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**Designation: E1225 − 13**

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

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  $(\varepsilon)$  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,  $\lambda$ , 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 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 changes of thickness or cross-sectional area by more than  $\pm 5\%$ . For<br>composites or heterogeneous systems consisting of slabs or plates bonded<br>together, the specimen should be more than 20 units wide and 20 units<br> $\frac{3.$ 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 thick, respectively, where a unit is the thickness of the thickest slab or<br>plate, so that diameter or length changes of one-half unit will affect the<br>**expandant** in the direction permeaning that are not proposed or apparent  $\lambda_4$  by less than  $\pm 5$ %. For systems that are non-opaque or partially transparent in the infrared, the combined error due to inhomopartially transparent in the infrared, the combined error due to inhomo-<br>geneity and photon transmission should be less than  $\pm 5$  %. Measurements<br>on highly transparent solids must be accompanied with infrared ebecation on highly transparent solids must be accompanied with infrared absorption coefficient information, or the results must be reported as apparent thermal conductivity,  $λ_A$ .

Nore 2—This test method may also be used to evaluate the contact  $- a61d-489600$  -6209e33478d4/astm-e1225-13 thermal conductance/resistance of materials. 3.1.2 *Symbols:*

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 and health 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 Recom-* *mendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

# **2. Referenced Documents**

2.1 *ASTM Standards:*<sup>2</sup>

E230 [Specification and Temperature-Electromotive Force](https://doi.org/10.1520/E0230) [\(EMF\) Tables for Standardized Thermocouples](https://doi.org/10.1520/E0230)

# **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 **ASTM E122 thermal conductivity for the material tested.** 

$$
\lambda_M(T) = \text{thermal conductivity of meter bars (reference materials) as a function of temperature, (W/ (m·K)),}
$$

- *λM*  $=$  thermal conductivity of top meter bar (W/ (*m*·K)),
- *λM*  $=$  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)),
- $λ'$ <sub>s</sub> $(T)$ = thermal conductivity of specimen calculated by ignoring heat exchange correction, (W/ (*m*·K)),
- *λI*  $=$  thermal conductivity of insulation as a function of temperature, (W/(*m*·K)),

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee [E37](http://www.astm.org/COMMIT/COMMITTEE/E37.htm) on Thermal Measurements and is the direct responsibility of Subcommittee [E37.05](http://www.astm.org/COMMIT/SUBCOMMIT/E3705.htm) on Thermophysical Properties.

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<sup>2</sup> 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.

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## **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,  $\lambda_{\rm S}$ , but having an estimated thermal conductance of  $\lambda_s / l_s$ , is mounted between two meter bars of improved processes in the standard standard in the thorough standards in the standard standard in the thorough standard in the thorough standard in the thoroug known thermal conductivity,  $\lambda_M$ , of the same cross-section and similar thermal conductance,  $\lambda_M / l_M$ . A more complex but apparatus are outlined in Social state arrangement is a column consisting of a disk heater suitable arrangement is a column consisting of a disk heater with a specimen and a meter bar on each side between heater with a specimen and a meter bar on each side between heater<br>and heat sink. Approximately one-half of the power would then<br>then the power would then<br> $\frac{1}{2}$  and the preview between the power would then<br> $\frac{1}{2}$  and the 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- $22$  the n 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,  $T<sub>g</sub>(z)$ . 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  $T<sub>o</sub>(z)$  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>3</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  $\lambda_s$ , as uncorrected for heat shunting) can then be determined using the following equation where the notation is shown in Fig. 1:

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

$$
\lambda_{s} = \frac{Z_{4} - Z_{3}}{T_{4} - T_{3}} \cdot \frac{\lambda_{M}}{2} \cdot \left( \frac{T_{2} - T_{1}}{Z_{2} - Z_{1}} + \frac{T_{6} - T_{5}}{Z_{6} - Z_{5}} \right)
$$
(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.

# **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 uncerectional dimensions are larger than the thickness it is  $_{\odot}$  atainty in  $\lambda_M$ , it is preferable to use standards available from a National Metrology Institute. 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 some of the recognized reference materials. 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 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  $\lambda_I$ 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's thermal conductivity  $\lambda_{\rm s}$  is between the thermal conductivity values of two types of reference materials, the reference material 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 exists for reducing both radial heat flow in the column-guard annulus and surrounds, and for heat shunting



**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**

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 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. distinct advantages.

6.3.3 When thermocouples are employed, they should be **(https://standards.iteh.ai)** a fabricated from wires which are 0.1 mm diameter or less. A constant temperature reference shall always be provided for all constant temperature reference shall always be provided for all cold junctions. This reference can be an ice-cold slurry **(3)**, a **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

> 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.

