



Designation: E1363 – 16

# Standard Test Method for Temperature Calibration of Thermomechanical Analyzers<sup>1</sup>

This standard is issued under the fixed designation E1363; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 This test method describes the temperature calibration of thermomechanical analyzers from  $-50$  to  $1500^{\circ}\text{C}$ . (See [Note 1](#).)

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 is similar to ISO 11359–1 but addresses a larger temperature range and utilizes additional calibration materials.

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.* Specific precautionary statements are given in [Section 7](#) and [Note 11](#).

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

[E473 Terminology Relating to Thermal Analysis and Rheology](#)

2.2 *Other Standards:*<sup>3</sup>

[ISO 11359–1 Thermomechanical Analysis \(TMA\)-Part 1: General Principles](#)

## 3. Terminology

3.1 *Definitions:*

3.1.1 The terminology relating to thermal analysis appearing in Terminology [E473](#) shall be considered applicable to this document.

<sup>1</sup> 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.

Current edition approved Dec. 1, 2016. Published January 2017. Originally approved in 1990. Last previous edition approved in 2013 as E1363 – 13. DOI: 10.1520/E1363-16.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

## 4. Summary of Test Method

4.1 An equation is developed for the linear correlation of the experimentally observed program temperature and the actual melting temperature for known melting standards. This is accomplished through the use of a thermomechanical analyzer with a penetration probe to obtain the onset temperatures for two melting point standards. An alternate, one-point method of temperature calibration is also given for use over very narrow temperature ranges. (See [Note 2](#).)

**NOTE 1**—This test method may be used for calibrating thermomechanical analyzers at temperatures outside this range of temperature. However, the accuracy of the calibration will be no better than that of the temperature standards used.

**NOTE 2**—It is possible to develop a more elaborate method of temperature calibration using multiple (more than two) fusion standards and quadratic regression analysis. Since most modern instruments are capable of heating rates which are essentially linear in the region of use, the procedure given here is limited to a two-point calibration.

## 5. Significance and Use

5.1 Thermomechanical analyzers are employed in their various modes of operation (penetration, expansion, flexure, etc.) to characterize a wide range of materials. In most cases, the value to be assigned in thermomechanical measurements is the temperature of the transition (or event) under study. Therefore, the temperature axis (abscissa) of all TMA thermal curves must be accurately calibrated either by direct reading of a temperature sensor or by adjusting the programmer temperature to match the actual temperature over the temperature range of interest.

## 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 or Platform*, of inert, low expansivity material ( $<1 \mu\text{m m}^{-1} \text{K}^{-1}$ ) to center the specimen in the furnace and to fix the specimen to mechanical ground.

6.1.2 *A Rigid (expansion compression, flexure, tensile, etc.) Probe*, of inert, low expansivity material ( $<1 \mu\text{m m}^{-1} \text{K}^{-1}$ ) that contacts with the specimen with an applied compressive or tensile force. For this test method, the use of a penetration probe is recommended.

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

6.1.3 *A Sensing Element*, linear over a minimum range of 2 mm to measure the displacement of the rigid probe to  $\pm 50$  nm resulting from changes in the length/height of the specimen.

6.1.4 *A Weight or Force Transducer*, to generate a constant force of  $50 \pm 5$  mN ( $5.0 \pm 0.5$  g) that is applied through the rigid probe to the specimen.

NOTE 3—The recommendation of a 5.0 g load (or a force of 50 mN) is based on the use of penetration probes commonly used in the commercially available thermomechanical analyzers. These probes have tip diameters ranging from 0.89 to 2.0 mm and lead to pressures from 80 to 16 kPa when using the recommended 5.0 g load. The use of probes which differ greatly from this range of tip diameters may require different loading (or force).

6.1.5 *A Furnace*, capable of providing uniform controlled heating (cooling) at a rate of  $1^\circ\text{C min}^{-1}$  to  $10 \pm 1^\circ\text{C min}^{-1}$  of a specimen to a constant temperature within the applicable temperature range of this method.

NOTE 4—The temperature range of operation of commercial thermomechanical analyzers vary by manufacturer and mode. The complete range of temperature of an instrument is sometimes achieved by the use of two different furnaces. In this case, temperature calibration must be carried out for each furnace.

6.1.6 *A Temperature Controller*, capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change of  $10 \pm 1^\circ\text{C min}^{-1}$ .

6.1.7 *A Temperature Sensor*, that may be positioned in close proximity to the test specimen to provide an indication of the specimen/furnace temperature to within  $\pm 0.1^\circ\text{C min}^{-1}$ .

