Designation: E 1530 – 99

Standard Test Method for Evaluating the Resistance to Thermal Transmission of Materials by the Guarded Heat Flow Meter Technique[1]

This standard is issued under the fixed designation E 1530; 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 a steady-state technique for the determination of the resistance to thermal transmission (thermal resistance) of materials in thicknesses of less than 25 mm. For homogeneous opaque solid specimens of a representative thickness, thermal conductivity can be determined (see Note 1). This test method is useful for materials having a thermal resistance in the range from 10 to 400 $\times$ 10^{-4} m^2·K/W thermal conductivity in the approximate range, $0.1 < \lambda < 30$ W/(m·K) over the approximate temperature range from 150 to 600 K. It can be used ouside these ranges with reduced accuracy for thicker specimens and for thermal conductivity values up to 60 W/(m·K).

NOTE 1—A body is considered homogeneous when the preceding property is found by measurement to be independent of specimen dimensions.

1.2 This test method is similar in concept to Test Method C 518, but is modified to accommodate smaller test specimens, having a higher thermal conductance. In addition, significant attention has been paid to ensure that the thermal resistance of contacting surfaces is minimized and reproducible.

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

2. Related Documents

2.1 *ASTM Standards:*

C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus[2]

C 1045 Practice for Calculating Thermal Transmission Properties Under Steady-State Test Conditions[2]

E 220 Method for Calibration of Thermocouples by Comparison Techniques[3]

E 1142 Terminology Relating to Thermophysical Properties[4]

E 1225 Test Method for Thermal Conductivity of Solids by Means of the Guarded-Comparative-Longitudinal Heat Flow Technique[4]

F 104 Classification System for Nonmetallic Gasket Materials[5]

F 433 Practice for Evaluating Thermal Conductivity of Gasket Materials[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *heat flux transducer (HFT)*—a device that produces an electrical output that is a function of the heat flux, in a predefined and reproducible manner.

3.1.2 *thermal conductance (C)*—the time rate of heat flux through a unit area of a body induced by unit temperature difference between the body surfaces.

3.1.2.1 *average temperature*—the average temperature of a surface is the area-weighted mean temperature of that surface.

3.1.3 *thermal conductivity (λ)—(of a solid material)*—the time rate of heat flow, under steady conditions, through unit area, per unit temperature gradient in the direction perpendicular to the area:

3.1.3.1 *apparent thermal conductivity*—When other modes of heat transfer through a material are present in addition to conduction, the results of the measurements performed in accordance with this test method will represent the apparent or effective thermal conductivity for the material tested.

3.1.4 *thermal resistance (R)*—the reciprocal of thermal conductance.

3.2 *Symbols:*

3.2.1 λ—thermal conductivity, W/(m·K) (Btu·in./h·ft^2·°F).

3.2.2 *C*—thermal conductance, W/(M^2·K) (Btu/(h·ft^2·°F).

3.2.3 *R*—thermal resistance, m^2·K/W (h·ft^2·°F/Btu).

3.2.4 Δ*x*—specimen thickness, mm (in.).

3.2.5 *A*—specimen cross-sectional area, m^2(ft^2)

[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.05 on Thermophysical Properties.

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[2] *Annual Book of ASTM Standards*, Vol 04.06.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

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

[5] *Annual Book of ASTM Standards*, Vol 09.02.

3.2.6 Q—heat flow, W (Btu/h).

3.2.7 ϕ—heat flux transducer output, mV.

3.2.8 N—heat flux transducer calibration constant, W/(m^2)·mV (Btu/h·ft^2·mV).

3.2.9 $N\phi$—heat flux, W/m^2(Btu/h·ft^2).

3.2.10 ΔT—temperature difference,° C °F.

3.2.11 Tg—temperature of guard heater, °C °F.

3.2.12 Tu—temperature of upper heater, °C (°F).

3.2.13 Tl—temperature of lower heater, °C (°F).

3.2.14 T_1—temperature of one surface of the specimen, °C (°F).

3.2.15 T_2—temperature of the other surface of the specimen, °C (°F).

3.2.16 Tm—mean temperature of the specimen, °C (°F).

3.2.17 $_s$—unknown specimen.

3.2.18 $_r$—known calibration or reference specimen.

3.2.19 $_o$—contacts.

