Designation: E 1952 – 01

Standard Test Method for Thermal Conductivity and Thermal Diffusivity by Modulated Temperature Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation E 1952; 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 determination of thermal determination of thermal conductivity of homogeneous, nonporous solid materials in the range of 0.10 to 1.0 W/(K • m) by modulated temperature differential scanning calorimeter. This range includes many polymeric, glass, and ceramic materials. Thermal diffusivity, which is related to thermal conductivity through specific heat capacity and density, may also be derived. Thermal conductivity and diffusivity can be determined at one or more temperatures over the range of 0 to 90 °C.

1.2 Electronic instrumentation or automated data analysis and reduction systems or treatments equivalent to this test method may be used.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information purposes only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 473 Terminology Relating to Thermal Analysis[2]

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

E 968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters[2]

E 1142 Terminology Relating to Thermophysical Properties[2]

E 1231 Practice for Calculation of Hazard Potential Figures-of-Merit for Thermally Unstable Materials[2]

3. Terminology

3.1 *Definitions:*

3.1.1 Specific technical terms used in this document are defined in Terminologies E 473 and E 1142.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *modulated temperature differential scanning calorimeter*—a version of differential scanning calorimetry that provides a sinusoidally varying temperature program to the test specimen in addition to the traditional isothermal or temperature ramp programs. Results from analysis shall include apparent and specific heat capacity.

4. Summary of Test Method

4.1 The heat capacity of a test specimen may be determined using the modulated temperature approach in which an oscillatory or periodically repeating temperature program (around an average temperature) is imposed upon a test specimen producing an oscillatory (periodic) heat flow into or out of the specimen. The heat capacity of the test specimen may be obtained from the amplitude of the resultant heat flow divided by the amplitude of the oscillatory (periodic) temperature that produces it. Specific heat capacity is obtained by normalizing the heat capacity to specimen mass.

4.1.1 The accuracy of the heat capacity thus obtained depends upon experimental conditions. When a thin test specimen encapsulated in a specimen pan of high thermal conductivity is treated with temperature oscillations of long period (low frequency), the test specimen is assumed to achieve a uniform temperature distribution and the resultant heat capacity information will be comparable with those of other non-oscillatory test methods.

4.1.2 When one end of a thick test specimen is exposed to the temperature oscillations of short period (high frequency), the test specimen will achieve a temperature distribution over its length related to its thermal diffusivity.

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

Current edition approved August 10, 2001. Published October 2001. Originally published as E 1952 – 98. Last previous edition E 1952 – 98.

The process described in this test method is covered by a patent (Marcus, S. M. and Reading, M., U. S. Patent 5 335 993, 1994) held by TA Instruments, Inc., 109 Lukens Drive, New Castle DE 19720. Interested parties are invited to submit information regarding the identification of acceptable alternatives to this patented method to the Committee on Standards, ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken PA 19428-2959. Your comments will receive careful considerations at a meeting of the responsible technical committee which you may attend.

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

4.1.3 The apparent heat capacity information thus obtained is lower than that of the uniform temperature distribution case described above and is proportional to the square root of thermal conductivity of the test specimens. [3] The thermal conductivity of the test specimen may be derived from the apparent heat capacity of a thick specimen, the actual heat capacity of a thin specimen, and a series of geometric and experimental constants.

4.2 If the thermal conductivity of the test specimen is low, approaching that of the purge gas surrounding it, a correction to the measured thermal conductivity is required to compensate for heat losses from the thick test specimen.

4.3 Thermal diffusivity is derived from the determined thermal conductivity, specific heat capacity, and density of the test specimen.

5. Significance and Use

5.1 Thermal conductivity is a useful design parameter for the rate of heat transfer through a material.

5.2 The results of this test method may be used for design purposes, service evaluation, manufacturing control, research and development, and hazard evaluation. (See Practice E 1231.)

6. Interferences

6.1 Because the specimen size used in thermal analysis is on the order of 10 to 100 mg, care must be taken to ensure it is homogeneous or representative of the material, or both.

6.2 The calculation of thermal conductivity requires knowledge of this specimen geometry. This test method requires a specific specimen size and shape. Other geometries may be used with the appropriate modifications to the calculating equations.

7. Apparatus

7.1 A modulated temperature differential scanning calorimeter consisting of:

7.1.1 *A Differential Scanning Calorimetry (DSC) Test Chamber,* of (1) a furnace to provide uniform controlled heating/cooling of a specimen and reference to a constant temperature or at a constant rate within the applicable range of this test method; (2) a temperature sensor (or other signal source) to provide an indication of the specimen temperature readable to 0.01°C; (3) a differential sensor to detect a heat flow difference between the specimen and reference equivalent to 0.001 mW; and (4) a means of sustaining a test temperature environment of inert nitrogen purge gas at a rate of 50 mL/min± 10 mL/min.

