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Standard Test Method for Evaluating the Resistance to Thermal Transmission of Materials by the Guarded Heat Flow Meter Technique¹

This standard is issued under the fixed designation E1530; 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 <u>covers_describes</u> a steady-state technique for the determination of the resistance to thermal transmission (thermal resistance) of materials <u>of thicknesses having a thickness of less</u> than 25 mm. For homogeneous <u>opaque solid specimens</u> of a representative thickness, thermal conductivity can be determined Thermal conductivity may be determined for homogeneous <u>opaque solid specimens</u> (see Note 1). This test method is <u>particularly useful for homogeneous</u>, multilayer, and composite specimens having a thermal resistance in the range from 10 to(cm)²·K·W⁻¹ 400 × 10to⁻⁴ m 400 (cm)²·K·W⁻¹, which eanmay be obtained from materials of with an approximate thermal conductivity in the approximate range from 0.1 range 0.1 W·m⁻¹·K⁻¹ to 30 W·m⁻¹·K⁻¹ over the approximate temperature range from 150 K to 600 K. It can be used outside these ranges with reduced accuracy for thicker specimens and for thermal conductivity values up to 60 W·m⁻¹·K⁻¹.

Note 1—A body is considered homogeneous when the property to be measured is found to be independent of specimen dimensions.

- 1.2 This test method is similar in concept to Test Method C518, 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. The additional values are mathematical conversions to inch-pound units that are provided for information only and are not considered No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.5 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. Related Documents

2.1 ASTM Standards:²

C518 Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus

C1045 Practice for Calculating Thermal Transmission Properties Under Steady-State Conditions

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E1142 Terminology Relating to Thermophysical Properties

E1225 Test Method for Thermal Conductivity of Solids Using the Guarded-Comparative-Longitudinal Heat Flow Technique

F104 Classification System for Nonmetallic Gasket Materials

F433 Practice for Evaluating Thermal Conductivity of Gasket Materials

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

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 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- 3.1.1 heat flux transducer (HFT)—(HFT), n—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)—(C), n—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 of a surface—surface, n—the area-weighted mean temperature of that surface.
- 3.1.2.2 average (mean) temperature of a specimen (disc shaped)—shaped), n—the mean value of the upper and lower face temperatures.
- 3.1.3 thermal conductivity (λ)—(of a solid material)—material), n—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—conductivity, n*—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)—(R), n—the reciprocal of thermal conductance.
 - 3.2 Symbols:

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= thermal conductivity, W·m<sup>-1</sup>·K<sup>-1</sup>
           or Btu·in.·h-1·ft-2·oF-1
       = thermal conductivity, W·m<sup>-1</sup>·K<sup>-1</sup>
λ
\epsilon
       = thermal conductance, W·m<sup>-2</sup>·K<sup>-1</sup> or Btu·h<sup>-1</sup>·ft<sup>-2</sup>·°F<sup>-1</sup>
          thermal conductance, W·m<sup>-2</sup>·K<sup>-1</sup>
<u>C</u>
       = thermal resistance, m<sup>2</sup>·K·W<sup>-1</sup> or h·ft<sup>2</sup>·°F·Btu<sup>-1</sup>
R
          thermal resistance, \underline{m}^2 \cdot \underline{K} \cdot W^{-1}
R
          specimen thickness, mm or in
\Delta x
<u>∆x</u>
          specimen thickness, mm
A
          specimen cross-sectional area, m<sup>2</sup> or ft<sup>2</sup>
          specimen cross-sectional area, m<sup>2</sup>
\underline{A}
          heat flow, W or Btu·h-1
\overline{\varrho}
Q
          heat flow, W
          heat flux transducer output, mV
          heat flux transducer calibration constant, W·m<sup>-2</sup>·mV<sup>-1</sup> or Btu·h<sup>-1</sup>·ft<sup>-2</sup>·mV<sup>-1</sup>
₩
          heat flux transducer calibration constant, W·m<sup>-2</sup>·mV<sup>-1</sup>
          heat flux, W·m<sup>2</sup> or Btu·h<sup>-1</sup>·ft<sup>2</sup>
N\varphi
      = heat flux, W·m<sup>2</sup>
N\varphi
      temperature difference, C or Ftandards/sist/74c2fce8-8a3d-426e-8760-db89826b7a0d/astm-e1530-19
\Delta T
      <u>=</u> temperature difference, °C
\frac{\Delta T}{T_g}
\frac{T_g}{T_u}
\frac{T_u}{T_l}
\frac{T_u}{T_l}
\frac{T_u}{T_l}
\frac{T_u}{T_l}
\frac{T_u}{T_m}
       = temperature of guard heater, °C or °F
       <u>=</u> temperature of guard heater, °C
       = temperature of upper heater, °C or °F
       <u>=</u> temperature of upper heater, °C
       = temperature of lower heater, °C or °F
       = temperature of lower heater, °C
       = temperature of one surface of the specimen, °C or °F
       = temperature of one surface of the specimen, °C
       = temperature of the other surface of the specimen, °C or °F
       = temperature of the other surface of the specimen, °C
       = mean temperature of the specimen, °C or °F
       = mean temperature of the specimen, °C
       = unknown specimen
       = known calibration or reference specimen
       = contacts
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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, within an insulated guard tube, to produce a heat flow through the test stack. an uniform heat flow. A reproducible load is applied to the test stack by pneumatic or other means, to ensure that there is a reproducible contact resistance between the specimen and plate surfaces. A 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, On attaining steady state conditions, the difference in temperature between the surfaces contacting the specimen is measured with temperature sensors embedded in the plate surfaces, together with

