



Standard Test Method for Assignment of the DSC Procedure for Determining T_g of a Polymer or an Elastomeric Compound¹

This standard is issued under the fixed designation D7426; 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 the assignment of the glass transition temperatures (T_g) of materials using differential scanning calorimetry.

1.2 This test method is applicable to amorphous materials, including thermosets or semicrystalline materials containing amorphous regions, that are stable and do not undergo decomposition or sublimation in the glass transition region.

1.3 The normal operating temperature range is from -120 to 500°C . The temperature range may be extended, depending upon the instrumentation used.

1.4 Computer or electronic-based instruments, techniques, or data treatment equivalent to this test method may also be used.

NOTE 1—Users of this test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user of this standard to determine the necessary equivalency prior to use.

1.5 ISO 11357–2 is equivalent to this test method.

1.6 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.7 *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.*

2. Referenced Documents

2.1 ASTM Standards:²

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

Current edition approved Nov. 1, 2013. Published January 2014. Originally approved in 2008. Last previous edition approved in 2008 as D7426 – 08. DOI: 10.1520/D7426-08R13.

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.

E473 Terminology Relating to Thermal Analysis and Rheology

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E967 Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers

E1142 Terminology Relating to Thermophysical Properties

2.2 *ISO Standard:*³

ISO 11357–2 Differential Scanning Calorimetry (DSC)-Part 2 Determination of Glass Transition Temperature

3. Terminology

3.1 Definitions:

3.1.1 The following terms are applicable to this test method and can be found in Terminology **E473** and Terminology **E1142**: *differential scanning calorimetry (DSC)*, *differential thermal analysis (DTA)*, *glass transition*, *glass transition temperature (T_g)*, and *specific heat capacity*.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 There are commonly used transition points associated with the glass transition region. (See **Fig. 1**.)

3.2.2 *extrapolated end temperature (T_e)*, $^\circ\text{C}$, n —the point of intersection of the tangent drawn at the point of greatest slope on the transition curve with the extrapolated baseline following the transition.

3.2.3 *extrapolated onset temperature (T_p)*, $^\circ\text{C}$, n —the point of intersection of the tangent drawn at the point of greatest slope on the transition curve with the extrapolated baseline prior to the transition.

3.2.4 *inflection temperature (T_i)*, $^\circ\text{C}$, n —the point on the thermal curve corresponding to the peak of the first derivative (with respect to time) of the parent thermal curve. This point corresponds to the inflection point of the parent thermal curve.

3.2.5 *midpoint temperature (T_m)*, $^\circ\text{C}$, n —the point on the thermal curve corresponding to $1/2$ the heat flow difference between the extrapolated onset and extrapolated end.

