Designation: E1356 – 08

StandardTest Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation E1356; 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 of materials using differential scanning calorimetry or differential thermal analysis.

1.2 This test method is applicable to amorphous materials or to partially crystalline 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 ISO standards 11357–2 is equivalent to this standard.

1.6 *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

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:*

11357–2 Differential Scanning Calorimetry (DSC)-Part 2 Determination of Glass Transition Temperature[3]

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).

3.2.1.1 *extrapolated end temperature, (T_e), °C*—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.

3.2.1.2 *extrapolated onset temperature, (T_f), °C*—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.

3.2.1.3 *inflection temperature, (T_i), °C*—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.

3.2.1.4 *midpoint temperature, (T_m), °C*—the point on the thermal curve corresponding to ½ the heat flow difference between the extrapolated onset and extrapolated end.

3.2.1.5 *Discussion*—Midpoint temperature is most commonly used as the glass transition temperature (see Fig. 1).

3.2.2 *Two additional transition points are sometimes identified and are defined: —*

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

[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Calorimetry and Mass Loss.

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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.

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

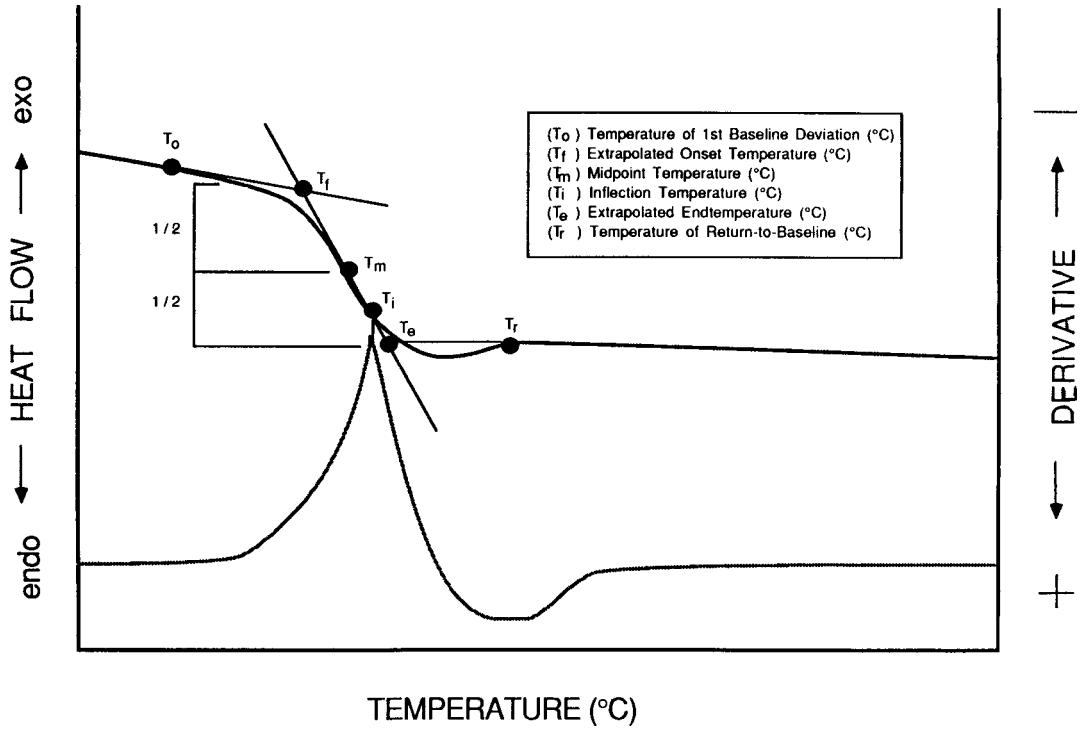

FIG. 1 Glass Transition Region Measured Temperatures

3.2.2.2 *temperature of return to baseline, (T_r), °C*—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 into, 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. The glass transition is manifested as a step change in specific heat capacity. For amorphous and semicrystalline materials the determination of the glass transition temperature may lead to important information about their thermal history, processing conditions, stability, progress of chemical reactions, and mechanical and electrical behavior.

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 particle size has an effect upon the detected transition temperature, the specimens to be compared should be of the same particle 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 ±0.1 °C, differential sensors to detect heat flow difference between the specimen and reference with a sensitivity of ± μW, a means of sustaining a *test chamber* environment of a purge gas of 10 to 100 mL/min within 4 mL/min, 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 20 °C/min constant to ± 0.5 °C/min.

7.2 *A Data Collection Device,* To provide a means of acquiring, storing, and displaying measured or calculated