Designation: E 1225 – 04

Standard Test Method for Thermal Conductivity of Solids by Means of the Guarded-Comparative-Longitudinal Heat Flow Technique[1]

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 (ϵ) 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, λ, of homogeneous-opaque solids (see Notes 1 and 2). This test method is for materials with effective thermal conductivities in the approximate range $0.2 < \lambda < 200$ W/(*m*·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]

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus

C 408 Test Method for Thermal Conductivity of Whiteware Ceramics

C 1045 Practice for Calculating Thermal Transmission Properties Under Steady-State Conditions

D 4351 Test Method for Measuring the Thermal Conductivity of Plastics by the Evaporation-Calorimetric Method

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques

E 230 Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples

F 433 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*·K)),
λ_M^1	=	thermal conductivity of top meter bar (W/(*m*·K)),
λ_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)),
$\lambda_I(T)$	=	thermal conductivity of insulation as a function of temperature, (W/(*m*·K)),
T	=	absolute temperature (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 E 1225–99.

[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.

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, λ_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 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 **(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:

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

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

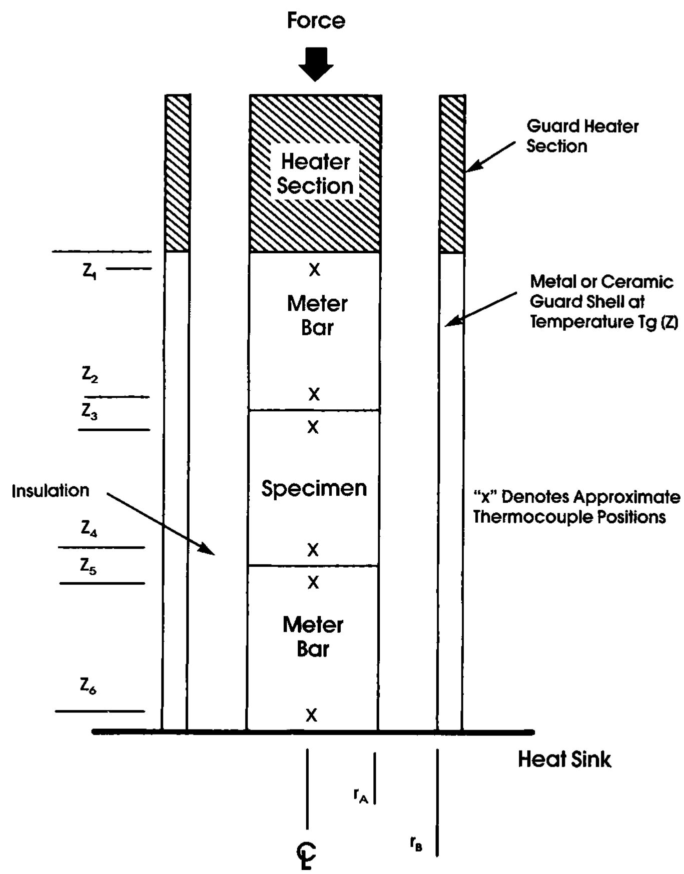


FIG. 1(a) Schematic of a Comparative-Guarded-Longitudinal Heat Flow System Showing Possible Locations of Temperature Sensors