4. Summary of Test Method

4.1 A specimen and a heat flux transducer (HFT) are sandwiched between two flat plates controlled at different temperatures, to produce a heat flow through the test stack. A reproducible load is applied to the test stack by pneumatic or hydraulic means, to ensure that there is a reproducible contact resistance between the specimen and plate surfaces. A cylindrical guard surrounds the test stack and is maintained at a uniform mean temperature of the two plates, in order to minimize lateral heat flow to and from the stack. At steady state, the difference in temperature between the surfaces contacting the specimen is measured with temperature sensors embedded in the surfaces, together with the electrical output of the HFT. This output (voltage) is proportional to the heat flow through the specimen, the HFT and the interfaces between the specimen and the apparatus. The proportionality is obtained through prior calibration of the system with specimens of known thermal resistance measured under the same conditions, such that contact resistance at the surface is made reproducible.

5. Significance and Use

5.1 This test method is designed to measure and compare thermal properties of materials under controlled conditions and their ability to maintain required thermal conductance levels.

6. Apparatus

6.1 A schematic rendering of a typical apparatus is shown in Fig. 1. The relative position of the HFT to sample is not important (it may be on the hot or cold side) as the test method is based on maintaining axial heat flow with minimal heat losses or gains radially. It is also up to the designer whether to choose heat flow upward or downward or horizontally, although downward heat flow in a vertical stack is the most common one.

6.2 *Key Components of a Typical Device* (The numbers 1 to 22 in parentheses refer to Fig. 1):

6.2.1 The compressive force for the stack is to be provided by either a regulated pneumatic or hydraulic cylinder (1) or a spring loaded mechanism. In either case, means must be provided to ensure that the loading can be varied and set to certain values reproducibility.

6.2.2 The loading force must be transmitted to the stack through a gimball joint (2) that allows up to 5° swivel in the plane perpendicular to the axis of the stack.

6.2.3 Suitable insulator plate (3) separates the gimball joint from the top plate (4).

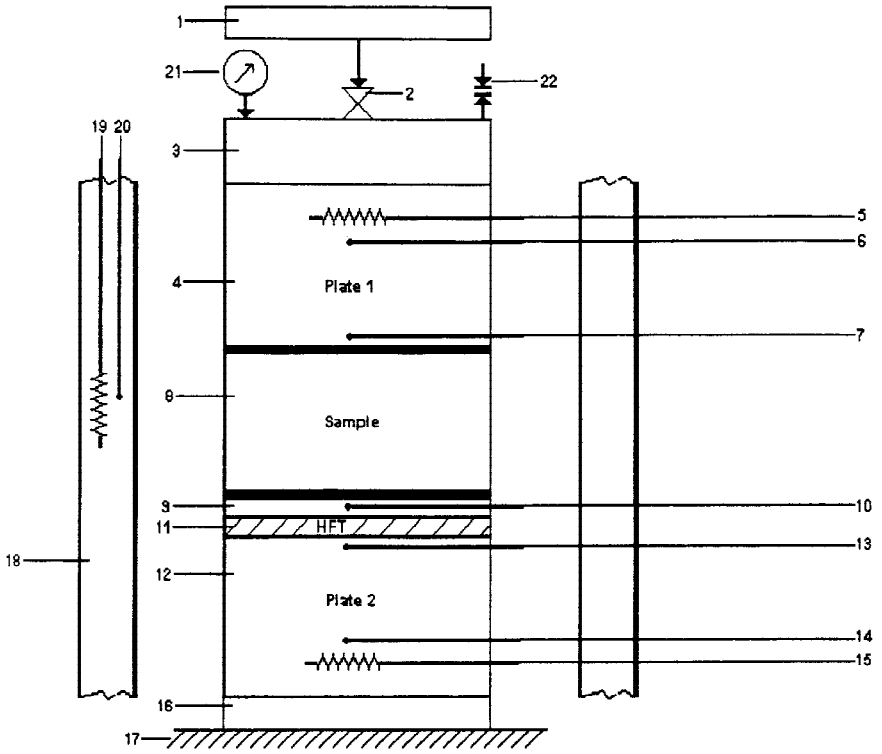

FIG. 1 Key Components of a Typical Device

6.2.4 The top plate (assumed to be the hot plate for the purposes of this description) is equipped with a heater (5) and control thermocouple (6) adjacent to the heater, to maintain a certain desired temperature. (Other means of producing and maintaining temperature may also be used as long as the requirements in 6.3 are met.) The construction of the top plate is such as to ensure uniform heat distribution across its face contacting the sample (8). Attached to this face (or embedded in close proximity to it) in a fashion that does not interfere with the sample/plate interface, is a temperature sensor (7) (typically a thermocouple, thermistor) that defines the temperature of the interface on the plate side.

