ASTM INTERNATIONAL

Designation: ~~E1867 – 18~~ E1867 – 22

Standard Test Methods 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 These test methods ~~describes~~describe the temperature calibration of dynamic mechanical analyzers (DMA) from ~~−100 °C to 300 °C.~~ −100 °C to 300 °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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
E473 Terminology Relating to Thermal Analysis and Rheology
E1142 Terminology Relating to Thermophysical Properties
E1970 Practice for Statistical Treatment of Thermoanalytical Data
E1640 Test Method for Assignment of the Glass Transition Temperature By Dynamic Mechanical Analysis
E2161 Terminology Relating to Performance Validation in Thermal Analysis and Rheology
E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus
E3301 Test Method for Temperature Calibration of Dynamic Mechanical Analyzers Using Thermal Lag

3. Terminology

3.1 *Definitions:*

3.1.1 The technical terms used in these test methods are defined in Terminologies E473, E1142, and E2161, including *dynamic mechanical analysis, frequency, stress, strain*, and *storage modulus*.

4. Summary of Test Methods

4.1 In dynamic mechanical analysis, often large (for example, 1 g to 10 g), low thermal conductivity test specimens are

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[1] These test methods are under the jurisdiction of ASTM Committee E37 on Thermal Measurements and are the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.
Current edition approved ~~Aug. 1, 2018~~Oct. 1, 2022. Published ~~August 2018~~October 2022. Originally approved in 1997. Last previous edition approved in ~~2016~~2018 as ~~E1867 – 16.~~E1867 – 18. DOI: ~~10.1520/E1867-18.~~10.1520/E1867-22.

[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.

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

characterized while being mechanically supported using high thermal conductivity materials, while a temperature sensor is free-floating in the atmosphere near the test specimen. Under temperature programming conditions, where the atmosphere surrounding the test specimen is heated or cooled at rates up to 5 °C/min, the temperature of the test specimen may lead or lag that of the nearby temperature sensor. It is the purpose of this standard to calibrate the dynamic mechanical analyzer temperature sensor so that the indicated temperature more closely approximates that of the test specimen. In Methods A, B, and C, this is accomplished by separating the calibration specimen (with its first order transition) from its mechanical supports and from the surrounding atmosphere using a low thermal conductivity material. In Method D, the thermal lag between the temperature sensor and the test specimen is determined as a function of heating rate. This value is then used to adjust the indicated temperature following calibration under isothermal ambient conditions.

4.2 An equation is developed for the linear correlation of experimentally observed program or sensor temperature and the actual melting temperature for known melting or glass transition of the reference material. This is accomplished in Method A by a melting point reference material loaded into a polymer tube, or in Method B by wrapping the calibration material with polymer tape or in Method C by placing the calibration material between glass or ceramic plates and subjecting this test specimen 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).

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 dynamic mechanical analysis thermal curves must be accurately calibrated by adjusting the apparent temperature scale to match the actual specimen temperature over the temperature range of interest.

5.2 This test method is useful for research, quality assurance, and specification acceptance.

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 These test methods do 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.

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 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 readable to within ± 0.1 °C, force to within ± 1 % and frequency to within ± 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 stable to within ± 0.1 °C.

7.1.1.5 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 dynamic mechanical analysis are storage modulus, loss modulus, tangent delta, temperature, and time.

NOTE 1—Some instruments, suitable for this test, may display only linear or logarithmic storage modulus while others may display both linear 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 For Method A, high-temperature polymer tubing such as *PTFE (Polytetrafluoroethylene) or PEEK (Polyetheretherketone)*, of 3-mm outside diameter and wall thickness of 0.5-mm (0.002 in.) (**1**)[3] may be used for low temperature standards (that is, less than 160 °C). The tubing may be sealed with suitable melting temperature wax plugs, or similar sealant. (See Appendix X2.)

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 dynamic mechanical analyzer signal within the range of the suggested calibrant materials. *PEEK* provides increased stiffness for ease of loading. For other temperature ranges, a suitable replacement for the high temperature polymer tubing may be used.

