Designation: E1225 – 20

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

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 U.S. 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 applicable to materials with effective thermal conductivities in the range $0.2 < \lambda < 200$ W/(*m*·K) over the temperature range between 90 K 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 and composites.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

1.4 *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. Referenced Documents

2.1 *ASTM Standards:*[2]

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *thermal conductivity, λ, n*—the time rate of heat flow, under steady conditions, through unit area, per unit temperature gradient in the direction perpendicular to the area.

3.1.2 *apparent thermal conductivity, n*—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.

3.2 *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)

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[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.

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)

4. Summary of Test Method

4.1 A test specimen is inserted under an applied load between two similar specimens of a material of known thermal properties and a thickness such that the thermal conductance is of similar order to that of the specimen. 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 similar 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 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 mechanical or pneumatic force is applied to the column to ensure good contact between specimens. The stack is surrounded by a thermal insulation material of thermal conductivity, λ_I, 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 usually by maintaining the top at a temperature, T_T, and the bottom at a lower 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 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:

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

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 precision is to be achieved.

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

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 (SRM or CRM) available from a national metrology institute.* Other reference materials are available due to numerous measurements of λ having been made by a number of organizations including national metrology institutes, and with general acceptance of the values. Table 1 lists the recommended reference materials and contain thermal conductivity values for the appropriate temperature range. Fig. 2 illustrates the approximate variation of λ_M with temperature.

6.1.2 Table 1 is not exhaustive and other materials may be used as references providing the property values that are used are those for the specific material referenced (**1**). 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 temperature sensor 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 (**2**), the reference which has a λ_M nearest to λ_S should be used for the meter bars.

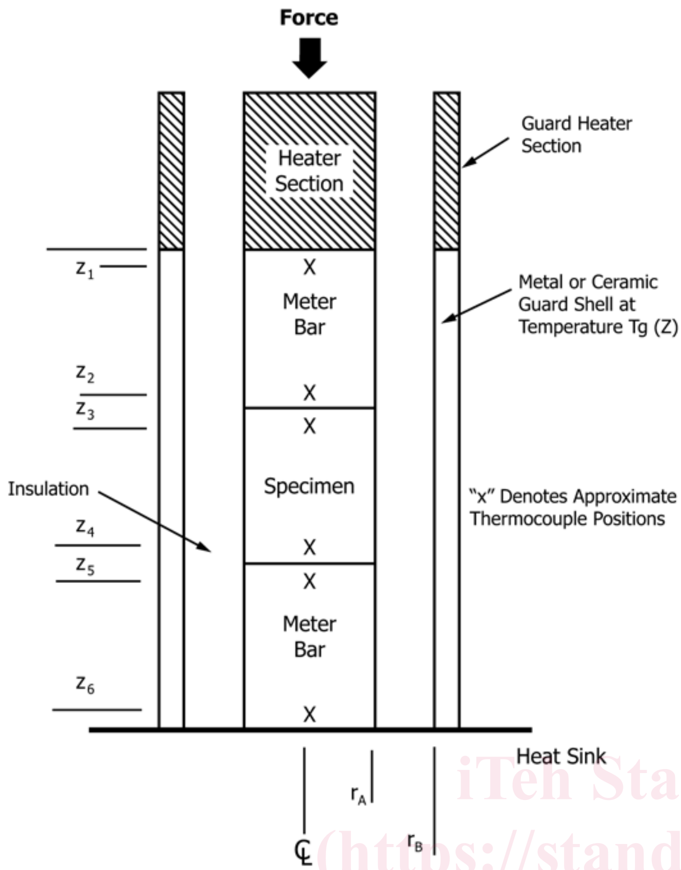

FIG. 1(a) Schematic of a Comparative-Guarded-Longitudinal Heat Flow System Showing Possible Locations of Temperature Sensors

