Designation: E 1356 – 98

Standard Test Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry or Differential Thermal Analysis[1]

This standard is issued under the fixed designation E 1356; 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 Computer or electronic-based instruments, techniques, or data treatment equivalent to this test method may also be used.

1.5 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.

1.6 The values stated in SI units are to be regarded as the standard.

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:*

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[2]

E 473 Terminology Relating to Thermal Analysis[2]

E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods[2]

E 967 Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers[2]

E 1142 Terminology Relating to Thermophysical Properties[2]

3. Terminology

3.1 *Definitions:*

3.1.1 The following terms are applicable to this test method and can be found in Terminology E 473 and Terminology E 1142: *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.

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.

[1] This test method is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

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[2] *Annual Book of ASTM Standards,* Vol 09.01.

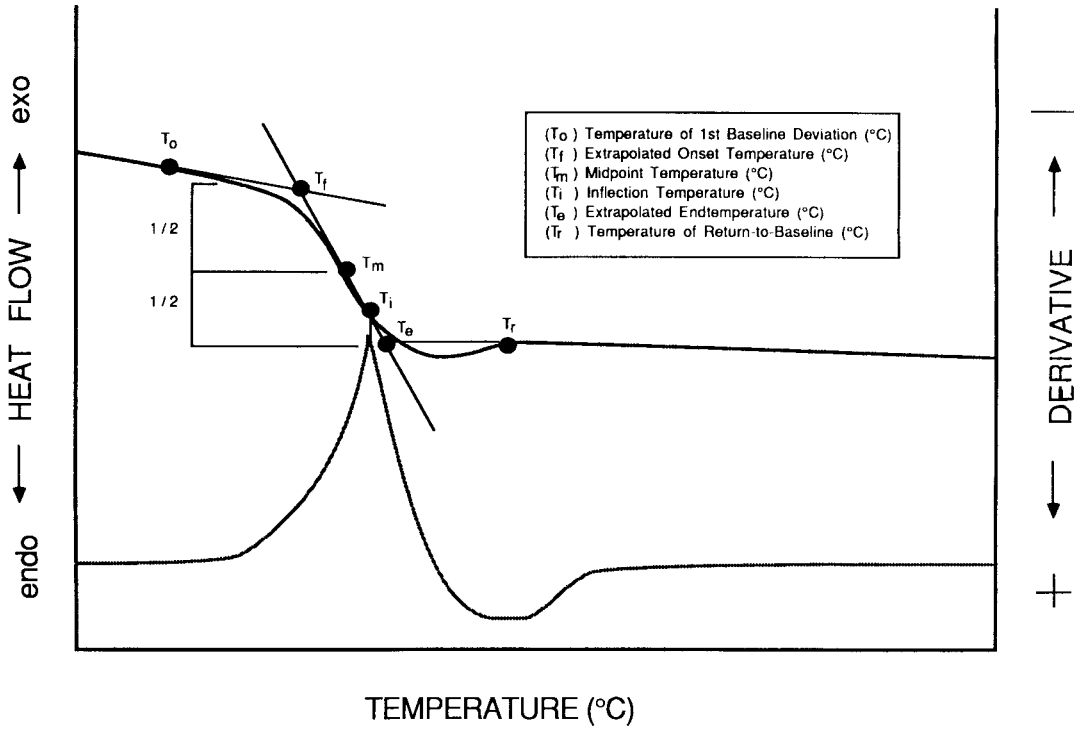

FIG. 1 Glass Transition Region Measured Temperatures

5. Significance and Use

5.1 Differential scanning calorimetry or differential thermal analysis 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 Apparatus shall be either type listed as follows:

7.1.1 *Differential Scanning Calorimeter,* capable of heating (or cooling) at rates up to at least 20°C/min and of automatically recording the differential heat flow input between a specimen and a reference material, both to the required sensitivity and precision, as given in Practice E 967.

7.1.2 *Differential Thermal Analyzer,* capable of heating (or cooling) at rates up to at least 20°C/min and of automatically recording the differential temperature between a specimen and a reference material, both to the required sensitivity and precision. Typically, the differential temperature sensitivity should be sufficient to provide specimen temperature readability to at least ±1°C.

7.2 *Specimen Capsules,* composed of aluminum or an inert material of high thermal conductivity, are used for DSC. For DTA, sample cups or tubes composed of borosilicate glass, alumina, or quartz may be used. The specimen capsules, pans, or tubes must not react with the specimen.

7.3 For ease of interpretation, an inert reference material with an heat capacity approximately equivalent to that of the specimen may be used. The inert reference material may often be an empty specimen capsule or tube.

7.4 *Nitrogen,* or other inert purge gas supply, of purity equal to or greater than 99.9 %.

7.5 *Analytical Balance,* with a capacity greater than 100 mg, capable of weighing to the nearest 0.01 mg.

8. Specimen Preparation

8.1 *Powders or Granules*—Avoid grinding if a preliminary thermal cycle as outlined in 10.2 is not performed. Grinding or