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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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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.

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 $\pm 1\%$.

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.