ASTM

Designation: E 967 – 97

Standard Practice 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 practice covers the temperature calibration of differential thermal analyzers and differential scanning calorimeters over the temperature range from –40 to +2500°C.

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

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

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:*
E 473 Terminology Relating to Thermal Analysis[2]
E 968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters[2]
E 1142 Terminology Relating to Thermophysical Properties[2]

3. Terminology

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

4. Summary of Practice

4.1 This practice consists of heating the calibration materials at a controlled rate in a controlled atmosphere through a region of known thermal transition. The difference in heat flow or temperature between the calibration material 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 and quantitative differential thermal analyzers 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 practice 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.

6.1.2 *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 *Specimen Pans*, for DSC, composed of clean aluminum or of other high thermal conductivity material. For DTA, specimen cups or tubes composed of clean borosilicate glass, alumina, platinum, aluminum, or quartz may be used. Specimen containers composed of tungsten, graphite or silicon carbide/nitride may be used above 1600°C. The specimen pans or tubes must not react with the specimen and must not melt under the temperatures of the test.

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

6.4 *Analytical Balance*, with a capacity greater than 20 mg, capable of weighing to the nearest 0.1 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 Some differential thermal analyzers and differential

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scanning calorimeters have nonlinear temperature indication over some temperature regions of applications. This practice assumes linear temperature indication. Care must be taken in the application of this practice to ensure that calibration points are taken sufficiently close together so that linear temperature indication may be approximated.

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.

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 practice.

Calibration Material	Melting Temperature[A]	
	(°C)	(K)
Mercury	–38.862	234.288
Water	0.01[B]	273.16[B]
Phenoxybenzene	26.87	300.02
Benzoic Acid	122.37	395.52
Indium	156.60[B]	429.73[B]
Tin[C]	231.93[B]	505.08[B]
Bismuth	271.442	544.592
Cadmium[D]	321.108	594.258
Lead	327.502	600.652
Zinc	419.53[B]	692.68[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.
[D] Some materials (for example, cadmium) have an appreciable vapor pressure at their melting temperature and could be harmful to personnel and to apparatus.

9.1.2.3 Select the appropriate sensitivity for energy input or temperature difference axis to yield a 40 to 95 % deflection of the recorder.

NOTE 2—Preanalysis on a similar specimen may be run to yield this information.

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

9.1.2.5 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 3—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.

9.1.2.6 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 4—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 5—The available precision of the temperature measurements depends upon instrument capabilities and the temperature range of the test. Below 300°C, measurements to ±0.5°C are common while at greater than 700°C,± 2°C is reasonable.

NOTE 6—For high-purity crystalline materials (not polymers), T_e is taken as the transition temperature when measured by differential scanning calorimeters and other instruments where the test specimen is removed from the temperature sensor. For instruments in which the temperature sensor is in intimate contact with the sample, (such as some differential thermal analyzers), T_p is taken as the transition temperature.

9.1.3 Using the apparent transition temperatures thus obtained, calculate the slope (S) and intercept (I) of the calibration Eq 1 (see Section 10). The slope and intercept values reported should be mean values from duplicate determinations based on separate specimens.

9.2 *One-Point Calibration*:

9.2.1 If the slope value (S) previously has been determined in 9.1 (using the two-point calibration calculation in 10.2) to be sufficiently close to 1.0000, a one-point calibration procedure may be used.

NOTE 7—If the slope value differs by only 1 % from linearity (that is, $S < 0.9900$ or $S > 1.0100$), a 1°C error will be produced if the test temperature differs by 100°C from the calibration temperature.

9.2.2 Select a calibration material from Table 1. The calibration temperature should be centered as close as practical within the temperature range of interest.

9.2.3 Determine the apparent transition temperatures of the calibration material using steps 9.1.2.1-9.1.2.6.

9.2.4 Using the apparent transition temperature thus obtained, calculate the intercept (I) of the calibration equation using all available decimal places. The value reported should