



Designation: E 1225 – 99

# Standard Test Method for Thermal Conductivity of Solids by Means of the Guarded- Comparative-Longitudinal Heat Flow Technique<sup>1</sup>

This standard is issued under the fixed designation E 1225; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method describes a steady state technique for the determination of the thermal conductivity,  $\lambda$ , of homogeneous-opaque solids (see Notes 1 and 2). This test method is for materials with effective conductivities in the approximate range  $0.2 < \lambda < 200 \text{ W/m}\cdot\text{K}$  over the approximate temperature range between 90 and 1300 K. It can be used outside these ranges with decreased accuracy.

NOTE 1—For purposes of this technique, a system is homogeneous if the apparent thermal conductivity of the specimen,  $\lambda_A$ , does not vary with changes of thickness or cross-sectional area by more than  $\pm 5\%$ . For composites or heterogeneous systems consisting of slabs or plates bonded together, the specimen should be more than 20 units wide and 20 units thick, respectively, where a unit is the thickness of the thickest slab or plate, so that diameter or length changes of one-half unit will affect the apparent  $\lambda_A$  by less than  $\pm 5\%$ . For systems that are non-opaque or partially transparent in the infrared, the combined error due to inhomogeneity and photon transmission should be less than  $\pm 5\%$ . Measurements on highly transparent solids must be accompanied with infrared absorption coefficient information or the results must be reported as apparent thermal conductivity,  $\lambda_A$ .

NOTE 2—This test method may also be used to evaluate the thermal conductance/resistance of materials in contact.

1.2 *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:

- C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus<sup>2</sup>
- C 408 Test Method for Thermal Conductivity of Whiteware Ceramics<sup>3</sup>

- C 1045 Practice for Calculating Thermal Transmission Properties Under Steady-State Conditions<sup>2</sup>
- D 4351 Test Method for Measuring the Thermal Conductivity of Plastics by the Evaporation-Calorimetric Method<sup>4</sup>
- E 220 Test Method for Calibration of Thermocouples by Comparison Techniques<sup>5</sup>
- E 230 Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples<sup>5</sup>
- F 433 Practice for Evaluating Thermal Conductivity of Gasket Materials<sup>6</sup>

## 3. Terminology

### 3.1 Descriptions of Terms and Symbols Specific to This Standard:

#### 3.1.1 Terms:

3.1.1.1 *thermal conductivity*,  $\lambda$ —the time rate of heat flow, under steady conditions, through unit area, per unit temperature gradient in the direction perpendicular to the area;

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

#### 3.1.2 Symbols:

- $\lambda_M(T)$  = thermal conductivity of meter bars (reference materials) as a function of temperature, (W/m·K),
- $\lambda_M^1$  = thermal conductivity of top meter bar (W/m·K),
- $\lambda_M^2$  = thermal conductivity of bottom meter bar (W/m·K),
- $\lambda_S(T)$  = thermal conductivity of specimen corrected for heat exchange where necessary, (W/m·K),
- $\lambda'_S(T)$  = thermal conductivity of specimen calculated by ignoring heat exchange correction, (W/m·K),

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<sup>2</sup> Annual Book of ASTM Standards, Vol 04.06.

<sup>3</sup> Annual Book of ASTM Standards, Vol 15.02.

<sup>4</sup> Discontinued 1992; Vol 08.03.

<sup>5</sup> Annual Book of ASTM Standards, Vol 14.03.

<sup>6</sup> Annual Book of ASTM Standards, Vol 09.02.

- $\lambda_I(T)$  = thermal conductivity of insulation as a function of temperature, (W/m·K),
- $T$  = absolute temperature (K),
- $Z$  = position as measured from the upper end of the column, (m),
- $l$  = specimen length, (m),
- $T_i$  = the temperature at  $Z_i$ , (K),
- $q'$  = heat flow per unit area, (W/m<sup>2</sup>),
- $\delta\lambda, \delta T$ , etc. = uncertainty in  $\lambda, T$ , etc.,
- $r_A$  = specimen radius, (m),
- $r_B$  = guard cylinder inner radius, (m),
- $T_g(z)$  = guard temperature as a function of position,  $z$ , (K), and

**4. Summary of Test Method**

4.1 A test specimen is inserted under load between two similar specimens of a material of known thermal properties. A temperature gradient is established in the test stack and heat losses are minimized by use of a longitudinal guard having approximately the same temperature gradient. At equilibrium conditions, the thermal conductivity is derived from the measured temperature gradients in the respective specimens and the thermal conductivity of the reference materials.

