



Designation: C177 – 10

Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus¹

This standard is issued under the fixed designation C177; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

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

1. Scope

1.1 This test method 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 C1044, and on the line source apparatus, Practice C1043, 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/(m²K).

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 Method C1363 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 C1045.

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

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

Current edition approved June 1, 2010. Published August 2010. Originally approved in 1942. Last previous edition approved in 2004 as C177 – 04 . DOI: 10.1520/C0177-10.

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

modifying the apparatus, to become a designer or builder, or both, on whom the demands for fundamental understanding 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 standard. No other units of measurement are included in this standard.

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:

Section	Section
Scope	1
Referenced Documents	2
Terminology	3
Summary of Test Method	4
Significance and Use	5
Apparatus	6
Specimen Preparation and Conditioning	7
Procedure	8
Calculation of Results	9
Report	10
Precision and Bias	11
Keywords	12
Figures	
General Arrangement of the Mechanical Components of the Guarded-Hot-Plate Apparatus	Fig. 1
Illustration of Heat Flow in the Guarded-Hot-Plate Apparatus	Fig. 2
Example Report Form	Fig. 3
Annexes	
Importance of Thickness	A1.1
Measuring Thickness	A1.2
Limitations Due to Apparatus	A1.3
Limitations Due to Temperature	A1.4
Limitations Due to Specimen	A1.5
Random and Systematic Error Components	A1.6
Error Components for Variables	A1.7
Thermal Conductance or Thermal Resistance Error Analysis	A1.8
Thermal Conductivity or Thermal Resistivity Error Analysis	A1.9
Uncertainty Verification	A1.10

2. Referenced Documents

2.1 ASTM Standards:³

- C168 Terminology Relating to Thermal Insulation
- C518 Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus
- C687 Practice for Determination of Thermal Resistance of Loose-Fill Building Insulation
- C1043 Practice for Guarded-Hot-Plate Design Using Circular Line-Heat Sources
- C1044 Practice for Using a Guarded-Hot-Plate Apparatus or Thin-Heater Apparatus in the Single-Sided Mode
- C1045 Practice for Calculating Thermal Transmission Properties Under Steady-State Conditions
- C1058 Practice for Selecting Temperatures for Evaluating and Reporting Thermal Properties of Thermal Insulation
- C1363 Test Method for Thermal Performance of Building Materials and Envelope Assemblies by Means of a Hot Box Apparatus
- E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 ISO Standard:

- ISO 8302 Thermal Insulation—Determination of Steady-State Areal Thermal Resistance and Related Properties—Guarded-Hot-Plate Apparatus⁴

2.3 ASTM Adjuncts:ASTM

- Table of Theoretical Maximum Thickness of Specimens and Associated Errors⁵
- Descriptions of Three Guarded-Hot-Plate Designs⁵
- Line-Heat-Source Guarded Hot-Plate Apparatus⁶

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms and symbols used in this test method, refer to Terminology C168 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.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

⁵ Available from ASTM Headquarters, Order Adjunct: ADJC0177.

⁶ 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 C168.

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

- 3.3.1 ρ_m —specimen metered section density, kg/m³.
- 3.3.2 ρ_s —specimen density, kg/m³.
- 3.3.3 λ —thermal conductivity, W/(m K).
- 3.3.4 σ —Stefan-Boltzmann constant, W/m² K⁴.
- 3.3.5 A —metered section area normal to heat flow, m².
- 3.3.6 A_g —area of the gap between the metered section and the primary guard, m².
- 3.3.7 A_m —area of the actual metered section, m².
- 3.3.8 A_s —area of the total specimen, m².
- 3.3.9 C —thermal conductance, W/(m² 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.

