~~Designation:E2113–04~~ Designation: E 2113 – 09

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~~

1.1 This test 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 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 test 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:*[2]
E 473 Terminology Relating to Thermal Analysis and Rheology
E 831 Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis
E 1142 Terminology Relating to Thermophysical Properties
E 1363 ~~Test Method for Temperature Calibration of Thermomechanical Analyzers[2]~~ Test Method for Temperature Calibration of Thermomechanical Analyzers
E 2161 Terminology Relating to Performance Validation in Thermal Analysis
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 E473 and E1142.~~

3.1 Specific technical terms used in this test method are described in Terminologies E 473, E 1142, and E 2161 include 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 (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

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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:

~~6.1.1A rigid specimen holder~~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.

~~6.1.2A rigid expansion probe of inert, low expansivity material [<0.5 μm m~~

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 ~~s~~ Sensing ~~e~~ 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 ~~w~~ Weight or ~~force transducer~~ 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 ~~f~~ 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 ~~t~~ Temperature ~~c~~ 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 ~~t~~ Temperature ~~s~~ 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.

~~6.1.9A 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).~~

6.1.9 *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 ± 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 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.

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.

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

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

TABLE 1 Thermal Expansion Coefficients[A]

Temperature, °C	Aluminum [BCDEF] Mean Coefficient of Linear Thermal Expansion, μm/(m · °C)	Platinum [GHIJ] 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.

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 3—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 4—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 (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 5—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 and calibration coefficient retaining all available significant figures.

$$k = \alpha \cdot L \cdot \Delta T / \Delta L \tag{1}$$