Designation: E2206 – 11 (Reapproved 2015)

Standard Test Method for Force Calibration of Thermomechanical Analyzers[1]

This standard is issued under the fixed designation E2206; 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 the calibration or performance confirmation of the electronically applied force signal for thermomechanical analyzers over the range of 0 to 1 N.

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 There is no ISO method equivalent to this standard.

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 the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E4 Practices for Force Verification of Testing Machines
E473 Terminology Relating to Thermal Analysis and Rheology
E617 Specification for Laboratory Weights and Precision Mass Standards
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
E2113 Test Method for Length Change Calibration of Thermomechanical Analyzers
E2161 Terminology Relating to Performance Validation in Thermal Analysis and Rheology

3. Terminology

3.1 The technical terms used in this standard are defined in Terminologies E473, E1142, and E2161 including *calibration, conformance, precision, relative standard deviation, repeatability, reproducibility,* and *thermomechanical analyzer*.

4. Summary of Test Method

4.1 The electronic force signal generated by a thermomechanical analyzer is compared to that exerted by gravity on a known mass. The thermomechanical analyzer may be said to be in conformance if the performance is within established limits, typically 1 %. Alternatively, the force signal may be calibrated using a two-point calibration method.

5. Significance and Use

5.1 Most thermomechanical analysis experiments are carried out with some force applied to the test specimen. This force is often created electronically. It may be constant or changed during the experiment.

5.2 This method demonstrates conformance or calibrates the electronically applied force signal.

5.3 This method may be used for research and development, quality control, manufacturing or regulatory applications.

5.4 Other thermomechanical analyzer calibration functions include temperature by Test Method E1363 and length change by Test Method E2113.

6. Apparatus

6.1 *Thermomechanical Analyzer*—The essential instrumentation required to provide a minimum thermomechanical analysis or thermodilatometric capability for this method includes:

6.1.1 *Rigid Specimen Holder,* inert, low expansivity material [typically <0.6 μm/(m · K)] to center the specimen in the furnace and to fix the specimen to mechanical ground.

NOTE 1—Materials of construction with greater expansivity may be used but shall be reported.

6.1.2 *Rigid (Expansion or Compression) Probe,* inert, low expansivity material [typically <0.6 μm/(m · K)] which contacts the specimen with an applied compressive force (see Note 1).

6.1.3 *Sensing Element,* linear over a minimum range of 2 mm to measure the displacement of the rigid probe to ±1 μm resulting from changes in length of the specimen.

6.1.4 *Programmable Force Transducer,* to generate a constant force (±1.0 %) of up to 1.0 N that is applied through the rigid probe to 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.

NOTE 2—Other force ranges may be used but shall be reported.

6.1.5 *Furnace,* capable of providing uniform controlled heating (cooling) of the specimen to a constant temperature or at a constant rate within the temperature range of −100 to 600°C.

NOTE 3—Other temperature ranges may be used but shall be reported.

6.1.6 *Temperature Controller,* capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of change of up to 10°C/min constant to 0.1°C/min or at an isothermal temperature constant to ±0.5°C.

NOTE 4—Other heating rates may be used but shall be reported.

6.1.7 *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 temperature to ±0.1°C.

6.1.8 A means of sustaining an environment around the specimen of inert purge gas with a purge gas rate of 10 to 100 ± 5 mL/min.

NOTE 5—Typically, 99.9+ % pure nitrogen, argon, or helium 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 *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 50 to 100 g ± 0.002 % Class 4 or better *mass* (traceable to a national reference laboratory) in compliance with Specification E617.

7. Calibration

7.1 Prepare the thermomechanical analyzer for operation according to procedures recommended by the manufacturer of the thermomechanical analyzer as described in the Operations Manual.

7.2 Other calibration procedures which may be used, but which are not required in this standard include Test Methods E1363, E831, and E2113.

8. Procedure

8.1 With no specimen present, lower the probe so that it contacts the specimen holder. Zero the device so that no force (load) is applied by the probe to the specimen holder.

NOTE 6—The means for determining "no load" condition is specific to the instrument used. The user of this method should check the Instrument Operations Manual for this information.)

8.2 Apply a Class 4 or better (that is, Class 1, 2, 3, or 4) mass standard of 50 to 100 g to the probe. Record the (traceable) mass of the standard as M_1 in g. Apply a countering force to the force transducer so that no force is applied by the probe to the specimen holder. Record this force as F_2 in mN.

NOTE 7—Other masses may be used but shall be reported.

8.3 Calculate the force calibration constant (S) and conformity (C) using the equations of Section 9.

9. Calculations

9.1 For the purpose of this test method, it is assumed that the relationship between observed force (F_2) and the actual force (F_1) is linear and is governed by Eq 1:

$$F_1 = F_2 \cdot S \tag{1}$$

where:

S = force calibration constant (nominal value of 1.00000).