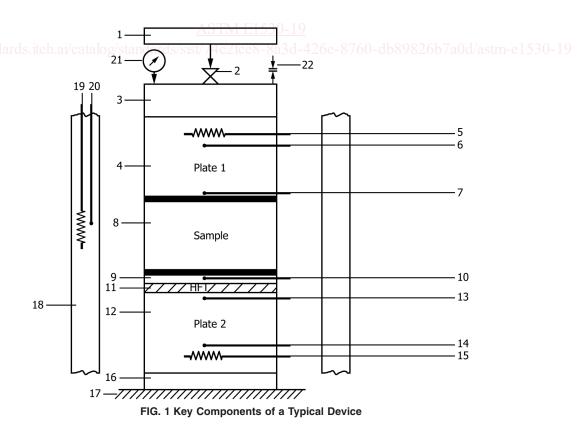
the electrical output of the HFT. This output (voltage) is proportional to the heat flow through the specimen, the HFT and the interfaces between the specimen and the apparatus. The proportionality is obtained through prior calibration of the system with specimens of known thermal resistance measured under the same conditions, such that contact resistance at the surfaces is can be made reproducible.

5. Significance and Use

5.1 This test method is designed describes a procedure to measure and compare the thermal properties of materials under controlled conditions and their ability to maintain required thermal conductance levels: resistance properties of specimens (less than 25 mm in thickness) under controlled conditions.

6. Apparatus

- 6.1 A schematic rendering of a typical apparatus is shown in Fig. 1. The relative position of the HFT relative to the specimen is not important (it(in may be on the hot or cold side) as the test-method is based on maintaining the maintenance of uniform axial heat flow with minimal radial heat losses or gains. It is also up to the designer whether designer/user of the application to choose heat flow upward or downward or horizontally, although downward upward, downward, or horizontal heat flow, although downwards heat flow in a vertical stack is the most common one commonly used.
 - 6.2 Key Components of a Typical Device (The numbers 1 to 22 in parentheses refer to Fig. 1):
- 6.2.1 The <u>adjustable</u> compressive force for the stack is to be provided by either a regulated pneumatic or hydraulic cylinder (1), dead weights or a spring loaded mechanism. In either case, means <u>mustshall</u> be provided to ensure that the loading can be varied and set to <u>eertain values reproducibly.selected reproducible values.</u>
- 6.2.2 The loading force mustshall 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 A suitable insulator plate (3) separates the gimball joint from the topupper plate (4).
- 6.2.4 The topupper plate (assumed to be the hot plate for the purposes of this description) is equipped with a heater (5) and control thermocouple temperature sensor (6) adjacent to the heater, to maintain a certain the desired temperature. (Other means of producing and maintaining temperature may also be used as long as providing the requirements in 6.3 are met.) The construction of the topupper plate is such as to ensure uniform heat distribution across its face contacting the face in contact with the specimen (8). Attached A temperature sensor (7) that defines the temperature of the interface on the plate side is attached to this face (or embedded in close proximity to it) in a fashion that such that it does not interfere with the specimen/plate interface, is a temperature sensor (7) (typically a thermocouple, resistance thermometer, or a thermistor) that defines the temperature of the interface on the plate side.interface.



