Designation: E1867 – 16

Standard Test Methods for Temperature Calibration of Dynamic Mechanical Analyzers[1]

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°C/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

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[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.

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

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 mechanic 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 ±0.1°C, force to ±1 % and frequency to ±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 sufficiently stable to permit measurement of specimen temperature to 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.

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.

NOTE 1—Some instruments, suitable for this test, may display only linear or logarithmic storage modulus while others may display linear or logarithmic storage modulus, or both. 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 X3.)

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 DMA 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.

7.3 For Method B, PTFE tape, to be used for wrapping metal point standards.

7.4 For Method C, sheet stock or coupons composed of one of the materials in Table 3, approximately 0.5 mm in thickness, and length and width similar to that of an unknown test specimen to be used.

7.5 *Calibration Materials*—One or more suitable materials presented in Table 2.

7.6 *Calipers* or other length measuring device capable of measuring dimensions (or length) within ±10 µm.

8. Reagents and Materials

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

9. Calibration and Standardization

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

TABLE 2 Calibration Materials

Material	Transition Temperature^A		Reference
	°C	K	
n-Heptane	–90.56	182.65	X1.2
Cyclohexane	–87.06	186.09	X1.3
n-Octane	–56.76	216.39	X1.1
n-Decane	–26.66	246.49	X1.1
n-Dodecane	–9.65	263.5	X1.1
Water	0.01	273.16	X1.4
Cyclohexane	6.54	279.69	X1.3
Indium	156.5985	495.7485	X1.4
Tin	231.928	505.078	X1.4

^{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 13.

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

TABLE 3 Insulating Sheet Stock

Material	Thermal Conductivity at 25°C, W/(m-K)	Reference	Room Temperature Thermal Diffusivity at 25 °C, mm2/s	Maximum Temperature, °C
Polytetrafluoroethylene	0.25	(**2**)		260 (**2**)
Polyimide	0.12 0.35^C	(**3**)		
Polyetheretherketone	0.25	(**4**)		400 (**4**)
Macor^A	1.46		0.84 0.73	
Soda Lime Glass	0.94		0.51	250
Pyrex^B Glass	1.45	(**5**)		

[A] Macor is a registered trademark of Corning, Inc., Corning, NY.

[B] Pyrex is a registered trademark of Corning, Inc., Corning, NY.

[C] At 40°C.

10. Procedure

10.1 *Two Point Calibration*—For the purposes of this procedure, it is assumed that the relationship between observed extrapolated onset temperature (T_o) and actual specimen temperature (T_t) is a linear one governed by the equation:

$$T_t = (T_o \times S) + I \tag{1}$$

where: S and I are the slope and intercept of a straight line, respectively.

10.2 Select two calibration standards near the temperature range of interest. The standards should be as close to the upper and lower temperature limits used for the subsequent test materials as practical.

NOTE 3—The purpose of the polymer encapsulation is to provide thermal resistance between the test specimen and the environment similar to that offered by polymer test specimens. In some testing geometries it may be possible to perform the test directly on the metal melting point reference materials without encapsulation. (See Appendix X2.)

10.3 *Method A—Calibration Using Materials that are Liquids at Ambient Temperature* and where the melting temperature does not exceed 100°C. (See Appendix X3.)

10.3.1 Fill the polymer tubing with the calibration material. Calibrant must extend to the ends of the clamping geometry and must have uniform dimensions with respect to width.

10.3.2 Mount the specimen in accordance with the procedure recommended by the manufacturer.

NOTE 4—For specimen clamping arrangements where the specimen is not gripped on either end (for example, free bending between knife edges) the specimen must be rigid enough at the test start temperature to sustain initial loading. Alternatively, the calibration specimen, without encapsulation, can be placed between the knife edge and a substrate.

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

10.3.4 Conduct the calibration experiments at the heating rate of interest, preferably 1°C/min but no greater than 5°C/min and a frequency of 1 Hz. Other heating rates and frequencies may be used but shall be reported. (See Appendix X2.)

