EXHIBIT 150
PART 5

CERTIFICATE

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

This standard is issued under the fixed designation C 177; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method establishes the criteria for the laboratory measurement of the steady-state heat flux through flat, homogeneous specimen(s) when their surfaces are in contact with solid, parallel boundaries held at constant temperatures using the guarded-hot-plate apparatus.

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

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

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

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

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

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

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

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

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

1.11 It is not practical for a test method of this type to establish details of design and construction and the procedures to cover all contingencies that might offer difficulties to a person without technical knowledge concerning theory of heat flow, temperature measurements and general testing practices. The user may also find it necessary, when repairing or modifying the apparatus, to become a designer or builder, or both, on whom the demands for fundamental understanding 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

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

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

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

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

1.15 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Note 22.

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

2. Referenced Documents

2.1 *ASTM Standards:*

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

C 168 Terminology Relating to Thermal Insulating Materials[3]

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

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

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

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

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

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

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

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

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

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

2.2 *ISO Standard:*

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

2.3 *ASTM Adjuncts:*

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

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

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

3. Terminology

3.1 *Definitions:*

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

3.2 *Definitions of Terms Specific to This Standard:*

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

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

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

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.

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

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

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

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

[7] Available from ASTM Headquarters, Request PCN No. 12-301770-00.

[8] Available from ASTM Headquarters, Request PCN No. 12-310430-61.

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

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

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

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

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

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

3.3.2 ρ_s—specimen density, kg/m^3.

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

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

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

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

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

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

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

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

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

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

3.3.13 L—in-situ specimen thickness, m.

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

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

3.3.16 m_s—mass of the specimen, kg.

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

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

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

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

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

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

3.3.23 R—thermal resistance, m^2 K/W.

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

3.3.25 T_c—cold surface temperature, K.

3.3.26 T_h—hot surface temperature, K.

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

4. Summary of Test Method

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

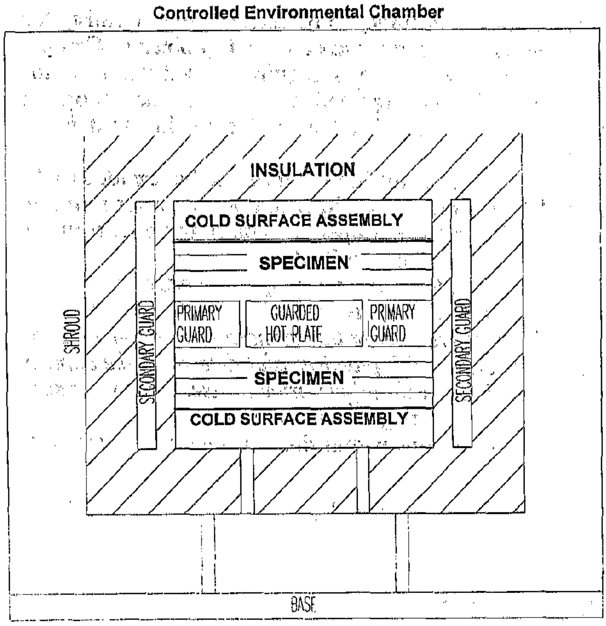

FIG. 1 General Arrangement of the Mechanical Components of the Guarded-Hot-Plate Apparatus

4.1.1 The guarded-hot-plate provides the power (heat flow per unit time) for the measurement and defines the actual test volume, that is, that portion of the specimen that is actually being measured. The function of the primary guard, and additional coplanar guard where applicable, of the guarded-hot-plate apparatus is to provide the proper thermal conditions within the test volume to reduce lateral heat flow within the apparatus. The proper (idealized) conditions are illustrated in Fig. 1 by the configuration of the isothermal surfaces and lines of constant heat flux within the specimen.

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

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,

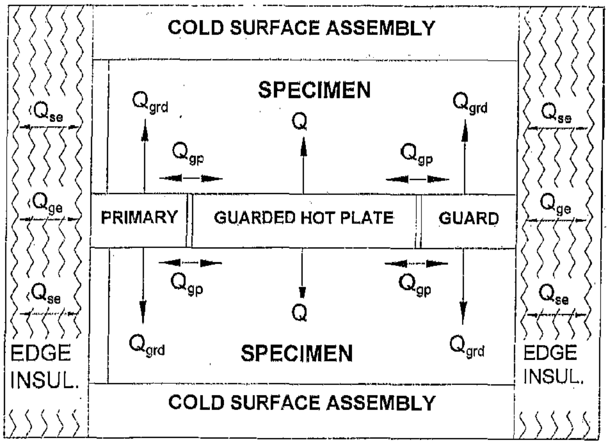

FIG. 2 Illustration of Idealized Heat Flow in a Guarded-Hot-Plate Apparatus

because the plates and specimens are of finite dimensions, and because the external controlled environment is often at a temperature different from the edge of the metered section, some lateral heat flow occurs. The primary guard for the guarded hot plate limits the magnitude of the lateral heat flow in the metered section. The effectiveness of the primary guard is determined, in part, by the ratio of its lateral dimension to that of the metered section and to the specimen thickness **(6,7,8,20,31)**.

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

5. Significance and Use

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

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

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

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

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

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

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

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

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

6. Apparatus

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

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

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

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

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

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

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

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

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

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

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

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

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

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

6.3.2.1 *Temperature Stability*—Materials are selected for the heaters and surface plates that are dimensionally and chemically stable and suitably strong to withstand warpage and distortion when a clamping force is applied. For modest temperatures, electric resistance heaters embedded in silicone have been successfully employed; at higher temperatures, heating elements sandwiched between mica sheets or inserted into a ceramic core have been used. Surface plates for hot surface assemblies used at modest temperatures have been fabricated from copper and aluminum. High purity nickel alloys have been used for higher temperature applications.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

NOTE 7—The temperature of the cold surface assembly may be maintained through the use of a temperature-controlled bath; in this instance, there is no need to install a cold surface heater. Care must be taken in this instance; the flow rate of the bath must be sufficient to satisfy the temperature uniformity requirements specified in 6.3.1.2 and 6.5.1.

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

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

6.5.3 *High Temperature Operation*—When the cold surface assemblies will be operated at high temperatures, several thin sheets of insulation may be inserted between the heat sink and cold surface heater. The addition of these insulation sheets will reduce the energy requirements to the cold surface heater and extend service life.

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

NOTE 8—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.

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

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

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

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

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

NOTE 10—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 11—Care shall be taken to ensure that there are no voids, pockets, or other extraneous sources of radiative heat transfer occurring at or near the guarded-hot-plate.

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

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

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

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

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

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

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

NOTE 12—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 13—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 inhomogenicities, generally increase as the temperature gradients within the measuring leads increase. For the same reason, junctions between dissimilar metal leads should not be made in the regions of appreciable temperature gradients. Low thermal emf switches should be used in the temperature measurement circuits. An insulated, isothermal box of heavy sheet metal can be used when joining leads of dissimilar metals in the thermocouple circuit. It is recommended that all connections of thermocouple wire to copper wire be accomplished within the isothermal box in order that the junctions are at the same temperature; then the copper, not the thermocouple, leads are connected to the needed switching devices and/or voltmeters.

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

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

NOTE 14—This method of deploying thermocouples is traditionally used for compressible specimens and for rigid specimens possessing flat surfaces that have a thermal resistance of greater than 0.2 m^2 K/W (1 h ft^2 F/Btu) at ambient conditions.

NOTE 15—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 16—It is recommended that one temperature sensor be placed in the center of the metered section and that additional sensor be uniformly distributed radially.

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

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

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

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

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

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

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

NOTE 17—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 $4_n T_m^3 (2/\epsilon-1)$. 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 C 518 can be used to check the similarity of the specimens' thermal characteristics.

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

7.1.2 *Size*—The specimen shall be sized to cover the entire metered section and guard area when possible. It is desirable to cover the gap between the guarded-hot-plate and the primary guard when sample size is limited. The guard portion of the volume between the heating and cooling plates should be filled with material having similar thermal conductance characteristics as the specimen. When the specimen has a high lateral conductance such as a dense solid, a gap between the metered section and the primary guard shall be provided within the specimen. Refer to 7.2.3 for special precautions.

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

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

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

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

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

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

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

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

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

NOTE 18—Specimens can be conditioned at different conditions in order to determine the effect on the thermal properties of the specimens. Conditioning environments shall be reported with the test results.

8. Procedure

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

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

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

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

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

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

8.7 Record the start time and date of the test. Begin data acquisition. The recorded data shall include: the date and time of data acquisition; power to the guarded-hot-plate; hot side guarded-hot-plate surface temperature; hot side guard temperatures; cold surface assembly temperatures; controlled environment ambient temperature and relative humidity; temperature difference or thermopile output across the gap between the guard and metered section; and calculated heat flux and estimated thermal property of interest.

NOTE 19—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 20—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 20). This combination of three runs shall be considered a valid test if each datum obtained for each measured variable meets the following criteria.

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

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

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

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

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

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

9. Calculation

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

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

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

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

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

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

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

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

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

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

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

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

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

or:

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

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

10. Report

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

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

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

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

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

NOTE 21—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 prop-

Test Report

Date: Test Report Number:

Operator: Duration of Test:

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

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

Experimental Results

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

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

FIG. 3 Example Test Report Form

erties, such as fill-gas and pressure, shall be specified when applicable,

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

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

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

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

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

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

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

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

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

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

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

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

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

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

NOTE 22: **Caution**—Where this test method might be specifically referenced in published test reports and published data claims, and where deviations from the specifics of the test method existed in the tests used to obtain said data, the following statement shall be required to accompany such published information: **"This test did not fully comply with following the provisions of Test Method C 177."** This statement shall be followed by a listing of specific deviations from this test method and any special test conditions that were applied.