3.2.5.1 *Discussion*—The inflection point temperature (T_i) is

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

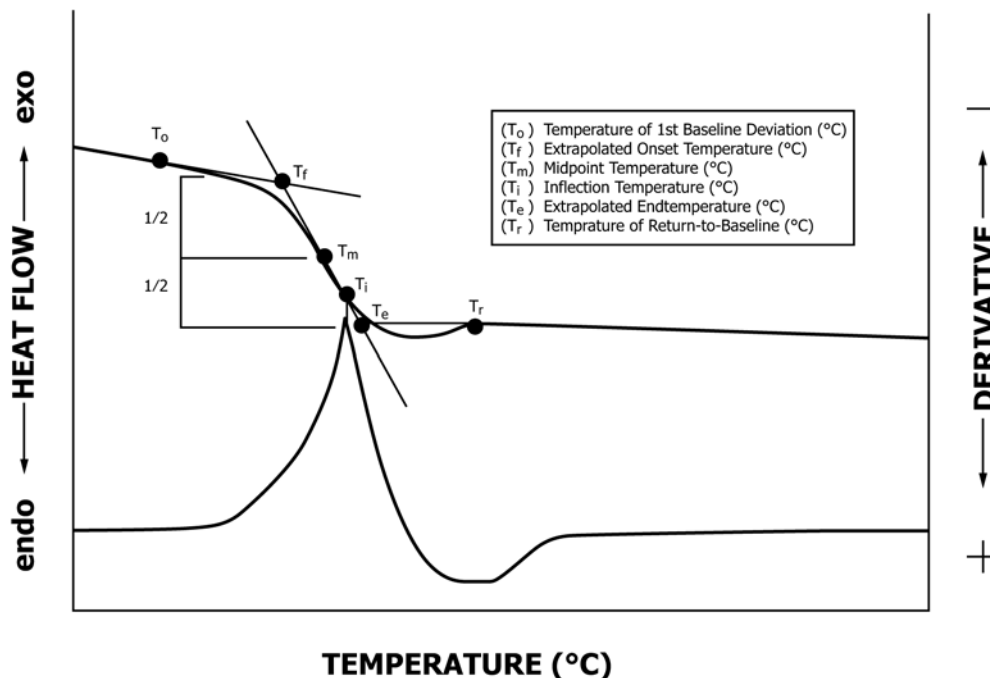


FIG. 1 Glass Transition Region Measured Temperatures

most easily determined from the first derivative curve and will be used as the glass transition temperature (see Fig. 1).

3.2.6 Two additional transition points are sometimes identified and are defined as follows:

3.2.7 *temperature of first deviation* (T_o), °C, n —the point of first detectable deviation from the extrapolated baseline prior to the transition.

3.2.8 *temperature of return to baseline* (T_r), °C, n —the point of last deviation from the extrapolated baseline beyond the transition.

4. Summary of Test Method

4.1 This test method involves continuously monitoring the difference in heat flow, or temperature between, a reference material and a test material when they are heated or cooled at a controlled rate through the glass transition region of the test material and analyzing the resultant thermal curve to provide the glass transition temperature.

5. Significance and Use

5.1 Differential scanning calorimetry provides a rapid test method for determining changes in specific heat capacity in a homogeneous material or domain. The glass transition is manifested as a step change in specific heat capacity. For amorphous and semi-crystalline materials the determination of the glass transition temperature may lead to important information about their thermal history, processing conditions, stability of phases, and progress of chemical reactions.

5.2 This test method is useful for research, quality control, and specification acceptance.

6. Interferences

6.1 A change in heating rates and cooling rates can affect the results. The presence of impurities will affect the transition,

particularly if an impurity tends to plasticize or form solid solutions, or is miscible in the post-transition phase. If domain size has an effect upon the detected transition temperature, the specimens to be compared should be of the same domain size.

6.2 In some cases the specimen may react with air during the temperature program causing an incorrect transition to be measured. Whenever this effect may be present, the test shall be run under either vacuum or an inert gas atmosphere. Since some materials degrade near the glass transition region, care must be taken to distinguish between degradation and glass transition.

6.3 Since milligram quantities of sample are used, it is essential to ensure that specimens are homogeneous and representative, so that appropriate sampling techniques are used.

7. Apparatus

7.1 *Differential Scanning Calorimeter*—The essential instrumentation required to provide the minimum differential scanning calorimetric capability for this method includes a test chamber composed of a furnace(s) to provide uniform controlled heating (cooling) of a specimen and reference to a constant temperature or at a constant rate over the temperature range from -120 to 500°C , a temperature sensor to provide an indication of the specimen temperature to $\pm 0.1^\circ\text{C}$, differential sensors to detect heat flow difference between the specimen and reference with a sensitivity of $\pm 50 \mu\text{W}$, a means of sustaining a test chamber environment of a purge gas of 10 to 100 mL/min within 4 mL/min, and a temperature controller capable of executing a specific temperature program by operating the furnace(s) between selected temperature limits at a rate of temperature change of up to $10^\circ\text{C}/\text{min}$ constant to $\pm 0.5^\circ\text{C}/\text{min}$.