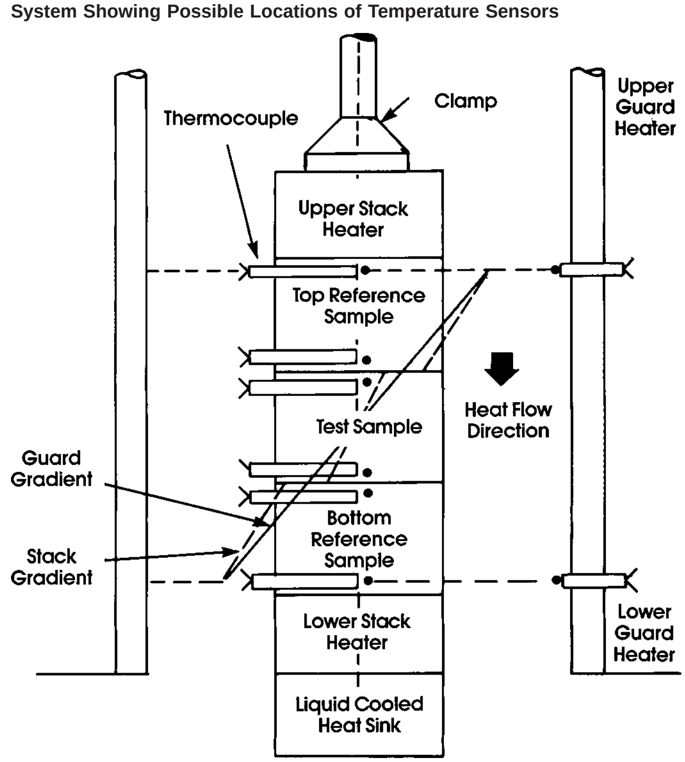
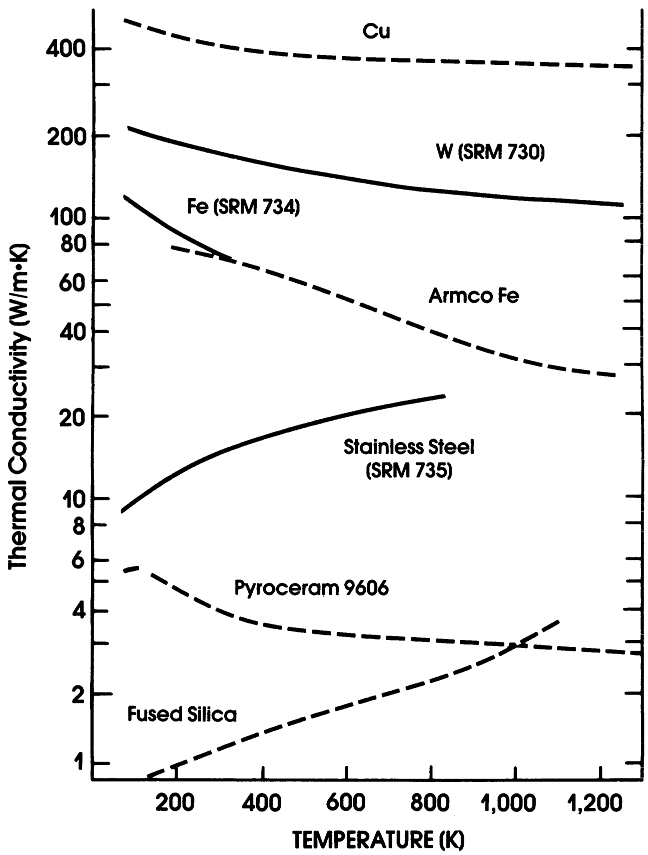


FIG. 1(b) Schematic of Typical Test Stack and Guard System Illustrating Matching of Temperature Gradients

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 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

TABLE 1 Reference Materials For Use as Meter Bars

Material	Temperature Range (K)	Percentage Uncertainty in λ (± %)	λ_M(*W/m·K*)	Material Source
Electrolytic Iron SRM 734	To 1000	2	[A]	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 %	$\lambda_M = 1.22T^{0.432}$ T > 200K[A]	NIST[A]
Iron	80 to 1200	2	λ_M should be calculated from measured values[BC]	...
Copper	90 to 1250	<2	$\lambda_M = 416.3 - 0.05904T + 7.087 \times 10^7/T^3$[D]	manufacturer
Pyroceram Code 9606	90 to 1200	...	[EF]	manufacturer
Fused Silica[G]	1300	<8 Up to 900 K	$\lambda_M = (84.7/T) + 1.484 + 4.94 \times 10^{-4}$ T $+ 9.6 \times 10^{-13}T^4$[HI]	manufacturer
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.
[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.
[I] 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[A]	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^3	0.044	0.13	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.

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)**.

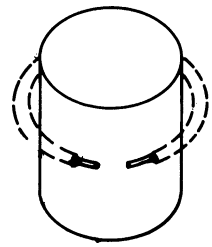
6.3.6 Since uncertainty in temperature sensor location leads to large errors, special care must be taken to determine the correct distance between sensors and to calculate the possible error resulting from any uncertainty.

6.4 *Reduction of Contact Resistance*:

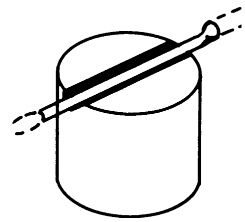
6.4.1 This test method requires uniform heat transfer at the meter bar to specimen interfaces whenever the temperature sensors are within a distance equal to r_A from an interface **(6)**. This requirement necessitates a uniform contact resistance across the adjoining areas of meter bars and specimens. 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.