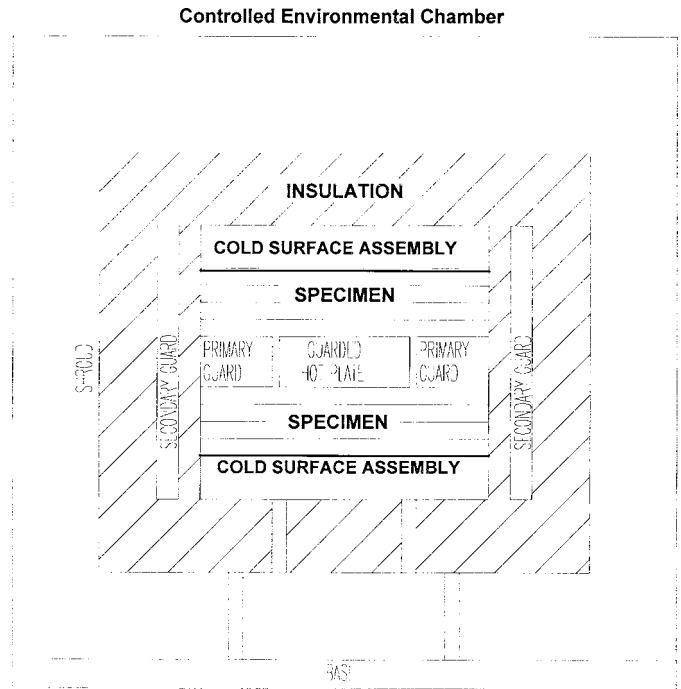


FIG. 1 General Arrangement of the Mechanical Components of the Guarded-Hot-Plate Apparatus

3.3.18 q —heat flux (heat flow rate per unit area), Q , through area, A , W/m².

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, m² 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, $(T_h + T_c)/2$.

3.3.27.1 *Discussion*— The Guarded-Hot-Plate Apparatus provides a means for measurement of steady state heat flux through insulation materials, that consists of a guarded heater unit, comprised of a center metering area and concentric separately heated guards, and an opposite, similarly sized cooling plate. Specimens are placed in the space between the heater plate and the cooling plate for testing. The guarded-hot-plate is operated as a single or double sided apparatus. Insulation thermal properties are calculated from measurements of metering area, energy input, temperatures, and thickness. The guarded-hot-plate, which provides an absolute measurement of heat flux, has been shown to be applicable for most insulating materials over a wide range of temperature conditions.

4. Summary of Test Method

4.1 Fig. 1 illustrates the main components of the idealized

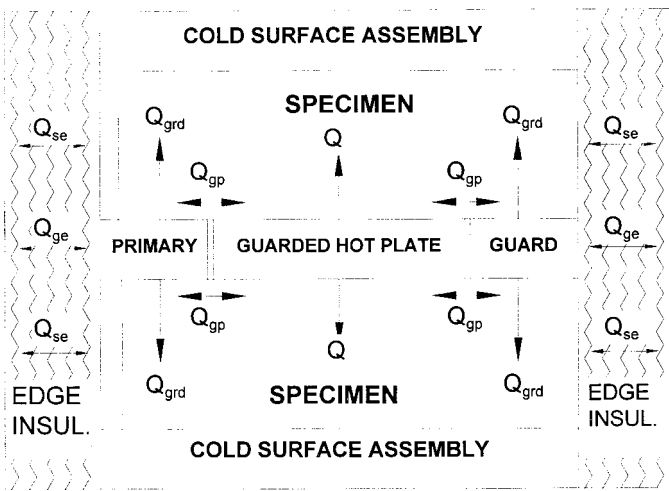


FIG. 2 Illustration of Idealized Heat Flow in a Guarded-Hot-Plate Apparatus

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 C1044. For guidance in the use of a guarded-hot-plate incorporating the use of a line source heater, refer to Practice C1043.

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.

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 C518 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 C518 may be comparable with that of this test method, Test Method C518 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 $\pm 10^\circ\text{C}$ 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 C1043.

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, 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 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 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 E230. 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 $0.2 \text{ m}^2 \text{ K/W}$ 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 $\pm 0.1 \text{ K}$ 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 $4\sigma T_m^3 / (2/\epsilon - 1)$. 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 $4\sigma T_m^3 / (2/\epsilon - 1)$. 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 C518 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 C1045. 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 C687 for details.

7.3 *Specimen Conditioning*—Condition the specimens either as stated in the material specification or where no guideline is given, at $22 \pm 5^\circ\text{C}$ and $50 \pm 10\%$ relative humidity for a period of time until less than a 1 % mass change in 24 h is observed.