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

7.3 For Method B, *PTFE* tape, to be used for wrapping metal point standards.

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

TABLE 2 Calibration Materials

Material	Transition Temperature[A]		Reference
	°C	K	
n-Heptane	−90.56	182.65	(**2**)
Cyclohexane	−87.06	186.09	(**3**)
n-Octane	−56.76	216.39	(**4**)
n-Decane	−26.66	246.49	(**4**)
n-Dodecane	−9.65	263.5	(**4**)
Water	0.01	273.16	(**5**)
Cyclohexane	6.54	279.69	(**3**)
Indium	156.5985	495.7485	(**5**)
Tin	231.928	505.078	(**5**)

[A] The values in this table were determined under special, highly accurate test conditions that are not attainable or applicable to these test methods. The actual precision of these test methods is given in Section 14.

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[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.

7.4 For Method C, sheet stock or coupons composed of one of the materials in Table 3, approximately 0.5 mm in thickness, and length and width similar to that of an unknown test specimen to be used.

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

7.5 *For Method D:*

7.5.1 *Calibration Material*—A high temperature polymer sheet stock or coupons, 0.5 mm to 1.0 mm in thickness with length and width similar to that of the unknown test specimen, with a well-defined glass transition.

Note 3—Polycarbonate or a fully cured thermoset composite have been found suitable.

7.5.2 *Thermometer, calibrated digital or analog,* capable of measuring temperature over the range of 15 °C to 30 °C readable to within 0.1 °C.

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

8. Reagents and Materials

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

Note 4—The same purge gas shall be used for calibration as for the determination of unknown specimens.

9. Calibration and Standardization

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

10. ~~Precedure—Methods~~Procedure—Methods A, B, or C

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:

$$T_t = (T_o \times S) + I \tag{1}$$

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.

TABLE 3 Insulating Sheet Stock

Material	Thermal Conductivity at 25 °C, W/(m-K)	Reference	Room Temperature Thermal Diffusivity at 25 °C, mm²/s	Maximum Temperature, °C
~~Polytetrafluorothylene~~	~~0.25~~	~~(6)~~		~~260~~ ~~(6)~~
Polytetrafluoroethylene	0.25	**(6)**		260 **(6)**
Polyimide	0.12 0.35^C	**(7)**		
Polyetheretherketone	0.25	**(8)**		200 **(8)**
Macor^A	1.46		0.84 0.73	
Soda Lime Glass	0.94		0.51	250
Pyrex^B Glass	1.45	**(9)**		

^A Macor is a registered trademark of Corning, Inc., Corning, NY.
^B Pyrex is a registered trademark of Corning, Inc., Corning, NY.
^C At 40 °C.

Note 5—The purpose of the polymer encapsulation is to provide thermal resistance between the test specimen and the environment similar to that offered by polymer test specimens. In some testing geometries it may be possible to perform the test directly on the metal melting point reference materials without encapsulation. (See Appendix X1.)

10.3 *Method A—Calibration Using Materials that are Liquids at Ambient Temperature* and where the melting temperature does not exceed 100 °C. (See Appendix X2.)

10.3.1 Fill the polymer tubing with the calibration material. Calibrant must extend to the ends of the clamping geometry and must have uniform dimensions with respect to width.

10.3.2 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.

10.3.3 Maximum strain amplitude shall be within the linear viscoelastic range of the specimens to be subsequently analyzed. Strains of less than 1 % are recommended and shall not exceed 3 %.

10.3.4 Equilibrate the test specimen for 5 min at a temperature 30 °C below the anticipated transition temperature.

10.3.5 Conduct the calibration experiments at the heating rate of interest, preferably ~~1 °C °C/min~~ /min but no greater than ~~5~~5 °C ~~°C/min~~ /min and a frequency of 1 Hz from ~~30 °C~~ 30 °C below to ~~20 °C~~ 20 °C above the anticipated melting temperature of the calibration material. Other heating rates and frequencies may be used but shall be reported. (See Appendix X1.)

Note 7—Calibration for temperature shall be performed under the conditions of heating rate and frequency at which the unknown specimens will be tested. This test method does not address the issues of frequency affects for polymeric transitions (such as the upwards shift of glass transition temperature with increasing frequency), and will only compensate for thermal lag within the measuring device.