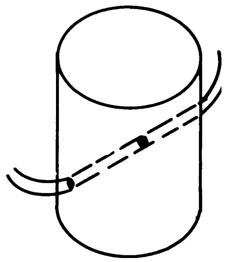
6.4.3 Means shall be provided for imposing a reproducible and constant load along the column with the primary purpose



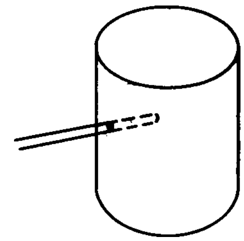
3a
Intrinsic weld with separate temperature elements welded to specimen or meter bars so that signal is through the material.



3b
Radial slots on the flat surfaces to hold a bare wire or ceramic insulated thermocouple sensor the may be bonded into slot.



3c
Small radial hole drilled through the specimen or meter bar and non-insulated (permitted if the material is an electrical insulator) or insulated thermocouple pulled through the hole.



3d
Small Radial hole drilled part way through the specimen or meter bar and a thermocouple pushed into the hole.

NOTE 1—In all cases the thermoelements should be thermally tempered and/or thermally grounded on the guard to minimize temperature measurement errors due to heat flow into or out of the hot junction.

FIG. 3 Thermocouple Attachments

of minimizing interfacial resistances at meter bar-specimen interfaces. Since the force applied to the column usually affects the contact resistance, it is desirable that this force be variable to ensure that λ_S does not change with force variation. This force can be applied either pneumatically, hydraulically, by spring action, or by putting a dead weight on the column. The above load mechanisms have the advantage of remaining constant with change in column temperature. In some cases, the compressive strength of the specimen might be so low that the applied force must be limited to the dead weight of the upper meter bar. In this case, special care must be taken to limit errors caused by poor contact, by judicious positioning of temperature sensors away from any heat flow perturbation at the interfaces.

6.5 *Guard Cylinder*:

6.5.1 The specimen-meter bar column shall be enclosed within a guard tube or pipe normally of right circular symmetry. This guard cylinder can be either a metal or a ceramic but its inside radius should be such that the ratio r_B/r_A will be between 2.0 and 3.5 **(1)**. This guard cylinder shall contain at least one heater for controlling the temperature profile along the guard.

6.5.2 The guard shall be constructed and operated so that the temperature of the guard surface is either isothermal and equal to the approximate mean temperature of the specimen or preferably has an approximately linear profile with the top and bottom ends of the guard matched to corresponding positions along the column. In each case, at least three temperature sensors shall be attached to the guard at known positions to measure the temperature profile.

6.6 *System Instrumentation*:

6.6.1 The combination of temperature sensor and the instrument used for measuring the sensor output shall be adequate to ensure a temperature measurement precision of ±0.04 K and an absolute error less than ±0.5 %.

6.6.2 Instrumentation for this technique shall be adequate to maintain the required temperature control and measure all pertinent output voltages with accuracy commensurate with the system capability. Although control can be manual, a technique of this general description can be automated so that a computer carries out all the control functions, acquires all pertinent voltages, and calculates the thermal conductivity **(7)**.

7. Sampling and Conditioning Test Specimens

7.1 *Test Specimens*—This test method is not restricted to a particular geometry. General practice is to use cylindrical or square cross-sections. The conduction area of the specimen and reference samples must be the same to within 1 % (see Note 3) and any difference in area shall be taken into account in the calculations of the result. For the cylindrical configuration, the radii of the specimen and meter bars must agree to within ±1 % and the specimen radius, r_A, must be such that r_B/r_A is between 2.0 and 3.5. Each flat surface of the specimen and reference must be flat with a surface finish equal to or better than 32− and the normal to each end shall be parallel with the specimen axis to within ±10 min.

NOTE 3—In some cases this requirement is not necessary. For example, some apparatus might consist of meter bars and specimen with high values of λ_M and λ_S so that thermal shunting errors would be small for long sections. These sections might be long enough to permit temperature sensor attachment to be far enough away from the interfaces to ensure that heat flow was uniform. The specimen length should be selected based on considerations of radius and thermal conductivity. When λ_M is higher than the thermal conductivity of SRM 735 (stainless steel), long specimens with length / r_A>>1 can be used. These long specimens permit the use of large distances between temperature sensors and this reduces the percentage error derived from the uncertainty in sensor position. When λ_M is lower than the thermal conductivity of SRM 735, the sample's length must be reduced because uncertainty due to the heat shunting becomes too large.

7.2 *Sampling and Conditioning*—Unless specifically required or prescribed, one representative specimen shall be prepared from the sample and no preconditioning has to be undertaken.