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



<sup>A</sup> SRM 8420 is available from National Institute of Standards and Technology (NIST), Gaithersburg, MD.<sup>*U*</sup><br><sup>*B* Hurst, J. G., and Lankford, A. B., "Report of Investigation, Research Materials 8420 and 8421, Electrolytic </sup> Function of Temperature from 2 to 1000K," National Institutes of Standards and Technology (nee National Bureau of Standards), Gaithersburg, MD, 1984.<br><sup>C</sup> Hurst, J. G., and Giarratano, P. J., Certificate, Standard Reference

National Bureau of Standards), Gaithersburg, MD, 1976.

*<sup>D</sup>* 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,USA, 1975.<br><sup>*E* Moore, J. P., Graves, R. S., and McElroy, D. L., "Thermal Conductivity and Electrical Resistivity of High-Purity Copper from </sup> Vol 45, 1967, pp. 3849–3865.

*<sup>F</sup>* Pyroceram is a trademark by Corning Incorporated, Corning, NY.

' Pyroceram is a trademark by Corning incorporated, Corning, NY.<br><sup>G</sup> Salmon, D. R., Roebben, G., and Brandt,R., "Certification of Thermal Conductivity and Thermal Diffusivity up to 1025 K of Glass-Ceramic Reference Materia EUR Report 21764, Institute for Reference Materials and Measurements (IRMM), Geel, Belgium, 2007.

*<sup>H</sup>* 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. ancaster, PA, 2005, pp. 382–390.<br>
ancaster, PA, 2005, pp. 382–390.<br>
ference Materials and Measurements (IRMM), Geel, Belgium.<sup>*U*</sup><br>
In Government Chemists (I.GC). Teddington Middlesey. IIK <sup>U</sup>

<sup>7</sup> BCR-2013 is available from the Institute for Reference Materials and Measurements (IRMM), Geel, Belgium.<sup>*U*</sup><br><sup>J</sup> BCR-724 is available from the Laboratory of the Government Chemists (LGC), Teddington, Middlesex, UK.<sup>U</sup>

*Thermal Conductivity 27/ Thermal Expansion 15*, H. Wang (ed.), DEStech Publications, Lancaster PA, 2005, pp. 372–381.<br><sup>/</sup> Above 700 K a large fraction of heat conduction in fused silica will be by radiation and the actual 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, Indiana.<br>
N Pyrex is a trademark by Corning Incorporated, Corning, NY.

<sup>O</sup> Tye, R. P., and Salmon, D. R., "Thermal Conductivity Certified Reference Materials: Pyrex 7740 and Polymethylmethacrylate," Thermal Conductivity 26 / Thermal<br>Expansion 14, R. Dinwiddie, ed., DEStech Publications, Lanca

*BCR*-39 is available from the Institute for Reference Materials and Measurements (IRMM), Geel, Belgium.<sup>*U*</sup>

**POCH-39 is avaluative indition and insulted in The Development Conductivity (in the assumption of the USIN Developments and Sources of Reference Materials,"** *Measurement* **Conductivity of Insulations Using Guarded Hot Plat** 

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.<br><sup>S</sup> Inconel is a trademark by Special Metals Corporation, Huntington WV.

*<sup>S</sup>* Inconel is a trademark by Special Metals Corporation, Huntington WV. *<sup>T</sup>* Nimonic is a trademark by Special Metals Corporation, Huntington WV.

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# 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 of minimizing interfacial resistances at meter bar-specimen

## **TABLE 2 Suitable Thermal Insulation Materials**



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



Thermal Conductivity — Tungsten, National Institute of Standards and Technology (nee National Bureau of Standards), 1976.

**TABLE 4 Thermal Conductivity of Tungsten***<sup>A</sup>*

4 154 6 231 8 306 10 377 12 444 14 503 16 553 18 591 20 618 30 585 40 438 50 330 60 275

Thermal Conductivity, (*W/m·K*)

Temperature,  $\frac{K}{4}$ 

800 41.96<br>900 37.12  $\frac{900}{37.12}$ 1000 32.98 *<sup>A</sup>* Hurst, J. G., and Lankford, A. B., Report of Investigation, Research Materials 8420 and 8421, Electrolytic Iron, Thermal Conductivity and Electrical Resistivity as

200 86.7 250 81.5 300 76.4 400 67.5 500 60.2 600 53.6 700 47.49

a Function of Temperature from 2 to 1000K, National Institute of Standards and Technology (nee Bureau of Standards), 1984.

interfaces. Since the force applied to the column usually affects the contact resistance, it is desirable that this force be variable to ensure that  $\lambda_s$  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.

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#### 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  $\pm 0.04$  K and an absolute error less than  $\pm 0.5$  %.

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)**.

7.1 *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  $\pm 1$  % 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  $\pm 10$  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  $\lambda_M$  and  $\lambda_S$  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  $\lambda_M$  is higher than the thermal conductivity of 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  $\lambda_M$  is lower than the thermal conductivity of stainless steal, the sample's length must be reduced because uncertainty due to the heat shunting becomes too large.