6.2.5 The sample (8) is in direct contact with the top plate on one side and an intermediate plate (9) on the other side.

6.2.6 The intermediate plate (9) is an optional item. Its purpose is to provide a highly conductive environment to the second temperature sensor (10), to obtain an average temperature of the surface. If the temperature sensor (10) is embedded into the face of the HFT, or other means are provided to define the temperature of the surface facing the sample, the use of the intermediate plate is not mandatory.

6.2.7 The heat flux transducer (HFT) is a device that will generate an electrical signal in proportion to the heat flux across it. The level of output required (sensitivity) greatly depends on the rest of the instrumentation used to read it. The overall performance of the HFT and its readout instrumentation shall be such as to meet the requirements in Section 13.

6.2.8 The lower plate (12) is constructed similarly to the upper plate (4), except it is positioned as a mirror image.

6.2.9 An insulator plate (16) separates the lower plate (12) from the heat sink (17). In case of using circulating fluid in place of a heater/thermocouple arrangement in the upper or lower plates, or both, the heat sink may or may not be present.

6.2.10 The entire stack is surrounded by a cylindrical guard (18) equipped with a heater (19) and a control thermocouple (20) to maintain it at the mean temperature between the upper and lower plates. A small, generally unfilled, gap separates the guard from the stack. For instruments limited to operate in the ambient region, no guard is required. A draft shield is recommended in place of it.

NOTE 2—It is permissible to use thin layers of high-conductivity grease or elastomeric material on the two surfaces of the sample to reduce the thermal resistance of the interface and promote uniform thermal contact across the interface area.

NOTE 3—The cross-sectional area of the sample may be any, however, most commonly circular and rectangular cross sections are used. Minimum size is dictated by the magnitude of the disturbance caused by thermal sensors in relation to the overall flux distribution. The most common sizes are 25 mm round or square to 50 mm round.

6.2.11 The instrument is to be equipped with suitable means (21) to measure the thickness of the sample, in situ, in addition to provisions (22) to limit compression when testing elastomeric or other compressible materials.

NOTE 4—This requirement is also mandatory for testing materials that soften while heated.

6.3 *Requirements*:

6.3.1 Temperature control of upper and lower plate is to be ± 0.1°C (0.18°F) or better.

6.3.2 Reproducible load of 0.28 MPa (40 psi) has been found to be satisfactory for solid samples. Minimum load shall not be below 0.07 MPa (10 psi).

6.3.3 Temperature sensors are usually fine gage or small-diameter sheath thermocouples, however, ultraminiature resistance thermometers and linear thermistors may also be used.

6.3.4 Operating range of a device using a mean temperature guard shall be limited to from −100 to 300°C, when using thermocoupules as temperature sensors, and from −180 to 300°C with platinum resistance thermometers.

7. Sampling and Conditioning

7.1 Cut representative test specimens from larger pieces of the sample material or body.

7.2 Condition the cut specimens in accordance with the requirements of the appropriate material specifications if any.

8. Test Specimen

8.1 The specimen to be tested should be representative for the sample material. The recommended specimen configuration is a 50.8± 0.25-mm (2 ± 0.010-in.) diameter disk, having smooth flat and parallel faces, ± 0.025 mm (± 0.001 in.), such that a uniform thickness within 0.025 mm (± 0.001 in.) is attained in the range from 0.5 to 25.4 (0.020 to 1.0 in.) For testing specimens with thicknesses below 0.5 mm, a special technique, described in Annex A1, has to be used.

9. Calibration

9.1 Select the mean temperature and load conditions required. Adjust the upper heater temperature (T_u) and lower heater temperature (T_l) such that the temperature difference at the required mean temperature is no less than 30 to 35°C and the specimen ΔT is not less than 3°C). Adjust the guard heater temperature (T_g) such that it is at approximately the average of T_u and T_l.