8. Calibration and Verification

8.1 There are many situations that call for equipment verifications before operations on unknown materials can be successfully accomplished. These include the following:

8.1.1 After initial equipment construction,

8.1.2 When the ratio of λ_M to λ_S is less than 0.3 or greater than 3 and it is not possible to match thermal conductance values,

8.1.3 When the specimen shape is complex or the specimen is unusually small,

8.1.4 When changes have been made in the system geometry,

8.1.5 When meter bar or insulation material other than those listed in 5.1 and 5.2 are considered for use, and

8.1.6 When the apparatus has been previously operated to a high enough temperature to change the properties of a component such as thermocouples' sensitivity.

8.2 These verification tests shall be run by comparing at least two reference materials in the following manner:

8.2.1 A reference material which has the closest thermal conductivity to the estimated thermal conductivity of the unknown sample should be machined according to 6.1, and

8.2.2 The thermal conductivity λ of the specimen fabricated from a reference material shall then be measured as described in Section 9, using meter bars fabricated from another reference material which has the closest λ to that of the specimen. For example, verification tests might be performed on a Pyroceram™ 9606 specimen using meter bars fabricated from SRM 735 stainless steel. If the measured thermal conductivity of the specimen disagrees with the value from Table 1 after applying the corrections for heat exchange, additional effort is required to find the error source(s).

9. Procedure

9.1 Where possible and practical, select the reference specimens (meter bars) such that the thermal conductance is of the same order of magnitude as that expected for the test specimen. After instrumenting and installing the proper meter bars, the specimen should be instrumented similarly. It should then be inserted into the test stack such that it is at aligned between the meter bars with at least 99 % of each specimen surface in contact with the adjacent meter bar. Soft foil or other contacting medium may be used to reduce interfacial resistance. If the system must be protected from oxidation during the test or if operation requires a particular gas or gas pressure to control λ_I, the system should be pumped and purged, and the operating gas and pressure established. The predetermined force required for reducing the effects of non-uniform interfacial resistance should be applied to the load column.

9.2 Heaters at either end of the column should be energized (see Note 4) and adjusted until the temperature differences between positions Z_1 and Z_2, Z_3 and Z_4, and Z_5 and Z_6 are between 200 times the imprecision of the ΔT measurements and 30 K, and the specimen is at the average temperature desired for the measurement. Although the exact temperature profile along the guard is not important for $r_B/r_A \geq 3$, the power to the guard heaters should be adjusted until the temperature profile along the guard, $T_g(z)$, is constant with respect to time to within ±0.1 K and either:

9.2.1 Approximately linear so that $T_g(z)$ coincides with the temperature along the sample column at a minimum of three places including the temperature at the top sensor on the top meter bar, the bottom sensor on the bottom bar, and the specimen midplane; or

9.2.2 Constant with respect to z to within ±5 K and matched to the average temperature of the test specimen.

NOTE 4—These heaters can either be attached to the ends of the meter bars or to a structure adjacent to the meter bar. The heaters can be powered with A.C. or D.C. Several different heater configurations are acceptable. The power to these heaters shall be steady enough to maintain short term temperature fluctuations less than ±0.03 K on the meter bar thermocouple nearest the heater. These two heaters, in conjunction with the guard shell heater and the system coolant shall maintain long term temperature drift less than ±0.05 K/h.

9.3 After the system has reached steady state (T drift <0.05 K/h), measure the output of all temperature sensors.

10. Calculation

10.1 *Approximate Specimen Thermal Conductivity*:

10.1.1 The outputs from the temperature sensors shall be converted to temperature, and the apparent heat flow per unit area, q', in the meter bars shall be calculated using the following:

$$q'_T = \lambda_M \cdot \frac{T_2 - T_1}{Z_2 - Z_1} \quad \text{top bar} \tag{2}$$

$$q'_B = \lambda_M \cdot \frac{T_6 - T_5}{Z_6 - Z_5} \quad \text{bottom bar} \tag{3}$$

In each of these equations, the λ_M value (see Note 5) to be inserted shall be obtained from the information of 6.1 for the average meter bar temperature. Although these two values, q'_T and q'_B, should agree with each other to within about ±10 % when heat exchange with the insulation is small, good agreement is not a sufficient condition (nor always a necessary condition) for low heat shunting error.

