Designation: C 177 – 97

Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus[1]

This standard is issued under the fixed designation C 177; 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 establishes the criteria for the laboratory measurement of the steady-state heat flux through flat, homogeneous specimen(s) when their surfaces are in contact with solid, parallel boundaries held at constant temperatures using the guarded-hot-plate apparatus.

1.2 The test apparatus designed for this purpose is known as a guarded-hot-plate apparatus and is a primary (or absolute) method. This test method is comparable, but not identical, to ISO 8302.

1.3 This test method sets forth the general design requirements necessary to construct and operate a satisfactory guarded-hot-plate apparatus. It covers a wide variety of apparatus constructions, test conditions, and operating conditions. Detailed designs conforming to this test method are not given but must be developed within the constraints of the general requirements. Examples of analysis tools, concepts and procedures used in the design, construction, calibration and operation of a guarded-hot-plate apparatus are given in Refs **(1-41)**.[2]

1.4 This test method encompasses both the single-sided and the double-sided modes of measurement. Both distributed and line source guarded heating plate designs are permitted. The user should consult the standard practices on the single-sided mode of operation, Practice C 1044, and on the line source apparatus, Practice C 1043, for further details on these heater designs.

1.5 The guarded-hot-plate apparatus can be operated with either vertical or horizontal heat flow. The user is cautioned however, since the test results from the two orientations may be different if convective heat flow occurs within the specimens.

1.6 Although no definitive upper limit can be given for the magnitude of specimen conductance that is measurable on a guarded-hot-plate, for practical reasons the specimen conductance should be less than 16 W/(m ^{2}K).

1.7 This test method is applicable to the measurement of a wide variety of specimens, ranging from opaque solids to porous or transparent materials, and a wide range of environmental conditions including measurements conducted at extremes of temperature and with various gases and pressures.

1.8 Inhomogeneities normal to the heat flux direction, such as layered structures, can be successfully evaluated using this test method. However, testing specimens with inhomogeneities in the heat flux direction, such as an insulation system with thermal bridges, can yield results that are location specific and shall not be attempted with this type of apparatus. See Test Methods C 976 or C 236 for guidance in testing these systems.

1.9 Calculations of thermal transmission properties based upon measurements using this method shall be performed in conformance with Practice C 1045.

1.10 In order to ensure the level of precision and accuracy expected, persons applying this standard must possess a knowledge of the requirements of thermal measurements and testing practice and of the practical application of heat transfer theory relating to thermal insulation materials and systems. Detailed operating procedures, including design schematics and electrical drawings, should be available for each apparatus to ensure that tests are in accordance with this test method. In addition, automated data collecting and handling systems connected to the apparatus must be verified as to their accuracy. This can be done by calibration and inputting data sets, which have known results associated with them, into computer programs.

1.11 It is not practical for a test method of this type to establish details of design and construction and the 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. The user may also find it necessary, when repairing or modifying the apparatus, to become a designer or builder, or both, on whom the demands for fundamental understanding

[1] This test method is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurement.

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

and careful experimental technique are even greater. Standardization of this test method is not intended to restrict in any way the future development of new or improved apparatus or procedures.

1.12 This test method does not specify all details necessary for the operation of the apparatus. Decisions on sampling, specimen selection, preconditioning, specimen mounting and positioning, the choice of test conditions, and the evaluation of test data shall follow applicable ASTM Test Methods, Guides, Practices or Product Specifications or governmental regulations. If no applicable standard exists, sound engineering judgment that reflects accepted heat transfer principles must be used and documented.

1.13 This test method allows a wide range of apparatus design and design accuracy to be used in order to satisfy the requirements of specific measurement problems. Compliance with this test method requires a statement of the uncertainty of each reported variable in the report. A discussion of the significant error factors involved is included.

1.14 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only. Either SI or Imperial units may be used in the report, unless otherwise specified.

1.15 *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.* Specific precautionary statements are given in Note 21.

1.16 Major sections within this test method are arranged as follows:

2. Referenced Documents

2.1 *ASTM Standards:*

C 167 Test Methods for Thickness and Density of Blanket or Batt Thermal Insulations[3]

C 168 Terminology Relating to Thermal Insulating Materials[3]

C 236 Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box[3]

C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus[3]

C 687 Practice for Determination of Thermal Resistance of Loose-Fill Building Insulation[3]

C 976 Test Method for Thermal Performance of Building Assemblies by Means of a Calibrated Hot Box[3]

C 1043 Practice for Guarded-Hot-Plate Design Using Circular Line-Heat Sources[3]

C 1044 Practice for Using the Guarded-Hot-Plate Apparatus in the One-Sided Mode to Measure Steady-State Heat Flux and Thermal Transmission Properties[3]

C 1045 Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements[3]

C 1058 Practice for Selecting Temperatures for Evaluating and Reporting Thermal Properties of Thermal Insulation[3]

E 230 Specification for Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

2.2 *ISO Standard:*

ISO 8302 Thermal Insulation—Determination of Steady-State Areal Thermal Resistance and Related Properties—Guarded-Hot-Plate Apparatus[6]

2.3 *ASTM Adjuncts:*ASTM

Table of Theoretical Maximum Thickness of Specimens and Associated Errors[7]

Descriptions of Three Guarded-Hot-Plate Designs[7]

Line-Heat-Source Guarded Hot-Plate Apparatus[8]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms and symbols used in this test method, refer to Terminology C 168 and the following subsections.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *auxiliary cold surface assembly*, *n*— the plate that provides an isothermal boundary at the outside surface of the auxiliary insulation.

3.2.2 *auxiliary insulation*, *n*—insulation placed on the back side of the hot-surface assembly, in place of a second test specimen, when the single sided mode of operation is used. (*Synonym*—backflow specimen.)

3.2.3 *cold surface assembly*, *n*—the plates that provide an isothermal boundary at the cold surfaces of the test specimen.

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[3] *Annual Book of ASTM Standards*, Vol 04.06.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[7] Available from ASTM Headquarters, Order Adjunct: ADJCO177.

[8] Available from ASTM Headquarters, Order Adjunct: ADJC1043.

3.2.4 *controlled environment*, *n*—the environment in which an apparatus operates.

3.2.5 *guard*, *n*—promotes one-dimensional heat flow. Primary guards are planar, additional coplanar guards can be used and secondary or edge guards are axial.

3.2.6 *guarded-hot-plate apparatus*, *n*—an assembly, consisting of a hot surface assembly and two isothermal cold surface assemblies.

3.2.7 *guarded-hot-plate*, *n*—the inner (rectangular or circular) plate of the hot surface assembly, that provides the heat input to the metered section of the specimen(s).

3.2.8 *hot surface/assembly*, *n*—the complete center assembly providing heat to the specimen(s) and guarding for the meter section.

3.2.9 *metered section*, *n*—the portion of the test specimen (or auxiliary insulation) through which the heat input to the guarded-hot-plate flows under ideal guarding conditions.

3.2.10 *mode, double-sided*, *n*—operation of the guarded-hot-plate apparatus for testing two specimens, each specimen placed on either side of the hot surface assembly.

3.2.11 *mode, single-sided*, *n*—operation of the guarded-hot-plate apparatus for testing one specimen, placed on one side of the hot-surface assembly.

3.2.12 *thermal transmission properties*, *n*— those properties of a material or system that define the ability of a material or system to transfer heat such as thermal resistance, thermal conductance, thermal conductivity and thermal resistivity, as defined by Terminology C 168.

3.3 *Symbols:Symbols*—The symbols used in this test method have the following significance:

3.3.1 ρ_m— specimen metered section density, kg/m^3.

3.3.2 ρ_s—specimen density, kg/m ^{3}.

3.3.3 λ—thermal conductivity, W/(m K).

3.3.4 σ—Stefan-Boltzmann constant, W/m^2 K^4.

3.3.5 A—metered section area normal to heat flow, m^2.

3.3.6 A_g—area of the gap between the metered section and the primary guard, m^2.

3.3.7 A_m—area of the actual metered section, m^2.

3.3.8 A_s—area of the total specimen, m^2.

3.3.9 C—thermal conductance, W/(m^2 K).

3.3.10 C_i—the specific heat of the *i*th component of the metered section, J/(kg K).

3.3.11 *dT/dt*—potential or actual drift rate of the metered section, K/s.

3.3.12 λ_g—thermal conductivity of the material in the primary guard region, W/(m K).

3.3.13 L—in-situ specimen thickness, m.

3.3.14 m—mass of the specimen in the metered section, kg.

3.3.15 m_i—the mass of the *i*th component, kg.

3.3.16 m_s—mass of the specimen, kg.

3.3.17 Q—heat flow rate in the metered section, W.

3.3.18 q—heat flux (heat flow rate per unit area), Q, through area, A, W/m^2.

3.3.19 Q_{ge}—lateral edge heat flow rate between primary Guard and Controlled Environment, *W*.

3.3.20 Q_{gp}—lateral heat flow rate across the gap, *W*.

3.3.21 Q_{grd}—guard heat flow through Specimen, *W*.

3.3.22 Q_{se}—edge heat flow between Specimen and Controlled Environment, *W*.

3.3.23 R—thermal resistance, m^2 K/W.

3.3.24 ΔT—temperature difference across the specimen, $T_h - T_c$.

3.3.25 T_c—cold surface temperature, K.

3.3.26 T_h—hot surface temperature, K.

3.3.27 T_m—mean temperature, K, $(T_h + T_c)/2$.

4. Summary of Test Method

4.1 Fig. 1 illustrates the main components of the idealized system: two isothermal cold surface assemblies and a guarded-hot-plate. The guarded-hot-plate is composed of a metered section thermally isolated from a concentric primary guard by a definite separation or gap. Some apparatus may have more than one guard. The test specimen is sandwiched between these three units as shown in Fig. 1. In the double-sided mode of measurement, the specimen is actually composed of two pieces. The measurement in this case produces a result that is the average of the two pieces and therefore it is important that the two pieces be closely identical. For guidance in the use of the one-sided mode of measurement, the user is directed to Practice C 1044. For guidance in the use of a guarded-hot-plate incorporating the use of a line source heater, refer to Practice C 1043.

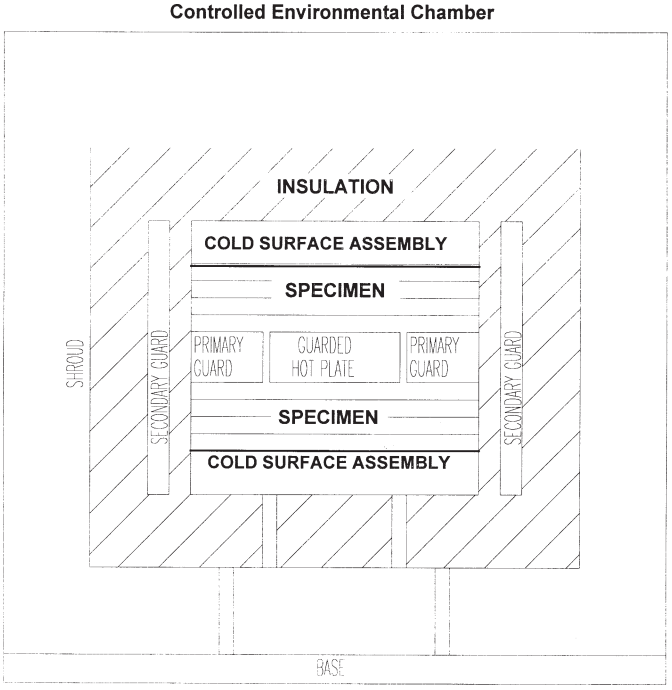

FIG. 1 General Arrangement of the Mechanical Components of the Guarded-Hot-Plate Apparatus

4.1.1 The guarded-hot-plate provides the power (heat flow per unit time) for the measurement and defines the actual test volume, that is, that portion of the specimen that is actually being measured. The function of the primary guard, and additional coplanar guard where applicable, of the guarded-hot-plate apparatus is to provide the proper thermal conditions within the test volume to reduce lateral heat flow within the

apparatus. The proper (idealized) conditions are illustrated in Fig. 1 by the configuration of the isothermal surfaces and lines of constant heat flux within the specimen.

4.1.2 Deviations from the idealized configuration are caused by: specimen inhomogeneities, temperature differences between the metered section and the guard (gap imbalance), and temperature differences between the outer edge of the assembly and the surrounding controlled environment (edge imbalance). These experimental realities lead to heat flow measurements that are too small or too large because the power supplied to the metered section is not exactly equal to that which flows through the specimen in the metered section. The resulting qualitative heat flows are depicted in Fig. 2.

