Designation: E 2113 – 02

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) over the temperature range from -100 to 600°C using the thermal expansion of a suitable reference material.

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

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

1.3 SI values are the standard.

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

1.5 *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., 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.

[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 Thermal.

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[2] *Annual Book of ASTM Standards,* Vol 14.02.

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

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.

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 between -100 and 600°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 2—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, either digital or analog, 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.

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 3—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 dimensions shall be between 3 and 9 mm. Other lengths and widths may be used but shall be noted in the report.

8. Calibration

8.1 Perform any calibration procedures described in the manufacturer's operations manual.

TABLE 1 Thermal Expansion Coefficients[A]

Temperature, °C	Aluminum [BCDEF]		Platinum [GHI]	
	Mean Coefficient of Linear Thermal Expansion, µm/(m · °C)	Linear Thermal Expansion, µm/m	Mean Coefficient of Linear Thermal Expansion, µm/(m · °C)	Linear Thermal Expansion, µm/m
800				7770
700			10.75	6680
600		16760	10.42	5620
550	35.3	14930		
500	33.2	13230	10.15	4595
450	31.8	11610		
400	30.5	10050	9.92	3590
350	29.2	8560		
300	27.8	7130	9.68	2610
250	26.8	5780		
200	26.2	4450	9.42	1655
150	25.5	3160		
100	24.5	1900	9.17	725
50	23.6	710	9.05	270
0	22.6	–460	8.85	–180
–50	20.9	–1550	8.53	–615
–100	18.8	–2550	8.10	–1035
–150		–3430		–1425

[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.

8.2 Calibrate the temperature sensor using Method E 1363.

9. Procedure

9.1 Measure the initial specimen length in the direction of the expansion test to within ± 25 µm at 23 ± 2°C.

9.2 Place the specimen on the specimen holder under the probe. Place the specimen temperature sensor within 2 mm but not touching the test specimen.

9.3 Move the furnace to enclose the specimen holder. If measurements at subambient temperatures are to be made, cool the test 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. A force between 1 and 50 mN (0.1 and 5 g) is adequate. The actual incremental force, mass or stress above that required to make contact with the zero force shall be noted in the report.

9.5 Heat the specimen at a rate of 5 ± 0.1°C/min over the desired temperature range and record the change in specimen length and temperature to all available decimal places. Other heating rates may be used but shall be noted in the report.

NOTE 4—For best results, specimen temperature gradients should be small. High heating rates, large specimen size and low specimen thermal conductivity may lead to large specimen temperature gradients.

NOTE 5—Intralaboratory testing indicates that no detectable increase in imprecision is observed for specimens size 2 to 20 mm in length, for heating rates between 2 and 10°C min^{-1} and for thermal conductivities between 0.2 and 400 Wm^{-1} K^{1}, if only one parameter is changed.

9.6 Determine the measurement instrument baseline by repeating steps 9.2-9.5 using the same test parameters but without a test specimen. The measured change of length (ΔL) of the specimen should normally be corrected by curve subtraction for this baseline especially for low expansion materials.

9.7 Select a ΔT from a smooth portion of the thermal curve in the desired temperature range. Then obtain the ΔL for this temperature range as depicted in Fig. 1.