9.2 Select at least two calibration specimens having thermal resistance values that bracket the range expected for the test specimens at the temperature conditions required.

9.3 Table 1 contains a list of several available materials commonly used for calibration together with corresponding thermal resistance (R_s) values for a given thickness. This

TABLE 1 Typical Thermal Resistance Values of Specimens of Different Materials

Material	Approximate Thermal Conductivity W/(m·K) at 30°C	Thickness, mm	Approximate Thermal Resistance, 10^{-4} m^2·K/W at 30°C
Vespel[6] Polyimide	0.4	20	500
Vespel[6] Polyimide	0.4	10	250
Vespel[6] Polyimide	0.4	1	25
Polyethylene	0.2	1	50
Polyethylene	0.2	0.5	25
Polyethylene	0.2	0.1	5
Pyroceram 9606[7]	4	20	50
Pyroceram 9606[7]	4	10	25
Pyrex 7740[7] Glass	1	20	200
Pyrex 7740[7] Glass	1	10	100
Pyrex 7740[7] Glass	1	1	10
304 Stainless Steel	14	20	14
304 Stainless Steel	14	10	7

information is provided to assist the user in selecting optimum specimen thickness for testing a material and in deciding which calibration specimens to use.

9.4 The range of thermal conductivity for which this test method is most suitable is such that the optimum thermal resistance range is from 10×10^{-4} to 400×10^{-4} $m^2 \cdot K/W$. The most commonly used calibration materials are the Pyrex 7740[6] and Pyroceram 9606[6], Vespel (polyimide) and stainless steel all having a well-established thermal conductivity as a function of temperature.[7]

10. Procedure

10.1 Measure the thickness of the specimen to 25 µm.

10.2 Coat both surfaces of a calibration specimen with a very thin layer of a compatible heat sink compound or place a thin layer of elastomeric heat-transfer medium on it to help minimize the thermal resistance at the interfaces of adjacent contacting surfaces.

10.3 Release the compressive load on the specimen stack, open the test chamber, and insert the calibration specimen. Care must be taken to ensure that all surfaces are free of any foreign matter.

10.4 Close the test chamber and clamp the calibration specimen in position between the plates at the recommended compressive load of 0.28 MPa.

10.5 Wait for thermal equilibrium to be attained. This should be seen when all the temperatures measured do not drift more than 0.1°C in 1 min. Read and record all temperatures and the output of the heat flux transducer.

NOTE 5—The time to attain thermal equilibrium is dependent upon the thickness of the specimen and its thermal properties. Experience shows that approximately 1 h is needed for thermal equilibrium to be attained, when testing a sample with the thermal conductivity within the optimum operating range of the instrument.

10.6 Repeat 10.1-10.5 with one or more calibration specimens, having different thermal resistance values covering the expected range for the test specimen.

10.7 *Thermal Conductivity of an Unknown Specimen*:

10.7.1 Tests shall only be conducted at a temperature in a range and under applied load conditions for which valid calibration data exists.

10.7.1.1 When automatic control of temperature of the heaters is involved, the controller settings should be checked to ensure that they are the same as those for the desired temperature level for the calibration.

10.7.2 Measure the thickness of the specimen.

10.7.3 Apply a thin layer of heat sink compound or place a thin layer of elastomeric heat transfer medium on the surfaces of the test specimen. This may be unnecessary for specimens of flexible materials.

NOTE 6—Care must be taken to ensure that any material applied to the surfaces of the specimen does not change its thermal properties, by soaking into it.

10.7.4 Repeat 10.3-10.5 using the test specimen. For compressible materials, it is mandatory to measure in situ the sample thickness under load, and to limit further compression by suitable mechanical stop.

10.8 *Thermal Conductivity of Thin Specimens*—For specimens less than approximately 0.5 mm (0.020 in.) in thickness (and less than 1 mm (0.040 in.) having λ greater than 0.5 W/(m·K)), a special stacking technique can be used. This is described in detail in Annex A1.

NOTE 7—Experience has indicated that for reliable measurements on a single specimen, the minimum thickness (mm) is given by $\Delta x \geq 3\lambda$ (W/(m·K)).

10.9 *Automated Systems:* Computerized or otherwise automated systems may require different operating steps, and may or may not provide intermediate readings described in 10.5. For these devices, follow the operating and calibrating procedures prescribed by the manufacturer.

