Designation: E3301 – 22

Standard Test Method for Temperature Calibration of Dynamic Mechanical Analyzers Using Thermal Lag[1]

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 °C to 300 °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 Dynamic 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 δ, 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 °C/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.

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

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

composite have be found suitable.

NOTE 5—By well-defined glass transition temperature is meant a glass transition temperature that is stable with repeated use. This requires conditioning the reference material by an initial run to erase any thermal history.

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

NOTE 6—The same protective purge gas shall be used with the calibration as with the test specimen.

9. Preparation of Apparatus

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

10. Procedure

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

10.2 Equilibrate the calibration specimen for 10 minutes at ambient temperature.

10.3 Using the calibrated thermometer placed close to the apparatus temperature sensor, measure and record the temperature of the test specimen. Adjust the temperature signal of the dynamic mechanical analyzer to indicate this value.

10.4 Perform a scouting (or pilot) run using Test Method E1640 to determine the approximate temperature range of the glass transition of the calibration material.

NOTE 7—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.

NOTE 8—Maximum strain amplitude shall be within the linear viscoelastic range of the specimen to be subsequently analyzed. Strains less than 1 % are recommended and shall not exceed 3 %.

10.5 Using Test Method E1640 and the estimated range of the glass transition from 10.4, determine the glass transition temperature of the calibration material at a minimum of 5 approximately equally distributed heating rates between 0.5 °C⁄min and 5 °C⁄min recording the measured heating rate (to within ± 0.1 °C⁄min and the glass transition temperature (to within ± 0.1 °C) at each rate.

NOTE 9—Heating rates of 0.5, 1.0, 2.0, 3.0, and 5.0 °C/min are commonly used.

10.6 Prepare a display of the 5 or more data sets from 10.5 with the glass transition temperature on the Y-axis and corresponding heating rate on the X-axis (see Fig. 1).

10.7 Using linear regression (see Practice E1970) determine the slope of the line through the four data sets (see Test Method E3142). Report this value as thermal lag ($\Delta T/\Delta\beta$). Determine the temperature offset (δ) for the heating rate to be used for the unknown specimen using Eq 1.

NOTE 10—A goodness-of-fit R value of >0.8 is considered acceptable. If less than 0.8, then add data points at additional heating rates to determine outliers or a non-linear response.

10.8 Apply the temperature offset value using Eq 2.

NOTE 11—The value of the temperature offset or the thermal lag may be a parameter for the data analysis portion of the data analyzer.

11. Calculation or Interpretation of Results

11.1 Using the value of thermal lag ($\Delta T/\Delta\beta$) from 10.7, determine the temperature offset (δ) for the heating rate (β) to be used:

$$\delta = (\Delta T / \Delta \beta) \times \beta \quad (1)$$

11.2 From the observed transition temperature of the unknown specimen (*To*), determine the actual transition temperature (*T*):

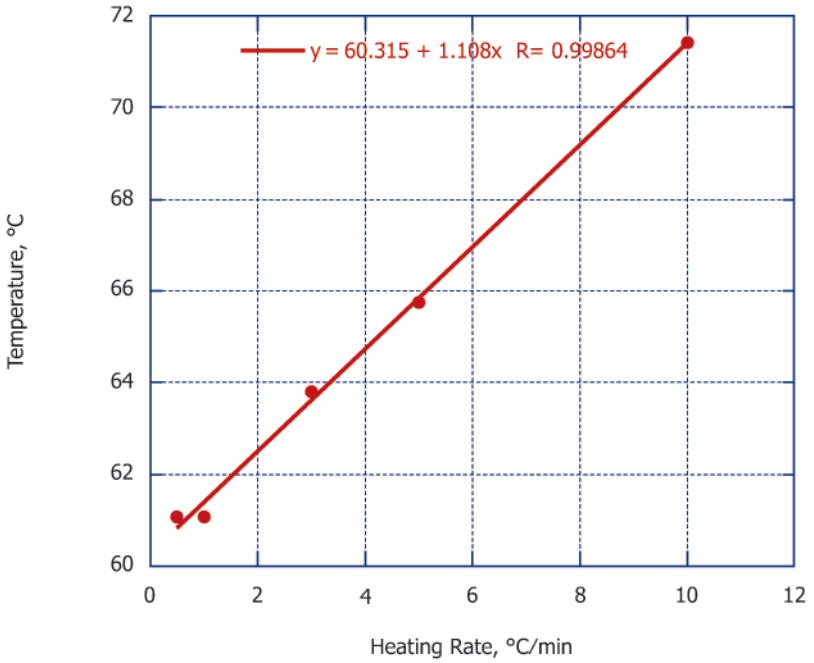

FIG. 1 Determination of Thermal Lag – Epoxy Resin (3)