9.8 Record the change in length (ΔL) for the test specimen over a corresponding change in temperature (ΔT). See Fig. 1.

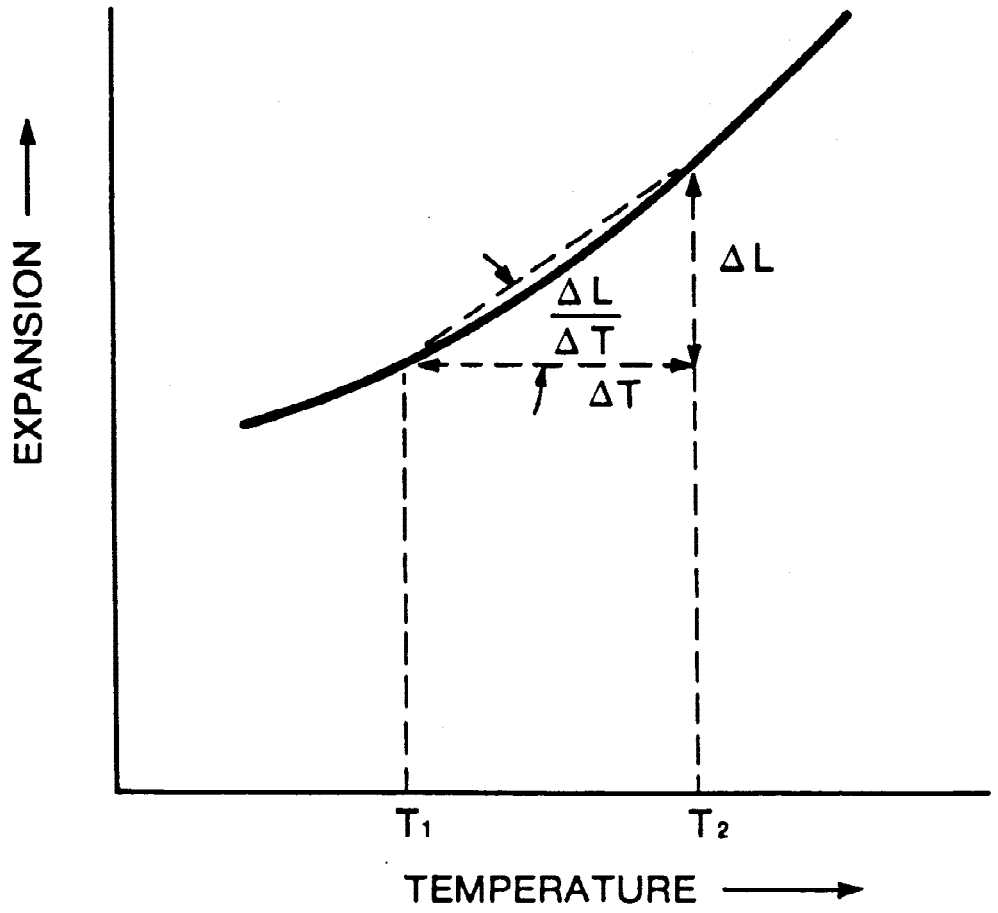

FIG. 1 Specimen Expansion Versus Temperature

FIG. 1 Specimen Expansion Versus Temperature

NOTE 6—For the best calibration results, values for ΔT should range between 50 and 100°C.

10. Calculation

10.1 Calculate the mean coefficient of linear thermal expansion for the desired temperature range retaining all available significant figures.

$$k = \alpha \cdot L \cdot \Delta T / \Delta L \quad (1)$$

where:

- α = mean coefficient of linear thermal expansion for the reference material at the midpoint of the ΔT range, in μm $m^{-1}°C^{-1}$.
- k = calibration coefficient, dimensionless
- L = length of the reference material at room temperature, in m
- ΔL = change in length of the reference material due to heating, in μm
- ΔT = temperature difference over which the change in specimen length is measured, in °C.

NOTE 7—The mean coefficient of linear thermal expansion described here is an approximation to the traditional coefficient of linear thermal expansion where the reference length is taken at the test temperature of interest. This approximation creates a bias on the order of about 0.015%.

10.2 The true length change (ΔL_t) of an unknown may be derived by multiplication of the observed length change (ΔL_o) by the calculation coefficient (k).

$$\Delta L_t = k \cdot \Delta L_o \quad (2)$$

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11. Report

11.1 Designation of the Reference Material for coefficient of linear thermal expansion including its source, lot number and chemical composition.

11.2 Dimensions of the specimen and any physical, mechanical or thermal pre-treatment and orientation with respect to the original part (if cut to size).

11.3 Designation of the thermomechanical analyzer by model number, serial number and specimen holder stage and probe type.

11.4 Experimental conditions including temperature range of test, heating rate, purge gas type and flow rate.

11.5 The calibration coefficient value determined the midpoint of the temperature range of calibration. For example: k = 1.001 at 100°C.

11.6 The specific dated version of this method used.

12. Precision and Bias

12.1 *Precision*

12.1.1 Precision of the calibration constant value may be estimated by the propagation of uncertainties method from estimates of the precision of the respective components of the calculation using:

$$\frac{\delta k}{k} = \left[\left(\frac{\delta \Delta L}{\Delta L}\right)^2 + \left(\frac{\delta L}{L}\right)^2 + \left(\frac{\delta \Delta T}{\Delta T}\right)^2\right]^{\frac{1}{2}} \quad (3)$$

where:

- k = calibration coefficient, dimensionless
- δk = imprecision in the measurement of k, dimensionless
- L = length of the reference material, in mm
- δL = imprecision in the measurement of L, in mm
- ΔL = change in specimen length due to heating, in μm
- $\delta \Delta L$ = imprecision of the measurement of L, in μm
- ΔT = temperature difference over which the change in specimen length is measured, in °C
- $\delta \Delta T$ = imprecision of the measurement of ΔT, in °C.

12.1.2 For example, if:

- L = 8.00 mm
- δL = 25 μm = 0.025 mm
- ΔL = 18.8 μm
- $\delta \Delta L$ = 0.10 μm
- ΔT = 100.0°C
- $\delta \Delta T$ = 0.10°C

$$\frac{\delta k}{k} = \left[\left(\frac{0.10\ \mu m}{18.8\ \mu m}\right)^2 + \left(\frac{0.025\ mm}{8.00\ mm}\right)^2 + \left(\frac{0.10\ °C}{100.0\ °C}\right)^2\right]^{\frac{1}{2}} \quad (4)$$

$$\delta k/k = [(0.005319)^2 + (0.003125)^2 + (0.001000)^2]^{\frac{1}{2}} \quad (5)$$

$$\delta k/k = (0.00002829) + (0.000009766) + (0.000001000)]^{\frac{1}{2}}$$
$$= [0.000039056]^{\frac{1}{2}} \quad (6)$$

$$\delta k/k = 0.006249 \quad (7)$$

or expressed as percent

$$\delta k/k = 0.6249\ \% \quad (8)$$

12.1.3 Intralaboratory precision measurements confirm the relationship in section 12.1.1.

12.1.4 An interlaboratory test involving eight laboratories and six instrument models was conducted in 1985. (Supporting data available from ASTM Headquarters. Request RR:E37-1000.) An aluminum calibration material, 8.0 mm in length was tested over a 100°C temperature range.

12.1.5 *Repeatability* —The standard deviation of results obtained by a single instrument and laboratory was ± 2.6%. Two results, each the mean value of duplicate determinations, should be considered suspect (95% confidence limit) if they differ by more than 7.3%.

12.1.6 Since the determination of the calibration coefficient is specific to a single instrument or laboratory, interlaboratory reproducibility has no meaning and is not reported.

12.2 *Bias*

12.2.1 The calibration constant determined by this method (i.e., its difference from unity) is, in itself, an estimation of bias. No further estimation is necessary.

13. Keywords

13.1 calibration; coefficient of thermal expansion; deflection; expansion; expansivity; thermal analysis; thermal expansion; thermomechanical analyzer (TMA)