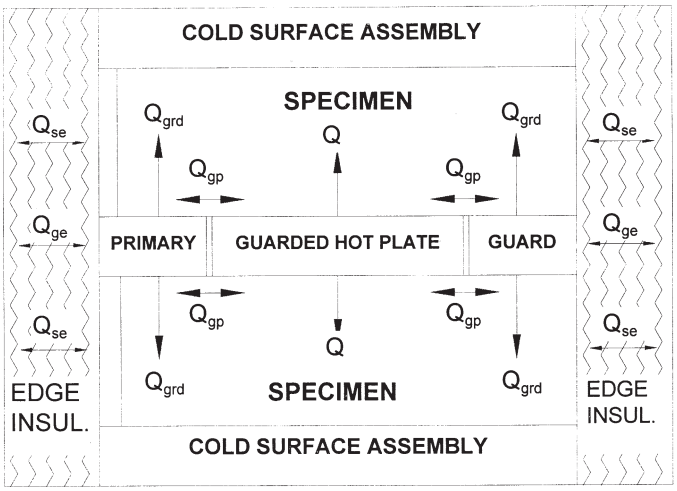

FIG. 2 Illustration of Idealized Heat Flow in a Guarded-Hot-Plate Apparatus

4.2 The three heating/cooling assemblies are designed to create isothermal surfaces on the faces of the specimens within the metered section. The two surfaces designated as the cold surface assemblies are adjusted to the same temperature for the double-sided mode of operation. In practice, because the plates and specimens are of finite dimensions, and because the external controlled environment is often at a temperature different from the edge of the metered section, some lateral heat flow occurs. The primary guard for the guarded hot plate limits the magnitude of the lateral heat flow in the metered section. The effectiveness of the primary guard is determined, in part, by the ratio of its lateral dimension to that of the metered section and to the specimen thickness **(6,7,8,20,31)**.

4.3 Compliance with this test method requires: the establishment of steady-state conditions, and the measurement of the unidirectional heat flow Q in the metered section, the metered section area A, the temperature gradient across the specimen, in terms of the temperature T_h of the hot surface and the temperature T_c of the cold surface, (or equivalently, the temperature T between the two surfaces), the thickness' L_1 and L_2 of each specimen, and guard balance between the metered section and primary guard.

5. Significance and Use

5.1 This test method covers the measurement of heat flux and associated test conditions for flat specimens. The guarded-hot-plate apparatus is generally used to measure steady-state heat flux through materials having a "low" thermal conductivity and commonly denoted as "thermal insulators." Acceptable measurement accuracy requires a specimen geometry with a large ratio of area to thickness.

5.2 Two specimens are selected with their thickness, areas, and densities as identical as possible, and one specimen is placed on each side of the guarded-hot-plate. The faces of the specimens opposite the guarded-hot-plate and primary guard are placed in contact with the surfaces of the cold surface assemblies.

5.3 Steady-state heat transmission through thermal insulators is not easily measured, even at room temperature. This is because heat may be transmitted through a specimen by any or all of three separate modes of heat transfer (radiation, conduction, and convection); any inhomogeneity or anisotropy in the specimen may require special experimental precautions to measure that flow of heat; hours or even days may be required to achieve the thermal steady-state; no guarding system can be constructed to force the metered heat to pass only through the test area of insulation specimen being measured; moisture content within the material may cause transient behaviour; and physical or chemical change in the material with time or environmental condition may permanently alter the specimen.

5.4 Application of this test method on different test insulations requires that the designer make choices in the design selection of materials of construction and measurement and control systems. Thus there may be different designs for the guarded-hot-plate apparatus when used at ambient versus cryogenic or high temperatures. Test thickness, temperature range, temperature difference range, ambient conditions and other system parameters must also be selected during the design phase. Annex A1 is referenced to the user, which addresses such issues as limitations of the apparatus, thickness measurement considerations and measurement uncertainties, all of which must be considered in the design and operation of the apparatus.

5.5 Apparatus constructed and operated in accordance with this test method should be capable of accurate measurements for its design range of application. Since this test method is applicable to a wide range of specimen characteristics, test conditions, and apparatus design, it is impractical to give an all-inclusive statement of precision and bias for the test method. Analysis of the specific apparatus used is required to specify a precision and bias for the reported results. For this reason, conformance with the test method requires that the user must estimate and report the uncertainty of the results under the reported test conditions.

5.6 Qualification of a new apparatus. When a new or modified design is developed, tests shall be conducted on at least two materials of known thermal stability and having verified or calibrated properties traceable to a national standards laboratory. Tests shall be conducted for at least two sets of temperature conditions that cover the operating range for the apparatus. If the differences between the test results and the national standards laboratory characterization are determined to be significant, then the source of the error shall, if possible, be identified. Only after successful comparison with the

certified samples, can the apparatus claim conformance with this test method. It is recommended that checks be continued on a periodic basis to confirm continued conformance of the apparatus.

5.7 The thermal transmission properties of a specimen of material: may vary due to the composition of the material; may be affected by moisture or other environmental conditions; may change with time or temperature exposure; may change with thickness; may change with temperature difference across the specimen; or may change with mean temperature. It must be recognized, therefore, that the selection of a representative value of thermal transmission properties for a material must be based upon a consideration of these factors and an adequate amount of test information.

5.8 Since both heat flux and its uncertainty may be dependent upon environmental and apparatus test conditions, as well as intrinsic characteristics of the specimen, the report for this test method shall include a thorough description of the specimen and of the test conditions.

5.9 The results of comparative test methods such as Test Method C 518 depend on the quality of the heat flux reference standards. The apparatus in this test method is one of the absolute methods used for generation of the reference standards. The accuracy of any comparative method can be no better than that of the referenced procedure. While the precision of a comparative method such as Test Method C 518 may be comparable with that of this test method, Test Method C 518 cannot be more accurate. In cases of dispute, this test method is the recommended procedure.

6. Apparatus

6.1 A general arrangement of the mechanical components of such a guarded-hot-plate apparatus is illustrated in Fig. 1. This consists of a hot surface assembly comprised of a metered section and a primary guard, two cold surface assemblies, and secondary guarding in the form of edge insulation, a temperature-controlled secondary guard(s), and often an environmental chamber. Some of the components illustrated in Fig. 1 are omitted in systems designed for ambient conditions, although a controlled laboratory environment is still required; edge insulation and the secondary guard are typically used only at temperatures that are more than ± 10°C (20°F) from ambient. At ambient conditions, the environmental chamber is recommended to help eliminate the effects of air movement within the laboratory and to help ensure that a dry environment is maintained.

6.1.1 The purpose of the hot surface assembly is to produce a steady-state, one-dimensional heat flux through the specimens. The purpose of the edge insulation, secondary guard, and environmental chamber is to restrict heat losses from the outer edge of the primary guard. The cold surface assemblies are isothermal heat sinks for removing the energy generated by the heating units; the cold surface assemblies are adjusted so they are at the same temperature.

6.2 *Design Criteria*—Establish specifications for the following specifications prior to the design. Various parameters influence the design of the apparatus and shall be considered throughout the design process, maximum specimen thickness; range of specimen thermal conductances; range of hot surface and cold surface temperatures; characteristics of the specimens (that is, rigidity, density, hardness); orientation of the apparatus (vertical or horizontal heat flow); and required accuracy.

6.3 *Hot Surface Assembly*—The hot surface assembly consists of a central metered section and a primary guard. The metered section consists of a metered section heater sandwiched between metered section surface plates. The primary guard is comprised of one or more guard heaters sandwiched between primary guard surface plates. The metered section and primary guard shall be thermally isolated from each other by means of a physical space or gap located between the sections. The hot surface assembly using a line-heat-source is covered in Practice C 1043.

NOTE 1—The primary guard, in some cases, is further divided into two concentric sections (double guard) with a gap separator to improve the guard effectiveness.

6.3.1 *Requirements*—The hot surface assembly shall be designed and constructed to satisfy the following minimum requirements during operation.

6.3.1.1 The maximum departure from a plane for any surface plate shall not exceed 0.025 % of the linear dimension of the metered section during operation.

NOTE 2—Planeness of the surface can be checked with a metal straightedge held against the surface and viewed at grazing incidence with a light source behind the straightedge. Departures as small as 2.5 μm are readily visible, and large departures can be measured using shim-stock, thickness gages or thin paper.

6.3.1.2 The average temperature difference between the metered section surface plate and the primary guard surface plate shall not exceed 0.2 K. In addition, the temperature difference across any surface plate in the lateral direction shall be less than 2 % of the temperature difference imposed across the specimen.

NOTE 3—When qualifying the apparatus, additional temperature sensors shall be applied to the surface plates of the metered section and primary guards that verify that the requirements of 6.3.1.2 are satisfied.

6.3.1.3 The surfaces of the metered and primary guard surface plates that are in contact with the test specimen shall be treated to maintain a total hemispherical emittance greater than 0.8 over the entire range of operating conditions.

NOTE 4—At high temperatures the importance of high emittance of the surfaces adjacent to the specimens cannot be stressed too strongly since radiative heat transfer predominates in many materials as the temperature increases.

6.3.1.4 The metered section and primary guard surface plates shall remain planar during the operation of the apparatus. See 6.3.1.1.

6.3.2 *Materials*—The materials used in the construction of the hot surface assembly shall be carefully chosen after considering the following material property criteria.

6.3.2.1 *Temperature Stability*—Materials are selected for the heaters and surface plates that are dimensionally and chemically stable and suitably strong to withstand warpage and distortion when a clamping force is applied. For modest temperatures, electric resistance heaters embedded in silicone have been successfully employed; at higher temperatures, heating elements sandwiched between mica sheets or inserted

into a ceramic core have been used. Surface plates for hot surface assemblies used at modest temperatures have been fabricated from copper and aluminum. High purity nickel alloys have been used for higher temperature applications.

6.3.2.2 *Thermal Conductivity*—To reduce the lateral temperature differences across the metered and primary guard surface plates, fabricate these plates from materials that possess a high thermal conductivity for the temperature and environmental conditions of operation. Copper and aluminum are excellent choices for modest temperature applications; at higher temperatures consider using nickel, high purity alumina or aluminum nitride. These are examples of materials used and the operator must fully understand the thermal conductivity versus temperature dependency of the materials selected.

6.3.2.3 *Emittance*—To obtain a uniform and durable high surface emittance in the desired range, select a surface plate material or suitable surface treatment, or both. For modest temperature applications, high emittance paints may be employed. Aluminum can be anodized to provide the necessary high emittance. For high temperature applications, most ceramics will inherently satisfy this requirement while nickel surface plates can be treated with an oxide coating.

6.3.2.4 *Temperature Uniformity*—Select a heating element design that will supply the necessary heat flux density for the range of specimen thermal conductances to be investigated. The design of the heating element shall also consider the heat flux distribution of the surface of the heating element. Most apparatus incorporate the use of a distributed electric resistance heating element dispersed uniformly across the metered section and the primary guard. The surface plates and heating elements shall be clamped or bolted together in a uniform manner such that the temperature difference requirements specified in 6.3.1.2 are satisfied. Bolting the composite constructions together has been found satisfactory.

6.3.2.5 The insertion of insulating sheets between the heating elements and surface plates (that is, to mount a gap temperature imbalance detector) is allowed. To satisfy the requirements of 6.3.1.2, similar sheets shall be mounted between the heating element and the opposing surface plate.

6.3.2.6 *Hot Surface Assembly Size*—Design criteria established in 6.2 will determine the size of the apparatus. The size of the metered section shall be large enough so that the amount of specimen material in contact with the metered section (and therefore being measured) can be considered representative of the material being tested.

6.3.2.7 After determining the maximum specimen thickness that will be tested by this design, refer to Adjunct, Table of Theoretical Maximum Thickness of Specimens and Associated Errors, regarding associated errors attributable to combinations of metered section size, primary guard width, and specimen thickness.

NOTE 5— Typically the width of the primary guard equal to approximately one-half of the linear dimension of the metered section has been found to reduce edge heat loss to acceptable levels.

6.3.2.8 *Heat Capacitance*—The heat capacity of the hot surface assembly will impact the time required to achieve thermal equilibrium. Selecting materials with a low specific heat will increase the responsiveness of the apparatus. The thickness of the surface plates needs to be carefully considered; thick plates assist in reducing lateral temperature distributions but reduce responsiveness. A balance between these requirements is needed.

