Designation: E 1363 – 03

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

This standard is issued under the fixed designation E 1363; 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 temperature calibration of thermomechanical analyzers from – 50 to 1100°C. (See Note 2.)

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

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

1.3 SI units are the standard.

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

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 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 10.

2. Referenced Documents

2.1 *ASTM Standards:*

E 473 Terminology Relating to Thermal Analysis[2]

2.2

11359–1 Thermomechanical Analysis (TMA)-Part 1: General Principles[3]

3. Terminology

3.1 *Definitions:*

3.1.1 The terminology relating to thermal analysis appearing in E 473 shall be considered applicable to this document.

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 3.)

NOTE 2—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 3—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 thermocouple 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 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 compression, flexure, tensile, etc) Probe*, of inert, low expansivity material (< 1 μm m^{-1} 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.

[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 Test Methods and Recommended Practices.

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

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

6.1.3 *A Sensing Element*—linear over a minimum range of 2 mm to measure the displacement of the rigid probe to ± 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 ± 5 mN (5.0 ± 0.5 g) that is applied through the rigid probe to the specimen.

NOTE 4—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 10 ± 1 °C min^{-1} of a specimen to a constant temperature within the applicable temperature range of this method

NOTE 5—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 ± 1 °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 ± 0.1 °C min

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

6.1.9 *A Recording Device*, capable of recording any displaying fraction (including noise) of the specimen dimension signal (TMA curve) on the Y-axis versus any fraction (including noise) temperature of the X-axis.

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.

NOTE 6—**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 10.1.2.1-10.1.2.8 of this test method, the measuring thermocouple 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 thermocouple position is changed or the thermocouple 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.)

TABLE 1 Recommended Melting Temperature Standards[A]

Calibration Material[B]	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
Zinc	419.527	692.677
Aluminum	660.323	933.473
Silver	961.78	1234.93
Gold	1064.18	1337.33
Copper	1084.62	1357.77

[A] 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 12.

[B] B. W. Mangnum and G. T. Furukawa, "Guidelines for Realizing the International Temperature Scale of 1990 (ITS-90)," National Institute of Standards and Technology Technical Note 1265, page 8, 1990.

NOTE 7—The melting temperatures of these materials have been selected as primary fixed points (see Table 1) for the International Practical Temperature Scale of 1990.[4]

NOTE 8—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 pretransition portion of the thermal curve to the point of intersection with a line drawn tangent to the steepest portion of the curve which

[4] Supporting data are available from ASTM Headquarters. Request RR:E37-1011.

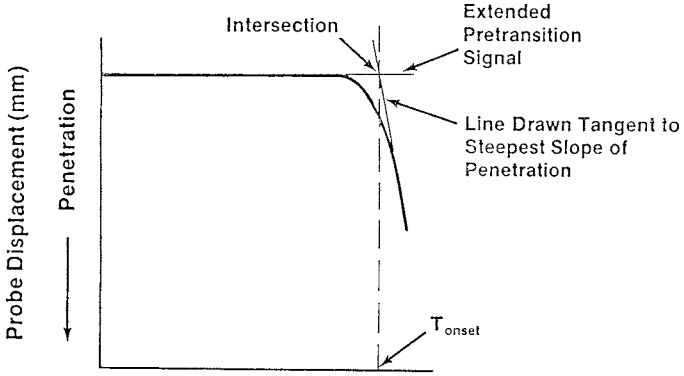

FIG. 1 Assignment of the Extrapolated Onset Temperature (T_o) from TMA Thermal Curve