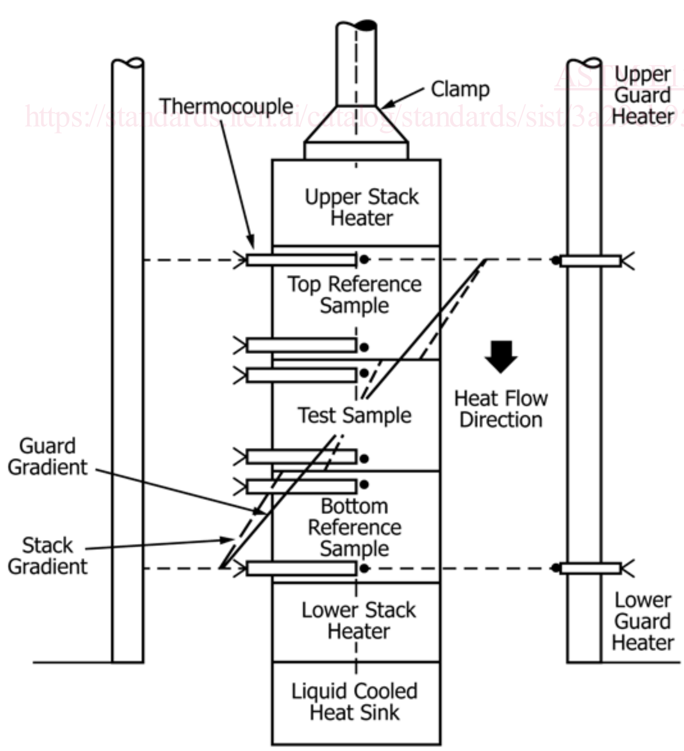

FIG. 1(b) Schematic of Typical Test Stack and Guard System Illustrating Matching of Temperature Gradients

FIG. 1 Schematics

6.1.4 If the thermal conductivity λ_s of the specimen is between the values for two reference materials, the reference material with the higher λ_M should be used in order to reduce the total temperature drop along the column.

6.2 *Insulation Materials:*

6.2.1 A 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 shall 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 be used.

6.2.2 Recommended thermal insulations are listed in Table 6.

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 (**3**) 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 of temperature grade materials and should be 0.1 mm diameter or less and with the individual wires in suitable twin-bore protective insulation. A constant temperature reference shall always be provided for all cold junctions. This reference can be an ice-cold slurry (**4**), a constant temperature zone box, or an electronic ice point reference. All thermocouples shall be fabricated from either calibrated thermocouple wire (**5**) 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.

TABLE 1 Reference Materials For Use as Meter Bars

Material	Temperature Range (K)	Percentage Uncertainty (± %)	Thermal Conductivity (W/*m*·K)
Electrolytic Iron[A,B]	2 to 1000	2	See Table 2.
Tungsten[C]	4 to 300	2	See Table 3.
	300 to 2000	2 to 5	
	>2000	5 to 8	
Austenitic Stainless[D]	200 to 1200	<5 %	See Table 4.
Copper[E]	85 to 1250	<2	$\lambda_M = 416.31 - 0.05904T + 7.0872 \times 10^7 / T^3$
Pyroceram[F,G,H,I,J,K]	298 to 1025 K	6.5	$\lambda = 2.332 + 515.2 / T$
		4 for $T > 300$ K	$\lambda = 3.65367 - 6.64042 \times 10^{-4} T - 218.937T^1 + 116163T^2$
Fused Silica[L,M]	1300	<8 Up to 900 K	$\lambda_M = (84.7 / T) + 1.484 + 4.94 \times 10^{-4} T + 9.6 \times 10^{-13} T^4$
Pyrex[N,K,O,P,Q]	90 to 600	<2 for $T > 200$ K	$\lambda = 1.1036 + 1.659 \times 10^{-3} (T - 273.15) - 3.982 \times 10^{-6} (T - 273.15)^2 + 6.746 \times 10^{-9} (T - 273.15)^3$ K
	140 to 470		
310 Stainless Steel[K,R]	300 to 1020	4	$\lambda = 12.338 + 1.781 \times 10^{-2} (T - 273.15)$
430 Stainless Steel[K,R]	300 to 770	4	$\lambda = 20.159 + 1.589 \times 10^{-2} (T - 273.15) - 1.283 \times 10^{-5} (T - 273.15)^2$
Inconel 600[S,K,R]	300 to 1020	4	$\lambda = 12.479 + 1.648 \times 10^{-2} (T - 273.15) + 3.741 \times 10^{-6} (T - 273.15)^2$
Nimonic 75[T,K,R]	300 to 1020	4	$\lambda = 11.958 + 1.657 \times 10^{-} (TT - 273.15) + 3.252 \times 10^{-6} (T - 273.15)^2$
Vespel SP1[V,W]	300 to 600	5	See Table 5.