11. Precision and Bias

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

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

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

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

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

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

11.5 *Ruggedness Tests*—The results of one ruggedness study for a 200 mm^2 guarded hot plate and two materials having different thermal conductivity's have been reported **(37)**. Matched pairs, 85 mm thick, of polyurethane foam and silicone rubber were measured at a mean temperature of 297 K and a temperature difference of 23 K. For each specimen, the width of edge insulation was set at one of five levels (0, 12.7, 25.4, 50.8, and 76.2 mm) while the ambient temperature was varied at one of three levels. The results indicate that the edge losses are reduced with edge insulation but only become zero when the ambient temperature is at one specific value. The optimum ambient temperature appears to be a function of specimen thickness and thermal conductivity, and edge insulation thickness.

NOTE 23—As noted in Section 8, the value of the ambient temperature is set to either the same value as the mean temperature of the test or a value slightly above the mean temperature. The user should determine the optimum value for their apparatus and test conditions by using the sensitivity analysis described in A4.2. This dependence may change appreciably for different specimens or apparatus conditions and, therefore, should be done under typical test conditions.

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

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

11.6.2 In 1985, results of a third round of interlaboratory comparisons were reported **(41)** for five large guarded-

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

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

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

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

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

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

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

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

11.8.2 *Systematic Error,* δ_s—A systematic error (bias) is a fixed deviation that is inherent in each and every measurement. If the magnitude and direction of the systematic error are known, the user can make appropriate correction(s) to the measured value. Under such circumstances a justification for the correction should be provided. In general, the magnitude of the error, $|\delta_s|$, is estimated by experience or judgment.

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

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

12. Keywords

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

ANNEX

(Mandatory Information)

A1. THICKNESS MEASUREMENT, LIMITATIONS AND MEASUREMENT UNCERTAINTY

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

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

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

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

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

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

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

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

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

A1.3 *Limitations Due to Apparatus:*

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

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

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

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

A1.4 *Limits to Temperature Difference:*

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

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

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

A1.4.4 *Maximum Specimen Thickness:*

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

A1.4.4.2 This is an approximation and the results do not necessarily imply the measurement of conductivity of each layer. The accuracy will remain close to that predictable for tests on homogeneous specimens. No guidelines can be supplied to assess measurement accuracy when the requirement of A1.4.4.1 is not met.

A1.4.5 *Minimum Specimen Thickness:*

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

A1.4.5.2 The metered area, that is, the area of the specimen traversed by the heat flow-rate fed by the metered section, is related to the specimen thickness and to the gap width. As the thickness tends to zero, the metered area tends to the area of the metered section, while for thick specimens the metered area is bounded by the line defining the centre of the primary guard gap. To avoid complex corrections, this definition can be retained, provided the thickness of the specimen is at least ten times the width of the gap.

A1.4.6 *Maximum Operating Temperature:*

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

A1.4.7 *Vacuum Conditions:*

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

A1.4.8 *Apparatus Size:*

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

A1.5 *Limitations Due to Specimen:*

A1.5.1 *Thermal Resistance or Thermal Conductance:*

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

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

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

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

A1.5.2 *Temperature-Difference Correlation:*

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

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

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

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

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

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

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

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

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

A1.5.7 *Dependence on Specimen Thickness:*

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

A1.5.9 *Warping*—Special care should be exercised with specimens with large coefficients of thermal expansion that warp excessively when subjected to a temperature gradient. The warping may damage the apparatus or may cause additional contact resistance that may lead to serious errors in the measurement. Specially designed apparatus may be necessary to measure such materials.

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

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

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

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

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

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

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

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

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

A1.7.3.1 It has been found that **(3,15,16)** the gap heat loss, Q_{gp} is linearly dependent on temperature unbalance across the gap, T_g, that is, $Q_{gp} = BT_g$. The proportionality constant, B, is dependent on the wires crossing the gap (number, size, and type), gap geometry (width and cross-sectional shape), the gap fill material (gas, insulation), the emittance of the gap surfaces and the material in the vicinity of the gap between the hot and cold plates. A reasonable approximation of this heat flow can be calculated from this information. It is recommended that this be done to confirm the value measured by the procedure described in the previous paragraph.

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

$$Q_d = dT/dt\, C_i M_i \qquad \text{(A1.1)}$$

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

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

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

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

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

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

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

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

A1.7.7 Specimen thickness error, ${}_sL$, and meter area error, ${}_sA$, are both systematic errors. The specimen thickness error is determined by the ability to measure the plate spacing (including variations of this thickness over the metered area) or, in the case of rigid specimens, the specimen thickness and the changes due to thermal expansion. The effect of bowing or warping at operating temperatures should be given attention. At relatively large thicknesses (above 5 cm) this error can be maintained below 0.5 %. At small thicknesses (below 0.5 cm) this error may become a dominating factor in the overall accuracy. The meter area error is usually small except for the assumption about what proportion of the gap area to include. This error is difficult to estimate for very thin specimens or when a discontinuity in the specimen occurs at the gap. The specimen thickness error will contain a random component, ${}_rL$, due to assembly and disassembly.

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

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

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

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

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

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

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

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

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

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

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

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

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CERTIFICATE

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By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

Document Name: ASTM C236: Standard Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box

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Standard Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box[1]

This standard is issued under the fixed designation C 236; 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. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

$^{\epsilon 1}$ NOTE—Section 12 was added editorially in September 1993.

1. Scope

1.1 This test method, known as the guarded hot box method, covers the measurement of the steady-state thermal transfer properties of panels. In distinction to Test Method C 177, which is primarily applicable to homogeneous samples, the guarded hot box method provides for the evaluation of thermal performance of building assemblies. This test method is suitable for building construction assemblies, building panels, and other applications of nonohomogeneous specimens at similar temperature ranges. It may also be used for homogeneous specimens.

1.2 This test method may be applied to any building construction for which it is possible to build a reasonably representative specimen of size appropriate for the apparatus.

NOTE 1—A calibrated hot box, Test Method C 976, may also be used for the described measurements and may prove more satisfactory for testing assemblies under dynamic conditions (nonsteady-state) and to evaluate the effects of water migration and air infiltration. The choice between the calibrated or the guarded hot box should be made only after careful consideration of the contemplated use.

1.3 In applying this test method, the general principles outlined must be followed; however, the details of the apparatus and procedures may be varied as needed.

1.3.1 The intent of this test method is to give the essential principles and the general arrangement of the apparatus. Any test using this apparatus must follow those principles. The details of the apparatus and the suggested procedures that follow are given not as mandatory requirements but as examples of this test method and precautions that have been found useful to satisfy the essential principles.

1.3.2 Persons applying this test method shall be trained in the methods of temperature measurement, shall possess a knowledge of the theory of heat flow, and shall understand the general requirements of testing practice.

1.3.3 *This standard does not purport to address all of the safety problems, 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.*

NOTE 2—While various units may be found for thermal properties, the International System of units is used exclusively in this test method. For conversion factors to inch-pound and kilogram-calorie systems, see Table 1.

2. Referenced Documents

2.1 *ASTM Standards:*

C 168 Terminology Relating to Thermal Insulating Materials[2]

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus[2]

C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus[2]

C 578 Specification for Preformed Cellular Polystyrene Thermal Insulation[2]

C 976 Test Method for Thermal Performance of Building Assemblies by Means of a Calibrated Hot Box[2]

C 1045 Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements[2]

E 178 Practice for Dealing With Outlying Observations[3]

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

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Terminology

3.1 *Definitions*— For definitions of terms used in this test method, refer to Terminology C 168.

3.2 *Symbols:*

3.2.1 The symbols used in this test method have the following significance:

λ = thermal conductivity, W/(m·K),

C = thermal conductance, W/(m^2·K),

h = surface conductance, W/(m^2·K),

U = thermal transmittance, W/(m^2·K),

q = heat flux (time rate of heat flow through Area A), W/m^2,

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

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

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

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

TABLE 1 Conversion Factors for Thermal Conductivity^A

	W/(m·K)^B	W/(cm·K)	cal/(s·cm·K)	kg-cal/(h-m·K)	Btu/(h·ft·°F)	Btu·in./(h·ft^2°F)
1 $W \cdot m^{-1} \cdot K^{-1}$ =	1.000	1.000×10^{-2}	2.388×10^{-3}	0.8598	0.5778	6.933
1 $W \cdot cm^{-1} \cdot K^{-1}$ =	100.0	1.000	0.2388	85.98	57.78	693.3
1 $cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}$ =	418.7	4.187	1.000	360.0	241.9	2903.00
1 $kg\text{-}cal \cdot h^{-1} \cdot h^{-1} \cdot K^{-1}$ =	1.163	1.163×10^{-2}	2.778×10^{-3}	1.000	0.6720	8.064
1 $Btu \cdot h^{-1} \cdot ft^{-1} \cdot °F^{-1}$ =	1.731	1.731×10^{-2}	4.134×10^{-3}	1.488	1.000	12.00
1 $Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$ =	0.1442	1.442×10^{-3}	3.445×10^{-4}	0.1240	8.333×10^{-2}	1.000

			Thermal Conductance^A		
	W/(m^2·K)^B	W/(cm^2·K)	cal/(s·cm^2·K)	kg-cal/(h·m^2·K)	Btu/h·ft^2·°F)
1 $W \cdot m^{-2} \cdot K^{-1}$ =	1.000	1.000×10^{-4}	2.388×10^{-5}	0.8598	0.1761
1 $W \cdot cm^{-2} \cdot K^{-1}$ =	1.000 × 104	1.000	0.2388	8598	1761
1 $cal \cdot s^{-1} \cdot cm^{-2} \cdot K^{-1}$ =	4.187×10^4	4.187	1.000	3.600×10^4	7373
1 $kg\text{-}cal \cdot h^{-1} \cdot m^{-2} \cdot K^{-1}$ =	1.163	1.163×10^{-4}	2.778×10^{-3}	1.000	0.2048
1 $Btu \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$ =	5.678	5.678×10^{-4}	1.356×10^{-4}	4.882	1.000

[A] Units are given in terms of (*1*) the absolute joule per second or watt, (*2*) the calorie (International Table) = 4.1868 J, or the British thermal unit (International Table) = 1055.06 J.