NOTE 8—For an automated system to meet the requirements of this test method, the calibration test methods, and calculation built into it shall at minimum include the steps or principles set forth in 10.1-10.8, and all applicable guidelines given in Sections 6, 9, 12, and 13.

11. Calculation

11.1 At equilibrium, the Fourier heat-flow equation applied to the system becomes as follows:

$$R_s = \frac{N\,(T_1 - T_2)}{Q} - R_o \tag{1}$$

and:

$$C_s = \frac{1}{R_s} \tag{2}$$

For homogeneous materials:

$$R_s = \frac{\Delta x}{\lambda} \tag{3}$$

11.1.1 In Eq 1, N and R_o are temperature— and load-dependent parameters obtained by calibration at each particular set of conditions. Once obtained, they should remain fixed for the particular settings used to attain the conditions.

NOTE 9—Since N is also determined by the particular HFT utilized, the calibration should be checked occasionally to ensure that continuous heating/cycling does not affect the HFT.

NOTE 10—The parameter R_o depends on the parallelism of the two surface plates and should be reproducible unless the test section is altered mechanically in any way. If this occurs, recalibration is necessary.

11.2 There are three methods of data analysis to determine R_s, C_s and λ. In each case, utilize relevant input parameters determined to the stated precision levels and use all available decimal places through the calculation stages to the final result. Calculate the thermal resistance R_s to the nearest whole number in practical units of 10^{-4} $m^2 \cdot K/W$ and derive values of thermal conductivity to the second significant figure level of precision.

11.2.1 *Graphical Method*—At each set of conditions, Eq 1 is represented by a straight line on a graph of R_s versus $(T_1 - T_2)/Q$. Plot the test result of several calibration specimens on the graph, and draw a best-fit straight line through the data points as illustrated in Fig. 2. When measuring the thermal conductivity of a test specimen, obtain R_s by drawing a vertical

[6] Vespel is a product and trademark of DuPont, Wilmington, DE.

[7] Pyrex 7740 and Pyroceram 9606 are products and trademarks of Corning Glass Co.