10.1.2 A value for the specimen thermal conductivity at temperature $(T_3 + T_4)/2$, as uncorrected for heat exchange with the insulation, can then be calculated using the following:

$$\lambda'_S = \frac{(q'_T + q'_B)(Z_4 - Z_3)}{2(T_4 - T_3)} \tag{4}$$

NOTE 5—This type of calculation procedure actually requires only two temperature sensors on each column section. In this case, the third sensor on each section serves as a test for consistency of the other two. Some calculation procedures require more than the two sensors to obtain more knowledge about *dT/dZ*.

10.2 *Corrections for Extraneous Heat Flow*:

10.2.1 Calculation of the specimen thermal conductivity by a simple comparison of temperature gradients in the meter bars to that in the specimen is less valid when the specimen or meter bars, or both, have low thermal conductivities relative to that of the insulation. The apparatus should be designed to minimize these errors. The deviation from uniform heat flow has been expressed as follows **(1)** :

$$\gamma = F_g F_\lambda \tag{5}$$

where F_g is a function of system dimensions, and F_λ is a function of λ_M, λ_I, and λ_S **(1).** The F_g term has a value between 2 and 3 for the ratio of guard radius to column radius specified for this system. The F_λ term is shown in Fig. 4 as a function of λ/λ_I for various values of λ_M/λ_I for a linear guard. At high ratios of λ_M/λ_I and λ_S/λ_I, corrections would not be necessary since the departure from ideal heat flow would be small. For example, the product of F_λ and F_g would be less than 0.10 (10 %) for all measurements where λ_M/λ_I and λ_S/λ_I are greater than 30. If the value of F_gF_λ is to be kept below 10 %, the ratios λ_M/λ_I and λ_s/λ_I must be within the boundaries on Fig. 4.

10.2.2 Measurements on materials where the ratios of λ_M/λ_I and λ_S/λ_I do not fall within these boundaries shall be accompanied with corrections for extraneous heat flow. These corrections can be determined in the following three different ways:

10.2.2.1 Use of analytical techniques as described by Didion **(1)** and Flynn **(8)**,

10.2.2.2 Using calculations from finite-difference or finite-element heat conduction codes, and

10.2.2.3 Determined experimentally by using several reference materials or transfer standards of different thermal conductance as specimens. The procedure must be used cautiously since all such specimens should have the same size as the specimen with an unknown thermal conductivity and have the same surface finish.

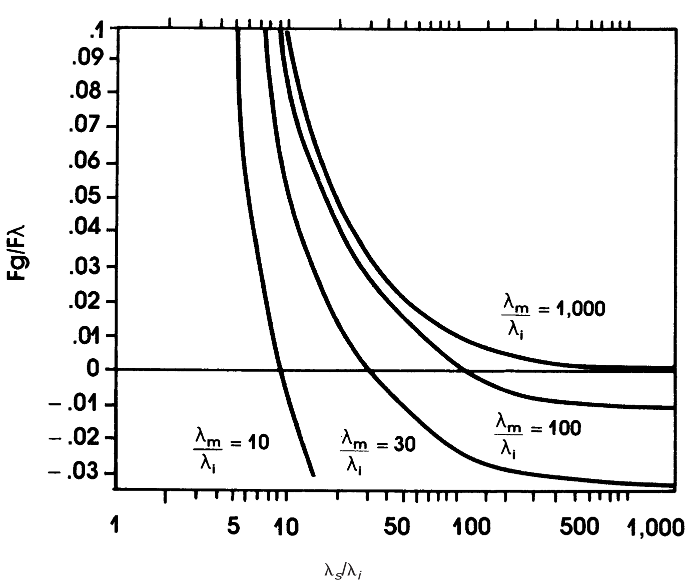


FIG. 4 Fractional Heat Exchange Between the Meter Bar-Specimen Column and Surrounding Insulation as a Function of λ_m/λ_i for Several Values of λ_s/λ_i

11. Report

11.1 The report of the test results shall include the following:

11.1.1 Complete specimen identification including shape and size,

11.1.2 Complete identification of insulation and source of λ_I values, gas, and gas pressure,

11.1.3 Statements of thermocouple type, size, and attachment procedure,

11.1.4 Complete listing of the geometrical dimensions of the system including r_A, r_B, specimen height, meter bar height, and distances between temperature sensors,

11.1.5 Column force,

11.1.6 Meter bar material and source of λ_M values if other than those listed in Table 1,

11.1.7 Reference to the use of this test method shall include a statement of the percentage variation of the qualification results about the true value. For example, "thermal conductivity results on Pyroceram™ 9606 using SRM 735 meter bars were within ±4 % of the accepted values for Pyroceram™ over the temperature range from 250 to 900 K,"