10.4 *Method B—Calibration Where the Material is a Solid at Ambient Temperature:*

10.4.1 The calibration material must extend to the ends of the clamping geometry and must have uniform dimensions with respect
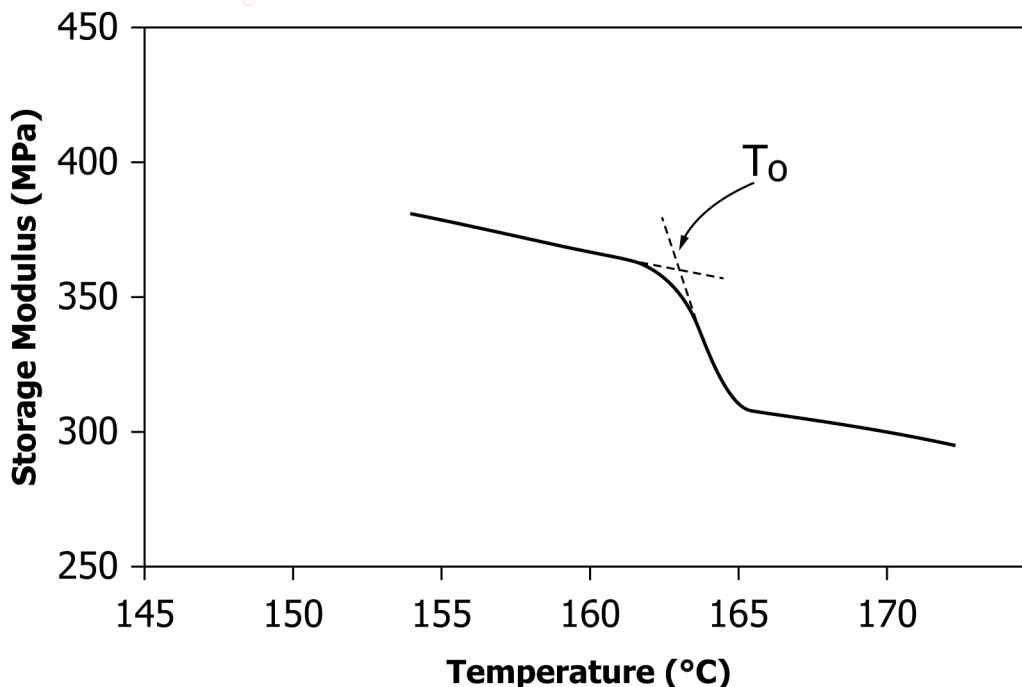
FIG. 1 Transition Temperature

to the width and thickness. Wrap the calibration material with polytetrafluoroethylene tape to a thickness of 0.5 mm. 0.5 mm. Other thicknesses may be used but shall be reported.

10.4.2 Mount the wrapped specimen into the apparatus according to the procedure recommended by the manufacturer as described in the operations manual.

NOTE 8—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.

10.4.3 Maximum strain amplitude shall be within the linear viscoelastic range of the specimen. Strain of less than 1 % is recommended and shall not exceed 3 %.

10.4.4 Equilibrate the test specimen for 5 min at a temperature 30 °C below the anticipated transition temperature.

10.4.5 Conduct the calibration experiments at the heating rate of interest, preferably 1 1 °C °C/min /min but no greater than 5 5 °C °C/min /min and a frequency of 1 Hz from 30 °C 1 Hz from 30 °C below to 20 °C 20 °C above the anticipated melting temperature of the calibration material. Other heating rates and frequencies may be used but shall be reported.

NOTE 9—Calibration for temperature shall be performed under the conditions of heating rate and frequency at which the unknown specimens will be tested. This test method does not address the issues of frequency affects for polymeric transitions (such as the upwards shift of glass transition temperature with increasing frequency), and will only compensate for thermal lag within the measuring device.

10.5 *Method C—Calibration Where Material is Solid at Ambient Temperature:*

10.5.1 The calibration material must extend to the ends of the clamping geometry and must have uniform dimensions with respect to the width and thickness. Place a 0.5 mm thick coupon of insulating material on either side of the calibration material. Other thicknesses may be used but shall be reported.