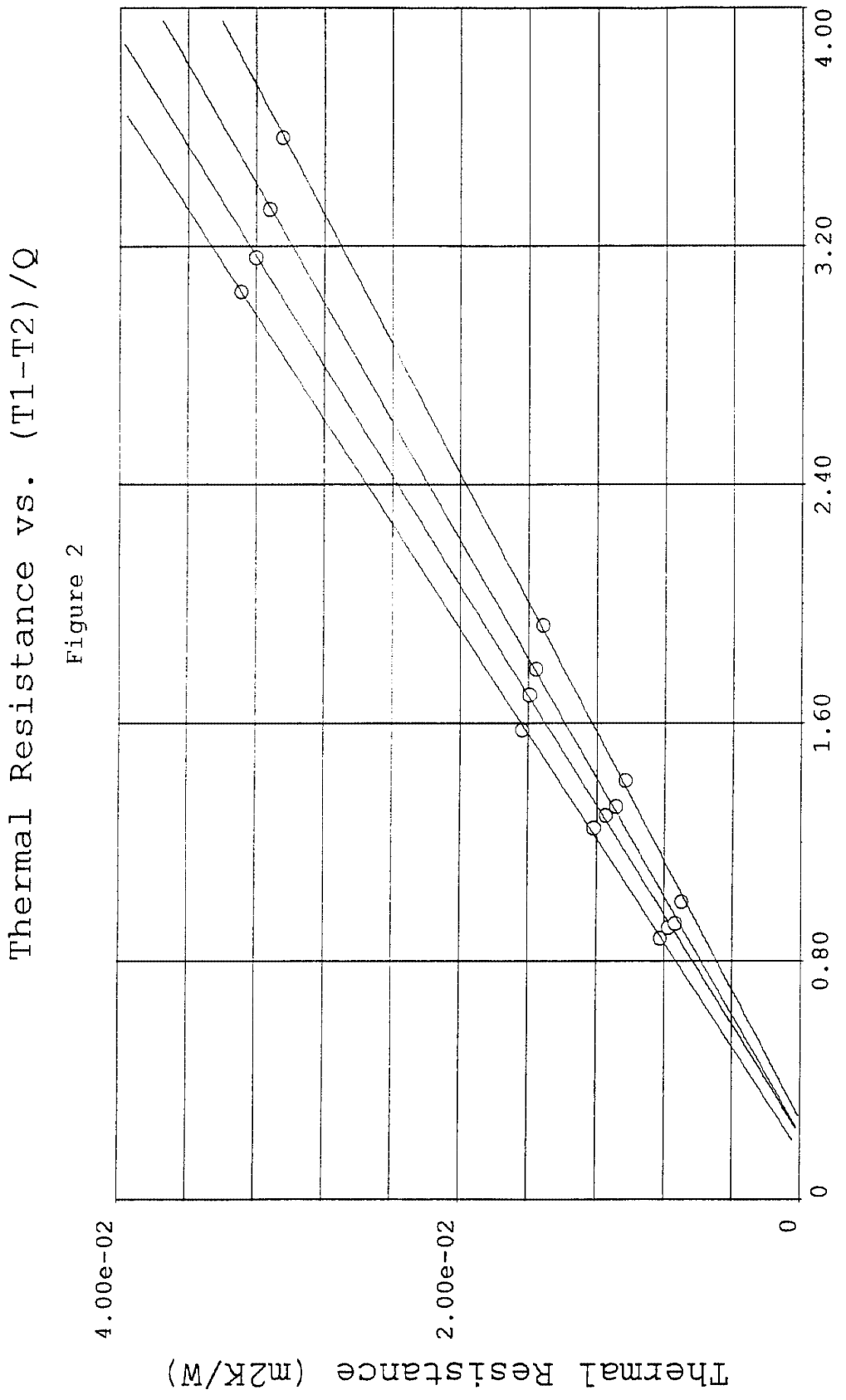

FIG. 2 Thermal Resistance Versus (T1–T2) / Q

line at the appropriate value of $(T_1 - T_2)/Q$ to intersect the calibration line. Obtain values of C_s and λ_s from Eq 2 and 3.

11.2.2 *Analytical Method*—At each set of conditions, solve Eq 1 mathematically for N and R_o after measuring a pair of reference specimens to yield two sets of data for R_s and $(T_1 - T_2)/Q$. Eq 1 can be used subsequently to determine R_s of the test specimen following measurement of T_1, T_2, and Q providing the calculated R_s falls within the calibration range corresponding to the particular pair of reference specimens in accordance with 9.2. By calibrating with additional reference specimens of different thermal resistances, several linear equations can be generated, each covering a part of the overall range.

11.2.3 *Computer-Aided Analysis*:

11.2.3.1 At each set of conditions, solve Eq 1 mathematically for N and R_o, using a linear regression analysis of the results for several sets of data for R_s and $(T_1 - T_2)/Q$ produced as a result of testing several calibration specimens. A similar series of tests carried out at the different temperatures provides new values of N and R_o.

11.2.3.2 Determine a polynomial relationship between N and temperature, and between R_o and temperature, so that Eq 1 becomes:

$$R_s = f_1(T)\cdot\frac{T_1 - T_2}{Q} - f_2(T) \quad (4)$$

where:
$f_1(T)$ = temperature dependent value of N,
$f_2(T)$ = temperature dependent value of R_o, and
T = test temperature.

11.2.3.3 The values of R_s and λ of the test specimen are calculated automatically, once T_1, T_2 and Q have been measured. Results are accurate provided that the test temperatures fall within the limits used during calibration, and that R_s does not fall outside the calibration range obtained with the reference specimens.

12. Report

12.1 Report the following information:

12.1.1 Complete identification and description of material and specimen including any conditioning procedure,

12.1.2 Details of reference specimen materials used for calibration,

12.1.3 Details of temperatures of appropriate surfaces, guard and ambient, °C (°F),

12.1.4 Applied load, Pa (psi),

12.1.5 Specimen thickness, mm (in.),

12.1.6 Mean temperature, °C (°F),

12.1.7 Measured thermal resistance to the nearest whole number in practical units, 10^{-4} $m^2 \cdot K/W$ ($h \cdot ft^2 °F/Btu$) and derived thermal conductivity to the second significant figure in $W/(m \cdot K)$ ($Btu \cdot in./(h \cdot ft^2 °F)$). Include details of the calculation method used (for manual instruments, omit for automated systems),

12.1.8 The sample's mean temperature and the direction and orientation of thermal transmission through the sample, since some bodies are not isotropic with respect to thermal conductivity, and

12.1.9 Designation of model/make in case a commercial device is used.

13. Precision and Bias

13.1 *Precision*—An interlaboratory study, summarized in Annex A2, involving four organizations and three materials having different thermal conductivity values in the applicable range of the test method has shown that a precision of ±5 % can be attained on a single specimen. If the specimen is in the form of two pieces clamped together, the precision is ±7 %.