NOTE 5—Calibration for temperature shall be performed under the conditions of heating rate and frequency at which the unknown specimens will be tested. This test method does not address the issues of frequency affects for polymeric transitions (such as the upwards shift of glass transition temperature with increasing frequency), and will only compensate for thermal lag within the measuring device.

10.3.5 Measure and record the ordinate signal, from 30°C below to 20°C above the melting point of the reference material. The calibration specimen may be equilibrated a minimum of 50°C below the melting transition, but adequate time to achieve thermal equilibrium in the specimen must be allowed.

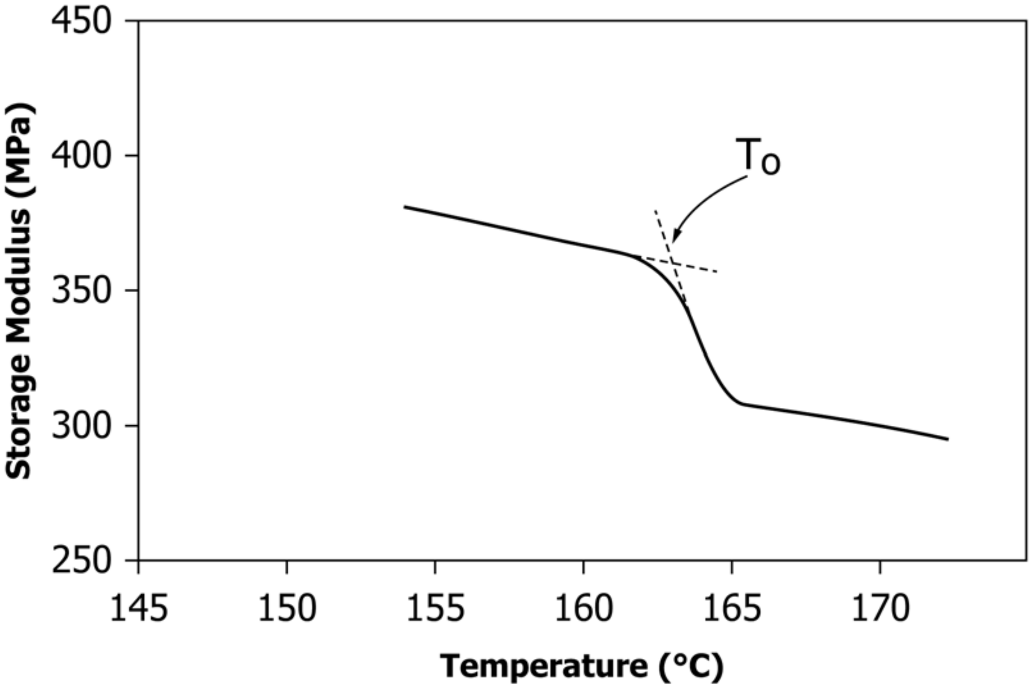

FIG. 1 Transition Temperature

10.4 *Method B—Calibration Where the Material is a Solid at Ambient Temperature:*

10.4.1 The calibration material must extend to the ends of the clamping geometry and must have uniform dimensions with respect to the width and thickness. Wrap the calibration material with polytetrafluoroethylene tape to a thickness of 0.5 mm. Other thicknesses may be used but shall be reported.

10.4.2 Mount the wrapped specimen into the apparatus according to the procedure recommended by the manufacturer as described in the operations manual.

NOTE 6—For specimen clamping arrangements where the specimen is not gripped on either end (for example, free bending between knife edges) the specimen must be rigid enough at the test start temperature to sustain initial loading. Alternatively, the calibration specimen, without encapsulation, can be placed between the knife edge and a substrate.

10.4.3 Maximum strain amplitude shall be within the linear viscoelastic range of the specimen. Strain of less than 1 % is recommended and shall not exceed 3 %.

