



Standard Test Method for Temperature Calibration of Dynamic Mechanical Analyzers¹

This standard is issued under the fixed designation E 1867; 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 covers methods for the temperature calibration of dynamic mechanical analyzers (DMA) from -150 to 500°C .

1.2 Electronic instrumentation or automated data analysis and reduction systems or treatments equivalent to this method may be used.

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 Note 7.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 473 Terminology Relating to Thermal Analysis²

E 1142 Terminology Relating to Thermophysical Properties²

3. Terminology

3.1 *Definitions:*

3.1.1 The technical terms used in this test method are defined in Terminology E 473 and Terminology E 1142.

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 standards. This is accomplished by loading a melting point standard into a polymer tube or aluminum foil and submitting 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 standards.

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.

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

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.

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 mechanical analyzers typically operate in one of several modes as outlined in Table 1.

7.1.1 The apparatus shall consist of the following:

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 $\pm 0.1^{\circ}\text{C}$, force to $\pm 1\%$ and frequency to $\pm 1\%$.

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.

7.1.1.4 *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 Where the melting material is to be confined to a tube

7.3 *Poly(Ether Ether Ketone) {PEEK} Tubing*, 0.5 mm (0.02 in.) inside diameter for low temperature standards, when containment is necessary. Tubing may be crimped with pliers or other tool.

NOTE 2—PEEK tubing is selected because it is inert to most solvents and has no low temperature transitions in its crystalline form that might interfere in the melting temperature range of liquid standards. It is readily available from most laboratory supply houses.

NOTE 3—PEEK must be in its crystalline form to have no temperature transitions. The PEEK tubing may be annealed at 300°C for 3 h to achieve this fully crystalline form.

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

7.5 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 Perform calibration of the instrument according to the manufacturers or designers recommended procedures. Note this calibration procedure in the report.

9.2 Calibration conditions (for example, heating rate, frequency and strain) should be identical to analysis conditions for subsequent specimens.

10. Procedure

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

TABLE 2 Calibration Materials

Material	Transition Temperature ^A		Reference
	°C	K	
Cyclopentane (solid-solid)	-151.16	121.99	2.2.1
Cyclopentane (solid-solid)	-135.06	138.09	2.2.1
<i>n</i> -Pentane	-132.66	140.49	2.2.2
<i>n</i> -Heptane	-90.56	182.65	2.2.3
Cyclohexane	-87.06	186.09	2.2.4
<i>n</i> -Octane	-56.76	216.39	2.2.3
<i>n</i> -Decane	-26.66	246.49	2.2.3
<i>n</i> -Dodecane	-9.65	263.5	2.2.3
Water	0.01	273.16	2.2.5
Cyclohexane	6.54	279.69	2.2.4
Diphenyl ether	26.87	300.02	2.2.6
Benzoic acid	122.37	395.62	2.2.6
Indium	156.5985	495.7485	2.2.5
Tin	231.928	505.118	2.2.5
Lead	327.502	600.652	2.2.6
Zinc ^B	419.527	692.677	2.2.5

^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.

extrapolated onset temperature (T_o) and actual specimen temperature (T_l) is a linear one governed by the equation:

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

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 4—In some sample mounting geometries (for example, parallel plate and three point bending and at some temperatures) it is possible to perform these tests directly on the specimen without encapsulation in PEEK tubing or aluminum foil.

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 PEEK tubing with the calibration material or wrap a solid calibration material with aluminum foil. Calibration material should extend to the ends of the package for a clamping geometry and foil package must have uniform dimensions with respect to width.

NOTE 5—Foil package is inert to most melting standards. Review Table 2 for standards that amalgamate with aluminum.

10.3 Measure the length and diameter (or width and thickness) of the foil package.

10.4 Mount the specimen in accordance with the procedure recommended by the manufacturer.

NOTE 6—For specimen clamping arrangements where the specimen is not gripped on either end (for example, free bending between knife edges) the specimen must be rigid enough at the test start temperature to sustain initial loading. Alternatively, the calibration specimen, without encapsulation, can be placed between the knife edge and a substrate.

10.5 Maximum strain amplitude should be within the linear viscoelastic range of the specimens to be subsequently analyzed. Strains of less than 1 % are recommended and should not exceed 5 %.

10.6 Conduct calibration runs at 1°C/min and a frequency of