[B] This is the SI unit.

Q = time rate of heat flow, total input to the metering box, W,
A = metering area normal to heat flow, m^2,
L = length of path of heat flow (thickness of specimen), m,
N = minimum number of thermocouples (see Eq 1, 6.5.1.1),
r = surface resistance, K·m^2/W,
R = thermal resistance, K·m^2/W,
R_u = overall thermal resistance, K·m^2/W,
t_h = average temperature of air 75 mm or more from the hot surface, K,
t_1 = area weighted average temperature of hot surface, K,
t_2 = area weighted average temperature of cold surface, K, and
t_c = average temperature of air 75 mm or more from cold surface, K.

4. Summary of Test Method

4.1 To determine the conductance, *C*, the thermal transmittance, *U*, and the thermal resistance, *R*, of any specimen, it is necessary to know the area, *A*, the heat flux, *q*, and the temperature differences, all of which must be determined under such conditions that the flow of heat is steady. The hot box is an apparatus designed to determine thermal performance for representative test panels and is an arrangement for establishing and maintaining a desired steady temperature difference across a test panel for the period of time necessary to ensure constant heat flux and steady temperature, and for an additional period adequate to measure these quantities to the desired accuracy. The area and the temperatures can be measured directly. The heat flux, *q*, however, cannot be directly measured, and it is to obtain a measure of *q* that the hot box has been given its characteristic design. In order to determine *q*, a five-sided metering box is placed with its open side against the warm face of the test panel. If the average temperature across the walls of the metering box is maintained the same, then the net interchange between the metering box and the surrounding space is zero, and the heat input to the metering box is a measure of the heat flux through a known area of the panel. The portion of the panel outside the meter area, laved by the air of the surrounding guard space, constitutes a guard area to minimize lateral heat flow in the test panel near the metering area. Moisture migration, condensation, and freezing within the specimen can cause variations in heat flow; to avoid this, the dew point temperature on the warm side must be kept below the temperature of the cold side when the warm surface is susceptible to ingress of moisture vapor. It is expected that, in general, tests in the guarded hot box apparatus will be conducted on substantially dry test panels, with no effort made to impose or account for the effect of the vapor flow through or into the panel during the test.

4.2 Since the basic principle of the test method is to maintain a zero temperature difference across the metering box walls, adequate controls and temperature-monitoring capabilities are essential. It is recognized that small temperature gradients could occur due to the limitations of controllers. Since the total wall area of the metering box is often more than twice the metering area of the panel, small temperature gradients through the walls may cause heat flows totaling a significant fraction of the heat input to the metering box. For this reason, the metering box walls may also be equipped to serve as a heat flow meter so that heat flow through them can be estimated and minimized by adjusting conditions during tests, and so that a heat flow correction can be applied in calculating test results.

5. Significance and Use

5.1 When the guarded hot box is constructed to test assemblies in the vertical orientation, it is suited for evaluating walls and other vertical structures. When constructed to test assemblies in the horizontal orientation, it is suited for evaluating roof, ceiling, floor, and other horizontal structures. Other orientations are allowable. The same apparatus may be used for both vertical and horizontal testing if it can be rotated or reassembled in either orientation.

NOTE 3—Horizontal structures that incorporate attic spaces between a ceiling and a sloping roof are highly complex constructions, and testing in the guarded hot box would be extremely difficult. Proper consideration must be given to specimen size, natural air movement, ventilation effects, radiative effect, baffles at the guard-meter demarcation, etc. All of these special conditions must be included in the report (10.1.1). Consideration should be given to the use of the calibrated hot box for such large, complex constructions.

5.2 For vertical specimens with air spaces that significantly affect thermal performance, the metering box height should ideally match the construction height. If this is not possible, horizontal convection barriers must be installed to prevent air exchange between meter and guard areas, unless it can be shown that the omission of such barriers does not significantly affect results.

5.3 For all specimens it is necessary to maintain a near zero lateral heat flow between the guard area and the meter area of the specimen. This can be achieved by maintaining a near zero temperature difference on the specimen surface between the metered and guard areas. In specimens incorporating an element of high lateral conductance (such as a metal sheet), it may be necessary to separate the metered and the guard areas of the highly conductive element by a narrow gap such as a saw cut.

5.4 Since this test method determines the total flow of heat through the test area demarcated by the metering box, it is possible to determine the heat flow through a building element smaller than the test area, such as a window or representative area of a panel unit, if the parallel heat flow through the remaining surrounding area or mask is determined (see Annex A1).

6. Apparatus

6.1 *Arrangement*—Fig. 1 (*a*) shows a schematic arrangement of the test panel and of various major elements of the apparatus; Fig. 1(*b*) and (*c*) show alternative arrangements. Still other arrangements, accomplishing the same purpose, may be preferred for reasons of convenience or ease of installing panels. In general, the size of the metering box determines the minimum size of the other elements.

6.2 *Metering Box:*

6.2.1 *Size*—The size of the metering box is largely governed by the metering area required to obtain a representative test area of panel. For example, for panels incorporating air spaces or stud spaces, the metering area, preferably, should exactly span an integral number of spaces. The height of the metering box should be not less than the width and is subject to the limitations as described in 5.2. The depth of the metering box should be not greater than that required to accommodate its necessary equipment.

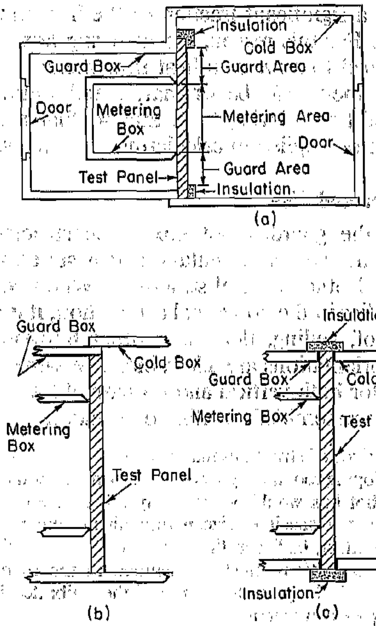

FIG. 1 General Arrangements of Test Box, Guard Box, Test Panel, and Cold Box

6.2.2 *Thermal Resistance*—The metering box walls shall have a thermal resistance of not less than 0.83 m^2 K/W. In order that the resistance of the box wall shall be uniform over the entire box area, a construction without internal ribs shall be used, for example, a glued balsa wood or a sandwich construction with aged urethane foam core. The edge in contact with the panel shall, if necessary, be narrowed on the outside only, to hold a gasket not more than 13 mm wide. If necessary, a wood nosepiece can be used to carry the gasket. The metering area of the panel shall be taken as the area included between the center lines of the gaskets. All surfaces that can exchange radiation with the specimen must have a total hemispherical emittance greater than 0.8.

6.2.3 *Heat Supply and Air Circulation*—Fig. 2 shows a possible arrangement of equipment in the metering box to assure an even, gentle movement of air over the metering

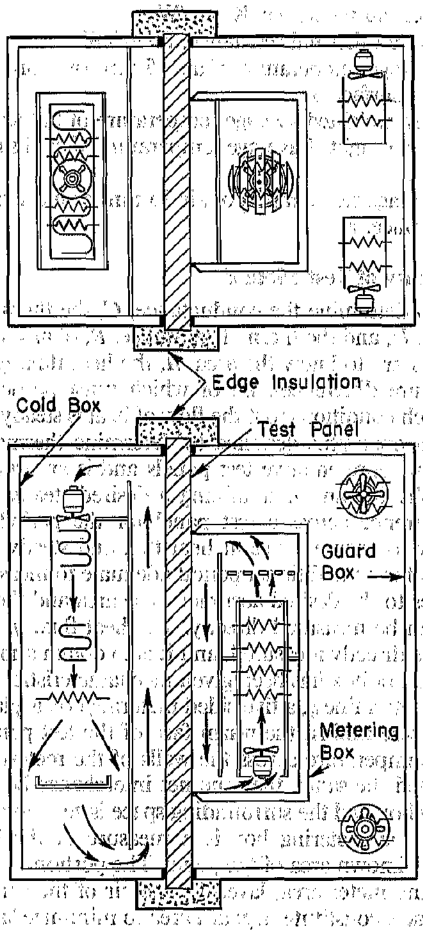

FIG. 2 Arrangement of Equipment During the Test

area of the panel. The electric heaters are mounted in a housing with walls of resistance not less than 0.83 m^2 K/W, and with a low emittance outside surfacing to minimize radiation heat transfer to the metering box walls. In this arrangement air is continuously circulated by a small fan upward through the cylindrical housing and downward between the baffle and the panel in accordance with the motion that would result from natural convection forces. A slat-type baffle is placed some distance above the outlet of the cylindrical housing to prevent impingement of a jet of heated air against the top inner surface of the metering box. For large meter boxes the cylindrical housing may cause concentrations of air flow. To direct the air properly across the specimen, other fan arrangements may be preferable. A curved vane is mounted at the top of the baffle to smooth the entrance of air into the baffle space. In a hot box apparatus used for testing panels in a vertical position only, the moderate circulation of air resulting from natural convection may be sufficient without the use of a fan. The change in temperature of the air as it moves along the surface of the panel will, in general, be greater with natural circulation than with a fan. If a fan is used, its motor should be within the metering box, its electrical input should be as small as feasible, and the input should be carefully measured. If it is necessary to locate the motor outside the metering box, the heat equivalent of the shaft power must be accurately measured, and air leakage into or out of the metering box around the shaft must be zero.