6.4 *The Gap*—The metered section and the primary guard shall be physically separated by a gap. The gap provides a lateral thermal resistance between these sections of the hot surface assembly. The area of the gap in the plane of the surface plates shall not be more than 5 % of the metered section area.

6.4.1 The heater windings from the metered section and primary guard heating elements shall be designed to create a uniform temperature along the gap perimeter.

6.4.2 The metered section area shall be determined by measurements to the center of the gap that surrounds this area, unless detailed calculations or tests are used to define this area more precisely.

6.4.3 Any connections between the metered section and the primary guard shall be designed to minimize heat flow across the gap. If a mechanical means is used to satisfy the requirements of 6.3.1.4, these connections shall be fabricated with materials having a high thermal resistance. Instrumentation or heater leads that cross the gap should be fabricated with fine-gage wire and traverse the gap at an oblique angle.

6.4.4 The gap may be filled with a fibrous insulation. Packing the gap with this insulation has been found to maintain the metered section and primary guard surface plates planar. An additional benefit of this practice for high temperature applications is that the densely packed insulation reduces the amount of heat conducted across the gap spacing.

6.5 *Cold Surface Assembly*—The cold surface assembly consists of a single temperature controlled section and is comprised of a cold surface heater sandwiched between cold surface plates and a heat sink. It is recommended that the size of the cold surface assembly be identical to the hot surface assembly, including the primary guard. Cold surface assemblies may be constructed with a gap where operation of the apparatus is susceptible to edge loss effects. This design is the ideal design, however, this assembly has traditionally been constructed without a gap with great success.

NOTE 6—The temperature of the cold surface assembly may be maintained through the use of a temperature-controlled bath; in this instance, there is no need to install a cold surface heater. Care must be taken in this instance; the flow rate of the bath must be sufficient to satisfy the temperature uniformity requirements specified in 6.3.1.2 and 6.5.1.

6.5.1 *Requirements*—The cold surface assemblies shall be designed and constructed to satisfy all of the requirements of 6.3.1 except that, since only one surface plate of each cold surface assembly is in contact with the test specimens, the requirement that specifies the temperature difference between the surface plates shall not apply.

6.5.2 *Materials*—The criteria to select materials that will be used in the construction of the cold surface assemblies are identical to the hot surface assembly and are listed in 6.3.2.

6.5.3 *High Temperature Operation*—When the cold surface assemblies will be operated at high temperatures, several thin sheets of insulation may be inserted between the heat sink and

cold surface heater. The addition of these insulation sheets will reduce the energy requirements to the cold surface heater and extend service life.

6.6 *Additional Edge Loss Protection*— Deviation from one-dimensional heat flow in the test specimen is due to non-adiabatic conditions at the edges of the hot surface assembly and the specimens. This deviation is greatly increased when the apparatus is used at temperatures other than ambient. When the guarded-hot-plate apparatus is operated at temperatures that deviate from ambient by more than 10°C (20°F), the apparatus shall be outfitted with additional components to reduce edge losses. These components are described in the following sections and shall be used if edge losses cannot be minimized.

NOTE 7—Another means of assessing whether edge insulation is required is to attach a temperature sensor to the mid-height of the exterior edge of the specimen. Sufficient edge insulation is present if the edge temperature, T_e, satisfies the following requirement.

$$(T_e - T_m)/\Delta T < 0.05 \quad (1)$$

6.6.1 *Secondary Guard*—To reduce heat exchange between the edges of the guarded-hot-plate and the environment, the guarded-hot-plate shall be outfitted with a co-axial temperature-controlled container referred to as the secondary guard. The secondary guard will be employed to adjust the ambient temperature to approximate the mean temperature of the test specimen.

6.6.1.1 *Size*—The secondary guard should have an inner dimension that is at least twice the dimension of the hot surface heater and the height should be equal to the thickness of the hot surface heater plus twice the thickness of the thickest specimen that will be tested.

6.6.1.2 *Materials*—The materials used in the construction of the secondary guard are not as critical as those selected for the hot and cold surface assemblies. However, the materials used in the design of the secondary guard shall be selected so that they are thermally stable over the intended temperature range, the heating element shall be capable of producing the necessary heat flux density to adjust the ambient temperature, and a means of cooling the secondary guard is required if the apparatus is intended for use at temperatures below the laboratory ambient. The use of high thermal conductivity metals is recommended for the construction since the secondary guard should be isothermal.

NOTE 8—Successful secondary guard designs consist of a sheathed heater wire or cable wrapped around an adequately-sized metal tube and pressed against the metal tube with another sheet of metal. For low-temperature operation, a cooling coil has been wrapped around the exterior surface of the secondary guard.

6.6.1.3 *Location*—The secondary guard shall be positioned around the hot surface assembly such that a uniform spacing is created between the components. The height of the secondary guard shall be adjusted such that the mid-height of the secondary guard is aligned with the center of the hot surface assembly thickness.

6.6.2 *Edge Insulation*—The interspace between the hot and cold surface assemblies, specimens and the secondary guard shall be filled with an insulating material. Due to the complex shapes of this interspace, a powder or fibrous insulation is recommended.

6.6.2.1 The selection of an edge insulation material will depend on the test conditions. Vermiculite is easy to use but should not be employed at temperatures above 540°C (1000°F) because it's thermal conductivity increases dramatically with temperature.

NOTE 9—Avoid the use of vermiculite when the guarded-hot-plate is used to evaluate specimens in different gaseous environments; vermiculite is extremely hygroscopic and the system is difficult to evacuate when it is used.

NOTE 10—Care shall be taken to ensure that there are no voids, pockets, or other extraneous sources of radiative heat transfer occurring at or near the guarded-hot-plate.

6.6.3 *Enclosure*—The guarded-hot-plate shall be placed inside an enclosure when the apparatus is used in to maintain a gaseous environment that is different than the laboratory ambient.

6.6.3.1 For low-temperature operation, a dry gas environment shall be used to prevent condensation from occurring on the cold surface assemblies and specimens.

6.6.3.2 For high temperature operation, it may be desirable to protect the apparatus from severe degradation by using a non-oxidizing gas.

6.6.3.3 The enclosure can also be used for substituting different gaseous environments and control of the ambient pressure.

6.7 *Clamping Force*—A means shall be provided for imposing a reproducible constant clamping force on the guarded-hot-plate to promote good thermal contact between the hot and cold surface assemblies and the specimens and to maintain accurate spacing between the hot and cold surface assemblies. It is unlikely that a force greater than 2.5 kPa (50 lb/ft ^{2}) will be required for the majority of insulating materials. In the case of compressible materials, a constant pressure arrangement is not needed and spacers between the plates may be necessary to maintain constant thickness.

6.7.1 A steady force, that will thrust the cold surface assemblies toward each other can be imposed by using constant-force springs or an equivalent method.

6.7.2 For compressible specimens, spacers are required if the test thickness can not be measured by other means. The spacers shall be small in cross-section and located near the exterior perimeter of the primary guard. Avoid placing spacers on surfaces where underlying sensors are being used to measure plate conditions.

NOTE 11—Because of the changes of specimen thickness possible as a result of temperature exposure, or compression by the plates, it is recommended that, when possible, specimen thickness be measured in the apparatus at the existing test temperature and compression conditions. Gaging points, or measuring studs along the outer perimeter of the cold surface assemblies, will serve for these measurements. The effective combined specimen thickness is determined by the average difference in the distance between the gaging points when the specimen is in place in the apparatus and when it is not in place.

6.8 *Temperature Measurements*:

6.8.1 *Imbalance Detectors*—A suitable means shall be provided to detect the average temperature imbalance between surface plates of the metering section and the primary guard.

6.8.1.1 *Sensors*—The gap region shall be instrumented with temperature sensors to monitor and control the average temperature imbalance across the gap. Fine-gage thermocouples connected as thermopiles are often used for this purpose, although other temperature control sensors, such as thermistors, have been used. Highly alloyed thermocouples, rather than pure metals, should be used to maximize the thermal resistance across the gap. Because of nonuniform heat flux within the surface plates, temperature imbalance is not always constant along the gap perimeter. It has been found that with proper design the thermal conductance of the wires crossing the gap can be made relatively small and, therefore, a large number of thermocouples can be used to increase the gap imbalance sensitivity. It is not uncommon to use ten or more sensing elements.

6.8.1.2 *Sensitivity*—The detection system shall be sufficiently sensitive to ensure that variation in measured properties due to gap temperature imbalance shall be restricted to not more than 0.5 % of the metered section power, as determined experimentally or analytically.

NOTE 12—The sensitivity of many temperature sensors is reduced drastically at temperatures below the laboratory ambient. Particular care must be used in designing thermopile measurement systems to operate under these conditions.

6.8.1.3 *Location*—When using only a minimum number of sensing elements along the gap, the most representative positions to detect the average balance for a square plate are those at a distance from the corners equal to one-fourth of the side of the metering area. The corners and the axes should be avoided. For a round plate, the sensors should be spaced equally around the gap.

6.8.1.4 Electrically isolated gap imbalance sensors should be placed on both surface plates of the guarded heating unit to average the imbalance on both faces of the heating unit.

6.8.1.5 Thermal junctions or other sensitive elements should each be located in similar areas of the hot surface assembly. It is suggested that all junctions should be located at points directly adjacent to the centers of the areas between heater windings. Any leads crossing the gap should be thermally anchored to the primary guard to provide a heat sink from external thermal variations. In some instances it may be desirable to provide a heat sink for these leads outside the primary guard to minimize any radial heat flow.

6.8.2 *Temperature Sensors*—Methods possessing adequate accuracy, such as thermistors, thermocouples, diodes and precision resistance thermometers may be used for the measurement of temperatures in the apparatus. Thermocouples are the most widely used detector due to their wide range of applicability and accuracy. The goal is to measure the temperature gradient within the specimen, and the method chosen (sensors mounted on the specimen surface, in grooves, or between interior layers) should be that which yields the highest accuracy in the measurement of the temperature gradient. A discussion of these alternatives is provided in 6.8.2.3 and 6.8.2.4.

6.8.2.1 *Use of Thermocouples*—Precautions should be used to minimize spurious voltages in temperature control and measuring circuits. Spurious voltages, due to wire inhomogeneities, generally increase as the temperature gradients within the measuring leads increase. For the same reason, junctions between dissimilar metal leads should not be made in the regions of appreciable temperature gradients. Low thermal emf switches should be used in the temperature measurement circuits. An insulated, isothermal box of heavy sheet metal can be used when joining leads of dissimilar metals in the thermocouple circuit. It is recommended that all connections of thermocouple wire to copper wire be accomplished within the isothermal box in order that the junctions are at the same temperature; then the copper, not the thermocouple, leads are connected to the needed switching devices and/or voltmeters.

6.8.2.2 *Accuracy*—Thermocouples whose outputs are used to calculate thermal transmission properties shall be fabricated from either calibrated thermocouple wire or wire that has been certified by the supplier, and shall have a standard limit of error equal to or less than the specifications of Tables E 230. The resulting error in temperature differences due to distortion of the heat flow around the sensor, to sensor drift, and other sensor characteristics shall be less than 1 %.

6.8.2.3 *Methods of Attachment*—The surface temperatures of the specimens are most often measured by means of permanently mounted thermocouples placed in grooves cut into the surface plates. Precautions shall be taken to ensure that the thermocouple is thermally anchored to the surface being measured. This method of instrumentation is employed when the contact resistance between the specimen and the surface plates is a small fraction of the specimen thermal resistance. The hot- and cold-surface assembly plate sensors on each side are sometimes connected differentially. Thermocouples mounted in this manner shall be made of wire not larger than 0.6 mm in diameter for large apparatus and preferably not larger than 0.2 mm for small apparatus.

NOTE 13—This method of deploying thermocouples is traditionally used for compressible specimens and for rigid specimens possessing flat surfaces that have a thermal resistance of greater than 0.2 m^2 K/W (1 h ft^2 F/Btu) at ambient conditions.

NOTE 14—For rigid specimens not satisfying the requirements of 6.8.2.2, two techniques for attaching temperature sensors are recommended. Small grooves may be cut into the surfaces of the specimens and thermocouples can be affixed into these grooves. As an alternative, thermocouples may be installed onto the surfaces of the specimen and thin sheets of a compressible homogeneous material interposed between the specimen and surface plates. In this latter case, an applied force should be used as indicated in 6.7 to ensure sufficient surface contact. For either of these applications, thermocouples shall be made of wire not larger than 0.2 mm in diameter.