10.5.2 Mount the sandwiched specimen into the apparatus according to the procedure recommended by the manufacturer as described in the operations manual.

NOTE 10—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.

10.5.3 Maximum strain amplitude shall be within the linear viscoelastic range of the specimen. Strain of less than 1 % is recommended and shall not exceed 3 %.

10.5.4 Equilibrate the test specimen for 5 min at a temperature 30 °C below the anticipated transition temperature.

10.5.5 Conduct the calibration experiments at the heating rate of interest, preferably 1 1 °C °C/min /min but no greater than 5 5 °C °C/min /min and a frequency of 1 Hz from 30 °C 1 Hz from 30 °C below to 20 °C 20 °C above the anticipated melting temperature of the calibration material. Other heating rates and frequencies may be used but shall be reported.

NOTE 11—Calibration for temperature shall be performed under the conditions of heating rate and frequency at which the unknown specimens will be tested. This test method does not address the issues of frequency affects for polymeric transitions (such as the upwards shift of glass transition temperature with increasing frequency), and will only compensate for thermal lag within the measuring device.

11. Procedure—Method D

NOTE 12—Method D is equivalent to Test Method E3301.

11.1 Mount the calibration specimen according to the procedure recommended by the apparatus manufacturer.

11.2 Equilibrate the calibration specimen for 30 min at ambient temperature.

11.3 Using the calibrated thermometer (7.5.2) measure and record the temperature of the test specimen as *I*.

11.4 Perform a scouting run using Test Method E1640 to determine the approximate temperature range of the glass transition region.

NOTE 13—Maximum strain amplitude shall be within the linear ~~viscoelastice~~viscoelastic range of the specimens to be subsequently analyzed. Strains less than 1 % are recommended and shall not exceed 3 %.

NOTE 14—This step erases any enthalpic relaxation present in the specimen and is a required step in this procedure even should the temperature range of the glass transition be known.

11.5 Using Test Method E1640, determine the glass transition temperature (T_g) of the calibration material at a minimum of 3 heating rates over the heating rate (β) range of 0.1 °C/min to 5 °C/min recording the heating rate and glass transition temperature at each rate.

11.6 Prepare a display of the 3 or more data sets from 11.5 displaying the glass transition temperature on the Y-axis and corresponding heating rate on the X-axis.

11.7 Using linear regression (see Practice E1970) determine the slope of the line through the three data sets as thermal lag ($\Delta T/\Delta \beta$) (according to Test Method E3142).

11.8 Determine the temperature offset (σ) for the heating rate to be used for the unknown test specimen using Eq 5.

11.9 Apply this temperature offset value using Eq 6.

12. Calculation

12.1 Take the transition temperature as the extrapolated onset to the sigmoidal change in the ordinate signal observed in the downward direction (see Fig. 1).

12.1.1 Construct a tangent to the ordinate signal curve below the transition temperature.

12.1.2 Construct a tangent to the ordinate signal curve at the inflection point approximately midway through the sigmoidal change associated with the transition.

12.1.3 Report the temperature at which these tangent lines intersect as reported as the observed transition temperature (T_o).

12.2 *Two Point Calibration:*

12.2.1 Using the standard temperature values from Table 2 and the corresponding onset temperatures obtained experimentally, calculate the slope and intercept using the following equations:

$$S = [T_r1 - T_r2]/[T_o1 - T_o2] \tag{2}$$

$$I = [(T_o1 \times T_r2) - (T_r1 \times T_o2)]/[T_o1 - T_o2] \tag{3}$$

where:
S = slope (nominal value = 1.0000),
I = intercept,
T_r1 = reference transition temperature for Standard 1 (in Table 2),
T_r2 = reference transition temperature for Standard 2 (in Table 2),
T_o1 = experimentally observed transition onset temperatures for Standard 1, and
T_o2 = experimentally observed transition onset temperature for Standard 2.

NOTE 15—The slope S is a dimensionless number whose value is independent of which temperature scale is used for I and $T.I$, in all cases, must have the same units as T_r1, T_r2, T_o1, and T_o2 that are, by necessity, consistent with each other.

12.2.2 S should be calculated to ± 0.0001 units while I should be calculated to ± 0.1 °C.