Designation: ~~E1952 – 17~~ E1952 – 23

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

This standard is issued under the fixed designation E1952; 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*~~Scope

1.1 This test method describes the determination of thermal conductivity of homogeneous, non-porous solid materials in the range of 0.10 ~~W/(K•m)~~W/(K·m) to 1.0 W~~/(K•m)~~/(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°C to 90°C.~~0 °C to 90 °C.

1.2 The values stated in SI units are to be regarded as standard. ~~The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.~~

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E473 Terminology Relating to Thermal Analysis and Rheology
E967 Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers
E968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters (Withdrawn 2023)[3]
E1142 Terminology Relating to Thermophysical Properties
E1231 Practice for Calculation of Hazard Potential Figures of Merit for Thermally Unstable Materials
E2161 Terminology Relating to Performance Validation in Thermal Analysis and Rheology

3. Terminology

3.1 *Definitions:*

3.1.1 Specific technical terms used in this test method are defined in Terminologies E473, E1142, and E2161 including *calibration,*

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*A Summary of Changes section appears at the end of this standard.

differential scanning calorimetry, heat capacity, modulated temperature, precision, reference material, relative standard deviation, repeatability, reproducibility, specific heat capacity, standard deviation, thermal analysis, thermal conductance, and *thermal conductivity*.

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.

4.1.3 The apparent heat capacity information thus obtained is lowers than that of the uniform temperature distribution case described above and is proportional to the square root of thermal conductivity of the test specimens (**1**).[4] 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 E1231.)

6. Interferences

6.1 Because the specimen size used in thermal analysis is on the order of 10 mg 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:

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[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.

7.1.1 A *Differential Scanning Calorimetry (DSC)(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;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,1 °C∕min, (*2*) holding at an isothermal temperature over the temperature range of 0°C to 90°C within ±0.1°C,0 °C to 90 °C within ±0.1 °C, and (*3*) sinusoidal varying temperature with an amplitude of ±0.2°C to 0.7°C±0.2 °C to 0.7 °C and a period of 60 s to 100 s (frequency of 10 mHz to 16 mHz).

Note 1—The upper thermal conductivity achievable by this method is extended to 4 W (K • m)(K·m) for instruments capable of 20 s periods (frequency of 50 mHz) (**2**).

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/K), specific heat capacity (preferably in units of J/(g • K)), J/(g·K)), and average test temperature to the required accuracy and precision.

7.1.4 A *Data Collecting Device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required are heat flow, temperature, time, heat capacity, specific heat capacity, and average temperature with a sensitivity of 0.001 mJ∕K for heat capacity, 0.001 J∕(g • K)(g·K) for specific heat capacity, 0.01°C0.01 °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.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 2—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*, 20 mg to 30 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 mm6.3 mm ± 0.2 mm in diameter and 3.5 ± 0.3 mm3.5 mm ± 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 mm6.3 mm ± 0.2 mm in diameter and 0.4 ± 0.1 mm0.4 mm ± 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°C to 100°C.−10 °C to 100 °C.

Note 3—Silicone oil with a viscosity of about 1 Pa • s1 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~~6.3 mm ± 0.2 mm in diameter and ~~3.5 ± 0.3 mm~~3.5 mm ± 0.3 mm in length.

7.9.2 *Polymeric 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~~6.3 mm ± 0.2 mm in diameter and ~~0.4 ± 0.1 mm~~0.4 mm ± 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 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 4—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 E967 using an indium reference material and a heating rate of ~~1°C/min.~~1 °C/min.

9.2 Calibrate the heat flow signal from the apparatus in accordance with Practice E968 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~~±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°C to 90°C.~~0 °C 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~~±0.5 °C and a period (P) of 80 s (12.5 mHz). (See Note 5.) 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.~~m1. 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 5—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).~~J/(g·K).

NOTE 6—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.

10.3.4 Weigh the thick (3.5 mm) polystyrene calibration disk (from 7.5); record the mass as ~~m,~~2; 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 Moisten the DSC sample and reference sensors with silicone oil. Place a thin aluminum disk over each sensor. Carefully place the thick sample (which has been moistened with oil on the bottom side) on the aluminum disk covering the sample sensor.

NOTE 7—Ensure that silicone oil does not change the characteristics of the test specimen. It may leave a residue that must be cleaned before alternative apparatus use. Xylene is a suitable solvent for cleaning.

NOTE 8—A cotton swab may be wetted with silicon oil and the pressed between the fingers to remove any excess oil. The "moist" cotton swab may be passed once over the surface to "wet" it with the oil.

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/K.

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 9—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 10—An example calculation is presented in 11.5.2.

NOTE 11—Typical values for D range from approximately ~~0.0100~~10 mW ~~W/(m · K) to 0.0500 W~~/(m·K) to 50 mW ~~/(m · K)~~/(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.~~m3. Enter this value into the experimental parameters screen of the apparatus.