**4.2 General Features of Test Method:**

4.2.1 The general features of the guarded longitudinal heat flow technique are shown in Fig. 1. A specimen of unknown thermal conductivity,  $\lambda_S$ , but having an estimated thermal conductance of  $\lambda_S/l_S$ , is mounted between two meter bars of known thermal conductivity,  $\lambda_M$ , of the same cross-section and similar thermal conductance,  $\lambda_M/l_M$ . A more complex but suitable arrangement is a column consisting of a disk heater with a specimen and a meter bar on each side between heater and heat sink. Approximately one-half of the power would then flow through each specimen. When the meter bars and specimen are right-circular cylinders of equal diameter the technique is described as the cut-bar method. When the cross-sectional dimensions are larger than the thickness it is described as the flat slab comparative method. Essentially any shape can be used as long as the meter bars and specimen have the same conduction areas.

4.2.2 A force is applied to the column to ensure good contact between specimens. The stack is surrounded by an insulation material of thermal conductivity,  $\lambda_I$ . The insulation is enclosed in a guard shell with a radius,  $r_B$ , held at the temperature,  $T_g(z)$ . A temperature gradient is imposed on the column by maintaining the top at a temperature,  $T_T$ , and the bottom at temperature  $T_B$ .  $T_g(z)$  is usually a linear temperature gradient matching approximately the gradient established in the test stack. However, an isothermal guard with  $T_g(z)$  equal to the average temperature of the specimen may also be used. An unguarded system is not recommended due to the potential very large heat losses, particularly at elevated temperatures (1).<sup>7</sup> At steady state, the temperature gradients along the sections are calculated from measured temperatures along the two meter bars and the specimen. The value of  $\lambda_S$  ( $\lambda_S$  as

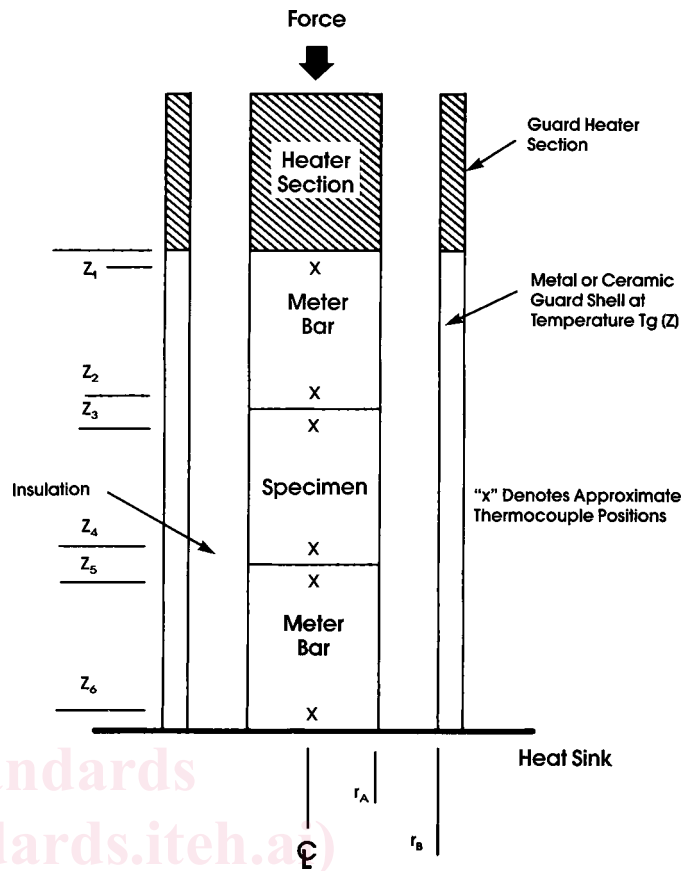


FIG. 1(a) Schematic of a Comparative-Guarded-Longitudinal Heat Flow System Showing Possible Locations of Temperature Sensors