10.4.4 Conduct the calibration experiments at the heating rate of interest, preferably 1°C/min but no greater than 5°C/min and a frequency of 1 Hz. Other heating rates and frequencies may be used but shall be reported.

NOTE 7—Calibration for temperature shall be performed under the conditions of heating rate and frequency at which the unknown specimens will be tested. This test method does not address the issues of frequency affects for polymeric transitions (such as the upwards shift of glass transition temperature with increasing frequency), and will only compensate for thermal lag within the measuring device.

10.4.5 Measure and record the ordinate signal, from 30°C below to 20°C above the melting point of the reference material. The calibration specimen may be equilibrated a minimum of 50°C below the melting transition, but adequate time to achieve thermal equilibrium in the specimen must be allowed.

10.5 *Method C—Calibration Where Material is Solid at Ambient Temperature:*

10.5.1 The calibration material must extend to the ends of the clamping geometry and must have uniform dimensions with respect to the width and thickness. Place a 0.5 mm thick coupon of insulating material on either side of the calibration material. Other thicknesses may be used but shall be reported.

10.5.2 Mount the sandwiched specimen into the apparatus according to the procedure recommended by the manufacturer as described in the operations manual.

NOTE 8—For specimen clamping arrangements where the specimen is not gripped on either end (for example, free bending between knife edges) the specimen must be rigid enough at the test start temperature to sustain initial loading. Alternatively, the calibration specimen, without encapsulation, can be placed between the knife edge and a substrate.

10.5.3 Maximum strain amplitude shall be within the linear viscoelastic range of the specimen. Strain of less than 1 % is recommended and shall not exceed 3 %.

10.5.4 Conduct the calibration experiments at the heating rate of interest, preferably 1°C/min but no greater than 5°C/min and a frequency of 1 Hz. Other heating rates and frequencies may be used but shall be reported.

NOTE 9—Calibration for temperature shall be performed under the conditions of heating rate and frequency at which the unknown specimens will be tested. This test method does not address the issues of frequency affects for polymeric transitions (such as the upwards shift of glass transition temperature with increasing frequency), and will only compensate for thermal lag within the measuring device.

10.5.5 Measure and record the ordinate signal, from 30°C below to 20°C above the melting point of the reference material. The calibration specimen may be equilibrated a minimum of 50°C below the melting transition, but adequate time to achieve thermal equilibrium in the specimen must be allowed.

11. Calculation

11.1 Take the transition temperature as the extrapolated onset to the sigmoidal change in the ordinate signal observed in the downward direction (see Fig. 1).

11.1.1 Construct a tangent to the ordinate signal curve below the transition temperature.

11.1.2 Construct a tangent to the ordinate signal curve at the inflection point approximately midway through the sigmoidal change associated with the transition.

11.1.3 Report the temperature at which these tangent lines intersect as reported as the observed transition temperature (T_o).

11.2 *Two Point Calibration:*

11.2.1 Using the standard temperature values from Table 2 and the corresponding onset temperatures obtained experimentally, calculate the slope and intercept using the following equations:

$$S = [T_r 1 - T_r 2]/[T_o 1 - T_o 2] \tag{2}$$

$$I = [(T_o\,1 \times T_r\,2) - (T_r 1 \times T_o 2)]/[T_o 1 - T_o 2] \tag{3}$$

where:

S = slope (nominal value = 1.0000),
I = intercept,
$T_r 1$ = reference transition temperature for Standard 1 (in Table 2),
$T_r 2$ = reference transition temperature for Standard 2 (in Table 2),
$T_o 1$ = experimentally observed transition onset temperatures for Standard 1, and
$T_o 2$ = experimentally observed transition onset temperature for Standard 2.

NOTE 10—The slope S is a dimensionless number whose value is independent of which temperature scale is used for I and T. I, in all cases, must have the same units as T_r1, T_r2, T_o1, and T_o2 that are, by necessity, consistent with each other.

