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StandardPractice for
Glass Transition Temperatures of Hydrocarbon Resins by Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation D6604; 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.

ε1 NOTE—Note 5 was added editorially in January 2009.

1. Scope

1.1 This practice covers determination of glass transition temperatures of hydrocarbon (HC) resins by differential scanning calorimetry (DSC).

1.2 This practice is applicable to HC resins as defined in Terminology D6640. The normal operating temperature range is from the cryogenic region to approximately 180°C. The temperature range can be extended.

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 Further discussion of glass transition can be found in Test Method D3418, and Test Method E1356.

1.5 *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]
D3418 Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry
D6640 Practice for Collection and Handling of Soils Obtained in Core Barrel Samplers for Environmental Investigations
E473 Terminology Relating to Thermal Analysis and Rheology

E1356 Test Method for Assignment of the Glass Transition Temperatures by Differential Scanning Calorimetry

3. Terminology

3.1 *Definitions:*

3.1.1 *differential scanning calorimetry (DSC), n*—A technique in which the difference in energy inputs into a substance and a reference material is measured as a function of temperature, while the substance and reference material are subjected to a controlled temperature program.

3.1.1.1 *Discussion*—The record is the DSC curve. Two modes, power-compensation DSC and heat-flux DSC, can be distinguished, depending on the method of measurement used.

3.2 For other definitions of terms relating to thermal analysis, see Terminology E473.

4. Summary of Practice

4.1 This practice consists of heating or cooling the test material at a controlled rate, in a controlled atmosphere, and continuously monitoring with a suitable sensing device, the difference in heat input between a reference material and a test material due to energy changes in the material. Absorption or release of energy marks a transition in the specimen resulting in a corresponding baseline shift in the heating or cooling curve.

5. Significance and Use

5.1 Thermal analysis provides a rapid method for determining transition temperatures in HC resins that possess them.

5.2 This practice is useful for both quality assurance and research.

6. Apparatus

6.1 *Differential Scanning Calorimeter*—An instrument capable of heating or cooling at rates up to 20 ± 1°C/minute and automatically recording the difference in input between the sample and a reference material to the required sensitivity and precision.

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[1] This practice is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.34 on Pine Chemicals and Hydrocarbon Resins.
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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.

6.2 *Sample Tubes or Pans*—Borosilicate glass tubes are used for certain applications and aluminum or other metal pans of high thermal conductivity for other applications.

6.3 *Reference Material*—Glass beads, indium, alumina, silicon carbide, or mercury in a hermetically sealed sample pan, or a material known to be unaffected by repeated heating and cooling and free from interfering transitions may be used. The thermal diffusivity should be as close as possible to that of the sample.

6.4 *Recording Charts or Software*—Temperature recording apparatus with suitable graduations for measurement of either temperature differential or energy differential versus temperature or time.

7. Reagents

7.1 *Nitrogen*—Inert gas for blanketing sample during testing.

7.2 *Indium,* (99.999 + % purity).

7.3 *Mercury,* (99.996 + % purity).

7.4 *Reagent Grade Benzoic Acid.*

8. Calibration

8.1 Using the same heating rate to be used for samples, calibrate the temperature scale of the apparatus with appropriate standard reference materials covering the temperature range of interest. For many commercial resins, the following substances cover this range:

Standards	Melting Point, °C
Benzoic acid	122.4
Indium	156.6 ± 0.4
Mercury	−38.9 ± 0.4

9. Sample Preparation

9.1 *Powdered or Granular Samples*—Avoid grinding if preliminary thermal cycle is not performed. (Grinding or similar techniques for size reduction often introduce thermal effects because of friction or orientation, or both, and thereby change the thermal history of the sample.)

10. Procedure

10.1 Use a sample weight appropriate for the material to be tested and the instrument used. In most cases, 10 to 20-mg sample weight is satisfactory.

NOTE 1—Since milligram quantities of sample are used, it is essential to ensure that samples are homogenous and representative. Also, particle size has an effect on the detected transition temperatures. Therefore particle size should be fairly consistent from sample to sample.

10.2 Perform and record a preliminary thermal cycle up to a temperature high enough to erase previous thermal and strain history, at a heating rate of 10°C/min.

NOTE 2—Use an inert gas purge such as nitrogen since the sample may react with oxygen during the temperature cycle causing an incorrect transition.

NOTE 3—An increase or decrease in heating rate from those specified may alter the results.

10.3 Hold this temperature for 10 min.

10.4 Quench cool to 50°C below the expected transition temperature of interest.

10.5 Hold this temperature for 10 min.

10.6 Repeat heating on the same sample at a rate of 10°C/min. and record the heating curve until all desired transitions have been completed.

11. Calculation

11.1 Measure corrected temperatures T_f, $T_e T$, T_m (see Fig. 1) as follows:

where:

T_f = extrapolated onset temperature °C,
T_e = extrapolated end temperature °C, and
T_m = midpoint temperature °C.

NOTE 4—A new baseline will probably be established after the transition. T_g can be defined as any of these measured values.

NOTE 5—In Fig. 1 the downward direction of the Heat Flow axis indicates an exotherm.

12. Report

12.1 Report the following information:

12.1.1 Complete identification and description of the material tested,

12.1.2 Description of the instrument used for the test,

12.1.3 Statement of the dimensions, geometry, and materials of the sample holder; and the average rate of linear temperature range,

12.1.4 Description of the temperature calibration procedure, and

12.1.5 State the type of T_g values reported.

13. Keywords

13.1 DSC; glass transition temperature; hydrocarbon resin; T_g