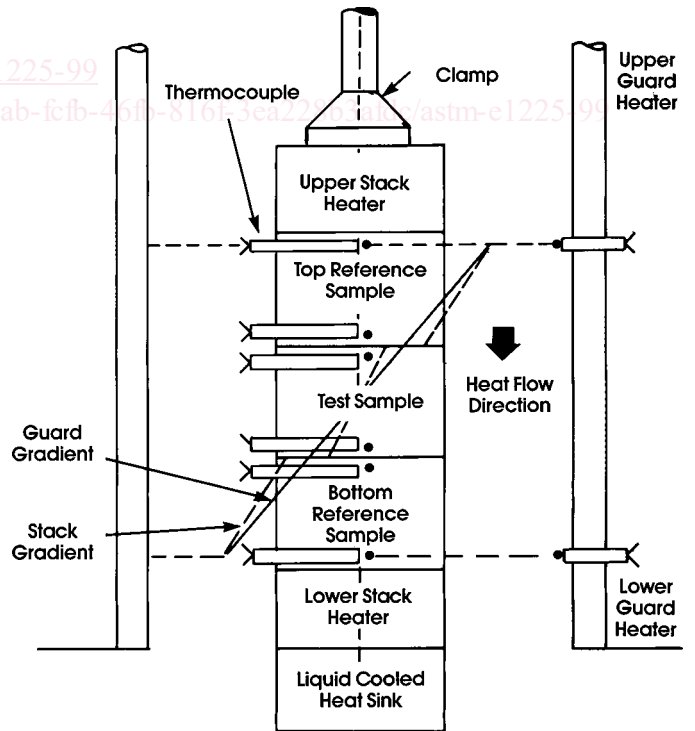


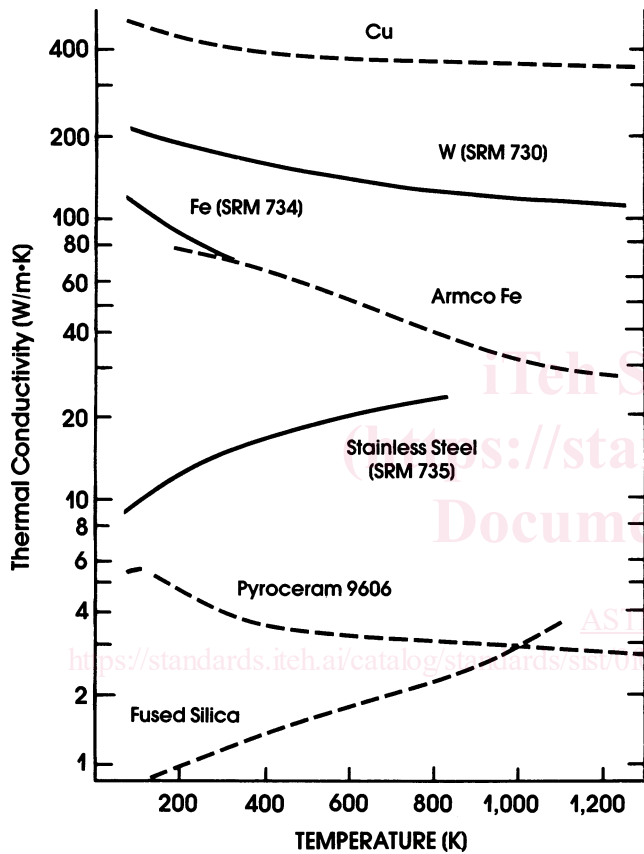
FIG. 1(b) Schematic of Typical Test Stack and Guard System Illustrating Matching of Temperature Gradients

<sup>7</sup> The boldface numbers in parentheses refer to a list of references at the end of this test method.

uncorrected for heat shunting) can then be determined using the following equation where the notation is shown in Fig. 1:

$$\lambda_s \left[ \frac{(Z_4 - Z_3) \lambda_M^1 (T_2 - T_1)}{(T_4 - T_3) 2 (Z_2 - Z_1)} + \left[ \frac{\lambda_M^2 (T_6 - T_5)}{2 (Z_6 - Z_5)} \right] \right] \quad (1)$$

This is a highly idealized situation, however, since it assumes no heat exchange between the column and insulation at any position and uniform heat transfer at each meter bar-specimen interface. The errors caused by these two assumptions vary widely and are discussed in Section 10. Because of these two effects, restrictions must be placed on this test method if the desired accuracy is to be achieved.



NOTE 1—The material selected for the meter bars should have a thermal conductivity as near as possible to the thermal conductivity of the unknown.

FIG. 2 Approximate Values for the Thermal Conductivity of Several Possible Reference Materials for Meter Bars

### 5. Significance and Use

5.1 The comparative method of measurement of thermal conductivity is especially useful for engineering materials including ceramics, polymers, metals and alloys, refractories, carbons, and graphites including combinations and other composite forms of each.

