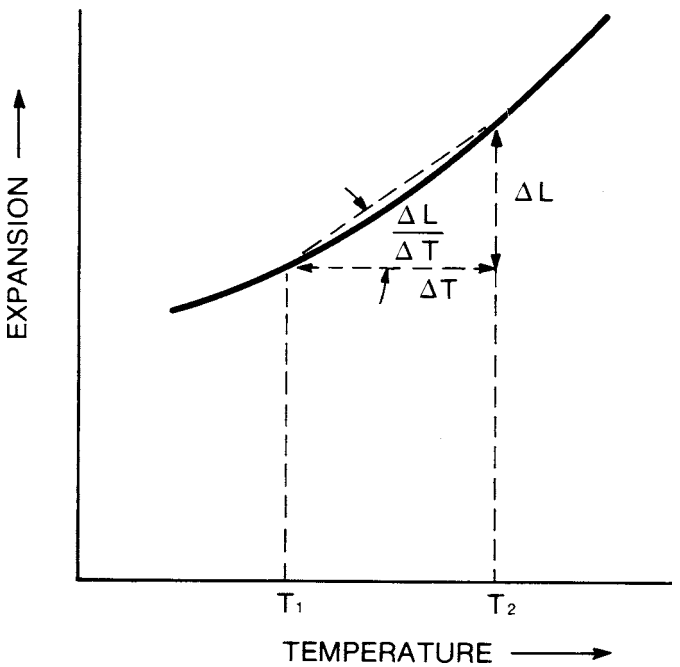


**FIG. 1 Specimen Expansion Versus Temperature**

$$\alpha_m = \frac{\Delta L_{sp} \times k}{L \times \Delta T} \quad (1)$$

$$k = \frac{\alpha_{ref} \times L_{ref} \times \Delta T_{ref}}{\Delta L_{ref}} \quad (2)$$

where:

$\alpha_{m-m}(T)$ = mean coefficient of linear thermal expansion,µm/(m·°C),
$\alpha_{ref}$ = mean coefficient of linear thermal expansion, for reference material, µm/(m·°C) (see 8.3),
$k$ = calibration coefficient, from Test Method E2113,
$L$ = specimen length at room temperature, m,
$\Delta L_{ref}$ = change of reference material length due to heating, µm,
$L_{ref}$ = reference material length at room temperature, m,
$\Delta L_{sp}$ = change of specimen length, µm,
$\Delta T_{ref}$ = temperature difference over which the change in reference material length is measured,° C, typically 100 °C,
$\Delta T$ = temperature difference over which the change in specimen length is measured, °C,
$T$ = midpoint temperature of the temperature range ΔT, and

NOTE 9—For low-expansion specimens, $\Delta L$ should be corrected for the expansion of the sample holder displaced by the specimen.

$$\Delta L_{sp} \ (or\ \Delta L_{ref}) = \Delta L_{obs} + L\,\Delta T\,\alpha_{holder} - \Delta L_{blank} \quad (3)$$

where:

$\Delta L_{obs}$ = measured change in length, µm,
$\alpha_{holder}$ = mean coefficient of linear thermal expansion for holder, µm/(m·°C), and
$\Delta L_{blank}$ = change of baseline due to heating, µm.

If the holder is composed of vitreous silica, $\alpha_{holder} = 0.52 \pm 0.02$ µm/(m·°C) from 20 to 700 °C.

10.2 Select a $\Delta T$ from a smooth portion of the thermal curves in the desired temperature range symmetically around the temperature of interest; then obtain $\Delta L$ as depicted in Fig. 1. The $\alpha_m$ shall not be calculated from a temperature range in which a transition point is noted.

NOTE 10—Values for $\Delta T$ generally range between 50 and 100 °C. Values less than 50 °C may lead to poor precision; values greater than 100 °C may mask small change in the expansion coefficient.

## 11. Report

11.1 The report shall include the following:

11.1.1 Designation of the material, including the name of the manufacturer and information on lot number and chemical composition when known,

11.1.2 Specimen orientation with respect to the original part or the direction of the oriented fiber fillers if a composite material is used,

11.1.3 Method of test specimen preparation,

11.1.4 Dimensions of the specimen,

11.1.5 Description of the thermomechanical analysis apparatus, including manufacturer and model number,

11.1.6 Purge gas and cooling medium, if used,

11.1.7 The midpoint temperature (T) and the temperature range (ΔT) at which the coefficient of linear thermal expansion has been determined,