6.8.2.4 *Electrical Isolation*—Temperature sensors can be either completely insulated electrically from the surface plates or grounded to the surface plate at one location. Consequently, thermocouples connected differentially can only have a single junction ground. Computations or experimental verifications, or both, shall be performed to confirm that other circuits do not affect the accuracy of the temperature measurements.

6.8.2.5 *Number of Sensors*—The number of temperature sensors on each side of the specimen in the metering area shall not be less than $10 \times \sqrt{A}$, or 2, whichever is greater.

NOTE 15—It is recommended that one temperature sensor be placed in the center of the metered section and that additional sensor be uniformly distributed radially.

6.9 *Thickness Measurements*—A means shall be provided for measuring the thickness of the specimen, preferably in the apparatus, to within 0.5 %.

6.10 *Metered Section Power Measurement*— Dc power is highly recommended for the metered section. Ac power may be used but the user should note that ac power determinations are more prone to error than dc measurements. The power to the metered section is determined with a wattmeter or from voltage and current measurements across the heater in the metered section. The voltage taps for this measurement should be placed to measure the voltage from the mid-point of the gap. The current can be determined from the voltage drop across a precision resistor placed in series with the metered section heater.

6.11 *Electrical Measurement System*— A measuring system having a sensitivity and accuracy of at least ± 0.1 K shall be used for measurement of the output of all temperature and temperature difference detectors. The system shall have sufficient sensitivity to measure the gap imbalance to a level equal to 1 % of the imbalance detector output that satisfies the requirement of 6.8.1.2. Measurement of the power to the metered section shall be made to within 0.2 % over the entire operating range.

6.12 *Performance Checks*—When a new apparatus is commissioned or an apparatus has undergone significant refurbishment, a series of careful checks shall be performed before initiating routine testing.

6.12.1 *Planeness*—The planeness of each surface plate shall be measured. See 6.3.1.1.

6.12.2 *Temperature Measurements*—With specimens installed in the apparatus, the coolant supply to the cold surface assembly shut off, and no electrical power being supplied to any of the heaters, mount the apparatus inside the enclosure. Allow the system sufficient time to come to thermal equilibrium. With no energy being supplied to the apparatus, note the output of all of the temperature sensors. The temperature sensors shall have an output that agrees to within the uncertainty prescribed in 6.8.2.2. The output of the imbalance detection circuit shall be within the noise level of the electrical measurement system.

6.12.3 *Imbalance Detection*—Determine the maximum imbalance that can be allowed that satisfies the requirements in 6.8.2.2. With the apparatus energized and operating normally, note the thermal resistance of a specimen and the imbalance detector output at equilibrium. Repeat the test at various levels of imbalance. Linearly fit the thermal resistance data as a function of bias. The slope of this relationship will define the maximum imbalance detector output that can be allowed during routine operation.

NOTE 16—The number of bias levels that need to be analyzed will depend on the quality of the curve fit; the scatter within the data set, as defined by twice the standard deviation, shall be less than the noise level of the electrical measurement system as defined in 6.11.

6.12.4 *Edge Heat Losses*—Edge heat losses give rise to the greatest measurement errors when the specimens approach the maximum specified thickness and thermal resistance. This series of experiments will determine which edge loss strategies must be employed to maintain edge losses to levels prescribed by this method.

6.12.4.1 Install specimens in the apparatus that approach the apparatus limits described above and instrument these specimens with the edge temperature sensors described in 6.6. Do not install any components described in 6.6 to reduce edge heat loss. While performing a test, verify that the difference between the specimen mean temperature and edge temperature satisfy the requirements of 6.6. Add additional edge loss apparatus components (edge insulation, secondary guard, enclosure) until the requirements of 6.6 are satisfied. These experiments will define the required levels of edge loss that shall be incorporated into the routine testing. In extreme cases, the secondary guard may have to be biased to satisfy these requirements; include these biases as part of the routine test procedure.

6.12.5 *Emittance of Surface Plates*—The emittance of the surfaces can be experimentally verified by testing an air gap, where the thickness of the air gap is limited to prevent the onset of convection. The heat flow rate per unit temperature difference is the sum of the thermal conductance of air and $4\sigma T_m^3 (2/\epsilon\text{-}1)$. A best fit of the plot of the heat flow rate per unit temperature difference and the inverse of the air space thickness supplies both the thermal conductivity of the air and $4_n T_m^3 (2/\epsilon\text{-}1)$. From this plot, the plate emittance can be verified **(42)**.

6.12.6 *Overall Design Verification*—When all of the other checks have been successfully completed, tests shall be performed on specimens that are traceable to a national standards organization. These tests shall cover the range of temperatures for which the apparatus has been designed. Verification of the apparatus may be limited by the temperature range of available standards. See 5.7.

7. Specimen Preparation and Conditioning

7.1 *Specimen Selection*—Only those specimen selection factors important to the performance of the apparatus are considered here. Factors related to the specimens' thermal properties are typically described in material specifications. When two specimens are required, the specimens should be selected to be as similar in thickness and thermal characteristics as possible. The use of Test Method C 518 can be used to check the similarity of the specimens' thermal characteristics.

7.1.1 *Thickness*—The maximum specimen thickness that can be measured to a given accuracy depends on several parameters, including the size of the apparatus, thermal resistance of the specimen, and the accuracy desired. To maintain edge heat losses below approximately 0.5 %, for a guard width that is about one-half the linear dimension of the metered section, the recommended maximum thickness of the specimen is one-third the maximum linear dimension of the metered section. For more specific quantitative information on this limitation see Refs **(1,5,7,8)** and adjunct material given in this test method.

7.1.2 *Size*—The specimen shall be sized to cover the entire metered section and guard area when possible. It is desirable to cover the gap between the guarded-hot-plate and the primary guard when sample size is limited. The guard portion of the

volume between the heating and cooling plates should be filled with material having similar thermal conductance characteristics as the specimen. When the specimen has a high lateral conductance such as a dense solid, a gap between the metered section and the primary guard shall be provided within the specimen. Refer to 7.2.3 for special precautions.

7.1.3 *Homogeneity*—Specimens exhibiting appreciable inhomogeneities in the heat flux direction shall not be tested with this method. There are two potential problems in attempting to determine the heat flux through highly inhomogeneous specimens. One is related to the interpretation and application of the resulting data, see Practice C 1045. The other is the degradation in the performance of the apparatus. If the specimen is highly inhomogeneous, that is, the heat flux varies appreciably over the metered section, several errors can be significantly increased. The plate temperature distribution can deviate appreciably from isothermal conditions which, in turn, can cause large uncertainties in the average temperature difference across the specimen. The increased plate temperature variations can also lead to increased gap and edge heat losses. The importance of measuring the plate or specimen surface temperatures at numerous points is greatly increased under such conditions.

7.2 *Specimen Preparation*—Prepare and condition the specimens in accordance with the appropriate material specification. Use the following guidelines when the material specification is unavailable. In general, the surfaces of the specimen should be prepared to ensure that they are parallel with and have uniform thermal contact with the heating and cooling plates.

7.2.1 *Compressible Specimens*—The surfaces of the uncompressed specimens may be comparatively uneven so long as surface undulations are removed under test compression. It may be necessary to smooth the specimen surfaces to achieve better plate-to-specimen contact. If the apparent thermal conductivity of the contact void is greater than that of the specimen, compressible or otherwise, the measured heat flux will be greater than the heat flux that would be obtained if the voids were absent. This may often be the case at higher temperatures where radiant heat transfer predominates in the void. For the measurement of compressible specimens, the temperature sensors are often mounted directly in the plate surfaces. Also, plate spacers may be required for the measurement of compressible specimens.

7.2.2 *Rigid and High Conductance Specimens*—The measurement of rigid specimens or high conductance specimens requires careful surface preparation. First, the surfaces should be made flat and parallel to the same degree as the guarded-hot-plate. If the specimen has a thermal resistance that is sufficiently high compared to the specimen-to-plate interface resistance, temperature sensors mounted in the plates may be adequate. However, for materials such as plastics or ceramics, when the thermal conductivity of the material exceeds 0.1 W/m·K, the following techniques shall be used to ensure accurate surface temperature measurement.

7.2.2.1 In some cases it is necessary to mount the temperature sensors directly on the specimen surfaces or in grooves in the specimens. Under vacuum conditions, the slightest space between plate and specimen is essentially an infinite thermal resistance (except for radiative heat transfer). Under these conditions extreme heat flux nonuniformities will occur. In any event the user should always try to minimize the ratio of contact resistance to specimen resistance and to strive for a constant ratio over the entire surface.

7.2.2.2 Another potential solution (that must be used with caution) is to mount a compressible thin sheet (for example, a soft rubber or thin fibrous pad) between the plates and specimen to improve the uniformity of the thermal contact. When this procedure is used, temperature sensors shall be instrumented in or on the surface of the specimens to ensure accurate temperature measurement of the specimen surface. An applied force should be used as in 6.7 to ensure sufficient surface contact.

7.2.3 *Anisotropic Specimens*—Specimens that have a high lateral to axial conductance ratio require that a low conductance gap be created in the specimen directly in line with the gap between the metered section and the primary guard.

7.2.4 *Loose-Fill Specimens*—The measurement of loose-fill specimens requires special handling, conditioning, and measurement techniques. The user is directed to Practice C 687 for details.

7.3 *Specimen Conditioning*—Condition the specimens either as stated in the material specification or where no guideline is given, at 22 ± 5°C (72 ± 3°F) and 50 ± 10 % relative humidity for a period of time until less than a 1 % mass change in 24 h is observed.

NOTE 17—Specimens can be conditioned at different conditions in order to determine the effect on the thermal properties of the specimens. Conditioning environments shall be reported with the test results.

8. Procedure

8.1 For a double sided test, select a pair of test specimens as outlined in Section 7.

8.2 Measure and record the specimen mass and dimensions. Also see 8.12.

8.3 Install the specimen into the apparatus at the desired test thickness.

8.4 Install the appropriate secondary guarding and an environmental chamber (as required).

8.5 If the test is to be conducted with gases other than air in the specimen-plate assembly, purge the environmental chamber and backfill with the desired gas. Care should be taken to limit the pressure of the fill-gas to below its condensation point at the lowest temperature expected within the chamber. Since the measured heat flux is dependent on both the type of fill gas and pressure, record both of these parameters.

8.6 Adjust the heating and cooling systems to establish the desired test conditions. For guidance in establishing test temperatures, refer to Practice C 1058. The ambient temperature should be the same as or slightly above the mean temperature of the test. This may require the use of a temperature controlled surrounding. This can be accomplished utilizing a controlled perimeter heater and insulation materials to aid in the control of the surrounding temperature.

8.7 Record the start time and date of the test. Begin data acquisition. The recorded data shall include: the date and time of data acquisition; power to the guarded-hot-plate; hot side

guarded-hot-plate surface temperature; hot side guard temperatures; cold surface assembly temperatures; controlled environment ambient temperature and relative humidity; temperature difference or thermopile output across the gap between the guard and metered section; and calculated heat flux and estimated thermal property of interest.

NOTE 18—Thermal steady-state is the time required for the test apparatus to stabilize. This varies considerably with the apparatus design, specimen to be measured, and test conditions. Generally, however, the stabilization time is on the order of hours. Stabilization times generally increase with thick specimens, specimens with low thermal diffusivity and is dependent on the mass of the metered section area. Measurements in a vacuum and on microporous materials create small monotonic changes over a long period of time and may take longer to stabilize.

8.8 Thermal steady state must be achieved for this test method to be valid. To determine if steady state is achieved, the operator must document steady state by time averaging the data, computing the variation and performing the following tests on the data taken in Section 8.

8.8.1 Thermal steady state for the purpose of this test method is defined analytically as:

8.8.1.1 The temperatures of the hot and cold surfaces are stable within the capability of the equipment at the test conditions. Ideally an error analysis will determine the magnitude of the allowable differences, however the difference is usually less than 0.1 % of the temperature difference.

8.8.1.2 The power to the metering area is stable within the capability of the equipment. Ideally an error analysis will determine the magnitude of the allowable differences, however the difference is usually less than 0.2 % of the average result expected.

