



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 describes 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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in [Note 10](#).

2. Referenced Documents

2.1 *ASTM Standards:*²

[E473 Terminology Relating to Thermal Analysis and Rheology](#)

[E1142 Terminology Relating to Thermophysical Properties](#)

[E2161 Terminology Relating to Performance Validation in Thermal Analysis and Rheology](#)

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 Method

4.1 In dynamic mechanical analysis, often large (for example, 1 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. This is accomplished by separating the test specimen from its mechanical supports and from the surrounding atmosphere using a low thermal conductivity material. Three test methods of providing this separation are provided.

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 reference materials. This is accomplished in Method A by a melting point reference materials 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). 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

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 Feb. 15, 2016. Published April 2016. Originally approved in 1997. Last previous edition approved in 2013 as E1867 – 13. DOI: 10.1520/E1867-16.

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.