[A] SRM 8420 is available from National Institute of Standards and Technology (NIST), Gaithersburg, MD.[U]
[B] Hurst, J. G., and Lankford, A. B., "Report of Investigation, Research Materials 8420 and 8421, Electrolytic Iron, Thermal Conductivity and Electrical Resistivity as a Function of Temperature from 2 to 1000K," National Institutes of Standards and Technology (nee National Bureau of Standards), Gaithersburg, MD, 1984.
[C] Hurst, J. G., and Giarratano, P. J., Certificate, Standard Reference Material 730, *Thermal Conductivity – Tungsten*, National Institutes of Standards and Technology (nee National Bureau of Standards), Gaithersburg, MD, 1976.
[D] Hurst, J. G., Sparks, L. L., and Giaarratano, P. J., Certificate, Standard Reference Material 735, *Thermal Conductivity – Austenitic Stainless Steel*, National Institutes of Standards and technology (nee National Bureau of Standards), Gaithersburg, MD, 1975.
[E] Moore, J. P., McElroy, D. L., and Graves, R. S., "Thermal Conductivity and Electrical Resistivity of High-Purity Copper from 78 to 400 °K," *Canadian Journal of Physics*, Vol 45, 1967, pp. 3849–3865.
[F] Pyroceram is a trademark by Corning Incorporated, Corning, NY.
[G] Salmon, D. R., Roebben, G., and Brandt, R., "Certification of Thermal Conductivity and Thermal Diffusivity up to 1025 K of Glass-Ceramic Reference Material BCR-720," EUR Report 21764, Institute for Reference Materials and Measurements (IRMM), Geel, Belgium, 2007.
[H] Stroe, D. E., Thermitus, M. A., and Jacobs – Fedore, R. A., "Thermophysical Properties of Pyroceram 9606," *Thermal Conductivity 27 / Thermal Expansion 15*, H. Wang, W. Porter, eds., DEStech Publications, Lancaster, PA, 2005, pp. 382–390.
[I] BCR-2013 is available from the Institute for Reference Materials and Measurements (IRMM), Geel, Belgium.[U]
[J] BCR-724 is available from the Laboratory of the Government Chemists (LGC), Teddington, Middlesex, UK.[U]
[K] Tye, R. P., and Salmon, D. R., "Development of New Thermal Conductivity Reference Materials: A Summary of Recent Contributions by National Physical Laboratory," *Thermal Conductivity 27 / Thermal Expansion 15*, H. Wang (ed.), DEStech Publications, Lancaster PA, 2005, pp. 372–381.
[L] 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.
[M] 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, IN. See *Thermophysical Properties of Matter*, Vol 3, Touloukian, Y., ed., Plenum Press, New York, 1970.
[N] Pyrex is a trademark by Corning Incorporated, Corning, NY.
[O] Tye, R. P., and Salmon, D. R., "Thermal Conductivity Certified Reference Materials: Pyrex 7740 and Polymethylmethacrylate," *Thermal Conductivity 26 / Thermal Expansion 14*, R. Dinwiddie, ed., DEStech Publications, Lancaster, PA, 2005, pp. 437–451.
[P] BCR-39 is available from the Institute for Reference Materials and Measurements (IRMM), Geel, Belgium.[U]
[Q] Salmon, D., "Thermal Conductivity of Insulations Using Guarded Hot Plates, including Recent Developments and Sources of Reference Materials," *Measurement Science and Technology*, Vol 12, 2001, pp. R89–R98.
[R] Clark, J., and Tye, R., "Thermophysical Properties Reference Data for Some Key Engineering Alloy," *High Temperatures – High Pressures*, Vols 35/36, 2003/2004, pp. 1–14.
[S] Inconel is a trademark by Special Metals Corporation, Huntington, WV.
[T] Nimonic is a trademark by Special Metals Corporation, Huntington, WV.
[U] This is the sole source of supply of this material known to the committee at this time. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.
[V] Vespel is a trademark by E.I. Dupont du Nemours, Wilmington, DE.
[W] Jacobs-Fedore, R. A., and Stroe, D. E., "Thermophysical Properties of Vespel SP1," *Thermal Conductivity 27 / Thermal Expansion 15*, H. Wang, W. Porter, eds., DEStech Publications, Lancaster, PA, 2005, pp. 231–238.