8.8.1.3 The required conditions above exist during at least four intervals 30 min in duration or four system time constants, whichever is longer.

NOTE 19—The thermal time constant of the system is the time required to come to within 1/*e* (37 %) of the fixed value after a step thermal disturbance of the system. The thermal time constant in the constant power mode is the time required to come to within 37 % of the final temperature. The thermal time constant in the constant temperature mode is the time required to come to within 37 % of the final power. The thermal time constant of a system can be approximated from the thermal diffusivities of the system components, but is generally determined experimentally.

8.9 After achievement of the desired steady-state as defined in 8.8.1, three successive repeat data acquisition runs shall be completed. These runs shall be conducted at intervals of at least 30 min and should not be less than the thermal time constant of the system (see Note 19). This combination of three runs shall be considered a valid test if each datum obtained for each measured variable meets the following criteria.

8.9.1 The data do not differ from the mean by no more than the uncertainty of that variable, see A1.5.

8.9.2 The data obtained does not change monotonically with time. This is determined by comparing the average result of the final three test periods to the averages of the previous four periods. Graphing of the test parameters versus time or monitoring the slope of the data are techniques for determining monotonic conditions.

8.9.3 If the data continues to drift, the test shall be considered incomplete and further data acquisition sets shall be conducted until thermal steady state is achieved. Drift, even at low levels, may indicate that either the specimen characteristics are changing or the system is not at steady-state. For further details see Refs **(3,12,13)**.

8.10 Prior to terminating the test, measure and record the pressure of the chamber.

8.11 Upon completion of the thermal test outlined above, remove the specimen and examine the system components, such as temperature sensor mounting, for proper placement and operation.

8.12 Determine the specimen thickness and weight after the test to ensure that they have not changed from the initial condition. Record any changes in the physical characteristics of the specimen.

9. Calculation

9.1 The primary data required for this test method include electrical power, surface temperatures, area, and thickness. Of these, only thickness is generally a directly measured quantity. The others are either calculated from other more fundamental measurements or are converted by an electrical device. The manner in which these variables can be obtained is discussed in 8.9 and below.

9.2 *Heat Flow*—The heat flow to be reported is that which passes through each specimen. This is equal to the power generated by the metered section heater. For the double-sided mode of operation, only one-half the power generated by the heater flows through each specimen. Determine the power, Q, from emf, E, and current, I, and calculate as follows:

$$Q = E \times I \tag{2}$$

9.3 *Metered Section Area*—Determine the metered section area, A, from the area, A_m, of the guarded-hot-plate and the gap area, A_g. If there is no discontinuity in specimen characteristics in the gap region, the metered area is calculated as follows:

$$A = A_m + \frac{A_g}{2} \tag{3}$$

For high precision measurements, this assumption that the gap contributes half of its area to the effective metered section area, A, may need to be verified for the particular apparatus used. If there is a discontinuity between the specimen in the metered section and the guard region, this equation is modified slightly, as in ISO 8302, to include the effect of heat flux distortion in the gap region:

$$A = A_m + \frac{A_g \lambda_g}{2\lambda} \tag{4}$$

Where significant expansion, or contraction, of the guarded-hot-plate is known during a test, appropriate corrections to the area shall be made.

9.4 *Heat Flux*—The heat flux is obtained from the ratio of the heat flow, Q, and the total metered section area, A, and is calculated as follows:

$$q = \frac{Q}{A} \tag{5}$$

9.5 *Temperature*—Electrical readings from the temperature sensors are normally converted to temperature using a mathematical equation based on either the sensor's calibration curve or an appropriate reference such as a thermocouple voltage table.

9.6 *Density*— The metered section area specimen density, ρ_m, or the sample density, ρ_s where metered section area density cannot be obtained, are to be reported as the average of the two pieces. The equation for density, is the following:

$$\rho_m = \frac{m}{A \times L} \tag{6}$$

or:

$$\rho_s = \frac{m_s}{A_s \times L}$$

9.7 *Thermal Transmission Properties*— These properties shall be reported only in accordance with the requirements and restrictions of Practice C 1045.

10. Report

10.1 To be in conformance with this test method, report the following:

10.1.1 The report shall be identified with a unique numbering system to allow traceability to the individual measurements taken during each test performed,

10.1.2 The average values as obtained from the test. Standard deviation about that average. The results may be reported in a form similar to that shown in Fig. 3,

10.1.2.1 Identification of the test organization, responsible person in charge, test operator (optional) and the test sponsor,

10.1.2.2 The generic name, or other identification required to provide a complete and detailed description of the tested material. For hygroscopic materials, such as concrete and wood, the moisture content should also be given,

NOTE 20—A generic description in addition to the brand name should be reported where possible.

Test Report

Date:	Test Report Number:
Operator:	Duration of Test:

Specimen Identification:	Product, name, manufacturers description.
Specimen Characteristics:	Unique characteristics such as degree of homogeneity or anisotropy, density (optional).
Specimen Conditioning:	Temperature, time, humidity.
Specimen Dimensions and Mass:	Before and after conditioning and after measurement.

Apparatus Description:	Size, shape and orientation of plates. Single or double-sided operation, description of secondary guarding, unique procedures.

Experimental Results

		Uncertainty	
Variable	**Measured Value**	**Systematic**	**Random**
Q, W			
Th, K			
Tc, K			
Tm, K			
$\Delta T, K$			
A, m^2			
L, m			
Fill gas pressure, Pa			
Other			

Derived thermal transmission properties including the applicable range of conditions shall be in conformance with Practice C 1045.

FIG. 3 Example Test Report Form

10.1.2.3 Information regarding the specimen preconditioning,

10.1.2.4 Variables that effect thermal transmission properties, such as fill-gas and pressure, shall be specified when applicable,

10.1.2.5 The dimensions of the metered section and guard(s) and their relationship to the overall specimen dimensions (m). The plate emittance,

10.1.2.6 Specimen orientation and the direction of heat transfer during the test,

10.1.2.7 The total area of the specimen (m^2),

10.1.2.8 The specimen density of the metered section area or sample density where metered section area density cannot be obtained (kg/m^3),

10.1.2.9 The thickness of the specimen(s) within the metered section (m),

10.1.2.10 The area averaged temperatures of both hot and cold specimen surfaces (K),

10.1.2.11 Net steady-state average heat flux through the specimen (W/m^2),

10.1.2.12 Any thermal transmission properties calculated and reported and their estimated error, and

10.1.2.13 The test date and time, the time required for steady temperature conditions, the time to reach steady-state, the data acquisition time period, frequency of data collection and the end date and time.

10.2 The following is optional information that may be included in the report:

10.2.1 Values for guard loss, back side energy loss and other losses included in the net energy calculation (W/m^2), and

10.2.2 A full description (or references) of test procedures and data analysis techniques used.

10.3 When certification of the test results is required, include the date of the latest apparatus verification and a description of the procedures used. References for the verification report(s) shall also be included. Where applicable, include a statement of laboratory accreditation of the test facility used, including date of latest inspection.

10.3.1 Where agreed upon between the customer and the test laboratory, less maybe reported but the remainder of the results shall be made available.

NOTE 21—**Caution:** Where this test method might be specifically referenced in published test reports and published data claims, and where deviations from the specifics of the test method existed in the tests used to obtain said data, the following statement shall be required to accompany such published information: **"This test did not fully comply with following the provisions of Test Method C 177."** This statement shall be followed by a listing of specific deviations from this test method and any special test conditions that were applied.

11. Precision and Bias

11.1 This section on precision and bias for the guarded hot plate apparatus includes a discussion of; general statistical terms; statistical control; factors affecting test results; ruggedness tests; interlaboratory comparisons conducted by ASTM Committee C-16; proficiency testing conducted under the auspices of the National Voluntary Laboratory Accreditation Program (NVLAP); and error propagation formulae.

11.2 *General Statistical Terms*—The accuracy of a test result refers to the closeness of agreement between the observed value and an accepted reference value. When applied to a set of observed values, the accuracy includes a random component (imprecision) and a systematic component (bias). The variability associated with the set of observed values is an indication of the uncertainty of the test result. Additional information on statistical terminology is available in Terminology E 456.

11.3 *Statistical Control*—The user of the guarded-hot-plate apparatus shall demonstrate that the apparatus is capable of performing in a consistent manner over time **(35)**. The use of control charts (see Manual 7 **(34)**) to monitor the operation of the guarded hot plate is one recommended way to monitor the control stability of the apparatus. When possible, it is recommended that a reference material traceable to a national stand ard s laboratory be used as the control specimen. Ideally, the long-term variation should be no greater than the short-term variability.

11.4 *Factors Affecting Test Results*— Experiments and theoretical analyses have identified two principal (systematic) errors that affect the operation of an idealized guarded hot plate apparatus. These errors are edge heat flows at the periphery of the specimens; and, heat flow across the gap due to a thermal imbalance. Other errors studied include the effect of gap width on the heat flow; and, the proper determination of the metered section area. These errors and others are discussed in detail in A1.3.

11.4.1 *Edge Loss Errors*—These have been found to depend on the size (and type) of the guard, the specimen thermal conductivity and thickness, and ambient temperature **(7,18,20,21,31,33)**. By using a sufficiently wide guard (see Section 6), appropriate levels of edge insulation, and proper selection of the ambient temperature (see Section 8), the edge loss error can be reduced to a negligible value relative to the specimen heat flow (see Annex A4.2). There is only limited experience (at room temperature) with measurement of apparent conductivity at large thickness' (above 30 cm), but errors may be expected to be above 2 %, especially if the user does not reduce the problems associated with long time constants and large lateral heat flows **(31)**.

11.4.2 *Gap Imbalance Error*—These have been found to depend on several parameters including the temperature difference, the gap geometry, the structural support system, the wires crossing the gap (number, size, and type), the gap fill material (gas or insulation), the emittance of the gap surfaces, and the specimen material in the vicinity of the gap **(5,6,8,18,22,36)**. The resulting heat flow due to a temperature imbalance can be obtained either by calculation based on the above parameters or empirical data. An empirical relationship for the gap heat flow can be determined by purposely introducing a temperature imbalance across the gap and measuring the resulting change in the specimen heat flow (see A1.4.3).

11.5 *Ruggedness Tests*—The results of one ruggedness study for a 200 mm^2 guarded hot plate and two materials having different thermal conductivity's have been reported **(37)**. Matched pairs, 85 mm thick, of polyurethane foam and silicone rubber were measured at a mean temperature of 297 K

and a temperature difference of 23 K. For each specimen, the width of edge insulation was set at one of five levels (0, 12.7, 25.4, 50.8, and 76.2 mm) while the ambient temperature was varied at one of three levels. The results indicate that the edge losses are reduced with edge insulation but only become zero when the ambient temperature is at one specific value. The optimum ambient temperature appears to be a function of specimen thickness and thermal conductivity, and edge insulation thickness.

NOTE 22—As noted in Section 8, the value of the ambient temperature is set to either the same value as the mean temperature of the test or a value slightly above the mean temperature. The user should determine the optimum value for their apparatus and test conditions by using the sensitivity analysis described in A4.2. This dependence may change appreciably for different specimens or apparatus conditions and, therefore, should be done under typical test conditions.

11.6 *Interlaboratory Tests*—The results of three published interlaboratory tests for guarded-hot-plate apparatus are discussed below. The results, where appropriate, state an index of precision (between laboratory) of two-standard deviation limits (2s). Certain aspects of the interlaboratory tests were not conducted completely in accordance with the requirements of Practice E 691, for example, the number of test laboratories was less than six in one study and none of the studies required replicates. Furthermore, a study involving a variety of materials is needed. Consequently, a general statement for the index of precision and bias that covers all conditions and materials is unavailable. In the interim, the user is directed to the interlaboratory tests if information on precision and bias is needed (see Practice C 687 for loose-fill materials).

11.6.1 In 1951, results of an interlaboratory comparison were reported **(38)** for 20 guarded-hot-plate apparatus from 17 laboratories. The plates ranged in size from 200 to 600 mm square. Different (numbered) pairs of corkboard (25 mm thick) were measured by each laboratory at a mean temperature from 266 to 322 K. The data from 15 of the 20 apparatus (75 %) were within ± 3 % of the mean value as determined by the National Bureau of Standards (now the National Institute of Standards and Technology). The maximum deviations were + 13 and – 16 %.