6.2.4 *Temperature Control*—To obtain reliable test results, accurate temperature control equipment must be utilized. Temperature controllers must be capable of controlling temperature within 0.25 K during the test period. The heaters should be the open-wire type of minimal heat capacity and lag.

6.2.5 *Gaskets*—The contact edges of the metering box should ensure, by a gasket or other means, a tight air seal against the surface of the test panel. For some panels special provisions may be necessary. The metering box should be pressed tightly against the panel by suitable means. Some gasket materials age with time and service. Periodic inspection of gaskets is recommended in order to confirm their ability to provide a tight seal under test conditions.

6.2.6 *Heat Flux Transducer*—To equip the metering box walls to serve as a heat flux transducer, a means of detecting the temperature difference across the metering box walls or the heat flux through the metering box walls shall be provided. One method found satisfactory for this purpose is to apply a number of differential thermocouples connected in a series to the inside and outside surfaces of the metering box walls to form a thermopile. Precautions must be taken when determining the number of differential thermocouples. Based on a survey of guarded hot box operators, the number of differential thermocouple pairs located on metering box walls shall be five pairs per square metre of specimen metered area located on the metering box sides. At no time shall there be less than 1 pair of differential thermocouples on each of the five sides of the metering box **(1)**.[5] Precautions must also be taken when determining locations of the differential thermocouples, as temperature gradients on the inside and outside of the metering box walls are likely to exist and have been found to be a function of metering and guard box air velocities and temperature. The junctions and the thermocouple wires for at least a 100-mm distance from the junctions shall be flush with, and in thermal contact with, the surface of the wall. The output of the thermocouple pairs shall be averaged.

6.2.7 *Thermopile emf and Heat Flow Relationship*—The relationship between thermopile emf and heat flow through the metering box walls shall be determined. This relationship shall be determined for each set of metering box conditions (temperature and air velocity). A suggested method of accomplishing this objective is outlined in Appendix X1.

6.3 *Guard Box:*

6.3.1 *Size*—It is recommended that the guard box be large enough so that there is a clear distance between its inner wall and the nearest surface of the metering box of not less than the thickness of the thickest panel to be tested, but in no case less than 150 mm.

6.3.2 *Thermal Conductance*—To assure that there shall be a temperature difference of no more than a few degrees between the guard box air and its inner surfaces, the walls shall have a thermal conductance not greater than 0.6 $W/(m^2 \cdot K)$. A low conductance is also desirable for operating reasons, to assure that the heat flow into or out of the guard box from outside will be only a small fraction of the heat flow through the guard area of the test panel.

6.3.3 *Heat Supply and Air Circulation*—One or more reflective-surfaced cylindrical heater units with a fan may be used to supply heat to the guard box air and also to circulate the air to avoid stratification. The fan air intake of at least one such heater unit should be located at the lowest point in the guard box, to prevent pooling of cool air at the bottom. The air discharged from the heater cylinder shall not impinge directly against either the metering box or the test panel.

6.3.4 *Temperature Control*—The guard box air temperature and heat input can be controlled by a differential thermopile such as that used on the metering box for a heat flow meter, or by a sensitive bridge circuit with opposed temperature-sensitive arms located in the guard and metering boxes. To avoid hunting due to the small periodic temperature variations of the metering box air, as its thermostat functions, it is desirable to put the temperature-sensitive element of the differential control in the metering box in good thermal contact with the inside surfaces of the metering box. The temperature-sensitive element in the guard box should be placed to avoid being directly in the air stream of the heater units and should be of minimum thermal lag. The control equipment used to maintain guard box temperatures must be capable of controlling to within 0.25 K.

6.4 *Cold Box:*

6.4.1 *Size*—The size of the cold box is governed by the size of the test panel or by the arrangement of boxes used, as illustrated in Fig. 1.

6.4.2 *Insulation*—The cold box should be heavily insulated to reduce the required capacity of the refrigerating equipment, and the exterior of the cold box should be provided with a good vapor barrier to prevent ingress of

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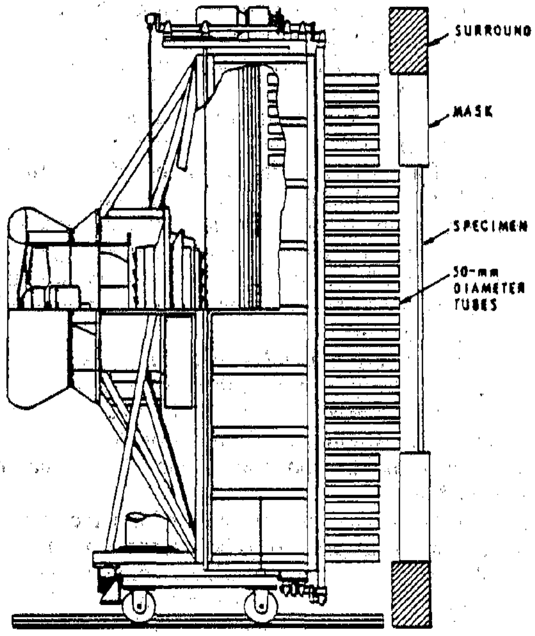
[5] The boldface numbers in parentheses refer to the list of references at the end of this test method.

vapor and heavy frost accumulations on the cooling coils.

6.4.3 *Temperature Control*—The cold box may be cooled in any manner that is capable of the close control of air temperature necessary during a test. An arrangement of equipment similar to that in the metering box may be used with a fan to force air downward through the enclosed refrigerating coils and upward through the space between a baffle and the test panel as indicated in Fig. 2. It has been found satisfactory with an arrangement of this sort to operate a unit refrigeration system continuously, with the evaporation temperature of the coil held constant by an automatic back-pressure regulating valve, and refrigerant supplied to the coil through an automatic expansion valve. An alternative method is to use an exterior located refrigeration system and insulated ducts to supply chilled air to the cold box. Liquid nitrogen in connection with a solenoid valve regulating its flow may also be used. For fine control of the cold box, installation of open wire electrical heaters in the blower duct or other fast moving part of the air circulation system and controlling these heaters by a sensor located in the discharge of the air circulation system is recommended. The use of desiccants to remove excessive moisture in the recirculating cold air may be useful. Temperature controllers for steady-state tests must be capable of controlling temperatures within ±0.25 K.

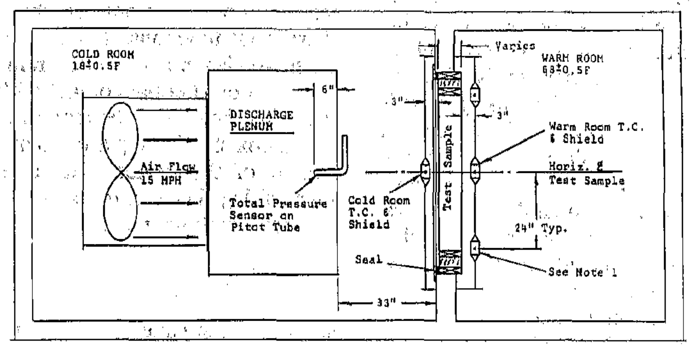
6.4.4 *Air Circulation*—High air velocities are permissible when their effect upon heat flow is to be determined. This may be accomplished by directing the airflow either parallel or perpendicular to the specimen cold surface. One method of obtaining parallel uniform velocity is to force air through a space between the specimen and a parallel baffle whose spacing may be adjustable to aid in changing the air curtain velocity. Parallel velocities, as provided in this test method, aid in obtaining uniform specimen surface temperatures and simulate the effect of cross winds. Velocities commonly used to simulate cross wind conditions are 3.35 m/s for summer conditions and 6.70 m/s for winter conditions. Perpendicular velocities, simulating direct wind impingment require moving larger amounts of air than most parallel situations, with corresponding larger power requirements. Also, the baffle should be placed further from the specimen surface and should have a porous section (a screen or honeycomb flow straightener) that directs the wind at the specimen surface (see Fig. 3 and 4). Velocities commonly used to simulate wind conditions are 3.35 m/s for summer conditions and 6.70 m/s for winter conditions. Air leakage through the specimen should be eliminated by sealing all cracks and joints with tape, caulk, or foam strips.

6.4.4.1 After construction of the air circulation system a velocity scan across the air curtain is required to verify that a uniform air curtain is formed. The apparatus should provide a means for determining air velocity past the specimen surface. One method is to locate velocity sensors directly in the air curtain.



NOTE—One inch is equal to 25.4 mm.

FIG. 3 DBR Wind Machine



NOTE 1—Thermocouples and shields on the warm side are movable to maintain 3 in. spacing to test sample.
NOTE 2—Overall chamber length may vary.
NOTE 3—One inch is equal to 25.4 mm.
NOTE 4—Thirty-two degrees farenheit is equal to 0°C.

FIG. 4 Thermal Chamber Diagram

6.5 *Temperature-Measuring Equipment:*

6.5.1 *Surface Temperatures*—Thermocouples of wire not larger in size than 0.25 mm (No. 30 AWG gage), and which meet or are calibrated to the special limits of error specified in Tables E 230, are recommended for measuring surface temperatures in the apparatus (larger thermocouples can be used if it can be shown that there is no difference in bias); for this purpose the thermocouple junction and the adjoining lead wires for a distance of at least 100 mm should be taped, or preferably cemented, tightly to the surface. The emittance of the surfacing material tape or cement should be close to the emittance of the surface.

6.5.1.1 If the specimen (and therefore its thermal resistance) is uniform, or nearly so, over the area and thus the surface temperatures vary only slightly at lower air velocities, a minimum number of thermocouples spaced uniformly and symmetrically over the surface is sufficient. This minimum number depends on the specimen size. Experience has shown that the required minimum number of thermocouples, N, can be determined from the relation that:

$$N = A/(0.07 + 0.08\sqrt{A}) \tag{1}$$

where A is the metering area in m^2. If the number of thermocouples used is within 10 % of the number determined by this relation, then the requirements of this section are judged to be met.