7.1.2 *A Temperature Controller,* capable of executing a specific temperature program by (1) operating the furnace between selected temperature limits at a rate of temperature change of 1°C/min, (2) holding at an isothermal temperature over the temperature range of 0 to 90 °C within ± 0.1°C, and (3) sinusoidal varying temperature with an amplitude of ±0.2 to 0.7°C and a period of 60 to 100 seconds (frequency of 10 to 16 mHz).

7.1.3 *A Calculating Device,* capable of transforming the experimentally determined modulated temperature and modulated specimen heat flow signals into the required continuous output forms of heat capacity (preferably in units of mJ/°C), specific heat capacity (preferably in units of J/(g°C)), and average test temperature to the required accuracy and precision.

7.1.4 *A Recording Device,* to record and display heat capacity, specific heat capacity, and average temperature on the ordinate (Y axis) and elapsed time (preferably in units of minutes) on the abscissa (X axis) with a sensitivity of 0.001 mJ/K for heat capacity, 0.001 J/(g • K) for specific heat capacity, 0.1°C for average temperature, and 0.1 min for time.

7.1.5 *A Coolant System,* to provide oscillatory heating and cooling rates of at least 3°C/min.

7.1.6 *Inert Nitrogen,* or other low conductivity purge gas flowing at a rate of 50 mL/min (see 7.1.1).

NOTE 1—Helium, a commonly used purge gas, is unacceptable for this purpose, due to its very high thermal conductivity which results in reduced range, precision, and accuracy.

7.2 *A Balance,* with a range of at least 200 mg to weigh specimens or containers, or both, (pans, crucibles, etc.) to ±0.01 mg.

7.3 *Calipers* or other length-measuring device with a range greater than 4 mm, readable to 0.01 mm.

7.4 *Sapphire Disk Calibration Material*, 10 to 15 mg.

7.5 *Polystyrene Thermal Conductivity Calibration Material,* of known thermal conductivity and specific heat capacity, in the shape of a right circular cylinder, 6.3 ± 0.2 mm in diameter and 3.5 ± 0.3 mm thickness.

7.5.1 *Polystyrene Specific Heat Capacity Reference Material,* composed of the same material as the thermal conductivity calibration material, in the shape of a right circular cylinder or disk, 6.3 ± 0.2 mm in diameter and 0.4 ± 0.1 mm in thickness.

7.6 *Circular Aluminum Disk*, 6.3 mm in diameter and 0.01 mm or thinner in thickness.

7.7 *Containers* (pans, crucibles, etc.) that are inert to the specimen and are of suitable structural shape and integrity to contain the specimen in accordance with the specific requirements of this test method.

7.8 *Silicone Heat Transfer Fluid,* with no thermal transitions over the temperature range from -10 to 100°C.

NOTE 2—Silicone oil with a viscosity of about 1 Pa • s (10 poise) has been found satisfactory for this application.

7.9 While not required, users may find the following optional apparatus and materials useful for this determination.

7.9.1 *Polymeric Thermal Conductivity Performance Material*, a right circular cylinder, 6.3 ± 0.2 mm in diameter and 3.5 ± 0.3 mm in length.

7.9.2 *Polymetric Specific Heat Capacity Reference Material,* composed of the same material as the thermal conductivity standard reference material, a right circular cylinder or disk, 6.3 ± 0.2 mm in diameter and 0.4 ± 0.1 mm in thickness.

8. Sampling

8.1 Select two right circular cylinders, both nominally 6.3 mm in diameter. The first of these test specimens is nominally

[3] Marcus, S.M., and Blaine, R.L., *Thermochim. Acta*, Vol 243, 1994, pp. 231-239.

0.4 mm thick and the second is nominally 3.5 mm thick. These test specimens are most conveniently obtained by cutting from 0.25 in. diameter rod, a common material form.

NOTE 3—Other fabrication techniques, such as cutting from sheet stock using cork borers, machining from stock, or molding may also be used.

8.1.1 Polish the circular end surfaces of the test specimens smooth and parallel to within ± 30 μm with 600 grit emery paper.

9. Calibration

9.1 Calibrate the temperature signal from the apparatus in accordance with Practice E 967 using an indium reference material and a heating rate of 1 °C/min.

9.2 Calibrate the heat flow signal from the apparatus in accordance with Practice E 968 using an indium reference material.