11.6.2 In 1985, results of a third round of interlaboratory comparisons were reported **(41)** for five large guarded-hot-plate apparatus ranging from 610 to 1219 mm^2 and 1016 mm diameter (the last apparatus mentioned being a circular line-heat-source guarded-hot-plate). The same specimens of fibrous-glass blanket (16 kg/m^3) were circulated to each laboratory. Matched pairs were tested at 297 K and thicknesses of 25.4, 50.8, 76.2, and 101.6 mm. Imprecision of the data versus a semi-empirical model for a density range of 11 to 20 kg/m^3 were 1.9, 2.3, 2.6, 2.9 % (2s level) at thicknesses of 25.4, 50.8, 76.2, 101.6 mm, respectively.

11.6.3 In 1988, results of a interlaboratory comparison were reported **(30)** for seven high-temperature guarded-hot-plate apparatus. The plates ranged in size from 203 to 406 mm in diameter and 300 to 610 mm^2. Different matched pairs of fibrous alumina-silica and calcium silicate were measured by each laboratory over a mean temperature range from 330 to 701 K. Reference equations based on NIST-Boulder corrections were fit to the data. Imprecision in the deviations from the model were 15 and 16 % (2s level) for fibrous alumina-silica and calcium silicate, respectively. It was established that a significant percentage of the standard deviation in this comparison was due to material variability and not apparatus error.

11.7 *Proficiency Tests*—In 1985, the results of a series of proficiency tests conducted for NVLAP over a four-year period were reported **(39)** for guarded-hot-plate apparatus (plate size not reported). Different specimens of four thermal insulation materials were distributed to each laboratory for testing. The materials were expanded polystyrene; foam board; low-density glass-fiber batt (8 to 16 kg/m^3); and, high-density glass-fiber batt, foil-faced (64 kg/m^3). Each laboratory reported a single test result, that is, no replicates were conducted. Results of the proficiency tests are summarized in Table 1. The index of precision (between laboratory) is expressed as a percentage for the one-standard deviation limit(s) divided by the mean of the test result, or one-coefficient of variation (CV %).

11.8 *Error Propagation*—Several formulae are available **(40)** for determining the apparatus uncertainty by error propagation. For guidelines on using a standard procedure, the user is referred to ISO Guide to the Expression of Uncertainty in Measurement **(32)**. Strictly speaking, determining a statement of uncertainty for a test result requires treating random and systematic errors separately. A description of random and systematic errors and possible sources of error are discussed below.

11.8.1 *Random Error,* δ_r— In a measurement, random errors (imprecision) are considered to be the sum total of all the small (negligible) independent errors that are uncontrolled, for example small fluctuations in environmental conditions or plate temperatures. Random errors are assumed normally distributed, uncorrelated, and preferably small. In general, random errors are a function of the capabilities of the control system and, to a lesser extent, the measurement system.

11.8.2 *Systematic Error,* δ_s—A systematic error (bias) is a fixed deviation that is inherent in each and every measurement. If the magnitude and direction of the systematic error are known, the user can make appropriate correction(s) to the

TABLE 1 NVLAP Proficiency Tests for Guarded-Hot-Plate Apparatus Ref (39)

Material	Nominal Thickness, mm	Thermal Conductivity Group Mean, W/(m K)	Number of Labs	Coefficient of Variation, %	Round
Expanded polystyrene board	25	0.037	6	1.80	10
Foam Board, rigid	25	0.040	9	2.52	4
Glass-fiber batt	25	0.040	10	2.15	5
Glass-fiber batt	25	0.040[A]	6[A]	2.26[A]	7[A]
Glass-fiber batt	25	0.039[A]	7[A]	2.82[A]	3B[A]
Glass-fiber batt	25	0.040	9	3.28	3A
Glass-fiber batt	25	0.040	7	3.43	7
Glass-fiber batt	25	0.040	9	4.66	3B
Glass-fiber batt, foil faced	25	0.032	9	0.98	6
Glass-fiber batt, foil-faced (stacked)	50	0.033	7	1.45	9
Glass-fiber batt, foil faced	25	0.032	8	1.95	8

[A] Recalculation with one or more laboratories excluded from the group statistics because their test results deviated from the pre-characterized value by more than 6 %.

measured value. Under such circumstances a justification for the correction should be provided. In general, the magnitude of the error, $|\delta_s|$, is estimated by experience or judgment.

11.8.3 *Statement of Uncertainty*—The statement of uncertainty requires an expression having credible limits for its inaccuracy. Different traditions and usage have resulted in different expressions of uncertainty that may be summarized as follows: both imprecision and bias negligible; imprecision negligible, bias not negligible; neither imprecision nor bias negligible; and, imprecision not negligible, bias negligible.

11.8.4 *Sources of Errors*—The uncertainty of the apparatus as determined by propagation of errors should consider the error in each of the separate measurements used to determine the test result. For a guarded-hot-plate apparatus, these errors in measurements are the uncertainty in: heat flow δQ; temperature difference, $\delta\Delta T$; metered section area, δA; and specimen thickness, δL. These errors and an example are discussed in A1.3.

12. Keywords

12.1 error analysis; guarded-hot-plate; heat flow; heat flux; steady-state; thermal conductivity; thermal resistance; thermal transmission; thermal conductance; thermal testing

ANNEX

(Mandatory Information)

A1. THICKNESS MEASUREMENT, LIMITATIONS AND MEASUREMENT UNCERTAINTY

A1.1 *Importance of the Thickness of the Insulation Specimens in Guarded-Hot-Plate Measurements*—The thickness of the specimen as installed in the apparatus determines both the density of the material and the temperature gradient applied to it during the measurement of the thermal property. If the thickness of a specimen is changed from its room-temperature value by thermal effects (thermally reversible expansion or contraction, or thermally induced irreversible shrinkage or expansion of the specimen), or by compression, then use of the room-temperature thickness outside the apparatus will lead to error in the determination of the apparent conductivity (or resistivity) of the specimen. A given relative (percentage) error in the thickness leads to an equal relative error in the determination of the conductivity. For measurements of thermal properties at mean specimen temperatures near room temperature the error in neglecting any changes in thickness may be negligible, but this can be ascertained only by observation in the specific case at hand.

A1.2 *Suggested Ways to Measure Thickness of Incompressible Specimens*—In determining the thickness of a specimen, one assumes that it is properly shaped, so that the measured thickness is valid. However, two different situations may sometimes occur to affect the thickness measurement. The shape of the specimen may be distorted by warping or bowing at the time it is first installed in the apparatus. In this case, either the (flexible) specimen should be compressed enough to remove the distortion when installed, (or, preferably, a specimen of better quality should be selected). Independent of, the specimen may undergo a change of shape as it is subjected either to high mean temperatures or to large temperature gradients, due to chemical changes occurring in the specimen at high temperatures. In this case it is difficult to define what the thickness of the specimen actually is during the measurement. The thickness of the specimen should be measured both before and after the thermal transmission property is measured, to show whether such dimensional changes are occurring. Any warping or bowing of the specimen, before or during measurement of thermal properties, adds to the uncertainty in the value of thickness. Some materials such as polymers have large coefficients of expansion and the material tends to bow unless a small thickness and temperature difference across the specimen is used.

A1.2.1 The recommended procedure for measuring specimen thickness is to measure the thickness while installed in the apparatus. This is necessary if the correct temperature gradient actually applied to the specimen during the measurement of the thermal property is to be obtained. Rigid rods may be securely installed extending laterally from the outer edges of the metered area/primary guard assembly, at two or three equally spaced locations along the circumference of the plate. The portion of the rod extending from the plate should be smooth and parallel to the plane of the plate surface. Alternatively, the plates may be machined with flat, horizontal plates extending from the circumference. Similar rods (or plates) are likewise located on each auxiliary heater plate, at the same circumferential positions, vertically (within 5° of arc) above or below the rods on the metered area/primary guard assembly.

A1.2.1.1 With no specimens installed, with the heater plates contacting each other in their usual order, and taking care not to change the plate separation, measure the separation between each vertical pair of rods on two adjacent plates with a vernier calliper. Compute the arithmetic mean of the plate separation for each pair of adjacent plates. Then, with specimens installed between the plates in the apparatus, and with the usual mechanical loading applied, measure the separation between the pairs of rods on adjacent plates, taking care not to change the plate separation. Compute the arithmetic mean. Subtract the mean separation obtained with no specimen from the mean separation with the specimen present, for corresponding pairs of plates, to obtain the as-installed thickness of each specimen. The standard deviation about the average of values from repeated measurements of the plate separation, starting from total disassembly, gives a statistical measure of the reproducibility. If contact cannot be made between the plates, standard spacers can be inserted between the plates. Bringing the plates

in contact with the spacers can determine the adjustment in specimen measured thickness required.

A1.2.1.2 The accuracy of this procedure is equal to the imprecision with which the vernier can be read. The accuracy of this test method depends on the precision with which the rods are mounted in a true horizontal orientation, and on not changing the plate separation during the measurement. The standard deviation about the average of values from repeated measurements of the plate separation, starting from total disassembly, gives a statistical measure of the reproducibility.

A1.2.2 An alternative is to place the specimen on a flat surface and measure the thickness at various points across the specimen with a thickness gage mounted above the specimen. The zero is first established by resting the foot of the gage on the flat surface. The specimen is then measured. This procedure has the advantage that specimen flatness and warp can be measured. Thickness is measured typically in at least five different locations across the full specimen and within the metered section to establish the metered thickness within the apparatus. The thickness, when applicable, should be measured after the test to monitor any significant changes that may have effected the results.

A1.2.2.1 The accuracy of this test method is equal to the imprecision with which the gage can be read. The accuracy and reproducibility of this test method depends on the ability of the operator to reproduce the amount of force exerted on the specimen especially in the case of compressible specimens.

A1.2.3 Another alternative is to use a micrometer or vernier calliper. This assumes that the specimen is not bowed or warped, that should of course be ascertained. During a measurement of thickness with a calliper, prevent the narrow jaws of the measuring tool from penetrating into the surface of the specimen. Cut two small pieces of flat, rigid rectangular metal sheet, about 20 by 40 mm and 0.5 to 1.0 mm thick. Measure the combined thickness of the two metal rectangles; then measure the thickness of the specimen while holding one metal piece under each jaw, between the surface of the specimen and the jaws of the micrometer or calliper. Be sure to subtract the combined thickness of the two metal plates from the total thickness of specimen plus metal pieces, to obtain the net specimen thickness. By this method measure the thickness at eight different, equally spaced locations around the outer margin of the specimen.

A1.2.3.1 The accuracy of this procedure is equal to the precision with which the vernier (or micrometer) can be read. The accuracy and reproducibility of this test method is lower than that described above in A1.2.1 and A1.2.2, due to the variable pressure used by different people in measuring the specimen between the jaws of the micrometer or calliper.

A1.3 *Limitations Due to Apparatus:*

A1.3.1 *Limitations Due to Contact Resistances*—When testing a rigid specimen of high thermal conductance (that is, specimens of a material too hard and unyielding to be appreciably altered in shape by the pressure of the heating and cooling units), even small, non-uniformities of the surface of both the specimen and the apparatus (surfaces not perfectly flat) will allow contact resistances not uniformly distributed between the specimens and the plates of the heating and cooling units.

A1.3.1.1 These will cause nonuniform heat flow-rate distribution and thermal field distortions within the specimens; moreover, accurate surface temperature measurements will be difficult. For specimens having thermal resistances less than 0.1 m^2 K/W, special techniques for measuring surface temperatures will be required. Metal surfaces should be machined or cut flat and parallel and stress-relieved.

A1.3.2 *Upper Limits for the Thermal Resistance*:

A1.3.2.1 The upper limit of thermal resistance that can be measured is limited by the stability of the power supplied to the metered section, the ability of the instrumentation to measure power level and the extent of the heat losses or gains due to temperature imbalance errors between the central and guard sections of the specimens and of the metered section.

A1.4 *Limits to Temperature Difference:*

A1.4.1 Providing uniformity and stability of the temperature of the hot and cold surfaces of the plates, the noise, resolution and temperature measurements can be maintained within the limits outlined in Section 6, temperature differences as low as 5 K, when measured differentially, can be used. Lower temperature differences shall be reported as not complying with this standard. See Practice C 1058.

A1.4.2 If temperature measurements of each plate are made by means of thermocouples with independent reference junctions, the accuracy of the calibration of each thermocouple may be the limiting factor in the accuracy of measured temperature differences. In this case, it is recommended that temperature differences of at least 10 K to 20 K are used in order to minimize temperature-difference measurement errors.