6.5.1.2 If the specimen is of nonuniform construction, the number of thermocouples specified in 6.5.1.1 may still be sufficient. In this case the thermocouples shall be judiciously located to represent each of the construction elements. Such representation shall be distributed approximately uniformly and symmetrically over the specimen surface.

6.5.1.3 If the surface temperatures are expected to be greatly nonuniform, additional thermocouples must be used to sample adequately the different temperature areas so that reliable weighted mean temperatures may be obtained.

6.5.1.4 With some nonhomogeneous walls, such as concrete, it may be advisable to use copper shim stock under the thermocouples to average the temperature. Large aggregates in the concrete can create biased temperature readings.

6.5.1.5 At least two surface thermocouples shall be placed on the guard area of the specimen at suitable locations to indicate the effectiveness of the guard area.

6.5.1.6 Surface temperatures on the cold side of the test panel shall be measured by surface thermocouples placed directly opposite those on the warm side.

6.5.2 Air temperatures may be measured by thermocouples, temperature sensitive resistance wires, or other sensors. Air thermocouples shall be made of wire not larger than 0.51 mm (No. 24 AWG).

6.5.2.1 If thermocouples or other point sensors are used, they shall be located in the metering box area in the same quantity and spacing as that specified for surface thermocouples in 6.5.1.1. The thermocouple shall be located midway between the face of the panel and the baffle, if one is used, but in no case less than 75 mm from the face of the panel. The junctions of the thermocouples shall have bright metallic surfaces and shall be as small as possible to minimize radiation effects. Another method is to shield the thermocouple junction. The thermocouples may be placed directly opposite the surface thermocouples; in any case they should be located as uniformly as possible over the metering area.

6.5.2.2 Thermocouples shall also be placed in the guard space at suitable locations, to indicate the degree of uniformity of guard space air temperatures; preferably, one should be placed opposite each guard area surface thermocouple, but not less than 75 mm from the panel.

6.5.2.3 Air temperatures on the cold side of the panel shall be measured by one thermocouple placed directly opposite each of the warm side air temperature thermocouples and located in a plane parallel to the specimens surface and spaced far enough away that they are unaffected by temperature gradients in the boundary layer. The thermocouples shall be located midway between the face of the panel and the baffle, if one is used. For low velocities, a minimum spacing of 75 mm from the specimen surface is required. At higher velocities the required minimum spacing is less but in no case less than 20 mm. No thermocouples need be placed in the cold space opposite guard space thermocouples remote from the panel surface.

6.5.2.4 If air temperatures are to be measured by means of resistance wire grids, the wire shall be distributed uniformly to indicate approximately the average temperature of the air on both sides of the panel at a plane midway between the baffle and the panel but in no case less than 75 mm from the panel.

6.5.2.5 It is recommended that the surface temperature of the baffles on the hot and cold sides be measured by placing thermocouples on all surfaces the specimen can see.

NOTE 4—This is not a requirement of this test method but is highly recommended. There are several reasons for the recommendation: (*1*) this indicates any difference between the baffle surface and air temperatures; (*2*) it will allow corrections to be made to the radiation component of the surface conductances due to differences in these temperatures; and (*3*) it is necessary to do this for specimens such as glass which have a high-thermal conductance.

6.6 *Instruments:*

6.6.1 All thermocouples or other temperature sensors for observing surface and air temperatures shall have their leads brought out individually to suitable measuring instruments capable of indicating temperatures to within ±0.05 K.

6.6.2 Total average power (or integrated energy over a specified time period) for all energy to the meter box shall be accurate to within ±0.5 % of reading under conditions of use. Power measuring instruments must be compatible with the power supplied whether ac, dc, on-off proportioning, etc. Voltage stabilized power supplies are strongly recommended.

6.6.3 Velocities of air over both surfaces of the panel should be measured with suitable instruments or be calculated from a heat balance between the rate of loss or gain of heat as it moves through the baffle space, as indicated by its temperature change, and the rate of heat flow through the test panel, average values of which can be determined from the test data. It should be recognized that radiant transfer between the baffle and the specimen can affect the calculation if the radiation is significant. For this reason direct velocity measurement is desirable.

NOTE 5—It is recommended that a central control location be established, that automatic scanning and recording equipment for unattended operation be used, and that data be computer processed.

6.7 *Verification*—When a new or modified apparatus is constructed, verification tests shall be conducted on panels made from materials of known conductance that does not exceed 1.5 W/($m^2 \cdot K$) as determined in Test Methods C 177 or Test Method C 518[6]. Any differences in results should be carefully analyzed and corrective measures taken. Further periodic checks are recommended.

7. Sampling and Test Specimens

7.1 Specimens shall be representative of the construction to be investigated but may be modified if necessary for test purposes as mentioned in 5.2 and 5.3. It must be recognized that modifications to the construction may result in conditions that do not represent true field conditions. In many cases conduction and convection paths have considerable effect on the performance of the specimen and must be left intact. Other considerations are:

7.1.1 *Sensors*—Install temperature sensors as directed in 6.5. When desired, temperature and other sensors may be installed throughout the interior of the specimen for special investigations.

7.1.2 *Conditioning*—The usual pre-test conditioning is in ambient air long enough to come to practical equilibrium. Assemblies that may have significant moisture content, which can influence test results, must be allowed to reach steady-state moisture conditions. Since the specimen size will probably preclude oven drying, concrete wall specimens may require 6 to 8 weeks of room temperature aging.

7.1.3 *Edge Insulation*—When a test panel is installed, its edges shall, if necessary, be insulated to prevent edge effect from overtaxing the guarding effect of the guard area of the panel. For this purpose, the edges of the panel may be protected against heat loss or gain by a thickness of insulation with an R of 1 or 1.25 K·m^2/W. It may be necessary to vapor-proof the insulation to prevent condensation of moisture in the edges of the panel, if a test arrangement similar to that shown in Fig. 1(*c*) is used. The edge of the specimen should be well sealed to prevent air infiltration between the guard and the cold box.

8. Procedures

8.1 Test conditions of temperature and orientation should be chosen to correspond as closely as possible to the circumstances of use of the construction to be tested. This test method is primarily designed for the temperatures encountered in normal building use; however, it is recognized that the method may find application in testing conditions that are outside this normal range. It is recommended that a minimum temperature differential of approximately 25 K be maintained for accurate measurement.

8.2 The required stabilization and test periods are as follows:

8.2.1 Impose steady-state conditions for at least 4 h prior to final data collection. This condition is satisfied when, over this 4-h period, the average surface temperature did not vary by more than ±0.06°C (±0.1°F) and the average power in the meter area did not vary by more than ±1 % and the data did not change unidirectionally. During this period, data shall be collected at intervals of 1 h or less.

8.2.2 After the conditions in 8.2.1 have been satisfied, continue the test period at least 8 h, but do not terminate the test until two or more successive 4-h periods produce results that do not differ by more than 1 %. During this period take data at intervals of 1 h or less. The average of the data for the two or more successive 4-h periods that agree within 1 % are used in calculating the final results. In testing panels that are heavily insulated, very massive, or both, it may be necessary to extend the duration of the test beyond the minimum period of two consecutive 4-h periods in order to be assured that conditions are steady, as it has been observed that continuing but small incremental changes can give a premature appearance of stability.

8.2.3 The calculation of a time constant, generated from apparatus measurements (Note 6) combined with an estimate of the thermal properties of the specimen, will help in estimating the time required for the test set-up to reach equilibrium. (2) It is also suggested that C and U values be calculated for the test specimen, utilizing known properties of the components. This will serve as general check of the measured results and avoid serious errors in measurement.

NOTE 6—The thermal mass of the apparatus may be the major factor contributing to the time constant of the system.

8.3 Data to be determined include:

8.3.1 The total net energy or average power through the specimen during a measurement interval. This includes all meter box heating and power to fans or blowers, and any corrections for meter box wall heat flow.

8.3.2 All air and surface temperatures specified in 6.5.1 and 6.5.2 (Note 7).

8.3.3 The effective dimensions of the metered area.

NOTE 7—In 6.5 the locations of thermocouples or temperature-measuring elements at various points are stipulated, for example, in the guard space and on the guard area of the test panel. The temperatures indicated by such thermocouples are of great value in evaluating the uniformity of temperatures prevailing in the guard space and on the test panel surfaces, but it is not feasible to stipulate generally the limits within which certain of these measured temperatures must agree. It must, therefore, be the responsibility of the test engineer to observe and weigh the significance of these temperatures to ascertain their effect upon the validity of a particular test measurement.

9. Calculation

9.1 Calculate the final test results by means of the following equations using the average data obtained in 8.2.2 for the two 4-h periods that agree within 1 %:

$$
\begin{aligned}
U &= Q/A(t_h - t_c) \\
C &= Q/A(t_1 - t_2) \\
R &= (t_1 - t_2)A/Q \\
R_u &= (t_h - t_c)A/Q = r_c + R + r_h \\
r_h &= (t_h - t_1)A/Q \\
r_c &= (t_2 - t_c)A/Q \\
h_h &= Q/A\,(t_h - t_1) \\
h_c &= Q/A\,(t_2 - t_c) \\
\lambda &= QL/A\,(t_1 - t_2)
\end{aligned}
$$

9.1.1 For a relatively uniform but nonhomogeneous specimen such as normal walls, floors, ceilings, etc., the properties that may be calculated are transmittance U, conductance C, resistance R, overall resistance R_u, surface resistances and surface conductances, h.

9.1.2 For uniform and homogeneous specimens all of the

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[6] Practice C 1045 must be used in conjunction with Test Methods C 177 and Test Method C 518.

properties listed in 9.1.1 may be calculated plus thermal conductivity λ.