9.2 Calculate the force exerted by the standard mass in air using Eq 2:

$$F_1 = M\,a\,f \tag{2}$$

where:

M = mass of the weight, g,
a = standard acceleration due to gravity, (= 9.8065 m s^{-2}),
f = correction factor for local values of gravity and air buoyancy taken from Table 1, dimensionless, and
F_1 = force exerted by the standard mass, mN.

9.3 Calculate the calibration constant (S) using the values from 8.2 and 9.2 and Eq 2.

$$S = \frac{F_1}{F_2} \tag{3}$$

TABLE 1 Unit Force Exerted by a Unit Mass in Air at Various Latitudes[A]

Latitude, (°)	Elevation Above Sea Level, m (ft)					
	−30.5 to 152 (−100 to 500)	152 to 457 (500 to 1500)	457 to 762 (1500 to 2500)	762 to 1067 (2500 to 3500)	1067 to 1372 (3500 to 4500)	1372 to 1676 (4500 to 5500)
20	0.9978	0.9977	0.9976	0.9975	0.9975	0.9974
25	0.9981	0.9980	0.9979	0.9979	0.9978	0.9977
30	0.9985	0.9984	0.9983	0.9982	0.9982	0.9981
35	0.9989	0.9988	0.9987	0.9987	0.9986	0.9985
40	0.9993	0.9993	0.9992	0.9991	0.9990	0.9989
45	0.9998	0.9997	0.9996	0.9996	0.9995	0.9994
50	1.0003	1.0002	1.0001	1.0000	0.9999	0.9999
55	1.0007	1.0006	1.0005	1.0005	1.0004	1.0003

[A] Taken from Practice E4.

9.4 Calculate the percent conformity (C) of the instrument force signal using the value for S from 9.3 and Eq 4.

$$C = (S - 1.00000) \times 100\,\% \quad (4)$$

NOTE 8—The percent conformity is usually a very small number and expressing it as a percent may be inconsistent with SI notation. Because of common use and its effect on the experiment, however, it is expressed as a percent in this procedure.

9.4.1 Conformity may be estimated to one significant figure using the following criteria:

9.4.1.1 If S lies:

(1) Between 0.9999 and 1.0001, the conformity is better than 0.01 %,

(2) Between 0.9990 and 0.9999, or between 1.001 and 1.0010, then conformity is better than 0.1 %,

(3) Between 0.9900 and 0.9990 or between 1.0010 and 1.0100, then conformity is better than 1 %, and

(4) Between 0.9000 and 0.9900 or between 1.0100 and 1.100, then conformity is better than 10 %.

9.5 Using the determined value for S, Eq 1 may be used to calculate the true force (F_1) from an observed force value (F_2).

10. Report

10.1 Report the following information:

10.1.1 A unique identification of the thermomechanical analyzer included manufacturer and model number,

10.1.2 The calibration constant (S), as determined in 9.3, reported to at least five places to the right of the decimal point,

10.1.3 Conformity (C) as determined in 9.4, and

10.1.4 The specific dated version of this method used.

11. Precision and Bias[3]

11.1 An interlaboratory test was conducted in 2005 in which 13 laboratories participated using four instrument models from two manufacturers.

11.2 *Precision:*

11.2.1 Within laboratory variability may be described using the repeatability value (r) obtained by multiplying the repeatability relative standard deviation by 2.8. The repeatability value estimates the 95 % confidence limit. That is, two results from the same laboratory should be considered suspect (at the 95 % confidence level) if they differ by more than the repeatability value.

11.2.2 The within laboratory repeatability relative standard deviation for the measurement of slope (S) was found to be 0.10 % with 48 degrees of experimental freedom.

11.2.3 Between laboratory variability (R) may be described using the reproducibility value (R) by multiplying the reproducibility relative standard deviation by 2.8. The reproducibility value estimates the 95 % confidence limit. That is, two results from different laboratories should be considered suspect (at the 95 % confidence level) if they differ by more than the reproducibility value.

11.2.4 The between laboratory reproducibility relative standard deviation for the measurement of slope (S) was found to be 2.8 % with 48 degrees of experimental freedom

11.3 *Bias*—This is a calibration document. Bias is defined in this standard by the value of percent conformity (C) determined.

11.3.1 The mean value of conformance was found to be +0.12 %.

NOTE 9—This value has no predictive qualities. It shall not be used to assess the performance of other instruments.

12. Keywords

12.1 calibration; conformity; force; thermal analysis; thermomechanical analysis

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1035. Contact ASTM Customer Service at service@astm.org.