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 (**6**).

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 (**7**). This requirement necessitates a uniform contact resistance

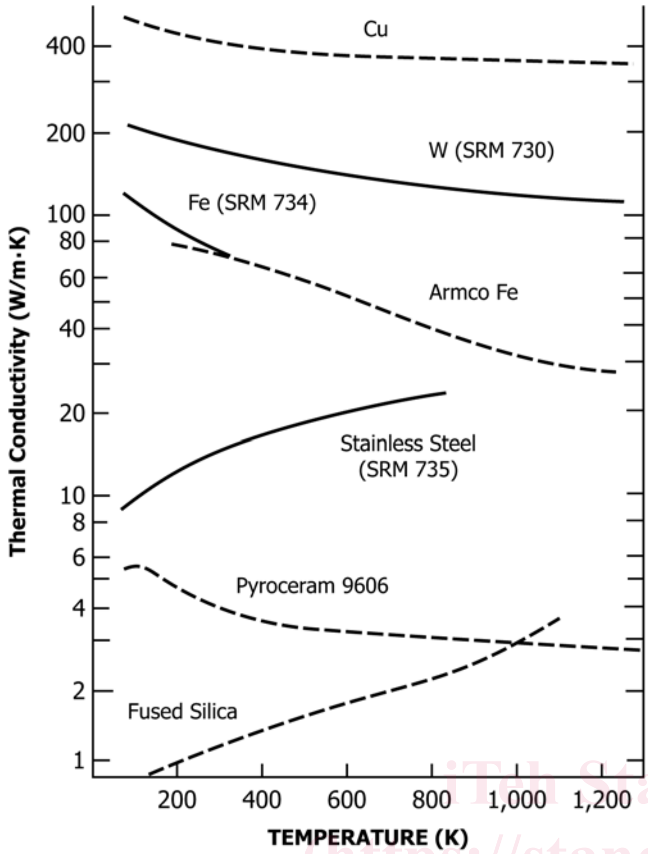

FIG. 2 Approximate for the Thermal Conductivity of Recommended Reference Materials

TABLE 2 Thermal Conductivity of Electrolytic Iron[A]

Temperature, K	Thermal Conductivity, (W/*m*·K)
80	123.8
90	115.4
100	108.9
150	92.7
200	86.7
250	81.5
300	76.4
400	67.5
500	60.2
600	53.6
700	47.49
800	41.96
900	37.12
1000	32.98

[A] Hurst, J. G., and Lankford, A. B., Report of Investigation, Research Materials 8420 and 8421, *Electrolytic Iron, Thermal Conductivity and Electrical Resistivity as a Function of Temperature from 2 to 1000K*, National Institute of Standards and Technology (nee Bureau of Standards), 1984.

TABLE 3 Thermal Conductivity of Tungsten[A]

Temperature, K	Thermal Conductivity, (W/*m*·K)
80	229
90	218
100	211
120	202
140	197
160	194
180	190
200	187
250	180
300	172
350	164
400	157
450	151
500	146
600	138
700	132
800	127
900	123
1000	120
1200	114

[A] Hurst, J. G., and Giarratano, P. J., Certificate, Standard Reference Material 730, *Thermal Conductivity — Tungsten*, National Institute of Standards and Technology (nee National Bureau of Standards), 1976.

TABLE 4 Thermal Conductivity of Austenitic Stainless Steel[A]

Temperature, K	Thermal Conductivity, (W/*m*·K)
80	8.27
85	8.55
90	8.80
95	9.04
100	9.25
110	9.65
120	9.99
130	10.3
140	10.6
150	10.9
160	11.1
170	11.4
180	11.6
190	11.9
200	12.1
250	13.2
300	14.3
350	15.3
400	16.2
450	17.1
500	17.9
600	19.3
700	20.6
800	21.9
900	23.0
1000	24.1
1100	25.1
1200	26.1

[A] Hurst, J. G., Sparks, L. L., and Giarratano, P. J., Certificate, Standard Reference Material 735, *Thermal Conductivity — Austenitic Stainless Steel, Thermal Conductivity as a Function of Temperature (5 to 1200K)*, National Institute of Standards and Technology (nee National Bureau of Standards), 1975.

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 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 (**8**).

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