- 6.2.5 The specimen (8) is in direct contact with the <u>topupper</u> 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 specimen, 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 significantly depends on the rest of the instrumentation used to read it. for its evaluation. 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. the mirror image. Both plates shall have a uniform thickness with the surfaces in contact with the specimen being flat and parallel and having a surface roughness of less than 10 µm maximum.
- 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 heater/temperature sensor 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 guard whose cross section tube, insulated from heat losses to the outside, with a cross section that is not too much different from that of the stack's stacks (18) equipped with a heater or cooling coils (19), or both, and a control thermocouple, resistance thermometer or thermistor similar control temperature sensor (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 but necessary although the use of a draft shield is recommended in place of it:recommended.
- Note 2—It-For materials in the higher thermal conductivity range, in particular, it is permissible to use a very thin layers of high-conductivity grease or elastomeric material on the two-layer of a suitable high conductivity contact medium on the contacting surfaces of the specimen to further reduce the thermal resistance of the interface and promote uniform thermal contact across the interface area. Calibration shall be performed under similar conditions of use or non-use of the contact medium.

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Note 3—Useful information on interfacial materials used for this test method is available from literature.³

Note 4—The cross-sectional area and the shape of the specimen maycan be any, however, most commonly circular and rectangular cross sections are most commonly 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 25 mm and 50 mm round disks.

6.2.11 The instrument measurement system is preferably equipped with suitable means (21) to measure the in situ thickness of the specimen, in situ, in addition to provisions specimen and provide a means (22) to limit compression when testing elastomeric or other compressible soft materials.

Note 5—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 $\pm 0.1^{\circ}$ 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 specimens. Minimum The minimum load shall not be below 0.07 MPa (10 psi).less than 0.07 MPa.
- 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 $-100 \, ^{\circ}\text{C}$ to $300 \, ^{\circ}\text{C}$, when using thermocouples as temperature sensors, and from $-180 \, ^{\circ}\text{C}$ to $300 \, ^{\circ}\text{C}$ when platinum resistance thermometers are used. Thermistors are normally present on more restricted allowable temperature range of use.

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 configurations of 25 mm or 50 mm ± 0.25 mm diameter disks or squares, shall have smooth flat and parallel faces, ± 0.025 mm (± 0.001 in.), surfaces, having a finish of 10 µm or less where possible, such that a uniform thickness within ± 0.025 mm (± 0.001 in.) is attained in the range from 0.5 mm to 25.4 mm (0.020 to 1.0 in.) For testing specimens with thicknesses below 0.5 mm, a special having a thickness less than 0.5 mm, an alternative technique, described in Annex A1, has to-is required beused. Other frequently favored sizes are 25.4 mm (1.00 in.) round or square cross section.

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_o) such that it is at approximately the average of T_u and T_l .

TABLE 1 Typical Thermal Resistance Values of Specimens of Different Materials

Material	Approximate Thermal Conductivity, W·m ⁻¹ ·K ⁻¹ at 30°G30 °C	Thickness, mm	Approximate Thermal Resistance, 10 (<u>cm</u>) ⁻⁴ - m ² -K·W ⁻¹ at 30°G 30 °C
Vespel ^A Polyimide SP1	0.4	20	500
Vespel ^A Polyimide SP1	0.4	10	250
Vespel ^A Polyimide SP1	0.4	1	25
Polyethylene	0.2	1	50
Polyethylene	0.2	0.5	25
Polyethylene	0.2	0.1	5
Pyroceram 9606 ^B	4	20	50
Pyroceram 9606 ^B	4	10	25
Pyrex 7740 ^B Glass	1	20	200
Pyrex 7740 ^B Glass	1	10	100
Pyrex 7740 ^B Glass	1	1	10
304 Stainless Steel	14	20	14
304 Stainless Steel	14	10	7

^A Vespel is a product and trademark of DuPont, Wilmington, DE.

^B Pyrex 7740 and Pyroceram 9606 are products and trademarks of Corning Glass Co.

³ Stacey, C., Sumpkin, A. J., and Jarrett, R. N., "Techniques for Reducing Thermal Contact Resistance in Steady-State Thermal Conductivity Measurements on Polymer Composites," *International Journal of Thermophysics*, Vol 37, 2016, p. 107.

9.1 Select at least three calibration specimens having thermal resistance values that bracket the range expected for the test specimens at the temperature conditions required. Calibration of the system shall be undertaken using a set of reference specimens having a range of thermal resistance achieved using different materials of known thermal conductivity or using different thickness of the same materials.

