Designation: E 1363 – 97ε1

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

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.

ε[1] NOTE—Editorial changes were made throughout in March 1998.

1. Scope

1.1 This test method covers 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 The values stated in SI units are to be regarded as the 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.* Specific precautionary statements are given in Section 7 and Note 9.

2. Referenced Documents

2.1 *ASTM Standards:*

E 473 Terminology Relating to Thermal Analysis[2]

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*, consisting of:

6.1.1 *Specimen Holder or Platform*, in which the specimen can be placed. Changes in the position of a probe (which may be either weighted or to which varying levels of force may be applied) must be sensed by the instrument. A variety of probe types may be used for such instruments. For this test method, the use of a penetration probe is recommended. A force of 50-mN (5-g) load should be employed with the penetration. (See Note 4.)

6.1.2 *Sensing Element*—Means for sensing movement of the probe resulting from changes in the vertical position of the probe due to penetration into the test specimen and for converting this movement into an electronic signal suitable for input to potentiometric recording devices. The sensing element should be capable of producing an electrical output of at least 1 mV per micrometre of probe movement.

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).

[1] This test method is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

Current edition approved February 10, 1997. Published March 1997. Originally published as E 1363 – 90. Last previous edition E 1363 – 95.

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

6.1.3 *Furnace*:

6.1.3.1 A means for uniformly heating the specimen at a predetermined rate over the temperature range of interest. Provisions should be made for precooling the furnace and specimen where near or sub-ambient temperature measurements are to be made. The instrument should be capable of providing heating rates up to 10°C per minute.

6.1.3.2 The temperature range of operation of commercial thermomechanical analyzers vary from manufacturer to manufacturer. 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.

6.1.4 *Thermocouple (or Other Temperature Measuring Device)*, that may be positioned in close proximity of the test specimen should be provided as a part of the TMA instrument. A chromel-alumel thermocouple is commonly used in commercial instruments.

6.1.5 *Pneumatic System*, that provides for purging the specimen with a dry inert gas. Nitrogen, argon, and helium are commonly used for this purpose.

6.1.6 *Data Handling Device*, that provides a means of recording changes in the analyzer probe position as a function of temperature.

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 5—**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.)

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 1968.[3]

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.

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.

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 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 some materials (for example, aluminum metal) which show pretransition probe displacement prior to the sharper downward deflection observed on melting. In this case, the pretransition 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 pretransition penetration.

10. Procedure

10.1 *Two-Point Calibration*—For the purposes of this procedure, it is assumed that the relationship between observed

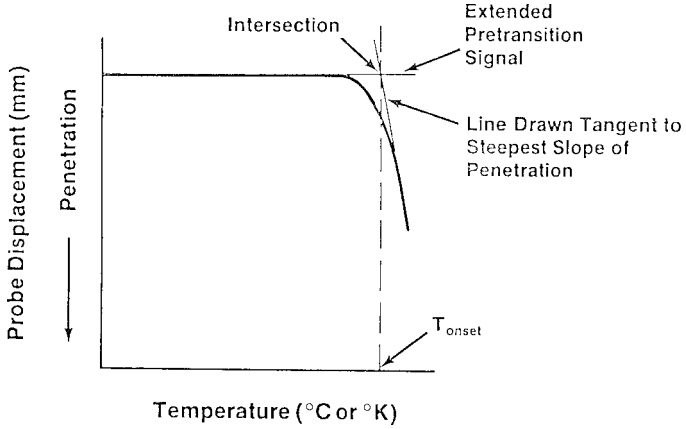

FIG. 1 Assignment of the Extrapolated Onset Temperature (T_o) from TMA Thermal Curve

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