



Designation: E3301 – 22

Standard Test Method for Temperature Calibration of Dynamic Mechanical Analyzers Using Thermal Lag¹

This standard is issued under the fixed designation E3301; 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 describes the temperature calibration of a dynamic mechanical analyzer using thermal lag over the temperature range of $-100\text{ }^{\circ}\text{C}$ to $300\text{ }^{\circ}\text{C}$.

1.2 This standard may be compared to Test Methods [E1867](#).

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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

[D4092](#) Terminology for Plastics: Dynamic Mechanical Properties

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

[E1142](#) Terminology Relating to Thermophysical Properties

[E1356](#) Test Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry

[E1640](#) Test Method for Assignment of the Glass Transition Temperature By Dynamic Mechanical Analysis

[E1867](#) Test Methods for Temperature Calibration of Dy-

amic Mechanical Analyzers

[E1970](#) Practice for Statistical Treatment of Thermoanalytical Data

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

[E2877](#) Guide for Digital Contact Thermometers

[E3142](#) Test Method for Thermal Lag of Thermal Analysis Apparatus

3. Terminology

3.1 *Definitions:*

3.1.1 The technical terms used in this test method are defined in Terminologies [D4092](#), [E473](#), [E1142](#), and [E2161](#) including *calibration*, *Celsius*, *damping*, *dissipative*, *elastic*, *frequency*, *loss modulus*, *peak*, *storage modulus*, *strain*, *stress* $\tan \delta$, *tan delta*, *tangent delta*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dew point, n*—the temperature below which condensation of water vapor begins when the atmosphere is cooled.

3.2.2 *relaxation, n*—in a glass or viscous liquid, the change in any material property (such as density, enthalpy, etc.) with time following a perturbation (such as a change in temperature, stress, etc.).

4. Summary of Test Method

4.1 In dynamic mechanical analysis, large (for example, 1 g to 10 g), low thermal conductivity test specimens are characterized. These specimens are mechanically supported using high thermal conductivity materials of construction. A free-floating temperature sensor is placed as close as practical to the specimen. Under conditions of temperature change, where the system atmosphere surrounding the specimen is heated or cooled at rates up to $5\text{ }^{\circ}\text{C}/\text{min}$, the temperature of the specimen will lead or lag that of the temperature sensor. It is the purpose of this standard to calibrate the dynamic mechanical analyzer temperature signal so that the measured temperature more closely approximates that of the assumed test specimen.

4.2 The thermal lag between the temperature sensor and the test specimen is determined as a function of temperature rate of change. This value is then used to adjust the indicated temperature following calibration under isothermal ambient temperature conditions.

¹ This test method is under the jurisdiction of ASTM Committee [E37](#) on Thermal Measurements and is 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.

5. Significance and Use

5.1 Dynamic mechanical analysis monitors changes in the viscoelastic properties (that is, storage modulus, loss modulus, tangent angle delta) of a material as a function of temperature and frequency, providing a means to quantify these changes. In many cases, the value to be assigned is the temperature of the transition or event under study. Therefore, the temperature axis (abscissa) of the dynamic mechanical analysis thermal curve must be accurately calibrated by adjusting the measured temperature scale to match the assumed specimen temperature over the temperature range of interest.

6. Interferences

6.1 Once the temperature calibration procedure has been executed, the temperature measuring 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 it is replaced, then the entire calibration procedure shall be repeated.

6.2 The temperature calibration is valid only for the specimen test geometry (bending, tension, and the like) used during the calibration process. If multiple geometries are used, then calibration shall be performed for each geometry.

6.3 Apparatus temperature calibration is known to be dependent upon the purge gas type (thermal conductivity) and flow rate and upon specimen size. These experimental conditions shall be the same for calibration as for the testing of unknown specimens.

6.4 Thermal lag is reported to be a linear function of temperature over the temperature range of this standard (1).³ In principle, the dependence of thermal lag on temperature may be determined using Test Method E3142 and a series of materials with differing glass transition temperatures.

NOTE 1—In addition to polycarbonate used here, polystyrene and polyimide have been found suitable by some users (see 8.1).

6.5 The glass transition is a kinetic event and will increase in temperature with increasing temperature-rate-of-change (heating rate) and with oscillatory frequency. For low heating rates used in dynamic mechanical analysis, the increase in temperature is assumed to be insignificantly small. In one case, the thermal lag effect was observed to be 0.25 min while the DMA calibration offset was 1.15 min (2). The actual influence of these effects may be assessed using a more closely coupled thermoanalytical technique such as differential scanning calorimetry (see Test Method E1356).

6.6 The effects of test specimen specific heat capacity and thermal conductivity on the thermal lag are not known. For best results, the size, shape, thermal conductivity, and specific heat capacity of the calibration material should be as similar to that of the test specimen as practical.

7. Apparatus

7.1 *Dynamic Mechanical Analyzer*—The essential instrumentation required to provide the minimum dynamic mechanical capability for this method includes:

7.1.1 A *drive motor*, to apply force or displacement to the specimen in a periodic manner capable of frequency of 1 ± 0.1 Hz. This motor may also be capable of providing static force or displacement on the specimen.

NOTE 2—Dynamic mechanical analyzers often have a frequency range of several decades. Other frequencies may be used but shall be reported. Thermal lag of the glass transition is constant over a 30-fold range from 1 Hz to 30 Hz (2).

7.1.2 A *coupling shaft*, or other means to transmit the force or displacement from the motor to the specimen.

7.1.3 A *clamping system(s)* to fix the specimen between the coupling shaft and the stationary clamp(s).

7.1.4 A *position sensor*, to measure the changes in position of the specimen during dynamic motion readable to 1 % of full scale, or

7.1.5 A *force sensor*, to measure the force developed by the specimen readable to 1 % of full scale.

7.1.6 A *temperature sensor*, to provide an indication of the specimen temperature readable to 0.1 °C.

7.1.7 A *furnace*, to provide controlled heating or cooling of a specimen at a constant temperature or at a constant rate within the applicable temperature range of -100 °C to $+300$ °C.

7.1.8 A *temperature controller*, capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change of up to 5 °C/min constant to within 0.1 °C/min or at an isothermal temperature constant to 0.1 °C.

7.1.9 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 angle delta, frequency, temperature, and time.

NOTE 3—Some instruments, suitable for this test, may display only linear or logarithmic storage modulus while others may display both linear and logarithmic storage modulus. Care shall be taken to use the same modulus scale when comparing specimens and the comparison of results from one instrument to another.

7.1.10 *Data analysis capability*, to provide storage modulus, loss modulus, tangent angle delta, or other useful parameters derived from the measured signals.

7.2 *Thermometer, calibrated, digital or analog*, class D (see Guide E2877) capable of measuring temperature over the range of 15 °C to 35 °C readable to 0.1 °C.

7.3 *Calipers* or other length measuring device capable of measuring length of up to 10 cm to within 10 µm.

8. Reagents and Materials

8.1 *Calibration Material*—A high temperature polymer sheet stock or coupon, with a well-defined, stable glass transition temperature, with length, thickness, and width similar (within 25 %) to that of the unknown specimen.

NOTE 4—Thermoplastic polycarbonate or a fully cured thermoset

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