5.2 Proper design of a guarded-longitudinal system is difficult and it is not practical in a method of this type to try to establish details of construction and procedures to cover all contingencies that might offer difficulties to a person without technical knowledge concerning theory of heat flow, tempera-

ture measurements, and general testing practices. Standardization of this test method is not intended to restrict in any way the future development by research workers of new or methods or improved procedures. However, new or improved techniques must be thoroughly tested and requirements for qualifying an apparatus are outlined in Section 10.

### 6. Requirements

#### 6.1 Meter Bar Reference Materials:

6.1.1 Reference materials or transfer standards with known thermal conductivities must be used for the meter bars. Since the minimum measurement error of the method is the uncertainty in  $\lambda_M$ , it is preferable to use standards available from a national standards laboratory. Other reference materials are available because numerous measurements of  $\lambda$  have been made and general acceptance of the values has been obtained. Table 1 lists the currently available recognized reference materials including those available from National Institute of Standards and Technology. Fig. 2 shows the approximate variation of  $\lambda_M$  with temperature.

6.1.2 Table 1 is not exhaustive and other materials may be used as references. The reference material and the source of  $\lambda_M$  values shall be stated in the report.

6.1.3 The requirements for any reference material includes stability over the temperature range of operation, compatibility with other system components, reasonable cost, ease of thermocouple attachment, and an accurately known thermal conductivity. Since heat shunting errors for a specific  $\lambda_I$  increase as  $\lambda_M/\lambda_s$  varies from unity, (1) the reference which has a  $\lambda_M$  nearest to  $\lambda_s$  should be used for the meter bars.

6.1.4 If a sample has a  $\lambda_s$  between two reference materials, the reference with the higher  $\lambda_M$  should be used to reduce the total temperature drop along the column.

#### 6.2 Insulation Materials:

6.2.1 A large variety of powder, particulate, and fiber materials exist for reducing both radial heat flow in the column-guard annulus and surrounds and heat shunting along the column. Several factors must be considered during selection of the most appropriate insulation. The insulation must be stable over the anticipated temperature range, have a low  $\lambda_I$ , and be easy to handle. In addition, the insulation should not contaminate system components such as the temperature sensors, it must have low toxicity, and it should not conduct electricity. In general, powders and particulates are used since they pack readily. However, low density fiber blankets can also be used.

6.2.2 Some candidate insulations are listed in Table 2.

#### 6.3 Temperature Sensors:

6.3.1 There shall be a minimum of two temperature sensors on each meter bar and two on the specimen. Whenever possible, the meter bars and specimen should each contain three sensors. The extra sensors are useful in confirming linearity of temperature versus distance along the column or indicating an error due to a temperature sensor decalibration.

6.3.2 The type of temperature sensor depends on the system size, temperature range, and the system environment as controlled by the insulation, meter bars, specimen, and gas within the system. Any sensor possessing adequate accuracy may be used for temperature measurement (2) and be used in large

**TABLE 1 Reference Materials For Use as Meter Bars**

Material	Temperature Range (K)	Percentage Uncertainty in $\lambda$ ( $\pm$ %)	$\lambda_M$ (W/m-K)	Material Source
Electrolytic Iron SRM 734	To 1000	2	<sup>A</sup>	NIST <sup>A</sup>
Tungsten SRM 730	4 to 300	2	$\lambda_M$ Dependent on T <sup>A</sup>	NIST <sup>A</sup>
	300 to 2000	2 to 5		
	>2000	5 to 8		
Austenitic Stainless SRM 735	4 to 1200	<5 %	$\lambda_M = 1.22T^{0.432}$ T > 200K <sup>A</sup>	NIST <sup>A</sup>
Iron	80 to 1200	2	$\lambda_M$ should be calculated from measured values <sup>BC</sup>	...
Copper	90 to 1250	<2	$\lambda_M = 416.3 - 0.05904T + 7.087 \times 10^7/T^{3D}$	manufacturer
Pyroceram Code 9606	90 to 1200	...	<sup>EF</sup>	manufacturer
Fused Silica <sup>G</sup>	1300	<8	$\lambda_M = (84.7/T) + 1.484 + 4.94 \times 10^{-4} T + 9.6 \times 10^{-13}T^{4HI}$	manufacturer
Pyrex 7740	90 to 600	6	<sup>EF</sup>	manufacturer

<sup>A</sup> National Institute of Standards and Technology, Washington, D.C. 20234. See Special Publications 260-52 and 260-46.

<sup>B</sup> Fulkerson W., et al., *Physics Review* 167, p. 765, (1968).

<sup>C</sup> Lucks C. F., *Journal of Testing and Evaluation*, ASTM 1 (5), 422 (1973).

