Designation: ~~E831 – 19~~ E831 – 24

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.

1. ~~Scope~~ Scope*

1.1 This test method determines the technical coefficient of linear thermal expansion of solid materials using thermomechanical analysis techniques.

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~~12).

1.4 This test method is applicable to the temperature range from −120 °C to 900 °C. The temperature range may be extended depending upon the instrumentation and calibration materials used.

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

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

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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D3386 Test Method for Coefficient of Linear Thermal Expansion of Electrical Insulating Materials (Withdrawn 2005)[3]

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

E473 Terminology Relating to Thermal Analysis and Rheology

[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.

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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.

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

***A Summary of Changes section appears at the end of this standard**

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
E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

3. Terminology

3.1 *Definitions*—Thermal analysis terms in Terminologies E473 and E1142 shall apply to this test method, including *coefficient of linear thermal expansion, thermodilatometry*, and *thermomechanical analysis*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *mean coefficient of linear thermal expansion, (α_m), n*—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.

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.

5.3 This test method may be used in research, specification acceptance, regulatory compliance, and quality assurance.

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 coefficient of expansion 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 coefficient of expansion 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~~ 2 mm to measure the displacement of the rigid expansion probe readable 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 mN to 100 mN (0.1 g 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 °C/min and 10 °C/min within the applicable temperatures range of between –150 °C and ~~1000 °C.~~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 °C/min 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~~ readable 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 mL/min to ~~50~~ 50 mL ~~mL/min./~~min.

NOTE 1—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 *Data Collection Device,* to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for thermomechanical analysis are a change in linear dimension, temperature and time.

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.~~mm, readable to ±25 µm, to determine specimen dimensions.

7. Test Specimens

7.1 Specimens shall be between 2 mm and 10 mm in length and have flat and parallel ends to within ±25 µm. Lateral dimensions shall not exceed 10 mm.

NOTE 2—Specimens of other dimensions may be used but dimensions shall be reported.

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~~.~~ at the same heating rate as that used for the test specimen (see section 9.5). (See Appendix X1).

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).

NOTE 5—Calibration or calibration verification of all signals is recommended at last annually.

9. Procedure

9.1 Measure the initial specimen length in the direction of the expansion test to ±25 µm at 20 °C to 25 °C.

NOTE 6—Direct readout of zero position and specimen length (*L*) 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 mN and 100 mN (0.1 g 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 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.

NOTE 7—Other heating rates may be used but shall be noted in the ~~report.~~report (see Appendix X1).

NOTE 8—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.

NOTE 9—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.

9.6 Measure the measurement instrument baseline by repeating 9.3 through 9.5 using the same test parameters but without a test specimen, that is, with the probe in contact with the specimen holder. The measured *ΔL* for the specimen should normally be corrected for this instrument baseline, especially for low expansion specimens.

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:

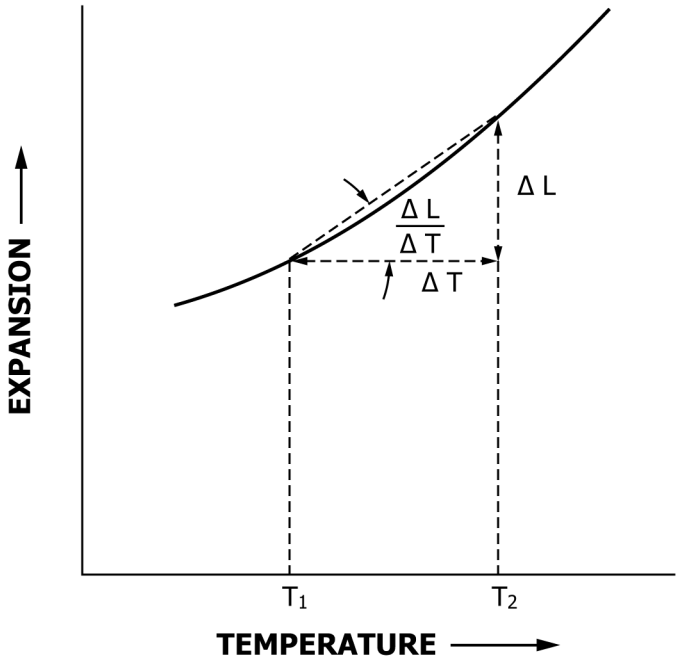

FIG. 1 Specimen Expansion Versus Temperature