Designation: E 2113 – 04

Standard Test Method for Length Change Calibration of Thermomechanical Analyzers[1]

This standard is issued under the fixed designation E 2113; 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 method describes calibration of the length change (deflection) measurement or thermal expansion of thermomechanical analyzers (TMA) within the temperature range from -150 to 1000 °C using the thermal expansion of a suitable reference material.

1.2 SI values are the standard.

1.3 This method differs from ISO standard 11359-1 by providing an alternative calibration procedure.

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

2. Referenced Documents

2.1 *ASTM Standards:*

E 473 Terminology Relating to Thermal Analysis[2]

E 831 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis[2]

E 1142 Terminology Relating to Thermophysical Properties[2]

E 1363 Test Method for Temperature Calibration of Thermomechanical Analyzers[2]

2.2 *Other Standards:*

ISO 11359-1 Plastics—Thermomechanical analysis (TMA)—Part 1: General principles[3]

3. Terminology

3.1 Specific technical terms used in this method are described in Terminologies E 473 and E 1142.

4. Summary of Test Method

4.1 Thermomechanical analyzers (TMAs) or related devices are commonly used to determine coefficient of linear thermal expansion of solid materials (e.g., Test Method E 831). The test specimen is heated at a linear rate over the temperature range of interest and the change in length (dimension) is electronically recorded.

4.2 Performance verification or calibration of the length change measurement is needed to obtain accurate coefficient of thermal expansion data.

4.3 The thermal expansion of a reference material is recorded using a thermomechanical analyzer. The recorded thermal expansion is compared to the known value of the reference material. The resultant ratio, a calibration coefficient, may then be applied to the determination of unknown specimens to obtain accurate results.

5. Significance and Use

5.1 Performance verification or calibration is essential to the accurate determination of quantitative dimension change measurements.

5.2 This method may be used for instrument performance validation, regulatory compliance, research and development and quality assurance purposes.

6. Apparatus

6.1 *Thermomechanical Analyzer (TMA)*— The essential instrumentation required to provide the minimum thermomechanical analytical or thermodilatometric capability for this method includes:

6.1.1 A rigid specimen holder of inert, low expansivity material [<0.5 μm m^{-1} K^{-1}] to center the specimen in the furnace and to fix the specimen to mechanical ground.

6.1.2 A rigid expansion probe of inert, low expansivity material [<0.5 μm $m^{-1}K^{-1}$] which contacts the specimen with an applicable compressive or tensile force.

6.1.3 A sensing element, linear over a minimum of 2 mm, to measure the displacement of the rigid probe to within ± 10 nm resulting from changes in length/height of the specimen.

[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] Available from American National Standards Institute, 11 W 42nd Street, 13th Floor, New York, NY 10036.

6.1.4 A weight or force transducer to generate a constant force between 1 and 100 mN (0.1 and 10 g) applied through the rigid probe to the specimen.

6.1.5 A furnace capable of providing uniform controlled heating (cooling) of a specimen to a constant temperature or at a constant rate within the applicable temperature range of this method.

6.1.6 A temperature controller capable of executing a specific temperature program by operating the furnace over any suitable temperature range between -150 and 1000 °C at a rate of temperature change of 5 K/min constant to within ± 0.1 K/min.

6.1.7 A temperature sensor that can be attached to, in contact with, or reproducibly positioned in close proximity to the specimen to provide an indication of the specimen/furnace temperature to within ± 0.1 K.

6.1.8 A means of sustaining an environment around the specimen of an inert purge gas at a rate of 10 to 50 ± 5 mL/min.

NOTE 1—Typically, 99.9+% pure nitrogen, helium or argon is employed, 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 A recording device, capable of recording and displaying any fraction of the specimen dimension change signal (TMA curve) including the signal noise on the ordinate (Y-axis) versus temperature on the abscissa (X-axis).

6.2 Micrometer, calipers or other length measurement device capable of measuring linear dimensions up to 10 mm with readability of ± 25 µm.

6.3 While not required, the user may find useful software that performs the calculations described in this method.

6.4 Thermal expansion reference material of 8 ± 2 mm length, the linear coefficient of expansion of which is known to ± 0.1 µm m^{-1} K^{-1}. The coefficient of thermal expansion should be between 9 and 40 µm m^{-1} K^{-1}.

6.4.1 Reference materials of known value traceable to a National Reference laboratory are available from a number of suppliers. Contact ASTM Headquarters for list of such potential suppliers[4].

6.4.2 In the absence of primary or secondary reference materials, high purity aluminum or platinum may be used along with the values for coefficient of thermal expansion presented in Table 1.

NOTE 2—The linear expansion of high purity aluminum, commonly supplied by instrument manufactures, is useful as a working reference material. Coefficient of thermal expansion values for pure aluminum are presented in Table 1 along with those for platinum.

7. Test Specimen

7.1 Specimens shall be between 6 and 10 mm in length and have flat and parallel ends to within ± 25 µm. Lateral

[4] A Research Report is available from ASTM Headquarters. Request RR:E37–1033.

TABLE 1 Thermal Expansion Coefficients[A]

Temperature, °C	Aluminum [BCDEF]	Platinum [GHIJ]
	Mean Coefficient of Linear Thermal Expansion, µm/(m · °C)	Mean Coefficient of Linear Thermal Expansion, µm/(m · °C)
1100		12.33
1000		11.87
900		11.26
800		11.08
700		10.75
600		10.45
550	35.3	10.31
500	33.2	10.18
450	31.8	10.05
400	30.5	9.92
350	29.2	9.80
300	27.8	9.67
250	26.8	9.64
200	26.2	9.45
150	25.5	9.38
100	24.5	9.18
50	23.6	9.01
0	22.6	8.85
–50	20.9	8.59
–100	18.8	8.19
–150		7.37

[A] Mean coefficient of linear thermal expansion values are calculated for ± 50 °C from the indicated temperature except in the case of platinum where values are for ± 100 °C of the indicated temperature for the range of 200 to 700 °C.
[B] Nix, F. C., and MacNair D., *Physical Review*, Vol 60, 1941, p. 597.
[C] Simmons, R. O., and Balluffi R. W., *Physical Review*, Vol 117, 1960, p. 52.
[D] Fraser, D. B., and Hollis Hallet, A. C., *7th International Conference on Low-Temperature Physics*, 1961, p. 689.
[E] Altman, H. W., Rubin, T., and Johnson, H. L., Ohio State University, Cryogenic Laboratory Report OSU-TR-264–27 (1954) AD 26970.
[F] Hidnert, P., and Krider, H. S., *Journal of Research National Bureau of Standards*, Vol 48, 1952, p. 209.
[G] Nix, F. C., and MacNair, D., *Physical Review*, Vol 61, 1942, p. 74.
[H] White, G. K., *Journal of Physics*, Vol 2F, 1972, p. 130.
[I] Hahn, T. A., and Kirby, R. K., *AIP Conference Proceedings*, No. 3, Vol 87, 1972.
[J] Kirby, R. K., *Thermal Conductivity 24/Thermal Expansion 12,*, Technomic Publishing, Lancaster, PA 1997, pp. 655–661.