9.1.3 For elements smaller than the metering area, the properties that apply to the element, according to the distinctions of 9.1.1 and 9.1.2 may be calculated if tests have been run that allow the element heat flow to be determined. Annex A1 presents considerations for these calculations.

10. Report

10.1 Report the following information:

10.1.1 Name, and any other identification or description of the test construction, including if necessary a blueprint showing important details, dimensions, and all modifications made to the construction, if any, and specimen orientation. Description of the test construction and a complete and detailed description of all materials. This includes the generic name of the material and its density. (For hygroscopic materials, such as some concrete materials and wood, the moisture content should also be given). If the thermal conductivities of these materials, at the test conditions, have been measured in a hot box facility (Test Method C 236 or Test Method C 976), a guarded hot plate (Test Method C 177) or a heat flow meter (Test Method C 518), these values should also be included.

NOTE 8—By generic description, the name of the material in addition to the brand name should be given (for example, preformed, cellular polystyrene Type VIII with a density of 22 kg/m^3; spruce-pine-fir with a moisture content of 12 % and a dry density of 486 kg/m^3).

10.1.2 Pertinent information in regard to preconditioning of the test panel.

10.1.3 Size and dimensions of the metering and guard areas of the test panel.

10.1.4 Average values during the test period of the temperatures and velocities of the air on both sides of the metering area of the panel, and of the temperature of the surfaces on both sides. (If significant, give the average values of the temperature of specific areas of the surface of the panel.)

10.1.5 Average rate of net heat input to the metering box.

10.1.6 Any thermal transmission properties calculated in 9.1 and the known precision of the equipment. Precision of the equipment should be checked using the propagation of errors theory.

NOTE 9—Discussions of this method can be found in many textbooks on engineering experimentation and statistical analysis (3).

10.1.7 Test duration and date.

10.2 All values shall be reported in both SI and inch-pound units unless specified otherwise by the client.

10.3 Where this test method is 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 the following provisions of Test Method C 236" (followed by a listing of specific deviations from this test method and any special test conditions that were applied).

11. Precision and Bias

11.1 *Background*—A round robin for guarded and calibrated hot boxes was conducted in accordance with Practice E 691. This round robin involved 21 different laboratories of which 16 had guarded hot boxes **(4)**. Data were reported for 100-mm (4-in.) thick homogeneous specimens of expanded polystyrene board (Specification C 578). Each laboratory received material from a special manufacturer's lot that was controlled to maintain a uniform density. Data reduction and analysis using Practice E 178 identified one of the 16 laboratories as a statistical outlier. Results from the other 15 laboratories showed that at a mean temperature (t) of 24°C (75°F), the average R value was determined to be 2.78 K·m^2/W (15.77 F ft^2 h/Btu). The regression equation for the data set was:

$$R = 3.146 - 0.016\,t\ (R \text{ in K}\cdot\text{m}^2/\text{W and } t \text{ in °C}) \quad (2a)$$

$$R = 17.867 - 0.028\,t\ (R \text{ in F ft}^2\text{h/Btu and } t \text{ in °F}) \quad (2b)$$

over the mean temperature range from 4°C to 43°C (40°F to 110°F). The mean specimen density ranged from 20.2 to 23.9 kg/m^3 (1.26 to 1.49 lbs/ft^3).

11.2 *Precision*—At a specimen thermal resistance of R = 2.78 K·m^2/W (15.76 F ft^{2}h/Btu) and on the basis of test error alone, the difference in absolute value of two test results obtained in different laboratories on the same specimen materials will be expected to exceed the reproducibility interval only 5 % of the time according to Table 2. For example, measurements from two different laboratories on the same specimen could differ by up to ±7.8 % at a mean temperature of 24°C (75°F) 95 % of the time.

11.3 *Bias*—Based on guarded hot plate data (Test Method C 177) from the National Institute of Standards and Technology—Center for Building Technology and supported by measurements from other laboratories, the true value for the round-robin specimen is a thermal resistance of 2.81 K·m^2/W (15.94 F ft^{2}h/Btu). The mean value measured by the guarded hot box differed by −1.07 %.

NOTE 10—Another test series was conducted on homogeneous common lot specimens in three guarded hot boxes at different laboratories. (5, 6) R-values of the specimens ranged from approximately 0.5 to 2.1 K·m^2/W (3 to 11.8 F ft^{2}h/Btu) at mean temperatures of 4, 24, and 43°C (40, 75, and 110°F). This series indicated that results with precision of ±5 % may be achieved.

NOTE 11—Both round robins used a homogeneous specimen, an ideal wall section. Actual wall sections will be nonhomogeneous. The precision and bias has not yet been determined for nonhomogeneous specimens. The above statements provide a bound.

12. Keywords

12.1 building assemblies; guarded hot box; test method; thermal performance; thermal resistance

TABLE 2 Precision for Test Method C 236

Mean Temperature, °C (°F)	Thermal Resistance, K·m^2/W (Fft^{2}h/Btu)	Reproducibility Interval, %	Change in R, K·m^2/W(Fft^{2}h/Btu)
4 (40)	2.95 (16.75)	±7.3	±0.22 (±1.23)
24 (75)	2.78 (15.77)	±7.8	±0.22 (±1.23)
43 (110)	2.60 (14.79)	±8.6	±0.22 (±1.27)

ANNEX

(Mandatory Information)

A1. USING THE GUARDED HOT BOX TO DETERMINE HEAT TRANSFER THROUGH A BUILDING ELEMENT SMALLER THAN THE METERING AREA

A1.1 General Considerations

A1.1.1 In this use, the building cement area (A_a) is located centrally in the metering area (A_b) demarcated by the hot box, and is surrounded by a masking partition which extends homogeneously beyond the area A_b into the guard area. The area of the mask (A_m) within the metering area equals ($A_b - A_a$). The total heat flow rate Q_b determined by the hot box measurement consists of two heat flow rates in parallel, in accordance with the equation

$$Q_b = Q_r + Q_m \tag{A1.1}$$

where Q_r is the total through the building element area A_∞ and Q_m is that through the mask area A_m.

A1.1.2 In conducting a test to ascertain Q_r for a particular building element, Q_b is determined by the hot box measurement, and Q_m is inferred from the results of calibration measurements. The calibration is made by means of hot box tests of the masking partition either before the aperture for the building element is cut out or with a blank of known thermal conductance installed in place of the building element. The error in Q_e is evidently equal to the difference of the algebraic errors in Q_b and Q_m. The fractional error is given by

$$\Delta Q_e/Q_e = (\Delta Q_h - \Delta Q_m)/(Q_b - Q_m)$$
$$= [(\Delta Q_h/Q_h) - (\Delta Q_m/Q_b)]/(1 - Q/Q_b) \tag{A1.2}$$

where (ΔQ_e) is the algebraic error in $Q\infty$ etc. An estimate of the fractional error (ΔQ_m)/Q_b is dependent upon the method which is used to calibrate the mask. If the calibration is made before the aperture for the building element is cut out then

$$\Delta Q_m/Q_h = (\Delta Q'_h/Q_h) \times (A_m/A_b) \tag{A1.3}$$

where ($\Delta Q'_h$) is the error in heat flow measured during the calibration test. If a blank of known thermal conductance is used to calibrate the mask then

$$\Delta Q_m/Q_h = (\Delta Q'_h - \Delta Q_{b1})/Q_b \tag{A1.4}$$

where (ΔQ_{b1}) is the algebraic error in determination of heat flow through the blank. Little can be said in general about the magnitudes of the algebraic fractional errors (ΔQ_h)/Q_b and (ΔQ_m)/Q_b, since these depend on the quality and management of the particular hot box apparatus and upon the accuracy of determination of heat flow through the blank, but is is evident that the systematic portion of the error (ΔQ_r)/Q_r is reduced as Q_m/Q_b is made small. Also, as Q_m is made small, the term ($\Delta Q_m/Q_h$) is presumably also made less significant. Thus, the fractional systematic error possible in the determination of Q_r is reduced by increasing either the area of the building element (if feasible), or the total thermal resistance of the mask.

A1.1.3 The need to infer the mask heat flow Q_m accurately requires that the mask be designed to act as a heat flow meter with an emf output and temperature difference of Δ_t proportional to the total heat flow through it. This consideration is the basis for the specific recommendations which follow.

NOTE A1.1—Additional error may arise due to the possible influences of the building element in causing two or three dimensional heat flow at the boundary with the mask and thus affecting the mask heat flow in regions adjacent to the element. Thus mask heat flow, determined under a given set of conditions with a calibration standard in place, may change when the building element is installed, even though the test conditions remain unchanged. The user of this procedure should be aware of such possible errors and should attempt to evaluate their magnitude in relation to the desired accuracy of the test.

A1.2 Recommendations

A1.2.1 It is recommended that the mask be made of a suitable uniform thickness of a homogeneous and stable material of low thermal conductivity having adequate strength to support the weight of the building elements to be tested. Suitable materials are faced high-density glass fiber or polystyrene boards laminated together as necessary. Stronger masks can be fabricated by sandwiching layers of insulation between layers of rigid materials such as plywood. Such masks, though nonhomogeneous, are uniform in the direction perpendicular to the direction of heat flow and are calibrated in the same manner as homogeneous masks. It may be necessary in some cases to incorporate framing in the mask to support heavy building elements such as heavy-duty metal frame windows or masonry sections. Such nonuniform masks are necessarily calibrated using blanks of known thermal conductance. Framing members must be kept away from the juncture with the building element and with the boundary of the metering area so as not to contribute excessively to lateral heat transfer at these points. It is important that the mask be low in hygroscopicity to minimize changes in its thermal resistance with ambient humidity conditions, and that it be substantially impervious to air flow through it.

