



Designation: E 967 – 03

Standard Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers¹

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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 $+2500^{\circ}\text{C}$.

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

1.3 SI units are the standard.

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

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.* Specific precautionary statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

E 473 Terminology Relating to Thermal Analysis²

E 968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters²

E 1142 Terminology Relating to Thermophysical Properties²

E 1953 Practice for Description of Thermal Analysis Apparatus³

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.

¹ This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E 37.01 on Test Methods and Practices.

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² *Annual Book of ASTM Standards*, Vol 14.02.

³ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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.

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 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 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.

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 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. 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.

TABLE 1 Melting Temperature of Calibration Material

NOTE 1—The values in Table 1 were determined under special, highly accurate steady state conditions that are not attainable or applicable to thermal analysis techniques. The actual precision of this test method is given in Section 12 of this test method.

Calibration Material	Melting Temperature ^A	
	(°C)	(K)
Mercury	-38.834	234.316
Water	0.01 ^B	273.16 ^B
Phenoxybenzene	26.87	300.02
Gallium	29.765 ^B	302.915 ^B
Benzoic Acid	122.37	395.52
Indium	156.598 ^B	429.748 ^B
Tin ^C	231.928 ^B	505.078 ^B
Bismuth	271.442	544.592
Lead	327.502	600.652
Zinc	419.527 ^B	692.677 ^B
Antimony	630.74	903.89
Aluminum	660.32 ^B	933.47 ^B
Silver	961.78 ^B	1234.93 ^B
Gold	1064.18 ^B	1337.33 ^B
Copper	1084.62 ^B	1357.77 ^B
Nickel	1455	1728
Cobalt	1494	1767
Palladium	1554	1827
Platinum	1772	2045
Rhodium	1963	2236
Iridium	2447	2720

^A F. D. Rossini, *Pure Applied Chemistry*, Vol 22, 1970, pg. 557.

^B The melting temperatures of these materials have been selected as primary fixed points for the International Practical Temperature Scale of 1990. See *Guidelines for Realizing the International Practical Temperature Scale of 1990 (ITS-90)*, by B. W. Mangum and G. T. Furukawa, NIST Technical Note 1265.

^C Some materials have different crystalline forms (for example, tin) or may react with the container. These calibration materials should be discarded after their initial melt.

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 cm³/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.

where:

T_e = extrapolated onset temperature for fusion, °C

T_p = melting peak temperature, °C

NOTE 3—The actual temperature displayed on the temperature axis differs depending upon the instrument type; for example, sample temperature, program temperature, sample program temperature average. Follow the instructions of the particular instrument manufacturer to obtain sample temperature at the point of interest.

NOTE 4—The available precision of the temperature measurements depends upon instrument capabilities and the temperature range of the