6.1.8 A means of sustaining an environment around the specimen with an inert purge gas (for example, nitrogen, helium, argon, etc.) at a purge gas flow rate of  $20 \text{ mL min}^{-1}$  to  $50 \text{ mL min}^{-1}$ .

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 for TMA are a change in linear dimension, temperature, and times.

## 7. Hazards

7.1 This test method may involve the use of hazardous materials, operations, and equipment. It is the responsibility of the user of this test method to establish appropriate safety practice and to determine the applicability of regulatory limitations prior to use. (**Warning**—Toxic or corrosive effluents, or both, may be released when heating some materials and could be harmful to personnel and the apparatus.)

7.2 Once this calibration procedure has been executed as described in 10.1.2.1 – 10.1.2.7 of this test method, the measuring temperature sensor position should not be changed, nor should it be in contact with the sample or sample holder in a way that would impede movement. If for some reason the temperature sensor position is changed or the temperature sensor is replaced, then the entire calibration procedure should be repeated.

## 8. Calibration

8.1 For the temperature range covered by many applications, the melting transition of 99.99 % pure materials may be used for calibration. (See Table 1.)

NOTE 5—The values in Table 1 were determined using special 99.9999 % pure materials and highly accurate steady-state conditions that are not attainable with this method. The actual precision of this test method is given in Section 13.

NOTE 6—The melting temperatures of these materials have been selected as primary fixed points (see Table 1) for the International Practical Temperature Scale of 1990.<sup>4</sup>

NOTE 7—Some materials have different crystalline forms (for example, tin) or may react with the container. Such calibration materials should be discarded after their initial melt.

## 9. Assignment of the Penetration Onset Temperature

9.1 The assignment of the TMA penetration onset temperature is an important procedure since, when using this method, temperature calibration of the thermomechanical analyzer is directly dependent upon it. The temperature standards given in Table 1 will give a downward deflection on the thermal curve, similar to that shown in Fig. 1, when placed under a weighted TMA penetration probe and heated to their respective melting temperatures.

9.2 The extrapolated onset temperature for such a penetration thermal curve is obtained by extending the pre-transition portion of the thermal curve to the point of intersection with a line drawn tangent to the steepest portion of the curve which describes the probe displacement. The temperature corresponding to this point of intersection is the penetration onset temperature. This is shown graphically in Fig. 1. There are

<sup>4</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1011. Contact ASTM Customer Service at service@astm.org.

**TABLE 1 Recommended Melting Temperature Reference Materials<sup>A</sup>**

Calibration Material <sup>B</sup>	Melting Temperature	
	(°C)	(K)
Mercury	-38.8344	234.3156
Water	0.01	273.16
Gallium	29.7646	302.9146
Indium	156.5985	429.7485
Tin	231.928	505.078
Bismuth	271.402	544.552
Cadmium	321.069	594.219
Lead	327.462	600.612
Zinc	419.527	692.677
Antimony	630.628	903.778
Aluminum	660.323	933.473
Silver	961.78	1234.93
Gold	1064.18	1337.33
Copper	1084.62	1357.77
Nickel	1455	1728
Cobalt	1495	1768

<sup>A</sup> The values in Table 1 were determined using special, 99.9999 % pure materials, and highly accurate steady-state conditions that are not attainable or applicable to thermal analysis techniques. The actual precision of this test method is given in Section 13.

<sup>B</sup> Della Gatta, G., Richardson, M. J., Sarge, S. M., and Stolen, S., "Standards, Calibration, and Guidelines in Microcalorimetry, Part 2: Calibration Standards for Differential Scanning Calorimetry," *Pure and Applied Chemistry*, Vol 78, No. 7, 2006, pp.1455–1476.

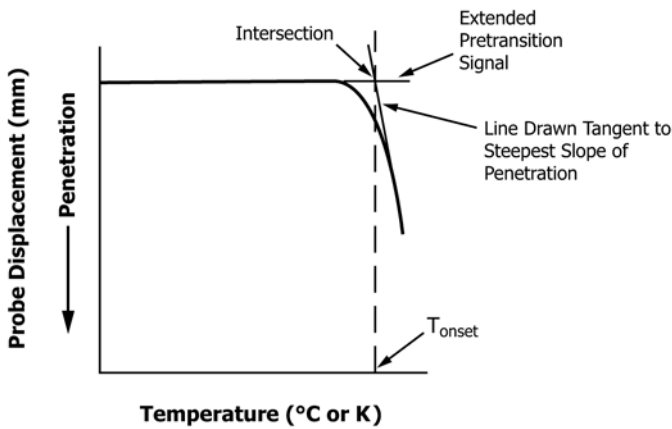


FIG. 1 Assignment of the Extrapolated Onset Temperature ( $T_o$ ) from TMA Thermal Curve

some materials (for example, aluminum metal) which show pre-transition probe displacement prior to the sharper downward deflection observed on melting. In this case, the pre-transition baseline is extended from the point which represents the highest temperature the material reaches prior to exhibiting significant or measurable softening under the conditions of the experiment. Fig. 2 describes the assignment of the extrapolated onset temperature for a specimen which exhibits pre-transition penetration.