11.2.2 S should be calculated to ±0.0001 units while I should be calculated to ±0.1°C.

11.2.3 Using the determined values for S and I, Eq 1 may be used to calculate the actual specimen transition temperature (T_t) from any experimentally observed transition temperature (T_o) for the particular DMA instrument employed.

11.3 *One Point Calibration:*

11.3.1 In this abbreviated procedure, a relationship between the extrapolated onset temperature as observed and the temperature as assigned by a temperature sensor is established. The operator should choose a calibration standard that is near the temperature of the transition or phenomenon under study.

11.3.2 Using the specimen handling techniques in 10.2 through 10.5, obtain the DMA curve for the calibration standard chosen from Table 2.

11.3.3 From the known melting temperature of the calibration material (see Table 2), calculate the value and sign of σ from the following equation:

$$\sigma = T_r - T_o \tag{4}$$

where:

T_r = reference transition temperature for standard (in Table 2),

T_o = experimentally observed transition onset temperature for standard, and

σ = correction factor for converting the observed temperature sensor temperature to actual sample temperature.

11.3.4 For the purpose of this abbreviated procedure, it is assumed that the relationship between the observed extrapolated onset temperature (T_o) and the actual specimen temperature is constant over the temperature range of interest. The value of σ is thus added to all observed measurements of transition temperatures for the particular instrument employed. That is:

$$T_t = T_o + \sigma \tag{5}$$

where:

T_t = temperature of transition to be assigned.

12. Report

12.1 Report the following information:

12.1.1 Complete description of the instrument (manufacturer and model number) as well as the data handling device used in these tests,

12.1.2 Complete identification of the temperature reference materials including source and purity,

12.1.3 Description of the dimensions, geometry, and material of the specimen. A description of the specimen holder should be specified as to composition, geometry and dimensions,

12.1.4 Complete description of the experimental conditions including identification of sample environment by gas flow rate, purity and composition, heating rate and description of any coolant used, and

12.1.5 Results of the calibration procedure including values for *S* and *I*. If the abbreviated one point calibration procedure was used, then the value of σ is given.

12.1.6 The specific dated version of these test methods.

13. Precision and Bias

13.1 The precision and bias of these test methods will be determined in an interlaboratory test program currently scheduled for 2016–2023. Anyone wishing to participate in the interlaboratory test should contact the ASTM International E37 Staff Manager.

14. Keywords

14.1 calibration; dynamic mechanical analyzer; temperature; thermal analysis

APPENDIXES

(Nonmandatory Information)

X1. ADDITIONAL PUBLICATIONS

X1.1 Aston, J. G., et al., "The heat capacity and enthalpy, heat of transition, fusion and vaporization, and vapor pressure of cyclopentane. Evidence for a non-planar structure," *Journal of the American Chemical Society*, Vol 65, 1943, p. 341.

X1.2 Finke, H. L., et al., "Low-temperature thermal data for the nine normal paraffin hydrocarbson from octane to hexadecane," *Journal of the American Chemical Society*, Vol 76, 1954, p. 33.

X1.3 Aston, J. G., et al., "The heat capacity and entropy, heats of transition, fusion and vaporization of cyclohexane. The vibrational frequencies of alicyclic ring systems," *Journal of the American Chemical Society*, Vol 65, 1943, p. 1135.

X1.4 Preston-Thomas, H., "The International Temperature Scale of 1990 (ITS-90)," *Metrologia*, Vol 27, 1990, pp. 3–10.

X1.5 Bedford, R. E., et al., "Recommended values of temperature on the International Temperature Scale of 1990 for a selected set of secondary reference points" *Metrologia*, Vol 33, 1996, pp. 133–154.

X2. ADDITIONAL NON-MANDITORY INFORMATION

X2.1 Sucseska, M., Liu, Z.-Y., Musanic, S.M., and Fiamengo, I., "Numerical modeling of sample-furnace thermal lag in dynamic mechanical analyzer," *Journal of Thermal Analysis and Calorimetry*, Vol 100, 2010, pp. 337–345, offers the following qualitative and quantitative observations about temperature calibration of dynamic mechanical analyzers.

