Designation: E2113 – 23

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

This standard is issued under the fixed designation E2113; 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 describes calibration of the length change (deflection) measurement or thermal expansion of thermomechanical analyzers (TMAs) within the temperature range from –150 °C to 1000 °C using the thermal expansion of a suitable reference material.

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

1.3 *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.*

1.4 *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]

E473 Terminology Relating to Thermal Analysis and Rheology

E831 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis

E1142 Terminology Relating to Thermophysical Properties

E1363 Test Method for Temperature Calibration of Thermomechanical Analyzers

E2161 Terminology Relating to Performance Validation in Thermal Analysis and Rheology

E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

3. Terminology

3.1 Specific technical terms used in this test method are described in Terminologies E473, E1142, and E2161 including *calibration, Celsius, coefficient of linear thermal expansion, Kelvin, reference material, repeatability, reproducibility,* and *thermomechanical analysis*.

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 (for example, Test Method E831). 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 test 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 test method includes:

6.1.1 *A Rigid Specimen Holder,* of inert, low expansivity material [<0.5 µm m^{-1} $°C^{-1}$] 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} $°C^{-1}$] 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 E37.10 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.

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

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 mN and 100 mN (0.1 g 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 test 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 °C and 1000 °C at a rate of temperature change of 5 °C/min constant to within ±0.1 °C/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 readable to within ±0.1 °C.

6.1.8 A means of sustaining an environment around the specimen of an inert purge gas at a rate of 10 mL/min to 50 mL/min ± 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 Data Collection Device,* to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required are dimension (length) change, temperature, and time.

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 test method.

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

6.4.1 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.

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 °C to 700 °C.

[B] Nix, F. C., and MacNair, D., "The Thermal Expansion of Pure Metals: Copper, Gold, Aluminum, Nickel, and Iron," *Physical Review*, Vol 60, 1941, pp. 597–605.

[C] Simmons, R. O., and Balluffi, R. W., "Measurements of Equilibrium Vacancy Concentrations in Aluminum," *Physical Review*, Vol 117, 1960, pp. 52–31.

[D] Fraser, D. B., and Hollis Hallet, A. C., "The Coefficient of Linear Expansion and Gruneisen γ of Cu, Ag, Au, Fe, Ni, and Al from 4°K to 300°K," *Proceedings of the 7th International Conference on Low-Temperature Physics*, 1961, pp. 689–692.

[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., "Thermal Expansion of Aluminum and Some Aluminum Alloys," *Journal of Research National Bureau of Standards*, Vol 48, 1952, pp. 209–220.

[G] Nix, F. C., and MacNair, D., "The Thermal Expansion of Pure metals. II: Molybdenum, Palladium, Silver, Tantalum, Tungsten, Platinum, and Lead," *Physical Review*, Vol 61, 1942, pp. 74–78.

[H] White, G. K., "Thermal Expansion of Platinum at Low Temperature," *Journal of Physics*, Vol 2F, 1972, pp. L30–L31.

[I] Hahn, T. A., and Kirby, R. K., "Thermal Expansion of Platinum from 293 to 1900 K," *American Institute of Physics Conference Proceedings*, 3, 1972, pp. 87–95.

[J] Kirby, R. K., "Platinum – A Thermal Expansion Reference Material," *Thermal Conductivity 24/Thermal Expansion 12*, Technomic Publishing, Lancaster, PA 1997, pp. 655–661.

7. Test Specimen

7.1 Specimens shall be between 6 mm and 10 mm in length and have flat and parallel ends to within ±25 µm. Lateral dimensions shall be between 3 mm 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.

8.2 Calibrate the temperature sensor using Test Method E1363 at the same heating rate to be used for the test specimen (see Test Method E3142 and Appendix X1).

NOTE 3—Committee E37 recommends calibration, or calibration verification, of all signals at least annually.

9. Procedure

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

9.2 Place the specimen on the specimen holder under the probe and adjust so that no load is applied. 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.

NOTE 4—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 mN and 50 mN (0.1 g 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 °C/min ± 0.1 °C/min over the desired temperature range and record the change in specimen length and temperature to all available decimal places.

NOTE 5—Other heating rates may be used but shall be noted in the report.

NOTE 6—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.

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 (that is, the probe is placed on the empty specimen holder) especially for low expansion materials.

9.7 Select a temperature change range (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.

NOTE 7—For the best calibration results, values for ΔT should range between 50 °C and 100 °C.

10. Calculation

10.1 Calculate the mean coefficient of linear thermal expansion for the desired temperature range and calibration coefficient 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, and
ΔT = temperature difference over which the change in specimen length is measured, in °C.

NOTE 8—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).

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

11. Report

11.1 A complete description of the Reference Material used including dimensions of the specimen and any physical, mechanical or thermal pre-treatment and orientation with respect to the original part (if cut to size).

11.2 A complete description of the thermomechanical analyzer being calibrated.

11.3 A complete description of the experimental conditions including temperature range of test, heating rate, purge gas type and flow rate.

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

11.5 The specific dated version of this test 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:

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