11.1.8 Average value of the coefficient of linear thermal expansion inμ m/(m·°C) as determined from three specimens,

11.1.9 Expansion curves obtained, and

11.1.10 The specific dated version of this test method used.

## 12. Precision and Bias [6]

12.1 Precision of thermal expansion measurements depends on the length of the specimen, the temperature range of interest, and the change in the specimen length. Maximum imprecision in the calculated coefficient of linear thermal expansion may be estimated from the imprecisions in the individual measurements by the following equation.

$$\delta_\alpha/\alpha_m = [(\delta\Delta L/\pm\Delta L)^2 + (\delta L/L)^2 + (\delta\Delta T/\Delta T)^2]^{1/2} \quad (4)$$

where:

$\alpha_m$ = mean coefficient of linear thermal expansion, μm/(m·°C),
$\delta_\alpha$ = imprecision in the measurement of α, μm/(m·°C),
$\Delta L$ = change of specimen length due to heating, μm,
$\delta\Delta L$ = imprecision in the measurement of ΔL, μm,
$L$ = specimen length at room temperature, m,
$\delta L$ = imprecision in the measurement of L, m,
$\Delta T$ = temperature difference over which the change in specimen length is measured, °C, and
$\delta\Delta T$ = imprecision in the measurement of ΔT, °C.

12.1.1 *Example:*

$$L = 8 \text{ mm} \quad (5)$$

$$\delta L = \pm 25 \text{ μm} \quad (6)$$

$$\Delta L = 60 \text{ μm} \quad (7)$$

$$\delta\Delta L = \pm 1 \text{ μm} \quad (8)$$

$$\Delta T = 100 \text{ °C} \quad (9)$$

$$\delta\Delta T = \pm 0.5 \text{ °C} \quad (10)$$

$$\delta_\alpha/\alpha_m = [(1 \text{ μm}/60 \text{ μm})^2 + (25 \times 10^{-6}\text{m}/8 \times 10^{-3}\text{m})^2 + (0.5 \text{ °C}/100 \text{ °C})^2]^{1/2} \quad (11)$$

or expressed as percent:

$$\delta_\alpha/\alpha_m = \pm 1.8\% \quad (12)$$

12.1.2 Intralaboratory precision measurements confirm the relationship above.

12.2 Interlaboratory precision of this method for coefficient of thermal expansion was determined from the results of a round robin in which eight laboratories using six instrument models participated.

12.2.1 For materials with α > 20 μm/(m·°C), interlaboratory precision on specimens 8 mm in length, measured over a 100°C temperature range, was ±2.6 % with a maximum deviation of 3.7 %.

12.2.2 For materials with 20 > α > 5 μm/(m·°C), interlaboratory precision on specimens 8 mm in length, measured over a 100°C temperature range, was ±5.1 % with a maximum deviation of 7.8 %.

12.2.3 For materials with 5 > α > 1 μm/(m·°C), interlaboratory precision on specimens 8 mm in length, measured over a 100°C temperature range, was ±12 % with a maximum deviation of 61 %.

12.2.4 For materials with α > 5 μm/(m·°C), intralaboratory precision on specimens 8 mm in length, measured over a 100°C temperature range, was ±1.8 % with a maximum deviation of 5.8 %.

12.3 The following criteria should be used for judging the acceptability of results:

12.3.1 (Single Instrument)—The standard deviation of results obtained by the same instrument and laboratory has been estimated to be 0.97 μm/(m·°C) at 32 degrees of freedom. Two results should be considered suspect (95 % confidence level) if they differ by more than 2.7 μm/(m·°C).

12.3.2 (Multi-Instrument)—The standard deviation of results (each the average of duplicates) obtained by different instruments or laboratories, has been estimated to be 1.1 μm/(m·°C) at 4 degrees of freedom. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 3.1 μm/(m·°C).

12.4 Accuracy is anticipated, from calculations, to be a function of specimen size and magnitude of the coefficient of thermal expansion varying from <±2 % for α > 20 μm/(m·°C) to >±15 % for α < 5 μm/(m·°C).

12.4.1 An estimation of the accuracy of the coefficient of thermal expansion measurement was obtained by comparing the mean of triplicate determinations performed in a single laboratory with values reported in the literature. Based on this comparison, the accuracy of the measurement is estimated to be + 5.4 %.

| Material | Testing | Coefficient of Expansion, μm/(m·°C), at 50 °C Literature |
|---|---|---|
| Copper | 17.8± 0.9 | 16.91 |
| Lead | 30.9 ± 1.3 | 29.33 |

## 13. Keywords

13.1 linear expansion; solid materials; thermal expansion coefficient; thermomechanical analysis

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1000.

## REFERENCES

(1) Gaskill, R., and Barrall, E. M., *Thermochimica Acta*, Vol 12, 1975, p. 102.

(2) Barton, J. M., *Thermochimica Acta*, Vol 29, 1979, p. 188.