X2.1.1 The temperature offset between actual and indicated temperature is a linear function of heating rate.

X2.1.2 The temperature offset is a function of and increases with temperature. Offset range from 3.0°C per each °C/min of heating rate at 32°C to 0.9°C per each °C/min of heating rate at –84°C.

X2.1.3 There is a small variation of temperature across the specimen thickness on the order of 0.2°C variation per each °C/min of heating rate.

X2.1.4 A minimum of 5 minutes of time at an isothermal temperature is required to achieve temperature equilibrium.

X3. ADDITIONAL NON-MANDATORY INFORMATION

INTRODUCTION

The following information is offered by C. Lotti and S. V. Canevarolo, in their paper "Temperature Calibration of a Dynamic-Mechanical Thermal Analyzer" (**1**).

X3.1 Selection of Plastic Encapsulating Tubing

X3.1.1 Plastic encapsulating tubes should be chosen for being inert and stable in the applicable temperature range and for having the ability to close the ends.

X3.1.2 Polypropylene (PP) was found to be useful for sub-ambient temperatures.

X3.1.3 Polytetrafluoroethylene (PTFE) was found to be useful for super-ambient temperatures.

X3.2 Tubing with an outer diameter of 3 mm and a wall thickness of 0.5 mm were found satisfactory.

NOTE X3.1—The cross sectional area should be greater than 44 % test specimen.

X3.3 Preparation of a Liquid Test Specimen in Polypropylene Tubing

X3.3.1 One end of the tube is sealed by squeezing it with heated pliers.

X3.3.2 Using a syringe, the tube is completely filled with the reference material liquid.

NOTE X3.2—Air bubbles trapped inside the tube should be avoided.

X3.3.3 The open end of the tube is sealed by squeezing it with heated pliers.

X3.4 Preparation of a Solid Test Specimen in Polytetrafluoroethylene Tubing

X3.4.1 One end of the tube is sealed with silicone glue, which acts as a plug.

X3.4.2 The solid reference material is inserted into the tube with the help of tongs.

X3.4.3 The whole tube is heated until the reference material is melted.

X3.4.4 Additional reference material is added and melted until the tube is nearly full.

NOTE X3.3—Air bubbles trapped inside the tube should be avoided.

X3.4.5 The open end of the tube is sealed with silicone glue.

REFERENCES

(1) Lotti, C., and Canevarolo, S. V., "Temperature Calibration of a Dynamic-Mechanical Thermal Analyzer," *Polymer Testing*, Vol 17, 1998, pp. 523–530.

(2) Kerbow, D. L., Poly(tetrafluoroethylene), in *Polymer Data Handbook*, J. E. Mark, Editor, 1999, Oxford university Press: New York, pp. 842–847.

(3) Tan, L.-S., Poly(pyromellitimide-1,4-diphenyl ether), in *Polymer Data Handbook*, J. E. Mark, Editor, 1999, Oxford University Press: New York, pp. 802–809.

(4) Fried, J. R., Poly(ether ether ketone), in *Polymer Data Handbook*, J.E. Mark, Editor, 1999, Oxford University Press: New York, pp. 466–470.

(5) Tye, R. P., and Salmon, D. R., "Thermal conductivity certified reference materials: Pyrex 7740 and polymethymethacrylate," in *Thermal Conductivity 26—Thermal Expansion 14*, R. B. Dinwiddie and R. Mannello, Editors, 2005, DEStech Publications: Lancaster, PA, pp. 437–451.

SUMMARY OF CHANGES

Committee E37 has identified the location of selected changes to this standard since the last issue (E1867 – 13) that may impact the use of this standard. (Approved Feb. 15, 2016.)

(1) Revised 1.1 to lower the temperature limit from –150°C to –100°C.

(2) Revised Table 2 to remove cyclopentane with its solid-solid transition.

(3) Modified Section 10 to include three test methods.

(4) Added Table 3 and its references.

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