

Designation: E1225 - 04

Standard Test Method for Thermal Conductivity of Solids by Means of the Guarded-Comparative-Longitudinal Heat Flow Technique¹

This standard is issued under the fixed designation E1225; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method describes a steady state technique for the determination of the thermal conductivity, λ , of homogeneous-opaque solids (see Notes 1 and 2). This test method is for materials with effective thermal 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, λ_A , does not vary with changes of thickness or cross-sectional area by more than ± 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 λ_A by less than ± 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 ± 5 %. Measurements on highly transparent solids must be accompanied with infrared absorption coefficient information, or the results must be reported as apparent thermal conductivity, λ_A .

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

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:²

C177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus

C408 Test Method for Thermal Conductivity of Whiteware Ceramics

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

D4351 Test Method for Measuring the Thermal Conductivity of Plastics by the Evaporation-Calorimetric Method

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples

F433 Practice for Evaluating Thermal Conductivity of Gasket Materials

3. Terminology

- 3.1 Descriptions of Terms and Symbols Specific to This Standard:
 - 3.1.1 Terms:
- 3.1.1.1 *thermal conductivity*, λ —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/ $(m \cdot K)$)

 λ_M^{-1} = thermal conductivity of top meter bar (W/ $(m \cdot K)$),

 λ_M^2 = thermal conductivity of bottom meter bar $(W/(m \cdot K))$,

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.

Current edition approved May 1, 2004. Published June 2004. Originally approved in 1987. Last previous edition approved in 1999 as E1225–99. DOI: 10.1520/E1225-04.

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.

 $\lambda_S(T)$ = thermal conductivity of specimen corrected for heat exchange where necessary, (W/($m \cdot K$)),

 $\lambda'_{S}(T)$ = thermal conductivity of specimen calculated by ignoring heat exchange correction, (W/ $(m\cdot K)$).

 $\lambda_I(T)$ = thermal conductivity of insulation as a function of temperature, $(W/(m \cdot K))$,

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^2) ,

 $\delta \lambda$, δT , etc. = uncertainty in λ , 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, λ_S , but having an estimated thermal conductance of λ_{S}/l_{S} , is mounted between two meter bars of known thermal conductivity, λ_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 crosssectional dimensions are larger than the thicknesss 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, λ_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

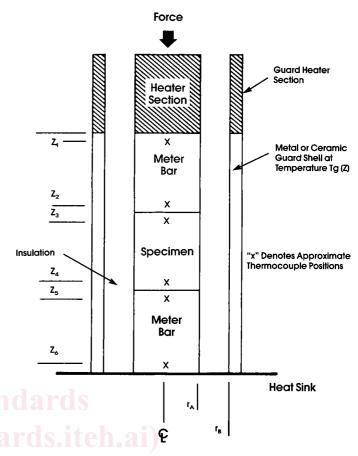


FIG. 1(a) Schematic of a Comparative-Guarded-Longitudinal Heat Flow System Showing Possible Locations of Temperature Sensors

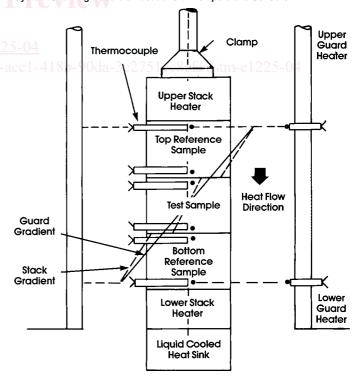
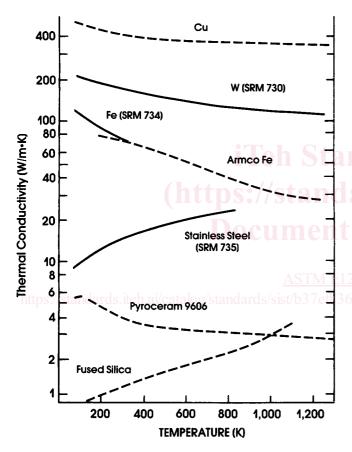


FIG. 1(b) Schematic of Typical Test Stack and Guard System Illustrating Matching of Temperature Gradients

(1).³ 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 λ_S , as uncorrected for heat shunting) can then be determined using the following equation where the notation is shown in Fig. 1:

$$\lambda_s = \frac{Z_4 - Z_3}{T_4 - T_3} \cdot \frac{\lambda_M}{2} \cdot \left(\frac{T_2 - T_1}{Z_2 - Z_1} + \frac{T_6 - T_5}{Z_6 - Z_5} \right) \tag{1}$$

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, temperature 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. 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 λ_M , it is preferable to use standards available from a national standards laboratory. Other reference materials are available because numerous measurements of λ 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 λ_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 λ_M values shall be stated in the report.
- 6.1.3 The requirements for any reference material include 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 λ_I increase as λ_M / λ_s varies from unity, (1) the reference which has a λ_M nearest to λ_S should be used for the meter bars.
- 6.1.4 If a sample's thermal conductivity λ_s is between the thermal conductivity values of two types of reference materials, the reference material with the higher λ_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 exists for reducing both radial heat flow in the column-guard annulus and surrounds, and for 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 λ_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

³ The boldface numbers in parentheses refer to a list of references at the end of this test method.

TABLE 1 Reference Materials For Use as Meter Bars

Material	Temperature Range (K)	Percentage Uncertainty in λ (\pm %)	λ _M (W/m-K)	Material Source
Electrolytic Iron SRM 734	To 1000	2	Α	NIST ^A
Tungsten SRM 730	4 to 300	2	λ_M Dependent on T ^A	NIST ^A
_	300 to 2000	2 to 5		
	>2000	5 to 8		
Austenitic Stainless SRM 735	4 to 1200	<5 %	$\lambda_M = 1.22 T^{0.432} T > 200 K^A$	NIST ^A
Iron	80 to 1200	2	λ_M should be calculated from measured values BC	
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	•••	EF	manufacturer
Fused Silica ^G	1300	<8	$\lambda_M = (84.7/T) + 1.484 + 4.94 \times 10^{-4}$	manufacturer
		Up to 900 K	$T + 9.6 \times 10^{-13} T^{4HI}$	
Pyrex 7740	90 to 600	6	EF	manufacturer

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

TABLE 2 Suitable Thermal Insulation Materials

Material ^A	Typical Thermal Conductivity (W/(m·K))			
Material	300K	800K	1300K	
Poured Powders	(Mttk	DS://	Stand	
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 ³	0.044	0.13	A S 0.33	
Zirconia 60-90 kg/m ³	0.039	0.09	0.25	

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

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 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 E230.

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

^B Fulkerson W., et al., *Physics Review 167*, p. 765, (1968).

^C Lucks C. F., Journal of Testing and Evaluation, ASTM 1 (5), 422 (1973).

^D Moore, J. P., Graves, R. S. and McElroy, D. L., Canadian Journal of Physics, 45, 3849 (1967).

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

F 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.

^G Hust J. G., Cryogenics Division; NBS, Boulder, Colorado 80302.

^H 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.

¹ 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.