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Standard Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers[1]

This standard is issued under the fixed designation E 967; 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 describes the temperature calibration of differential thermal analyzers and differential scanning calorimeters over the temperature range from −40 to +2500 °C.

~~1.2Computer or electronic based instruments, techniques, or data manipulation equivalent to this test method may also be used.~~

~~1.3SI units are the standard.~~

~~1.4This test method is similar to ISO standard 11357–1.~~

~~1.5~~

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 test method is similar to ISO standard 11357–1.

1.4 *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 Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E 473 ~~Terminology Relating to Thermal Analysis~~

~~E968Practice for Heat Flow Calibration of Differential Scanning Calorimeters[2]~~ Terminology Relating to Thermal Analysis and Rheology

E 1142 ~~Terminology Relating to Thermophysical Properties[2]~~

~~E1953Practice for Description of Thermal Analysis Apparatus~~ Terminology Relating to Thermophysical Properties

2.2 *ISO Standard:*

11357–1 Plastics-Differential Scanning Calorimetry (DSC)-Part 1: General Principles

3. Terminology

3.1 Specific technical terms used in this test method are defined in Terminologies E 473 and E 1142.

4. Summary of Test Method

4.1 This test method consists of heating the calibration materials at a controlled rate in a controlled atmosphere through a region of known thermal transition. The heat flow into the calibration material or the difference of temperature between the calibration material and a reference sample and a reference material is monitored and continuously recorded. A transition is marked by the absorption of energy by the specimen resulting in a corresponding endothermic peak in the heating curve.

NOTE 1—Heat flow calibrations are sometimes determined in conjunction with temperature calibration. Some differential scanning calorimeters permit both heat flow and temperature calibrations to be obtained from the same experimental procedure.

5. Significance and Use

5.1 Differential scanning calorimeters and differential thermal analyzers are used to determine the transition temperatures of materials. For this information to be meaningful in an absolute sense, temperature calibration of the apparatus or comparison of the resulting data to that of known standard materials is required.

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[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 Thermal Test Methods and Practices.

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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* ~~, Vol 14.02.~~volume information, refer to the standard's Document Summary page on the ASTM website.

5.2 This test method is useful in calibrating the temperature axis of differential scanning calorimeters and differential thermal analyzers.

6. Apparatus

6.1 Apparatus shall be of either type listed below:

6.1.1 *Differential Scanning Calorimeter (DSC)*, capable of heating a test specimen and a reference material at a controlled rate and of automatically recording the differential heat flow between the sample and the reference material to the required sensitivity and precision.

6.1.1.1 *A Furnace(s)*, to provide uniform controlled heating or cooling of a specimen and reference to a constant temperature or at a constant rate within the applicable temperature range of this test method.

6.1.1.2 *A Temperature Sensor*, to provide an indication of the specimen temperature.

6.1.1.3 *Differential sensors*, to detect a heat flow (power) difference between the specimen and reference.

6.1.1.4 *Test Chamber Environment*~~—a~~, a means of sustaining a test chamber environment of nitrogen or other inert purge gas at a purge rate of 10 to 50 mL/min.

6.1.1.5 *A Temperature Controller*~~—capable~~, capable of executing a specific temperature program by operating the furnace(s) between selected temperature limits at a rate of temperature change of 10K/min.

6.1.1.6 ~~*A Recording Device*—capable of recording and displaying on the Y-axis and fraction of the heat flow signal (DSC curve) including the signal noise as a function of any fraction of the temperature (or time) signal on the X-axis including the signal noise.~~ Data Collection Device, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for DSC are heat flow, temperature, and time.

6.1.2 *Differential Thermal Analyzer (DTA)*, capable of heating a test specimen and reference material at a controlled rate and of automatically recording the differential temperature between sample and reference material both to the required sensitivity and precision.

6.2 *Containers (pans, crucibles, vials, lids, closures, seals, etc.)*, that are inert to the specimen and reference materials and that are of suitable structural shape and integrity to contain the specimen and reference in accordance with the specific requirements of this test method.

6.3 *Nitrogen*, or other inert purge gas supply.

6.4 *A Balance*, to ~~wiegh~~weigh specimens or containers (pans, crucibles, vials, etc.), or both to ± 0.1 mg. The balance should have a capacity greater than 20 mg.

7. Precautions

7.1 Toxic or corrosive effluents, or both, may be released when heating some material and could be harmful to personnel and to apparatus.

7.2 This test method assumes linear temperature indication. Care must be taken in the application of this test method to ensure that calibration points are taken sufficiently close together so that linear temperature indication may be approximated. Linear temperature indications means that there exists a linear, or first order, dependence on the temperature determined by the instrument's temperature sensor on the true temperature of the sample material in its container and that this relation is adequately expressed by ~~Eq.~~Eq 1.

8. Calibration Materials

8.1 For the temperature range covered by many applications, the melting transition of >99.99 % pure materials in Table 1 may be used for calibration.

9. Procedure

9.1 *Two Point Calibration*:

9.1.1 Select two calibration materials from Table 1, with melting temperatures one above and one below the temperature range of interest. The calibration materials should be as close to the temperature range of interest as practical.

9.1.2 Determine the apparent transition temperature for each calibration material.

9.1.2.1 Into a clean specimen holder, place a 5 to 15-mg weighed amount of calibration material. Other specimen masses may be used but must be indicated in the report.

9.1.2.2 Load the specimen into the instrument chamber, purge the chamber with dry nitrogen (or other inert gas) at a flow rate of 10 to 50 cm^3/min throughout the experiment.

9.1.2.3 Heat (or cool) the calibration material rapidly to 30 °C below the calibration temperature and allow to stabilize.

9.1.2.4 Heat the calibration material at 10 °C/min through the transition until baseline is reestablished above the transition. Other heating rates may be used but must be noted in the report. Record the resulting thermal curve.

NOTE 2—Temperature scale calibration may be affected by temperature scan rate, specimen holder, purge gas and purge gas flow rate. The temperature calibration shall be made under the same conditions used for test specimens.

9.1.2.5 From the resultant curve, measure the temperatures for the desired points on the curve, T_e, T_p (see Fig. 1) retaining all available decimal places.