

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, it is possible that the secondary guard will 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/e-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/e-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. It is possible that



verification of the apparatus will 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**—It is possible that the surfaces of the uncompressed specimens will be comparatively

uneven so long as surface undulations are removed under test compression. It will potentially 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 is most likely 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, it is possible that plate spacers will 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.

**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 C1058. The ambient temperature should be the same as or slightly above the mean temperature of the test. It is possible that this will 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, has the potential to 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

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heater flows through each specimen. Determine the power,  $Q$ , from emf,  $E$ , and current,  $I$ , and calculate as follows:

$$Q = E \times I \quad (2)$$

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:

$$A = A_m + \frac{A_g}{2} \quad (3)$$

For high precision measurements, it is possible that this assumption that the gap contributes half of its area to the effective metered section area,  $A$ , will 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:

$$A = A_m + \frac{A_g \lambda_g}{2\lambda_s} \quad (4)$$

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:

$$q = \frac{Q}{A} \quad (5)$$

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,  $\rho_m$ , or the sample density,  $\rho_s$ , 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:

$$\rho_m = \frac{m}{A \times L} \quad (6)$$

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 C1045.

## 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.

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 for inclusion 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, it is acceptable that less be 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 C177." This statement shall be followed by a listing of specific deviations from this test method and any special test conditions that were applied.

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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

Variable	Measured Value	Uncertainty	
		Systematic	Random
Q, W			
Th, K			
Tc, K			
Tm, K			
$\Delta T, K$			
A, m <sup>2</sup>			
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

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 E456.

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 standard 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

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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 experience suggests that errors are some times 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<sup>2</sup> 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 E691, 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 materi-

als 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 C687 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  $\pm 3$  % 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<sup>2</sup> 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<sup>3</sup>) 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<sup>3</sup> 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 an 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<sup>2</sup>. 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<sup>3</sup>); and, high-density glass-fiber batt, foil-faced (64 kg/m<sup>3</sup>). 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

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 <sup>A</sup>	6 <sup>A</sup>	2.26 <sup>A</sup>	7 <sup>A</sup>
Glass-fiber batt	25	0.039 <sup>A</sup>	7 <sup>A</sup>	2.82 <sup>A</sup>	3B <sup>A</sup>
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

<sup>A</sup> 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%.

systematic errors separately. A description of random and systematic errors and possible sources of error are discussed below.

11.8.1 *Random Error,  $\delta_r$* —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,  $\delta_s$* —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 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 can 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  $\delta Q$ ; temperature difference,  $\delta \Delta T$ ; metered section area,  $\delta A$ ; and specimen thickness,  $\delta L$ . 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 it is possible that the error in neglecting any changes in thickness will 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. It is possible that the shape of the specimen will 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 this, it is possible that the specimen will 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 needs to be measured both before and after the thermal transmission property is measured, to show whether

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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. Install rigid rods securely 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 shall 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, is measured after the test to monitor any significant changes that have the potential to affect 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<sup>2</sup> 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 C1058.

A1.4.2 If temperature measurements of each plate are made by means of thermocouples with independent reference junctions, it is possible that the accuracy of the calibration of each thermocouple will 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 It is possible that 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 that 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 are potentially not representative of the bulk material, while specimens larger than 0.5 m have the potential to 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 has the potential to 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 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

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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  $\pm 1\%$ , 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,  $\Delta L$ , and increments of the thermal resistance,  $\Delta R$ .

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  $\lambda_m$ .

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 uncer-

ainties(32): uncertainty in heat flow,  $\delta Q$ ; uncertainty in temperature difference,  $\delta \Delta T = \delta(T_H - T_C)$ ; uncertainty in metered area,  $\delta A$ ; and, uncertainty in specimen thickness,  $\delta L$ .

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*,  $\delta_s$ —Systematic error,  $\delta_s$ , 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*,  $\delta_r$ —Random error,  $\delta_r$ , 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,  $\delta_s Q_e$** —Edge heat loss,  $\delta_s Q_e$ , 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,  $\delta_s Q_g$ , and random,  $\delta_r Q_g$ , 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,  $\delta Q_g$ , is linearly dependent on temperature unbalance across the gap,  $\Delta T_g$ , that is,  $\delta Q_g = B \Delta T_g$ . 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,  $\delta_s Q_d$ ; if not, the error is exhibited as random error,  $\delta_r Q_d$ . 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,  $\delta Q_d$  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:

$$\delta Q_d = dT/dt \Sigma C_p M_i \quad (A1.1)$$

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,  $\delta_s Q_p$ , and random,  $\delta_r Q_p$ , 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,  $\delta_s T$ , and random,  $\delta_r T$ , components. In addition, these errors are further subdivided according to the source of the error:

**A1.7.6.1 Calibration error,  $\delta_c T_s$** , 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,  $\delta T_m$** , 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

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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,  $\delta_p T_p$ , is systematic. A performance check that is helpful to determine the potential temperature error due to temperature nonuniformity is as follows: Assemble a multi-junction 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 multi-junction 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,  $\delta_s \Delta T$ , and random components,  $\delta_r \Delta T$ . 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  $\Delta T$  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 A1.7.6.3 is recommended to provide estimates of these error components.