A1.4.3 Higher temperature differences are limited only by the capability of the apparatus to deliver enough power while maintaining required temperature uniformity.

A1.4.4 *Maximum Specimen Thickness*:

A1.4.4.1 The boundary conditions at the edges of the specimens due to the effects of edge insulation, of auxiliary guard heaters and of the surrounding ambient temperature will limit the maximum thickness of specimen for any one configuration, as described in Section 6. For composite or layered specimens, the mean measurable thermal conductivity of each layer should be less than twice that of any other layer.

A1.4.4.2 This is an approximation and the results do not necessarily imply the measurement of conductivity of each layer. The accuracy will remain close to that predictable for tests on homogeneous specimens. No guidelines can be supplied to assess measurement accuracy when the requirement of 2.3 is not met.

A1.4.5 *Minimum Specimen Thickness*:

A1.4.5.1 The minimum specimen thickness is limited by contact resistances given in A1.3.1. Where thermal conductivity or thermal resistivity is required, the minimum thickness is also limited by the accuracy of the instrumentation for measuring the specimen thickness.

A1.4.5.2 The metered area, that is, the area of the specimen traversed by the heat flow-rate fed by the metered section, is related to the specimen thickness and to the gap width. As the

thickness tends to zero, the metered area tends to the area of the metered section, while for thick specimens the metered area is bounded by the line defining the centre of the primary guard gap. To avoid complex corrections, this definition can be retained, provided the thickness of the specimen is at least ten times the width of the gap.

A1.4.6 *Maximum Operating Temperature*:

A1.4.6.1 The maximum operating temperature of the heating and cooling units may be limited by oxidation, thermal stress or other factors that degrade the flatness and uniformity of the surface plate and by changes of electrical resistivity of electrical insulations which may affect accuracy of all electrical measurements.

A1.4.7 *Vacuum Conditions*:

A1.4.7.1 Care must be taken if a guarded hot plate is used for measurements under vacuum conditions. If a high vacuum is desired, the materials used in the design of the apparatus must be carefully selected to avoid excessive outgassing under such conditions. Under vacuum conditions, especially at lower temperatures, serious errors can arise if care is not taken when installing heater and temperature sensor leads so as to minimize extraneous heat flow-rates and temperature measurement errors.

A1.4.8 *Apparatus Size*:

A1.4.8.1 The overall size of a guarded hot plate will be governed by the specimen dimensions that typically range from 0.2 to 1 m diameter or square. Samples smaller than 0.3 m may not be representative of the bulk material, while specimens larger than 0.5 m may create considerable problems in maintaining the flatness of the specimens and plates, temperature uniformity, equilibrium time and total cost within acceptable limits.

A1.5 *Limitations Due to Specimen:*

A1.5.1 *Thermal Resistance or Thermal Conductance*:

A1.5.1.1 *Specimen Homogeneity*—In inhomogeneous specimens, the thermal flux density both within the specimen and over the faces of the metered section area may be neither unidirectional nor uniform. Thermal field distortions will be present within the specimen and can give rise to serious errors. The region in the specimen contiguous to the metered section area and especially near the edges of this area is most critical. It is hard to give reliable guidelines on the applicability of the method in such cases. The major risk is that the imbalance errors, edge heat loss errors, etc., now unpredictable, can vary in an unpredictable way when inhomogeneities take different relative positions within the specimen.

A1.5.1.2 One way to try to estimate the error is to compare the results for two specimens from the same sample, selected so that they have as widely different a structure near the edges or the metered section area. If the two extremes cannot be identified, a number of specimens may have to be tested.

A1.5.1.3 In some samples, the variation in structure may occur over small distances. This is true for many thermal insulations. In such cases, it may be possible to use a single specimen cut larger than the apparatus. This over-size specimen is tested twice, in each case with the specimen carefully positioned so that the edges of the test area are exposed to the two extremes in structure. The two results are then compared and the difference credited to distortion. The portion of the specimen(s) protruding from the apparatus should be well insulated in the two tests to reduce the possibility of the exposed section increasing edge losses. The size and thickness of the specimen affects the size of the variations in structure that can be accommodated. The larger the test area, the smaller the effect on the results. The effect of distortion may either increase or decrease with specimen thickness.

A1.5.1.4 Direct thermal short circuits may exist between the surfaces of the specimens in contact with the plates of the heating and cooling units. The largest effect occurs when sections of material which conduct heat readily, with extended surface area on each side of the specimen, are connected by a path of low thermal resistance relative to other paths. The effect can best be identified by breaking the thermal paths, especially when the collecting surfaces can be disconnected from the rest of the path. Sheets of thermally insulating materials can be used at the critical surfaces to provide the break. Sheets made of finely ground cork, or a similar material 2 mm or more thick, work well. The surfaces must be ground to the same degree of flatness as the heating unit. The thermal resistance of these sheets can be determined in separate measurements. The net change in thermal resistance of the specimen, due to thermal shorting, can thus be determined. If greater than 1 %, another measurement should be made with thicker sheets imposed.

A1.5.2 *Temperature-Difference Correlation*:

A1.5.2.1 Thermal resistance or thermal conductance are often a function of temperature differences across the specimen. In the report, the range of temperature differences that apply to the reported values of the two properties must be defined, or it must be clearly stated that the reported value was determined at a single temperature difference.

A1.5.3 *Mean Measurable Thermal Conductivity of a Specimen*:

A1.5.3.1 In order to determine the mean measurable thermal conductivity (or thermal resistivity) of a specimen, the criteria of A1.3.1 shall be fulfilled. The specimen shall be homogeneous. Homogeneous porous specimens shall be such that any inhomogeneity has dimensions smaller than one-tenth of the specimen thickness. In addition, at any one mean temperature, the thermal resistance shall also be independent of the temperature difference established across the specimen.

A1.5.3.2 The thermal resistance of a material is known to depend on the relative magnitude of the heat transfer process involved. Heat conduction, radiation and convection are the primary mechanisms. However, the mechanisms can combine or couple to produce non-linear effects that are difficult to analyze or measure even though the basic mechanisms are well researched and understood.

A1.5.4 The magnitude of all heat transfer processes depends upon the temperature difference established across the specimen. For many materials, products and systems, a complex dependence may occur at temperature differences which are typical of use. In these cases, it is preferable to use a temperature difference typical of use and then to determine an approximate relationship for a range of temperature differences. The dependence can be linear for a wide range of temperature differences.

A1.5.5 Some specimens, while being homogeneous, are anisotropic in that the thermal conductivity measured in a direction parallel to the surfaces is different to that measured in a direction normal to the surfaces. For such specimens, this can result in larger imbalance and edge loss errors. If the ratio between these two measurable values is lower than two, reporting according to this method is still possible if imbalance and edge heat loss errors are determined separately with anisotropic specimens mounted in the apparatus.

A1.5.6 *Thermal Conductivity or Thermal Resistivity of a Material*:

A1.5.6.1 In order to determine the thermal conductivity or thermal resistivity of a material, the criteria of A1.3.2 shall be fulfilled. In addition, adequate sampling must be performed to ensure that the material is homogeneous or homogeneous porous, and that the measurements are representative of the whole material product or system. The thickness of the specimens must be greater than that for which the thermal conductivity of the material product or system does not change by more than 2 % with further increase in thickness.

NOTE A1.1—Results obtained on specimens where thermal conductivity is still changing with specimen thickness are only applicable at that specific test thickness.

A1.5.7 *Dependence on Specimen Thickness:*

A1.5.7.1 Of the processes involved, only conduction produces a heat flow-rate that is directly proportional to the thickness of a specimen. The others result in a more complex relationship. The thinner and less dense the material, the more likely that the resistance depends on processes other than conduction. The result is a condition that does not satisfy the requirements of the definitions for thermal conductivity and thermal resistivity, both of which are intrinsic properties, since the transfer factor shows a dependence on the specimen thickness. For such materials, it may be desirable to determine the thermal resistance at conditions applicable to their use. There is believed to be a lower limiting thickness for all materials below which such a dependence occurs. Below this thickness, the specimen may have unique thermal transmission properties, but do not relate to the material. It remains, therefore, to establish this minimum thickness by measurements.

A1.5.7.2 Determination of minimum thickness above which thermal properties of the material may be defined.

A1.5.7.3 If the minimum thickness for which the thermal conductivity and resistivity can be defined is not known, it is necessary to estimate this thickness.

A1.5.7.4 In the absence of an established method, the procedure outlined below may be used to approximate the thickness and whether it occurs in the range of thickness in which a material is likely to be used.

A1.5.7.5 It is important to differentiate between added thermal resistance in measurements caused by the placement of the temperature sensors below the surfaces of the plates, added resistance caused by poor specimen surfaces, and added resistance caused by the coupling of the conduction and radiation modes of heat transfer in the specimens. All three can affect the measurements in the same way, and often the three may be additive.

A1.5.7.6 Select a sample uniform in density distribution, with the thickness L_5, equal to the greatest thickness of the material to be characterized or equal to the maximum allowable thickness for the test apparatus.

A1.5.7.7 Cut five sets of specimens in approximately equal increments from the sample ranging in thickness from the smallest likely to be used in practice. The set of specimens shall be designated s_1 to s_5 according to their respective thickness L_1 to L_5.

A1.5.7.8 For low density materials where heat is transferred by radiation and conduction mechanisms and where the absence of convection has been verified, the slope of a plot of thermal resistance versus thickness will very frequently diminish up to 1 to 2 cm and then will remain constant as the thickness increases. The reciprocal of this constant slope is the thermal conductivity to be assigned to high thickness specimens.

A1.5.7.9 Measure the thickness and thermal resistance of s_1, s_3, and s_5 at the same mean temperature and with the same temperature difference across the specimen. Plot the thermal resistance versus thickness. If these three values differ from a straight line relationship by less than ± 1 %, the slope of the straight line shall be computed. If the three values differ by more than 1 %, then similar measurements shall be made on s_2 and s_4 to check if there is a thickness above which the thermal resistance does not differ from a straight line by more than 1 %.

A1.5.7.10 If this thickness exists, the slope of the straight line shall be determined to compute a thermal conductivity $\lambda_m = \Delta L/\Delta R$ defined as the ratio between the increments of thickness, ΔL, and increments of the thermal resistance, ΔR.

A1.5.7.11 The thickness at which this occurs will vary according to the densities, types and forms of different materials, products and systems for different mean temperatures.

A1.5.7.12 Thermal conductivity and thermal resistivity then characterizes the material, product or system for thicknesses above which the transfer factor differs by less than 2 % from λ_m.

A1.5.7.13 Allowance for experimental errors must be made in the interpretation of results. Least-square curve fitting of R versus L may also help. A larger number of specimens may be used where greater definition is required.

A1.5.7.14 Thickness dependence may be a function of temperature difference across the specimens. For the purposes of this test method, the above checks, if performed at typical operating temperature differences, shall be adequate to indicate the degree of thickness dependence.

A1.5.8 *Method of Determining Dependence on Temperature Difference*—If the temperature-difference dependence of the thermal properties is not known for a material, a minimum of three measurements shall be made. These are made with widely differing temperature differences. A second-order dependence can be revealed by these measurements. When a simple linear relationship is known to occur, only two measurements, that is, one extra, need be made. This establishes the linear dependence for that particular sample.

A1.5.9 *Warping*—Special care should be exercised with specimens with large coefficients of thermal expansion that warp excessively when subjected to a temperature gradient.

The warping may damage the apparatus or may cause additional contact resistance that may lead to serious errors in the measurement. Specially designed apparatus may be necessary to measure such materials.

A1.6 *Measurement Uncertainty*—The uncertainty of the apparatus is based upon consideration of the random and systematic components of the following measurement uncertainties **(32)**: uncertainty in heat flow, Q; uncertainty in temperature difference, $T = (T_H - T_C)$; uncertainty in metered area, A; and, uncertainty in specimen thickness, L.

A1.6.1 Other specimen characterization and test condition data may need to be reported. The precision and bias of these data are to be reported to the extent they have a direct bearing on the accuracy of the results. Prescribed precision and bias of the primary data are not mandated by this test method. However, it is required that the user assess and report the precision and bias of the data. The discussion below provides guidelines to assist the user in performing this uncertainty assessment. A variety of helpful performance checks are included in this discussion. In the following discussion both random and systematic errors are considered. The subscript $_s$ is used to denote systematic, and the subscript $_r$ is used for the random components.