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., *Journal of Physical and Chemical Reference Data*, Vol 11, No. 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.

TABLE 2 Polystyrene Thermal Conductivity[A]

Temperature		Thermal Conductivity[B]
(°C)	(K)	(W/(m·K))
−13	260	0.1480
0	273	0.1506
7	280	0.1514
20	293	0.1529
27	300	0.1539
47	320	0.1562
67	340	0.1582
87	360	0.1605
97	370	0.1616

[A] Ho, C. Y., Desai, P. D., Wu, K. T., Havill, T. N., and Lee, T. Y., NBS Publication GCR-77-83, 1977.
[B] The values in this table were determined under special highly accurate steady-state conditions, which are not attainable by or applicable to this test method. The actual precision of this test method is given in Section 13.

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

10.4.3 Place the encapsulated test specimen on the DSC sample sensor. Place an empty aluminum container and lid on the DSC reference sensor.

NOTE 12—Matching the weights of the reference container and lid to the specimen container and lid within ±0.1 mg produces the best results.

10.4.4 Measure the specific heat capacity of the thin test specimen using the conditions in 10.2.1. Record the specific heat capacity (c_p) in units J/(g·K).

10.4.5 Weigh the thick test specimen, recording its mass as m4.

10.4.6 Measure and record the diameter (d) and length (L) of the test specimen.

10.4.7 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 position the thick test specimen (which has been wetted with oil on the bottom side) on the aluminum disk covering the DSC sample sensor (see Note 6 and Note 7).

10.4.8 Measure the apparent heat capacity of the test specimen in accordance with the conditions in 10.2.1. Record the apparent heat capacity (c) in units of mJ/K.

10.4.9 Using the value for P (from 10.2.1), c_p (from 10.4.4), and $M,L,$ and d (from 10.4.6 and 10.4.7), calculate the observed thermal conductivity (λ_o) for the test specimen using Eq 1.

NOTE 13—An example calculation is presented in 11.5.1.

10.4.10 Using the value for λ_o (from 10.4.9) and the value of D (from 10.3.10), calculate the value for λ of the test specimen using Eq 3.

NOTE 14—An example calculation is presented in 11.5.3.

10.4.11 Using the value for λ (from 10.4.10), the value of c_p (from 10.4.4), and the values of m and L (from 10.4.6 and 10.4.7), calculate the thermal diffusivity (a) for the test specimen using Eq 4.

NOTE 15—An example calculation is presented in 11.5.4.

10.4.12 Report values for thermal conductivity (λ) and thermal diffusivity (a) at temperature (T) as the mean values of duplicate determinations. Also report the range of measurements between the high and the low value.

10.5 *Performance Criteria for Test Apparatus:*

10.5.1 The apparatus used for this test method is considered adequate if the value for thermal conductivity for a suitable reference material is within ±10 %.

10.5.2 A suitable reference material is described in Section 12.

11. Calculation

11.1 Determine the observed thermal conductivity (λ_o) in accordance with Eq 1:

$$\lambda_o = (8\,L\,c^2)/(c_p\,m\,d^2\,P) \tag{1}$$

where:

$\cancel{\lambda_o}$ = ~~observed thermal conductivity (W/(K • m)),~~
$\underline{\lambda_o}$ = observed thermal conductivity (W/(K·m)),
L = specimen length (mm),
c = apparent heat capacity (mJ/K),
$\cancel{c_p}$ = ~~specific heat capacity (J/(g • K),~~
$\underline{c_p}$ = specific heat capacity (J/(g·K),
m = thick specimen mass (mg),
d = thick specimen diameter (mm), and
P = period (s).

NOTE 16—When performing these calculations, retain all available decimal places in the measured values and in intermediate values. The final result should be rounded to two significant places in the standard deviation, as indicated in Section 13.

11.2 Calculate the thermal conductivity calibration constant (*D*) in ~~W/(m • K)~~W/(m·K) using Eq 2:

$$D = (\lambda_o\,\lambda_r)^{\frac{1}{2}} - \lambda_r \tag{2}$$

where:

$\cancel{\lambda_r}$ = ~~reference thermal conductivity (W/(m • K))~~

$\underline{\lambda_r}$ = reference thermal conductivity (W/(m·K))

11.3 Calculate the test specimen thermal conductivity (λ) using Eq 3:

$$\lambda = \frac{\left[\lambda_o - 2\,D + (\lambda_o{}^2 - 4\,D\,\lambda_o)^{\frac{1}{2}}\right]}{2} \tag{3}$$

11.4 Calculate thermal diffusivity (*a*) using Eq 4 (X1).

$$a = (\pi\,\lambda\,d^2\,L)/(4\,c_p\,m) \tag{4}$$

11.5 *Example Calculations:*

11.5.1 If the following set of values were measured for a polystyrene thermal conductivity calibration material, the value for λ_o from Eq 1 becomes: