Designation: E1867 – 06

Standard Test Method for Temperature Calibration of Dynamic Mechanical Analyzers[1]

This standard is issued under the fixed designation E1867; 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 dynamic mechanical analyzers (DMA) from –150 to 500°C.

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 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 Note 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E473 Terminology Relating to Thermal Analysis and Rheology

E1142 Terminology Relating to Thermophysical Properties

3. Terminology

3.1 *Definitions*:

3.1.1 The technical terms used in this test method are defined in Terminology E473 and Terminology E1142.

4. Summary of Test Method

4.1 An equation is developed for the linear correlation of experimentally observed program or sensor temperature and the actual melting temperature for known melting reference materials. This is accomplished by loading melting point reference materials into a polymer tube, or wrapping them with polymer tape and subjecting it to a mechanical oscillation at either fixed or resonant frequency. The extrapolated onset of melting is identified by a rapid decrease in the ordinate signal (the apparent storage modulus, stress, inverse strain or probe position). This onset is used for temperature calibration with two melting point reference materials.

5. Significance and Use

5.1 Dynamic mechanical analyzers monitor changes in the viscoelastic properties of a material as a function of temperature and frequency, providing a means to quantify these changes. In most cases, the value to be assigned is the temperature of the transition (or event) under study. Therefore, the temperature axis (abscissa) of all DMA thermal curves must be accurately calibrated by adjusting the apparent temperature scale to match the actual temperature over the temperature range of interest.

6. Interferences

6.1 An increase or decrease in heating rates or change in purge gas type or rate from those specified may alter results.

6.2 Once the temperature calibration procedure has been executed, the measuring temperature sensor position shall not be changed, nor shall it be in contact with the specimen or specimen holder in a way that would impede movement. If the temperature sensor position is changed or is replaced, then the entire calibration procedure shall be repeated.

6.3 Once the temperature calibration has been executed, the geometry deformation (bending study, versus tensile, and the like) shall not be changed. If the specimen testing geometry differs significantly from that of the calibrants, then the calibration shall be repeated in the geometry matching that of specimen testing.

6.4 This method does not apply to calibration for shear or compressive geometries of deformation.

7. Apparatus

7.1 The function of the apparatus is to hold a specimen of uniform dimension so that the specimen acts as the elastic and dissipative element in a mechanically oscillated system. Dynamic mechanic analyzers typically operate in one of several modes as outlined in Table 1.

7.1.1 The apparatus shall consist of the following:

[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.

TABLE 1 Dynamic Mechanical Analyzer Modes of Operation

Mode	Mechanical Response			
	Tension	Flexural	Torsion	Compression
Free/dec[A]	. . .	. . .	X	. . .
Forced/res/CA[A]	. . .	X	X	. . .
Forced/fix/CA[A]	X	X	X	X
Forced/fix/CS[A]	X	X	. . .	X

[A] Free = free oscillation; dec = decaying amplitude; forced = forced oscillation; CA = constant amplitude; res = resonant frequency; fix = fixed frequency; CS = controlled stress.

7.1.1.1 *Clamps*—A clamping arrangement that permits gripping of the specimen. This may be accomplished by clamping at both ends (most systems), one end (for example, torsional pendulum) or neither end (for example, free bending between knife edges).

7.1.1.2 *Device to Apply Oscillatory Stress or Strain*—A device for applying an oscillatory deformation (strain) or oscillatory stress to the specimen. The deformation may be applied and then released, as in freely vibrating devices, or continually applied, as in forced vibration devices.

7.1.1.3 *Detector*—A device or devices for determining the dependent and independent experimental parameters, such as force (stress), deformation (strain), frequency, and temperature. Temperature shall be measurable with an accuracy of ± 0.1 °C, force to ± 1 % and frequency to ± 1 %.

7.1.1.4 *Temperature Controller and Oven*—A device for controlling the specimen temperature, either by heating, cooling (in steps or ramps), or by maintaining a constant experimental environment. The temperature programmer shall be sufficiently stable to permit measurement of specimen temperature to ± 0.1 °C.

7.1.1.5 *Output Device*—Capable of displaying the storage modulus (either linearly or logarithmically) on the *Y*-axis (ordinate) increasing in the upwards direction and temperature on the *X*-axis (abscissa) increasing to the right.

NOTE 1—Some instruments, suitable for this test, may display only linear or logarithmic storage modulus while others may display linear and/or logarithmic storage modulus. Care must be taken to use the same modulus scale when comparing unknown specimens, and in the comparison of results from one instrument to another.

7.2 *PTFE (Polytetrafluoroethylene)*, tubing of 3 mm diameter and wallthickness of 0.5 mm (0.002 in.) [3]inner diameter may be used for low temperature standards. The tubing may be sealed with suitable melting temperature wax plugs, or similar sealant.

NOTE 2—PTFE tubing is selected for its flexibility and inert nature for the solvents in use at the temperatures of interest. Furthermore its transitions should not produce any interference in the DMA signal within the range of the suggested calibrant materials. For other temperature ranges, a suitable replacement for the PTFE tubing may be used.

7.3 Where the melting material is to be confined to a tube

7.4 *PTFE Tape*, for wrapping metal point standards.

7.5 *Calibration Materials*—One or more suitable materials presented in Table 2.

[3] Lotti, C., Canevarolo. S.V., Polymer Testing, 1998, Vol 17, pp. 523–530. "Temperature Calibration of a Dynamic Mechanical Thermal Analyzer."

TABLE 2 Calibration Materials

Material	Transition Temperature[A]		Reference
	°C	K	
Cyclopentane (solid-solid)	-151.16	121.99	X1.1
Cyclopentane (solid-solid)	-135.06	138.09	X1.1
n-Heptane	-90.56	182.65	X1.2
Cyclohexane	-87.06	186.09	X1.3
n-Octane	-56.76	216.39	X1.1
n-Decane	-26.66	246.49	X1.1
n-Dodecane	-9.65	263.5	X1.1
Water	0.01	273.16	X1.4
Cyclohexane	6.54	279.69	X1.3
Indium	156.5985	495.7485	X1.4
Tin	231.928	505.078	X1.4
Lead	327.462	600.612	X1.5
Zinc[B]	419.527	692.677	X1.4

[A] The values in this table were determined under special, highly accurate test conditions that are not attainable or applicable to this test method. The actual precision of this test method is given in Section 13.

[B] Amalgamates with aluminum. Do not heat above 430°C.

7.6 *Calipers* or other length measuring device capable of measuring dimensions (or length) within ± 10 µm.

8. Reagents and Materials

8.1 Dry nitrogen, helium, or other inert gas supplied for purging purposes and especially to ensure that moisture condensation and ice formation is avoided when measurements involve temperatures below the dew point.

9. Calibration and Standardization

9.1 Prepare the instrument for operation as dexcribed by the manufacturer in the operations manual

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_t) is a linear one governed by the equation:

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

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

10.2 Select two calibration standards near the temperature range of interest. The standards should be as close to the upper and lower temperature limits used for the subsequent test materials as practical.

NOTE 3—In some testing geometries it may be possible to perform the test directly on the metal melting point reference materials without encapsulation.

10.2.1 Encapsulation technique for low temperature (liquid) standards where the melting temperature does not exceed 100 °C.

10.2.1.1 Fill the PTFE tubing with the calibration material or wrap a solid calibrant with PTFE tape. Calibrant must extend to the ends of the clamping geometry and must have uniform dimensions with respect to width.

NOTE 4—For solid calibrants, a wire of dimensions suitable for testing should be used.

10.3 Measure the length and for solid calibrants the diameter as well, of specimens.