A1.2.2 Thermocouples for measuring the temperature difference across the mask should be permanently installed uniformly flush with or just under its surfaces. These may be connected in series-differential for determination of the mask temperature difference, or as individual thermocouples for exploring the temperature distributions on the faces of the mask. It is recommended that there be at least eight thermocouple junctions on each face of uniform masks: four at positions bisecting the four lines from the corners of the building element aperture to the corresponding corners of the metering area, and four at positions bisecting the sides of the rectangle having the first four thermocouples at its corners. A suitable thermocouple arrangement would have to be chosen for nonuniform masks that would provide representative average surface temperatures. This is particularly important when natural convection is used and air temperatures and film coefficients vary over the metering surface. If framing members are used, an area-weighted average of temperatures measured over the members and away from them is necessary. The mask, as a heat flow meter, should be calibrated and used in terms of the average temperature (or thermocouple emf) difference across it indicated by the permanently installed thermocouples.

A1.2.3 To protect the surfaces of the mask and the permanently installed thermocouples, and if necessary to render the surfaces impervious to air, a permanent coating or thin facing on each face of the mask is desirable. However, the coating or facing must be of low conductance laterally so that it does not contribute excessively to lateral heat transfer at the juncture with the building element or at the boundary of the metering area. The emittance of the mask surfaces should be uniform, and unchanged after calibration of the mask; in cases where the transmittance (rather than the conductance) of the building element is of particular interest, it is preferable that the emittance of the mask surfaces be large.

A1.2.4 In view of the desirability of high thermal resistance of the mask relative to that of the building element, the uniform thickness of the mask should in general not be less than that of the building elements to be tested, and may be greater than that of the thinner elements. Mask thickness greatly exceeding that of the building element is to be avoided if possible because of lateral heat flow in the mask due to its exposure at uncovered areas of its aperture. (In special instances, for example, a window designed to be set a few inches outward from the plane of the inner surface of a wall, a special calibration of the mask as a heat flow meter may be necessary using a blank of known thermal conductance in the precise position of the window at the juncture with the mask aperture.)

A1.2.5 The mask aperture in which the building element is installed for test should fit the element specimen snugly. Cracks between them should be minimal in width, and should be filled completely with a good fibrous insulation and caulked or otherwise sealed at the mask surfaces to prevent air leakage. It is desirable that the insulation used to fill cracks have approximately the same conductivity as the mask material; it would then be possible, if the cracks aggregate an area significant in relation to the mask area, to compensate roughly for the increased virtual mask area by increasing the mask heat flow indicated by its temperature drop in proportion to the increase of area.

A1.2.6 It is probable that many building elements to be tested are inhomogeneous or nonuniform in construction for structural reasons, and in consequence that the local thermal conductances differ considerably at different frontal areas of the element. The variations are inherent, and the result of the test is an average conductance or transmittance value for the total construction, provided that the conductance variations at edges do not seriously impair the validity of using the mask as an adequate heat flow meter. This is a matter which varies with the case, and therefore must rest on the judgment and discretion of those conducting the test measurement. A useful guiding principle is that nothing should be incorporated in, or omitted from, a building element specimen being tested that would make it not representative of the assembly that would be found in actual installation in service. For example, if a metal window ordinarily is installed with inset wood framing, the test specimen should include just so much of the wood framing as is properly chargeable to it.

A1.3 Calibration of the Mask as a Heat Flow Meter

A1.3.1 The calibration of the mask is made by means of hot box tests either before the aperture for the building elements is cut out or with a blank of known thermal conductance installed in place of the building element. The mask must be fully prepared with the permanent differential thermocouples installed and any final facings or coatings applied. Several tests are made, adequately covering the range of mask mean temperatures (and perhaps mask temperature drops and box air velocities) at which the mask will be operated in tests of building elements. In each test, under steady-state conditions, the metering box heat flow Q'_t and the corresponding mask temperature drop Δ_t, indicated by its permanently installed thermocouples, are determined. The net mask heat flow Q'_m corresponding to Δt is calculated as $Q'_b(A_m/A_b)$ when the calibration is made before the aperture is cut, where A_m and A_b are as defined earlier, and as $(Q'_t - Q_{b1})$ for the calibrated-blank method where Q_{b1} is the calculated heat flow through the blank. In the latter method of calibration, a suitable blank must first be prepared and calibrated.

APPENDIX

(Nonmandatory Information)

X1. THERMOPILE EMF AND HEAT FLOW RELATIONSHIP (7, 8)

X1.1 The procedure given in this Appendix outlines the steps suggested to obtain the relationship between heat flow and thermopile output. This method addresses the technique that will yield the heat flow relationship as a function of the thermopile output and a thermopile offset, if present.

X1.1.1 It is essential that the number of fans and power input in the metering, guard, and environmental boxes be held constant along with all temperatures throughout the calibration (and measurement) phase. By holding the fan number and input along with the surface temperatures constant, the operator assures a constant heat transfer film coefficient to the specimen throughout testing. The E_o value associated with negligible net heat flow across the meter box walls is then obtained from the relationship between Q_m and E. The equation that describes the total heat flow drawn schematically in Fig. 4 is:

$$Q_F + Q_H + Q_m = Q_s = \frac{A\Delta T}{R} \qquad \text{(X1.1)}$$

where:

Q_F = heat flow due to fan, W,
Q_H = heat flow due to heater, W,
Q_m = heat flow through the metering box walls, W,
Q_s = heat flow through specimen, W,
R = thermal resistance of specimen, $m^2 \cdot K/W$,
A = heat flow metered area, m^2,
ΔT = temperature difference across specimen, K,
E_o = thermopile emf when net heat flow through metering box walls is negligible, and
E = thermopile emf.

The goal is to make Q_m equal to zero. Q_m can be described by:

$$Q_m = fn(E) = m\,E + b \qquad \text{(X1.2)}$$

X1.1.2 To quantify m, at least two test runs must be performed with differing levels of E. E must be held constant within each test. The specimen surface to surface temperature difference for all tests must be constant and of the same value. Q_s can be approximated by assuming the design R. It is not necessary to know the R of the specimen. Plot Q_m calculated from Eq X1.1 versus E. The slope of the line is m.

The next step is to quantify b in Eq 2. Set the temperature difference across the specimen surface equal to zero ($Q_s = 0$). Substituting Eq X1.2 into Eq X1.1 and setting $Q_s = 0$ reduces Eq X1.1 to:

$$Q_F + Q_H = -(m\,E + b) \qquad \text{(X1.3)}$$

X1.1.3 Set E to a value such that the fan wattage is at operational conditions and the heater wattage is at the minimum value that maintains temperature control. This will assure that no heat is flowing anywhere except through the meter box walls. During this test, lateral heat flow must still be negligible. Using m that was determined, Eq X1.3 will yield b. The thermopile emf value that pertains to negligible net heat flow through the meter box walls E_o can then be calculated using Eq X1.2:

$$E_o = -(b/m) \qquad \text{(X1.4)}$$

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CERTIFICATE

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* * *

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Standard Specification for Lightweight Aggregates for Structural Concrete[1]

This standard is issued under the fixed designation C 330; 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 specification covers lightweight or sand aggregates intended for use in structural concrete in which prime considerations are reducing the density while maintaining the compressive strength of the concrete. Procedures covered in this specification are not intended for job control of concrete.

1.2 The values stated in SI units are to be regarded as the standard. The values shown in parentheses are for information purposes only.

1.2.1 With regard to other units of measure, the values stated in inch-pound units are to be regarded as standard.

1.3 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

NOTE 1—This specification is regarded as adequate to ensure satisfactory lightweight aggregates for most concrete. It is recognized that it may be either more or less restrictive than needed for some conditions and for special purposes, such as fire resistance, fill, and concrete constructions, the use of which is based on load tests rather than conventional design procedures.

2. Referenced Documents

2.1 *ASTM Standards:*

C 29/C 29M Test Method for Unit Weight and Voids in Aggregate[2]

C 33 Specification for Concrete Aggregates[2]

C 39 Test Method for Compressive Strength of Cylindrical Concrete Specimens[2]

C 40 Test Method for Organic Impurities in Fine Aggregates for Concrete[2]

C 114 Test Methods for Chemical Analysis of Hydraulic Cement[3]

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates[2]

C 142 Test Method for Clay Lumps and Friable Particles in Aggregates[2]

C 151 Test Method for Autoclave Expansion of Portland Cement[3]

C 157 Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete[2]

C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory[2]

C 496 Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens[2]

C 567 Test Method for Unit Weight of Structural Lightweight Concrete[2]

C 641 Test Method for Staining Materials in Lightweight Concrete Aggregates[2]

C 666 Test Method for Resistance of Concrete to Rapid Freezing and Thawing[2]

D 75 Practice for Sampling Aggregates[4]

3. General Characteristics

3.1 Two general types of lightweight aggregates are covered by this specification, as follows:

3.1.1 Aggregates prepared by expanding, pelletizing, or sintering products such as blast-furnace slag, clay, diatomite, fly ash, shale, or slate, and

3.1.2 Aggregates prepared by processing natural materials, such as pumice, scoria, or tuff.

3.2 The aggregates shall be composed predominately of lightweight-cellular and granular inorganic material.

4. Chemical Composition

4.1 Lightweight aggregates shall not contain excessive amounts of deleterious substances, as determined by the following limits:

4.1.1 *Organic Impurities (Test Method C 40)*—Lightweight aggregates that, upon being subjected to test for organic impurities, produce a color darker than the standard shall be rejected, unless it is demonstrated that the discoloration is due to small quantities of materials not harmful to the concrete.

4.1.2 *Staining (Test Method C 641)*—An aggregate producing a heavy or very heavy stain shall be rejected when the

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[1] This specification is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C 09.21 on Lightweight Aggregates.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.01.

[4] *Annual Book of ASTM Standards*, Vol 04.03.

material making up the stain is found upon chemical analysis to contain an iron content, expressed as Fe_2O_3, equal to or greater than 1.5 mg/200 g of sample.