A1.7.7 Specimen thickness error,  $\delta_s L$ , and meter area error,  $\delta_p A$ , 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,  $\delta_r L$ , 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:

$$\delta C/C^2 = \delta Q/Q^2 + \delta \Delta T/\Delta T^2 + \delta A/A^2 \quad (A1.2)$$

where  $\delta Q/Q$  and  $\delta \Delta T/\Delta T$  and  $\delta A/A$  are the total relative uncertainties of heat flux, temperature difference, and meter area 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 C1045. 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. Note that the example below uses hypothetical values for  $\delta Q/Q$  and  $\delta \Delta T/\Delta T$ . The user must determine their own values for this calculation:

$$\delta C/C^2 = 0.5^2 + 0.25^2 + 0.01^2 = 0.31 \quad (A1.3)$$

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:

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

where  $\delta A/A$  and  $\delta L/L$  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 C1045. 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. Again the example below uses hypothetical values for these uncertainties. The user must obtain their own input values.

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

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 thickness reference materials or calibrated transfer specimens. Comparison of the measurement results with the accepted values will reveal whether the performance of the

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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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DOCENTE DEL CURSO : FECHA : 26.10.2014

Nº	INSTRUMENTO	CANT.	MARCA	COD. PATRIMONIAL	ESTADO	OBSERVACIONES
1	LADRILLOS	02				
2	VERNIER	01				
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Nº	CÓDIGO	NOMBRES Y APELLIDOS	FIRMA	DNI
1	009200396-H	Ronald Camino Quispe		42128342
2	012100710-B	Ricardo Camino Quispe		41219264
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DOCENTE DEL CURSO : Prof. Henry Enciso B. FECHA : 22.5.1.2014

Nº	INSTRUMENTO	CANT.	MARCA	COD. PATRIMONIAL	ESTADO	OBSERVACIONES
1	Equipo de compresión	01				
2	Worillas de 12 cm de diame.	02				
3	Placa metálica	01				
4	Embalse metálico	01				
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1	012100710-0	Richard Camino Quispe		41219264
2	009200396-4	Ronald Camino Quispe		42128343
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2	placas metalicas	03				
3	embase proctor	01				
4	bermet	01				
5	bedilejo	01				
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Nº	CÓDIGO	NOMBRES Y APELLIDOS	FIRMA	DNI
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TITULO DE LA PRACTICA : Topometria HORARIO : Sabado 7-13
DOCENTE DEL CURSO : Ing. Henry Enciso B. FECHA : 21.5.2019

Table with 7 columns: N°, INSTRUMENTO, CANT., MARCA, COD. PATRIMONIAL, ESTADO, OBSERVACIONES. Contains 3 rows of instrument data.

Table with 5 columns: N°, CÓDIGO, NOMBRES Y APELLIDOS, FIRMA, DNI. Contains 2 rows of user data.

Table with 2 columns: HORA DE ENTREGA, HORA DE DEVOLUCIÓN.

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TÍTULO DE LA PRÁCTICA : Succión de lamillas K.K. HORARIO : Sábado 7-1 pm  
DOCENTE DEL CURSO : Ing. Henry Enciso B. FECHA : 15.5.2014

Nº	INSTRUMENTO	CANT.	MARCA	COD. PATRIMONIAL	ESTADO	OBSERVACIONES
1	Bandeja	01				
2	Nivel	01				
3	barilla 5 mm de altura	02				
4	berner	01				
5	pipeta	01				
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Nº	CÓDIGO	NOMBRES Y APELLIDOS	FIRMA	DNI
1	012100710	Richard camino Quispe		41219264
2	009200386-A	Ronald Camino Quispe		42128343
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Nº	INSTRUMENTO	CANT.	MARCA	COD. PATRIMONIAL	ESTADO	OBSERVACIONES
1	Horno eléctrico	01				
2	balanza de precisión	01				
3	regla metálica	01				
4	recipientes	03				
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Nº	CÓDIGO	NOMBRES Y APELLIDOS	FIRMA	DNI
1	012100710-5	Ricardo Camino Quispe		41219264
2	009200396-H	Ronald Camino Quispe		42128373
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