11.1.8 Variations, if any, from this test method. If results are to be reported as having been obtained by this method, then all requirements prescribed by this method shall be met. Where such conditions are not met, the phrase, "All requirements of this method have been met with the exception of ..." shall be added and a complete list of the exceptions included;

11.1.9 Measured values of temperature and specimen thermal conductivity.

12. Precision and Bias

12.1 *Example of Error Estimation*:

12.1.1 Assumptions for a system where both meter bars and the specimen are of equal length is that the sensor spacings are all 13 mm and $\lambda_M \approx \lambda_S$:

$$\left| \frac{\delta\lambda_M}{\lambda_M} \right| = |\, 0.003\, | \tag{6}$$

$$Z_2 - Z_1 \sim Z_4 - Z_3 \sim Z_6 - Z_5 = 13 \text{ mm};$$
$$T_2 - T_1 \sim T_4 - T_3 \sim T_6 - T_5 = 10 \text{ K};$$
$$\delta(Z_2 - Z_1) \sim \delta(Z_4 - Z_3) \sim \delta(Z_6 - Z_5) = 0.2 \text{ mm; and}$$
$$\delta(T_2 - T_1) \sim \delta(T_4 - T_3) \sim \delta(T_6 - T_5) = 0.04 \text{ K}.$$

12.1.2 The maximum value of $\delta(Z_2 - Z_1)$ etc. was approximated by assuming an uncertainty of ±0.5 (sensor diameter) at each temperature measurement position. Therefore, if the diameter of each sensor is 0.2 mm, the uncertainty in the difference would be ±0.2 mm. The number for $\delta(T_2 - T_1)$ etc. was calculated based on the sensor absolute accuracy.

12.1.3 With these values the fractional uncertainty in λ'_S will be |0.069| or ±6.9 %.

12.2 *Indeterminate Errors*:

12.2.1 There are at least three other errors that can contribute to total system error and these are (1) non-uniform interfacial resistance, (2) heat exchange between the column and the guard, and (3) heat shunting through the insulation around the column. These three errors must be minimized or appropriate corrections applied to the data if the desired accuracy is to be obtained.

12.2.2 The contributions from the last two errors can be determined approximately using results from appropriate experiments carried out at different levels of guard temperature to specimen stack temperature out of balance.

12.3 *Overall*—An international, inter-laboratory round robin study also involving absolute methods **(9)** has shown that a precision of ±6.8 % can be attained over the temperature range 300 to 600 K. Although no definite bias could be established these are indications that the values were on the order of 2 % lower than those obtained by absolute methods. This cited paper is on file at ASTM as a research report.

REFERENCES

(**1**) Didion, D. A., "An Analysis and Design of a Linear Guarded Cut-Bar Apparatus for Thermal Conductivity Measurements," AD-665789, January, 1968, available from the National Technical Information Service, Springfield, VA.

(**2**) Finch, D. I., "General Principles of Thermoelastic Thermometry," in *Temperature, It's Measurement and Control in Science and Industry*, Vol. 3, Part 2, Section 1, Reinhold Publishing Corporation.

(**3**) Caldwell, F. R., "Temperatures of Thermocouple Reference Junctions in an Ice Bar," *J. Res. Nat. Bur. Std., 69C* (2) (1965).

(**4**) ASME: PTC 19.3, *Temperature Measurement*, Part 3, p. 1232, 1974.

(**5**) Anderson, R. L., and Kollie, T. G., "Problems in High Temperature Thermometry," July, 1976, pp. 171–221.

(**6**) Fried, E., *Thermal Conductivity*, Vol 2, Tye, R. P. (ed.), Academic Press, New York (1969).

(**7**) Morgan, M. T., and West, G. A., "Thermal Conductivity of the Rocks in the Bureau of Mines Standard Rock Site," *Thermal Conductivity*, 16, Larsen, D. C., ed., Plenum Press, NY, NY, 1983, pp 79–90.

(**8**) Flynn, D. R., "Thermal Conductivity of Ceramics," *Mechanical and Thermal Properties of Ceramics*, Special Publication 303, National Bureau of Standards, 1969.

(**9**) Hulstrom, L. C., Tye, R. P., and Smith, S. E., "Round-Robin Testing of Thermal Conductivity Reference Materials," *Thermal Conductivity*, 19, Yarbrough, D. W., ed., Plenum Press, New York. (See also High-Temp-High Pressures, Vol 17, p. 707, 1985).