



Standard Test Method for Thermal Conductivity and Thermal Diffusivity by Modulated Temperature Differential Scanning Calorimetry¹

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, non-porous 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 Analysis²

E 967 Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers²

E 968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters²

¹ This test method is under the jurisdiction of Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

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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.

E 1142 Terminology Relating to Thermophysical Properties²

E 1231 Practice for Calculation of Hazard Potential Figures-of-Merit for Thermally Unstable Materials²

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.

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 $\pm$ 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 $\pm$ 0.1°C, and (3) sinusoidal varying temperature with an amplitude of $\pm$ 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 modu-

lated 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 $\pm$ 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 0.4 mm thick and the second is nominally 3.5 mm thick. These test specimens are most conveniently obtained by cutting from

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