Note 6—At this writing (2017), there is a very limited number of appropriate reference materials available. Comprehensive information, including source, description, material type, range of application, availability, and properties is available.⁴

- 9.2 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 information is provided to assist the user in selecting optimum specimen thickness for testing a material and in deciding which calibration specimens to use.
- 9.3 The range of thermal conductivity for which thisthe test method is most suitable is such that the optimum thermal resistance range is from 10×1010^{-4} —to $(cm)^2 400 \times 10 \cdot \text{K} \cdot \text{W}^{-4-1}$ —mto 400—(cm) $^2 \cdot \text{K} \cdot \text{W}^{-1}$. The most commonly recommended and used ealibration materials for calibration are the Pyrex 7740 and Pyroceram 9606, (a glass) and Pyroceram 96063 (a ceramic), Vespel (polyimide) and stainless steel all having well-established thermal conductivity behaviors with temperature. (a polyimide, a polymer), polymethylmethacrylate (PMMA, a polymer), and one of several stainless steel (a metal).
- 9.4 Table 2 and Table 3 are listing <u>list</u> thermal conductivity values for selected reference materials, with the appropriate bibliographic references appearing in bold characters. The temperature range listed for each reference material corresponds to the temperature range mentioned in each particular cited work, and in some cases materials. Their temperature range of application,

TABLE 2 Thermal Conductivity Values of Selected Reference
Materials

Temperature (°C) —	Thermal Conductivity (W·m ⁻¹ ·K ⁻¹)			
	Vespel ^A SP1	Pyrex 7740 ^B	Pyroceram 9696 ^C	
-50		1.010		
0	lah Sto	1.104	1 02	
25	0.377	1.177	4.03	
2 <u>5</u>	0.377	1.177	4.06	
100	0.391	1.236	3.65	
100	0.391	1.236	3.71	
200	0.413	1.330	3.40	
200 300	0.413	1.330	3.42	
300	0.436	1.447 ^D	3.24	
300	0.436	1.447 ^D	3.23	
400			3.14	
<u>400</u>	<u></u>	<u></u>	3.10	
500	A\(\bar{\bar{\bar{\bar{\bar{\bar{\bar{	1530- -	3.05	
500	1 / * //= 4 0.0	0 0 2 = 426	3.00	
u/catalog 600 mdarc	ds/sist/ 2 4c2fce	8-8a3 Ē -426e	-8/6(2.98)898	
<u>600</u>	<u></u>	<u></u>	<u>2.92</u> 2.91	
700	==	==		
<u>700</u>	<u>=</u>	<u>=</u>	<u>2.86</u>	
800	==	=	2.84	
<u>800</u>	<u></u>	<u></u>	<u>2.81</u>	
900			2.77	
1000			2.71	

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^A Jacobs-Fedore, R.A., R. A., and Stroe, D.E., D. Thermophysical E., "Thermophysical Properties of Vespel SP1, SP1," in Thermal Conductivity 27+27: Thermal Expansion 15, DEStech Publications, Inc., Lancaster, PA, 2004, pp. 231–238.

^B Tye, R.P., and Salmon, D.R, Tye, R. P., Hume, Thermal Conductivity Certified Reference Materials: D., "Reference Materials for Thermal Pryrex 7740 and Transport Property Measurements," Polymethylmethacrylate, National Physical Laboratory report, 2004, Journal of Thermal Analysis and Calorimetry, Teddington, United Kingdom: 2017, pp. 1–11.

^C-Stroe, D.E., Thermitus, M.A., and Jacobs-Fedore, R.A., Salmon, D., Roebben, G., Lamberty, A., Thermophysical Properties of Pyroceram 9606",Brandt, R., "Certification of in-Thermal Conductivity 27 / Thermal Expansion 15, DESteeh Publications, Inc., 2004, pp. 382–390. and Thermal Diffusivity Up to 1025 K of a Glass-Ceramic Reference Material BCR-724," EUR Report 21764 EN, European Commission, Geel, Belgium, 2007.

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⁴ Tye, R. P., and Hume, D., "Reference Materials for Thermal Transport Property Measurements," Journal of Thermal Analysis and Calorimetry, 2017, pp. 1–11.

⁵ Pyrex 7740 and Pyroceram 9606 are products and trademarks of Corning Glass Co.

⁶ Vespel SP1 is a product and trademark of DuPont, Wilmington, DE.