<sup>D</sup> Moore, J. P., Graves, R. S. and McElroy, D. L., *Canadian Journal of Physics*, 45, 3849 (1967).

<sup>E</sup> "Thermal Conductivity of Selected Materials," Report NSRDS-NBS 8, National Bureau of Standards, 1966.

<sup>F</sup> L. C. Hulstrom, R. P. Tye, and S. E. Smith, *Thermal Conductivity* 19, Ed. D. W. Yarbrough, Plenum Press, New York, In Course of Publication (see also High Temperature-High Pressures, 17, 707, 1985).

<sup>G</sup> Hust J. G., Cryogenics Division; NBS, Boulder, Colorado 80302.

<sup>H</sup> Above 700 K a large fraction of heat conduction in fused silica will be by radiation and the actual effective values may depend on the emittances of bounding surfaces and meter bar size.

<sup>I</sup> Recommended values from Table 3017 A-R-2 of the *Thermophysical Properties Research Center Data Book*, Vol. 3, "Nonmetallic Elements, Compounds, and Mixtures," Purdue University, Lafayette, Indiana.

**TABLE 2 Suitable Thermal Insulation Materials**

Material <sup>A</sup>	Typical Thermal Conductivity (W/(m-K))		
	300K	800K	1300K
Poured Powders			
Diatomaceous Earth	0.053	0.10	0.154
Bubbled Alumina	0.21	0.37	0.41
Bubbled Zirconia	0.19	0.33	0.37
Vermiculite	0.07	0.16	...
Perlite	0.050	0.17	...
Blankets and Felts			
Aluminosilicate 60–120 kg/m <sup>3</sup>	0.044	0.13	0.33
Zirconia 60–90 kg/m <sup>3</sup>	0.039	0.09	0.25

<sup>A</sup> All materials listed can be used up to the 1300 K limit of the comparative longitudinal except where noted.

systems where heat flow perturbation by the temperature sensors would be negligible. Thermocouples are normally employed. Their small size and the ease of attachment are distinct advantages.

6.3.3 When thermocouples are employed they should be fabricated from wires which are 0.1 mm diameter or less. A constant temperature reference shall always be provided for all cold junctions. This reference can be an ice-cold slurry (3), a constant temperature zone box, or an electronic ice point reference. All thermocouples shall be fabricated from either calibrated thermocouple wire (4) or from wire that has been certified by the supplier to be within the limits of error specified in Table 1 of Standard E 230.

6.3.4 Thermocouple attachment is important to this technique in order to ensure that reliable temperature measurements are made at specific points. The various techniques are illustrated in Fig. 3. Intrinsic junctions can be obtained with metals and alloys by welding individual thermo-elements to the surfaces (Fig. 3a). Butt or bead welded thermocouples junctions can be rigidly attached by peening, cementing, or welding in fine grooves or small holes (Fig. 3b, 3c, and 3d).

6.3.5 In Fig. 3b, the thermocouple resides in a radial slot, and in Fig. 3c the thermocouple is pulled through a radial hole in the material. When a sheathed thermocouple or a thermocouple with both thermoelements in a two-hole electrical insulator is used, the thermocouple attachment shown in Fig. 3d can be used. In the latter three cases, the thermocouple should be thermally connected to the solid surface using a suitable glue or high temperature cement. All four of the procedures shown in Fig. 3 should include wire tempering on the surfaces, wire loops in isothermal zones, thermal wire grounds on the guard, or a combination of all three (5).

6.3.6 Since uncertainty in temperature sensor location leads to large errors, special care must be taken to determine the correct distance between sensors and to calculate the possible error resulting from any uncertainty.

#### 6.4 Reduction of Contact Resistance:

6.4.1 This test method requires uniform heat transfer at the meter bar to specimen interfaces whenever the temperature sensors are within a distance equal to  $r_A$  from an interface (6). This requirement necessitates a uniform contact resistance across the adjoining areas of meter bars and specimens. This is normally attained by use of an applied axial load in conjunction with a conducting medium at the interfaces. Measurements in a vacuum environment are not recommended unless the vacuum is required for protection purposes.

6.4.2 For the relatively thin specimens normally used for materials having a low thermal conductivity the temperature sensors must be mounted close to the surface and in consequence the uniformity of contact resistance is critical. In such cases, a very thin layer of a compatible highly conductive fluid, paste, soft metal foil, or screen shall be introduced at the interfaces.

6.4.3 Means shall be provided for imposing a reproducible and constant load along the column with the primary purpose