## 10. Procedure

10.1 *Two-Point Calibration*—For the purposes of this procedure, it is assumed that the relationship between observed extrapolated onset temperature ( $T_o$ ) and actual specimen temperature ( $T_i$ ) is a linear one governed by the equation:

$$T_i = (T_o \times S) + I \quad (1)$$

where  $S$  and  $I$  are the slope and intercept of a straight line, respectively.

10.1.1 Select two calibration reference materials near the temperature range of interest. The standards should be as close to the upper and lower temperature limits used in the actual analysis runs as is practical.

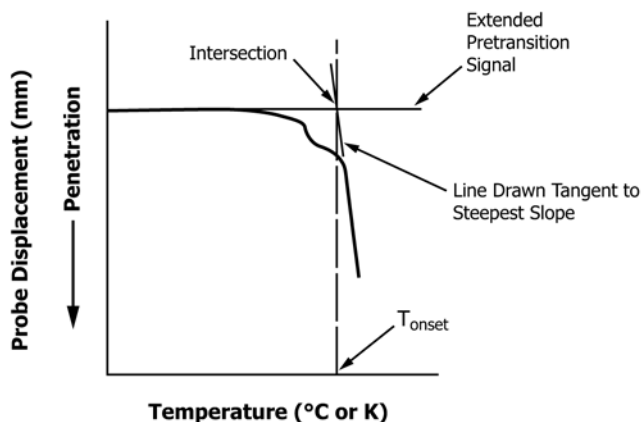


FIG. 2 Assignment of Extrapolated Onset Temperature ( $T_o$ ) from TMA Thermal Curve for Specimen Exhibiting Pre-transition Softening

10.1.2 Determine the apparent extrapolated onset temperature for the calibration reference material chosen, using a penetration-type probe with the TMA instrument.

10.1.2.1 Place a 10-mg to 20-mg specimen of one of the calibration reference materials on the sample platform (or holder, whichever is applicable).

NOTE 8—The specimen should have a smooth surface on both top and bottom. Avoid the use of specimens with sharp ridges and irregular surfaces. These can lead to false values for the onset temperatures. Powdered or liquid standards may be placed into a stable, inert container, if necessary.

10.1.2.2 Place a probe loaded with 5 g (or force of 50 mN) in contact with the test specimen.

10.1.2.3 Purge the specimen chamber area with inert gas at a flow rate that is appropriate to the dimensions of the apparatus throughout the experiment. Typical flow rates are from 20 mL/min to 50 mL/min. The same purge gas and flow rate should be maintained in both calibration runs and analysis runs.

10.1.2.4 Heat the calibration sample specimen to a temperature about 50°C below the calibration temperature and allow the TMA furnace to equilibrate for at least 1 min.

10.1.2.5 Heat the calibration specimen at 5 °C/min through the transition allowing the probe to reach a point of maximum penetration. (See Fig. 1.)

NOTE 9—Temperature calibration may be affected by heating rate, purge gas flow rate, and choice of purge gas.

NOTE 10—Other heating rates may be used but shall be reported.

10.1.2.6 From the TMA thermal curve obtained, assign the extrapolated onset temperature (see Fig. 1) to the required precision.

NOTE 11—Retain all available digits.

10.1.2.7 Repeat the procedure described in 10.1.2 – 10.1.2.5 using the second calibration reference material that was chosen.

## 11. Calculation

11.1 Using the reference material temperature values from Table 1 and the corresponding onset temperatures obtained experimentally, determine the slope and intercept using the following equations:

$$S = [T_{a1} - T_{a2}] / [T_{o1} - T_{o2}] \quad (2)$$

$$I = [(T_{o1} \times T_{a2}) - (T_{a1} \times T_{o2})] / (T_{o1} - T_{o2}) \quad (3)$$

where:

$S$  = slope (nominal value = 1.00),

$I$  = intercept,

$T_{a1}$  = reference transition temperature for Reference Material 1 taken from Table 1,

$T_{a2}$  = reference transition temperature for Reference Material 2 taken from Table 1,

$T_{o1}$  = experimentally observed transition onset temperature for Reference Material 1, and

$T_{o2}$  = experimentally observed transition onset temperature for Reference Material 2.

