



Designation: E1867 – 22

Standard Test Methods for Temperature Calibration of Dynamic Mechanical Analyzers¹

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 describe the temperature calibration of dynamic mechanical analyzers (DMA) from -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

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.

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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.

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 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 $5^{\circ}\text{C}/\text{min}$, 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).

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

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 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

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.

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.

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

TABLE 2 Calibration Materials

Material	Transition Temperature ^A		Reference
	°C	K	
<i>n</i> -Heptane	−90.56	182.65	(2)
Cyclohexane	−87.06	186.09	(3)
<i>n</i> -Octane	−56.76	216.39	(4)
<i>n</i> -Decane	−26.66	246.49	(4)
<i>n</i> -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](#).