A1.6.1.1 *Systematic Error,* $_s$—Systematic error, s, is any component of error that remains fixed during the runs that constitute a successful test. To simplify the discussion, this does not include any components of error that are known both in magnitude and sign. Under such circumstances, the user should make appropriate corrections to the conductivity measurements and supply the justification for them. The user may check for the presence of unexpected errors by using a reference specimen or transfer standard available from appropriate sources. If errors are discovered, their source should be identified and removed. A guarded hot plate cannot be calibrated. The task of estimating the remaining systematic errors is based on judgment and experience, including an awareness of the results of interlaboratory comparisons. The implications of such estimates is often that they are the maximum possible systematic errors. In this event the total maximum systematic error is the sum of the errors. It is, however, more likely that these estimates are probabilistic in nature and do not, in fact, represent the worst possible case. The total probable systematic errors are summed in the same manner as random errors, that is, the square root of the sum of squares. In the following discussion the latter approach is taken. However, the user must decide if the bias estimates are worst cases or probabilistic in nature, and sum them accordingly.

A1.6.1.2 *Random Error,* $_r$—Random error, r, is that component of error that may vary both in sign or magnitude during the runs that constitute a successful test. For simplicity, it is assumed that the variations are normally distributed and conventional statistical techniques are applicable. An estimate of random error components can be obtained by repeat measurements of each variable.

A1.6.1.3 It is important to distinguish between random and systematic errors for the following reason. The results reported in the test method are mean values derived from more than a single run. The uncertainties reported generally apply to these mean values. The uncertainty of a mean value due to the random error component decreases approximately as $1/\sqrt{n}$ where n is the number of repeat runs. In contrast to this, the uncertainty of the mean value due to the systematic error component does not decrease with repeat runs. Thus, it is recommended that the error components be treated separately. The total uncertainty is expressed by reporting both components separately.

A1.7 *Error Components*—In the following sections, the error components of each reported variable are discussed. The total random or systematic uncertainty for each variable is taken to be the square root of the sum of squares.

A1.7.1 *Heat Flow, Q*— The objective of the test method is to establish and measure uniaxial heat flow through the metered area of the specimen. Any deviation from this objective represents error in the reported heat flow. The following sources of error should be considered:

A1.7.2 *Edge Heat Loss,* $_sQ_{se}$—Edge heat loss, $_s\ Q_{se}$ is a systematic error as the conditions surrounding the plate-specimen stack remain constant throughout the test procedure. Although tests have been reported that shed some light on the magnitude of this error, the results generally are not proven to the point where corrections based on these results are universally accepted **(1,4,6,7,18-22)**. However, the results are considered sufficiently valid for the basis of defining the maximum specimen thickness. The optimum environmental temperature to minimize this error is a small fraction of T above the mean test temperature. To determine the sensitivity of this error to test conditions, the user should determine the heat flux as a function of secondary guard temperature. This dependence may change appreciably with specimen and apparatus characteristics and, therefore, should be done under typical test conditions.

A1.7.3 *Gap Heat Loss*— Gap heat loss is considered to be composed of both systematic, $_sQ_{gp}$, and random, $_r$Q $_{gp}$, components. The systematic component can be, in part, due to the fact that there may be a finite number of locations along the gap at which the imbalance is measured; reducing the temperature difference between a finite number of points on opposite sides of the gap to zero may not necessarily ensure that there is zero net flow of heat across the gap. Improper position of the sensors will lead to systematic error. Spurious emfs within the circuitry will result in a systematic imbalance. The random component is due to short-term control fluctuations. After estimating the probable imbalance across the gap in terms of temperature (or sensor voltage) one needs to determine the effect of this imbalance on the measured heat flow through the metered area. This can be done by measuring the dependence of metered area power on intentionally introduced gap imbalance. A typical way of addressing this is to run three tests, one with the guard balanced and one each biased positive and negative. The results are plotted, lambda versus gap balance, and the zero intercept is determined. The imbalance introduced should be large enough to yield an easily measured change in Q, but small enough to remain in the region where the dependence of Q upon imbalance is approximately linear.

A1.7.3.1 It has been found that **(3,15,16)** the gap heat loss, Q_{gp} is linearly dependent on temperature unbalance across the

gap, T_g, that is, $Q_{gp} = BT_g$. The proportionality constant, B, is dependent on the wires crossing the gap (number, size, and type), gap geometry (width and cross-sectional shape), the gap fill material (gas, insulation), the emittance of the gap surfaces and the material in the vicinity of the gap between the hot and cold plates. A reasonable approximation of this heat flow can be calculated from this information. It is recommended that this be done to confirm the value measured by the procedure described in the previous paragraph.

A1.7.4 *Effect of Drift of the Metered Area Heater*—A quasi-heat loss exists due to the changing heat content of the metered area heater as its temperature changes. Typical plates have a relatively high heat capacity and even for small drift rates can produce significant errors in measured heat flow. If the drift is monotonic, the error is systematic, ${}_sQ_d$; if not, the error is exhibited as random error, ${}_rQ_d$. Normally, the experiment is conducted so that there is no observable drift. Under this circumstance, the possible drift is determined by the detectability or control limit, dT/dt, of the system. One can compute the magnitude of this error, Q_d in watts, from a knowledge of the maximum possible dT/dt and the specific heats and masses of the various components of the metered section of the plate as follows:

$$Q_d = dT/dt\ C_iM_i \tag{A1.1}$$

The specimen heat capacity also contributes to the drift error, but for low-density insulations the heat capacity of the specimen is small compared to the plate. This error also can be determined by measuring the dependence of drift rate on measured heater power. Comparison of the calculated and measured results is advised to increase confidence in the reported result.

A1.7.5 Power determination error, composed of both systematic, ${}_sQ_p$ and random, ${}_rQ_p$, components. With high quality instrumentation these errors can be reduced to an insignificant level. The manufacturers' specifications on bias and precision will normally suffice to define these errors.

A1.7.6 *Temperature and Temperature Difference*—Temperature error is composed of systematic, ${}_sT$, and random, ${}_rT$, components. In addition, these errors are further subdivided according to the source of the error:

A1.7.6.1 Calibration error, ${}_sT_c$, is entirely systematic as long as the same calibration is used. It is, however, not necessarily the same for each temperature sensor. In the case of thermocouples, calibration is frequently performed for each spool of wire, not for each piece of wire from that spool. Therefore, systematic differences can occur as one progresses through the spool. The calibration is frequently represented by an equation which approximates the experimental calibration data taken at selected temperatures. If a digital read-out device is used that yields temperature directly, the calibration formulation is built into the device and the same basis for error exists.

A1.7.6.2 Instrumentation measurement error, T_m, occurs when the sensor output is measured. This error contains both systematic and random components. Each component should be estimated from equipment manufacturer's specifications and from estimated spurious circuit effects. In addition, temperature errors are introduced by long and short-term control fluctuations. A helpful procedure to assess the magnitude of these errors is as follows. Place the guarded metered area and primary guard(s) in thermal contact with the adjacent cold plates (insert high conductance plates in place of the specimens if the plates cannot be placed physically together). Adjust the cold plates to the desired temperature; control this temperature until steady-state is reached. The metered area heater should be off. Periodically read the isothermal surface temperatures to detect systematic differences and random variations over an extended time.

A1.7.6.3 Sensor positioning, a potentially significant source of error in temperature measurement can be caused by improper positioning of the sensor or the disturbance caused by the presence of or finite size of the sensor itself. It is intended that the average temperature of each specimen surface be measured. If the sensor is mounted in the plate surface, thermal contact resistance between the plate and specimen is a source of error. If the sensor is mounted in the specimen surface, sensor separation (specimen thickness) is a source of error. If the specimen is inhomogeneous across the metered area, surface temperature variations exist and the indicated temperature will depend on its location on the surface. If heat flows along the sensor leads from the external environment, the measured temperature will be in error because of the presence of the sensor. For a single test on a given specimen, this source of error, ${}_sT_p$, is systematic. A performance check that is helpful to determine the potential temperature error due to temperature nonuniformity is as follows: Assemble a multijunction thermocouple and place it between the specimen and plate in question. Establish steady-state at the desired test condition. Determine the variation in temperature across the plate from the multijunction thermocouple outputs.

A1.7.6.4 A helpful technique to estimate interface temperature errors is to mount sensors both within the plate and within the specimen surface. Then perform a test and calculate the difference between the two sets of data.

A1.7.6.5 Temperature difference error is also composed of systematic, ${}_sT$, and random components, ${}_tT$. Care must be exercised in estimating these components compared to the error components for temperature itself. The results can depend strongly on whether a differential measurement or two absolute measurements are performed. Because T is frequently small, large percentage errors can occur if care is not observed. For example, at a mean specimen temperature of 300 K, an error of 1 K in the mean temperature, that corresponds to an error of about 0.2 % in thermal resistance for typical insulations. However, this same error of 1 K in measurement of a specimen temperature difference of 25 K corresponds to a 4 % error in both T and in the value of the thermal resistance, independent of the mean temperature. The ad hoc experiment described in 9.5.1.3 is recommended to provide estimates of these error components.

A1.7.7 Specimen thickness error, ${}_sL$, and meter area error, ${}_sA$, are both systematic errors. The specimen thickness error is determined by the ability to measure the plate spacing (including variations of this thickness over the metered area) or, in the case of rigid specimens, the specimen thickness and the changes due to thermal expansion. The effect of bowing or warping at operating temperatures should be given attention.

At relatively large thicknesses (above 5 cm) this error can be maintained below 0.5 %. At small thicknesses (below 0.5 cm) this error may become a dominating factor in the overall accuracy. The meter area error is usually small except for the assumption about what proportion of the gap area to include. This error is difficult to estimate for very thin specimens or when a discontinuity in the specimen occurs at the gap. The specimen thickness error will contain a random component, $_rL$, due to assembly and disassembly.

A1.8 *Thermal Conductance or Thermal Resistance*—The relative uncertainty in thermal conductance, *C*, caused by either random errors or systematic errors of indeterminate sign, may be calculated from the following error propagation formula:

$$(\Delta C/C)^2 = (\Delta Q/Q)^2 + (\Delta T/T)^2 \quad \text{(A1.2)}$$

where *Q/Q* and *T/T* are the total relative uncertainties of heat flux and temperature difference, respectively. The same equation applies to thermal resistance. Included in the total relative uncertainties are those due to the measurement as well as those discussed in Practice C 1045. For example for fibrous glass insulation at 24°C mean temperature and a 40°F temperature difference across the specimen the following errors can be realized:

$$(\Delta C/C)^2 = (0.5)^2 + (0.25)^2 = 0.31 \quad \text{(A1.3)}$$

Therefore, the uncertainty in thermal conductance would be $\sqrt{0.31} = 0.56$ %.

A1.9 *Thermal Conductivity or Thermal Resistivity*—The relative uncertainty in thermal conductivity caused by either random or systematic errors may be calculated from the following error propagation formula:

$$\left(\frac{\Delta\lambda}{\lambda}\right)^2 = (\Delta Q/Q)^2 + (\Delta T/T)^2 + (\Delta A/A)^2 + (\Delta L/L)^2 \quad \text{(A1.4)}$$

where *A/A* and *L/L* are the total relative uncertainties of area and thickness, respectively. Again, the above total relative uncertainties include not only the measurement uncertainty, but also the effect of material variability and deviations from the definitions as discussed in Practice C 1045. In addition, it should be noted that the temperature to which each measured property is assigned also contains a measurement error that affects the uncertainty of the final result. The effect of this error increases as the temperature dependence of the measured property increases.

A1.9.1 For example for fibrous glass insulation at 24°C mean temperature and a 22°C temperature difference across the specimen the following errors can be realized.

$$\left(\frac{\Delta\lambda}{\lambda}\right)^2 = (0.5)^2 + (0.25)^2 + (0.01)^2 + (0.1)^2 = 0.32 \quad \text{(A1.5)}$$

Therefore, the uncertainty in thermal conductivity would be $\sqrt{0.32} = 0.57$ %.

A1.10 It is recommended that the user periodically confirm these calculated uncertainties by measuring specimens of established standard reference materials or calibrated transfer specimens. Comparison of the measurement results with the accepted values will reveal whether the performance of the guarded hot plate is of acceptable quality. The results of such comparative measurements are not to be used to obtain an apparatus "calibration" or "correction" factor. For further information on this see Refs **(23-29)**.

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