(Warning—The slope  $S$  is a dimensionless number whose value is independent of which temperature scale is used for  $I$

and  $T$ . In all cases,  $I$  must have the same units as  $T_{a1}$ ,  $T_{a2}$ ,  $T_{01}$ , and  $T_{02}$  and are, by necessity, consistent with each other.)

11.2  $S$  should be calculated to  $\pm 0.01$  units while  $I$  should be calculated to  $\pm 0.1$  °C.

11.3 Using the determined values for  $S$  and  $I$ , Eq 1 may be used to calculate the actual specimen transition,  $T_i$ , from any experimentally observed transition temperature,  $T_o$ , for the particular TMA instrument employed.

NOTE 12—The final result is rounded to the nearest 0.1°C consistent with the repeatability standard deviation reported in 13.2.1.

#### 11.4 One-Point Calibration:

11.4.1 In this abbreviated procedure, a relationship between the extrapolated onset temperature as observed from the use of a weighted penetration probe with one of the calibration reference materials (see Table 1) and the temperature as assigned by a temperature sensor is established. The operator should choose a calibration reference material that is near the temperature of the transition or phenomenon under study. For example, if one were interested in assigning the glass transition temperature of a polycarbonate specimen ( $T_g \approx 150^\circ\text{C}$ ), a good choice for the temperature calibration reference material would be indium metal ( $T_m = 156.6^\circ\text{C}$ ).

11.4.1.1 Using the sample handling techniques as described in 7.1 and 10.1.2, use a penetration probe to obtain the TMA thermal curves for the calibration standard chosen in Table 1.

11.4.1.2 From the known melting point of the calibration reference material (see Table 1), calculate the value and sign of  $\sigma$  from the following expression:

$$\sigma = T_a - T_o \text{ (}^\circ\text{C or K)} \quad (4)$$

where:

$T_a$  = known transition temperature for the calibration reference material,

$T_o$  = observed extrapolated onset temperature, and

$\sigma$  = correction factor for converting the observed thermocouple temperatures to actual sample temperatures.

See Note 12.

11.4.2 For the purpose of this procedure, it is assumed that the relationship between the observed extrapolated onset temperature ( $T_o$ ) and the actual specimen temperature is constant over the temperature range of interest. The value of  $\sigma$  is thus added to all observed measurements of transition temperatures for the particular instrument employed. That is:

$$T_i = T_o + \sigma \quad (5)$$

where:

$T_i$  = temperature of transition to be assigned

See Note 12.

## 12. Report

12.1 Report the following information:

12.1.1 Complete description of the instrument (manufacturer and model number) as well as the data handling device used for these tests.

12.1.2 Complete description of the temperature reference materials.

12.1.3 Complete description of the experimental conditions.

12.1.4 Results of the calibration procedure including values for  $S$  and  $I$ . If the abbreviated one-point calibration procedure was used, then the value of  $\sigma$  should be given.

12.1.5 The dated version of this method used.

## 13. Precision and Bias<sup>4</sup>

13.1 The precision of this test method was determined in an interlaboratory test in which ten laboratories participated using thermomechanical analyzers from four different manufacturers. In all cases the analyzers were of the vertical configuration. For this test method, two highly pure melting point calibration materials (diphenylether and tin) were used to obtain values for the calibration constants  $S$  and  $I$ . Using these constants, the melting point of a third highly pure material (indium), intermediate to these two calibration materials, was determined.

13.2 The following criteria may be used to judge the acceptability of actual sample temperature information determined using the two-point calibration procedure of this test method.

13.2.1 *Repeatability (Single Analyst)*—The standard deviation of results (each the average of duplicate determinations), obtained by the same analyst on different days, has been estimated to be 0.76 °C, with 27 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 2.1°C.

13.2.2 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicate determinations), obtained by the analysts in different laboratories, has been estimated to be 1.72°C, with 8 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 4.8°C.

13.3 The bias of actual sample temperature information determined by using the two-point calibration procedure of this test method was determined by comparing the results obtained for the intermediate temperature material with that judged to be the true value.

13.3.1 The average deviation of results (each the average of four determinations) from the true value was 0.07 °C with 10 df.

## 14. Keywords

14.1 calibration; temperature; thermal analysis; thermomechanical analysis

**SUMMARY OF CHANGES**

Committee E37 has identified the location of selected changes to this standard since the last issue (E1363 – 13) that may impact the use of this standard. (Approved Dec. 1, 2016.)

(1) Additions to **Table 1** and change in the literature reference to the values in **Table 1**.

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