13.2 *Bias*—Based on comparison with measurements made by an absolute method, there is no significant bias when measurements are made on single specimens.

14. Keywords

14.1 heat flow meter; heat flux transducer; thermal conductance; thermal conductivity; thermal resistance; thin specimen

ANNEXES

(Mandatory Information)

A1. TESTING OF THIN SPECIMENS LESS THAN 0.5 mm IN THICKNESS

A1.1 This technique involves evaluation of the thermal resistance of thin specimens by testing them stacked, providing that the thermal resistance of the interface between the layers is negligibly small. This assumption is valid for most flexible materials, such as: plastics, rubber, papers, and so forth, having relatively low thermal conductivity values.

A1.2 Several specimens have to be cut from the material to be tested, all of them having the same cross section with the instrument's stack.

A1.3 One or two stacked specimens have to be tested first, to evaluate if the thermal resistance falls within the calibration range of the instrument.

NOTE A1.1—No thermal compound or oil should be used between the layers of specimens or between the specimens and the instrument. Since the thermal resistance of these interfaces will be considered negligible, the number of specimens stacked should be reduced to the minimum necessary. Testing up to five or six layers is usually sufficient.

A1.3.1 If the thermal resistance values of one or two specimens tested together fall within the instrument's calibration range, the testing process should continue by testing three, four, and five layers of materials, stacked. It is recommended to have at least four different numbers of layers of material tested.

NOTE A1.2—If the specimens show a low thermal resistance value

when tested in one or two layers, but they have the tendency to stick to each other under compression, minimizing the thermal resistance between them, higher numbers of layers can be tested (for example, four, five, six, seven, and so forth).

A1.3.2 If the thermal resistance values of one or two specimens tested together are considerably lower than the minimum value of the instrument's calibration range, the specimens have to be stacked on a reference material and tested together with it. It is recommended that one of the reference materials used for the instrument's calibration should be considered for this particular application.

A1.4 Upon completion of the tests, determine the thermal resistance (R_s) of each set of specimens using an appropriate data reduction method, in accordance with 11. Plot the thermal resistance (R_s) versus the specimen thickness (total thickness of the stacked specimens, not including the thickness of the reference material, if used). The slope of the line obtained is the inverse of the thermal conductivity of the material tested.

A1.5 Due to the compressibility of the samples, in situ thickness measurement and compression limiting stop capabilities are mandatory requirements for the instrument used.

A2. SUMMARY OF INFORMATION FOR PRECISION AND BIAS STATEMENT

A2.1 An interlaboratory comparison was carried out on three different molding compounds by four organizations under the auspices of Semiconductor Equipment and Materials Institute, Inc. The four organizations involved were Fiberite Corp.; Holometrix, Inc.; Hysol Division of Dexter Corp.; and Plaskon Electronic Materials, Inc.

A2.2 The four materials were described as low, L; medium M; and high, H, thermal-conductivity molding compounds, respectively. Measurements using the guarded heat flow meter method were carried out at approximately 40°C on 12-mm thick specimens, 50-mm diameter disks each. For the L and M materials, measurements were also carried out on two 6-mm thick specimens stacked together. Separate measurements were made by one organization on other larger specimens cut from the same samples using an absolute method of measurement of thermal conductivity.

A2.3 The results obtained are summarized in Table A2.1

TABLE A2.1 Thermal Conductivity, W/m·K) of Four Molding Compounds

Organization	12 mm thick			2 samples, 6 mm thick each			Absolute Method		
	L	M	H	L	M	H	L	M	H
A	0.605	1.24	1.83	0.590	1.14	—	—	—	—
B	0.594	1.24	2.00	0.570	1.19	—	0.60	1.27	1.97
C	0.624	1.31	1.92	0.611	1.22	—	—	—	—
D	0.592	1.25	1.94	0.577	1.20	—	—	—	—