9.3 Calibrate the apparatus for heat capacity measurements in accordance with the instructions of the manufacturer as described in the instrument manual using isothermal temperature conditions (at the mid point of the temperature range of interest), the sapphire calibration material (from 7.4) ±0.5 °C amplitude and 80 s period (12.5 mHz frequency).

10. Procedure

10.1 Measure thermal conductivity under quasi-isothermal conditions at an operator-selected temperature within the range from 0 to 90 °C. If measurements at additional temperatures are desired, repeat the procedure at those additional temperatures.

10.2 A common set of experimental conditions are used for each measurement:

10.2.1 Select the modulated mode on the DSC and record the heat capacity signal. Equilibrate the apparatus at the test temperature selected by the operator. Modulate the temperature with an amplitude of ±0.5 °C and a period (*P*) of 80 s (12.5 mHz). After 15 min equilibration time, record the average test temperature (*T*) and the specific heat capacity (C_p) or apparent heat capacity (*C*) as called for in the appropriate section.

10.3 Determine the thermal conductivity calibration factor, *D*.

10.3.1 Weigh the thin (0.4 mm) polystyrene (or other) calibration disk (from 7.5.1); record the mass as *m*. Enter it as an experimental parameter into the apparatus calculator. Encapsulate the thin polystyrene calibration disk in a standard aluminum sample container with lid.

10.3.2 Place the encapsulated test specimen in the DSC on the specimen sensor. Use an empty aluminum container and lid on the reference side.

NOTE 4—Matching the combined weights of the reference container and lid to those of the specimen container and lid within ±0.1 mg produces the best results.

10.3.3 Measure the heat capacity of the thin polystyrene calibration material using the conditions of 10.2.1. Record the specific heat capacity (C_p) in units of J/(g • K).

NOTE 5—This value for the specific heat capacity of polystyrene may be compared against the literature values listed in Table 1 as a performance criteria test.

TABLE 1 Polystyrene Specific Heat Capacity[A]

Temperature		Specific Heat Capacity[B]
(°C)	(K)	(J/(g • K))
6.8	280.0	1.1326
16.8	290.0	1.1775
26.8	300.0	1.2230
36.8	310.0	1.2691
46.8	320.0	1.3156
50.0	323.2	1.3305
56.8	330.0	1.3626
66.8	340.0	1.4100
76.8	350.0	1.4577
86.8	360.0	1.5056
96.8	370.0	1.5539

[A] Gaur, U., and Wunderlich, B, *J. Phys. Chem. Ref. Data*, Vol 11 (2), 1982, p. 313.

[B] The values in this table were determined under special highly accurate test conditions that are not attainable by or applicable to this test method. The actual precision of this test method is given in Section 13.

10.3.4 Weight the thick (3.5 mm) polystyrene calibration disk (from 7.5); record the mass as *m*; and enter it into the experimental parameters screen on the measuring apparatus.

10.3.5 Measure and record the diameter (*d*) and length (*L*) of the polystyrene calibration test specimen.

10.3.6 Place a small drop of silicone oil on the DSC sample and reference sensors. Place a thin aluminum disk over each drop of oil. Carefully place the thick sample (which has been wetted with oil on the bottom side) on the aluminum disk covering the sample sensor.

NOTE 6—Ensure that silicone oil does not change the characteristics of the test specimen.

NOTE 7—Use the minimum amount of oil that will provide complete contact between the specimen disk and the DSC sensor. Wiping the surface with a cotton swab moistened with the oil is usually sufficient.

10.3.7 Measure the apparent heat capacity of the specimen in accordance with the conditions of 10.2.1. Record the apparent heat capacity (*C*) in the units of mJ/°C.

10.3.8 Using the values of *P* (from 10.2.1), C_p (from 10.3.3); and *m*, *L*, and *d* (from 10.3.4 and 10.3.5), calculate the observed thermal conductivity (λ_o) for polystyrene using Eq 1 (see 11.1).

NOTE 8—An example calculation is presented in 11.5.1.

10.3.9 Determine the value for thermal conductivity of polystyrene (λ_r) for the corresponding temperature (*T*) (from 10.2.1) from Table 2, linearly interpolating between values if necessary.

10.3.10 Using the values for λ_o from 10.3.8 and the value for λ_r from 10.3.9, calculate the thermal conductivity calibration constant (*D*) for temperature *T* using Eq 2.

NOTE 9—An example calculation is presented in 11.5.2.

NOTE 10—Typical values for *D* range from approximately 0.0100 to 0.0500 W/(m • K).

10.4 Determine the thermal conductivity of the test specimen.

10.4.1 Weigh the thin (0.4 mm) test specimen and record the mass as *m*. Enter this value into the experimental parameters screen of the apparatus.

10.4.2 Encapsulate the thin specimen in a standard aluminum sample container with lid.