4.1.3 *Loss on Ignition (Methods C 114)*—The loss on ignition of lightweight aggregates shall not exceed 5 %.

Note 2—Certain processed aggregates may be hydraulic in character, and may be partially hydrated during production; if so, the quality of the product is not usually reduced thereby. Therefore, consideration should be given to the type of material when evaluating the product in terms of ignition loss.

5. Physical Properties

5.1 Lightweight aggregate under test shall meet the following requirements:

5.1.1 *Clay Lumps*—The amount of clay lumps shall not exceed 2 % by dry weight.

5.1.2 *Grading*—The grading shall conform to the requirements shown in Table 1.

5.1.3 *Uniformity of Grading*—To ensure reasonable uniformity in the grading of successive shipments of lightweight aggregate, fineness modulus shall be determined on samples taken from shipments at intervals stipulated by the purchaser. If the fineness modulus of the aggregate in any shipment differs by more than 7 % from that of the sample submitted for acceptance tests, the aggregate in the shipment shall be rejected, unless the supplier demonstrates that it will produce concrete of the required characteristics.

5.1.4 *Bulk Density (Loose)*—The bulk density (loose) of the lightweight aggregates shall conform to the requirements shown in Table 2.

5.1.5 *Uniformity of Bulk Density (Loose)*—The reported bulk density (loose) of lightweight aggregate shipments, sampled and tested, shall not differ by more than 10 % from that of the sample submitted for acceptance tests, but the dry loose bulk density shall not exceed the limits in Table 2.

TABLE 2 Bulk Density (Loose) Requirements of Lightweight Aggregates for Structural Concrete

Size Designation	Maximum Dry Loose Bulk Density kg/m^3 (lb/ft^3)
Fine aggregate	1120 (70)
Coarse aggregate	880 (55)
Combined fine and coarse aggregate	1040 (65)

5.2 Concrete specimens containing lightweight aggregate under test shall meet the following requirements:

5.2.1 *Compressive Strength (Test Method C 39), Density (Test Method C 567), and Splitting Tensile Strength (Test Method C 496)*—Compressive strength and density shall be an average of three specimens and the splitting tensile strength shall be the average of eight specimens. It shall be possible to produce structural concrete using the lightweight aggregates under test, so that from the same batch of concrete one or more of the compressive strength requirements and splitting tensile strength requirements in the following table will be satisfied without exceeding the corresponding maximum unit weight values.

Average Air Dry 28-day Density max, kg/m^3 (lb/ft^3)	Average 28-day Splitting Tensile Strength, min, (MPa) psi	Average 28-day Compressive Strength, min, (MPa) psi
	All Lightweight Aggregate	
1760 (110)	2.2 (320)	28 (4000)
1680 (105)	2.1 (300)	21 (3000)
1600 (100)	2.0 (290)	17 (2500)
	Sand/Lightweight Aggregate	
1840 (115)	2.3 (330)	28 (4000)
1760 (110)	2.1 (310)	21 (3000)
1680 (105)	2.1 (300)	17 (2500)

Note 3—Intermediate values for strength and corresponding density values shall be established by interpolation. Materials that do not meet the minimum average splitting tensile strength requirement may be used provided the design is modified to compensate for the lower value.

5.2.2 *Natural Sand*— Natural sand, when used to replace part, or all, of the lightweight-aggregate fines shall comply with the applicable requirements of Specification C 330. The test report shall record the proportion of all ingredients and the characteristics of the natural sand to ensure compliance with these minimum requirements.

5.2.3 *Drying Shrinkage*— The drying shrinkage of concrete specimens prepared and tested as described in the method for preparation of samples for shrinkage of concrete shall not exceed 0.07 %.

5.2.4 *Popouts*—Concrete specimens prepared as described in the method for preparation of sample for shrinkage of concrete and tested in accordance with Test Method C 151 shall show no surface popouts.

5.2.5 *Resistance to Freezing and Thawing*—When required, the aggregate supplier shall demonstrate by test or proven field performance that the lightweight aggregate when used in

TABLE 1 Grading Requirements for Lightweight Aggregates for Structural Concrete

Size Designation	Percentages (Mass) Passing Sieves Having Square Openings								
	25.0 mm (1 in.)	19.0 mm (¾) in.)	12.5 mm (½ in.)	9.5 mm (⅜ in.)	4.75 mm (No. 4)	2.36 mm (No. 8)	1.18 mm (No. 16)	300 μm (No. 50)	150 μm (No. 100)
Fine aggregate:									
4.75 mm to 0	...	...	...	100	85–100	...	40–80	10–35	5–25
Coarse aggregate:									
25.0 m to 4.75 mm	95–100	...	25–60	...	0–10	...	...	...	...
19.0 mm to 4.75 mm	100	90–100	...	10–50	0–15	...	...	...	...
12.5 mm to 4.75 mm	...	100	90–100	40–80	0–20	0–10	...	...	...
9.5 mm to 2.36 mm	...	...	100	80–100	5–40	0–20	0–10	...	...
Combined fine and coarse aggregate:									
12.5 mm to 0	...	100	95–100	...	50–80	...	...	5–20	2–15
9.5 mm to 0	...	...	100	90–100	65–90	35–65	...	10–25	5–15

concrete, had the necessary resistance to freezing and thawing to perform satisfactorily in its intended use.

6. Sampling

6.1 Sample lightweight aggregates in accordance with Practice D 75.

7. Number of Tests

7.1 *Tests on Aggregate*—One representative sample is required for each test for organic impurities, staining, loss on ignition, grading, unit weight, and clay lumps.

7.2 *Tests on Concrete*—At least three specimens are required for each of the following tests of concrete: compressive strength, shrinkage, density, resistance to freezing and thawing, and presence of popout materials. At least eight concrete specimens are required for splitting tensile strength tests.

8. Test Methods

8.1 *Compressive Strength (Test Method C 39)*—Make test specimens in accordance with Practice C 192. Cure specimens in accordance with Practice C 192 until the time of test, or follow the curing procedures for the air dry density (Test Method C 567). When the latter procedure is used, remove the specimens from the moist curing at the age of 7 days and store at 23 ± 2°C (73.5 ± 3.5°F) with a relative humidity of 50 ± 5 % until the time of test.

8.2 *Splitting Tensile Strength*—Make 152 by 305 mm (6 by 12 in.) cylindrical test specimens in accordance with Practice C 192, cure, and test in accordance with Test Method C 496.

8.3 *Density of Concrete (Test Method C 567)*—Follow the procedures in Test Method C 567.

8.4 *Shrinkage of Concrete (Test Method C 157)*—Follow the procedures of Test Method C 157 with the following exceptions:

8.4.1 Prepare the concrete mixture using 335 kg of cement/m^3 (564 lb/yd^3), admixture (if any), and with an air content of 6 ± 1 %. Adjust the water content so as to produce a slump of 50 to 100 mm (2 to 4 in.). Thoroughly consolidate the concrete in steel molds not smaller than 50 by 50 mm (2 by 2 in.) nor larger than 100 by 100 (4 by 4 in.) in cross section, and long enough to provide a 250 mm (10 in.) gage length. The surface of the concrete shall be steel troweled.

8.4.2 *Curing*—To prevent evaporation of water from the unhardened concrete, cover the specimen with a nonabsorptive, nonreactive plate or sheet of tough, durable, impervious plastic or wet burlap. When wet burlap is used for covering, the burlap must be kept wet until the specimens are removed from the molds (see Note 4). Remove specimens from the molds not less than 20 nor more than 48 h after casting and store in a moist room maintained at 23 ± 2°C (73.5 ± 3.5°F) with a relative humidity of not less than 95 %. At the age of 7 days, remove the specimens from the moist room, measure for length, and store in a curing cabinet maintained at 37.8 ± 1.1°C (100 ± 2°F) with a relative humidity of 32 ± 2 %.

NOTE 4—Placing a sheet of plastic over the burlap will facilitate keeping it wet.

NOTE 5—The air immediately above a saturated solution of magnesium chloride (MgCl$_2$) at 37.8°C (100°F) is approximately 32 % relative humidity.

8.4.3 *Report*—After storage in the cabinet for 28 days, determine the change in length of each specimen to the nearest 0.01 % of the effective gage length. Report the change in length as the drying shrinkage of the specimen; report the average drying shrinkage of the specimens as the drying shrinkage of the concrete.

8.5 *Test for Popout Materials*—Prepare concrete specimens for the test for popout materials as described in method for preparation of samples for shrinkage of concrete. Cure and autoclave the specimens in accordance with Test Method C 151. Visually inspect the autoclaved specimens for the number of popouts that have developed on the surface. Report the average number of popouts per specimen.

8.6 *Test for Freezing and Thawing*— Make freezing and thawing tests of concrete, when required, in accordance with Test Method C 666, with the following modification to the section on Test Specimens and on Procedure. Unless otherwise specified, remove the lightweight aggregate concrete specimens from moist curing at an age of 14 days and allow to air dry for another 14 days exposed to a relative humidity of 50 ± 5 % and a temperature of 23 ± 2°C (73.5 ± 3.5°F). Then submerge the specimens in water for 24 hours, prior to the freezing and thawing test.

8.7 *Grading (Method C 136)*—Follow the procedures of Method C 136, except that the mass of the test sample for fine aggregate shall be in accordance with Table 3. The test sample for coarse aggregate shall consist of 2830 cm^3 (0.1 ft^3) or more of the material used for the determination of bulk density. Mechanical sieving of aggregate shall be for 5 minutes..

8.8 *Bulk Density (Loose) (Test Method C 29)*—The aggregate shall be tested in an oven dry condition utilizing the shoveling procedure.

8.9 *Clay Lumps and Friable Particles in Aggregates*—Test Method C 142.

9. Rejection

9.1 Material that fails to conform to the requirements of this specification shall be subject to rejection. Rejection shall be reported to the producer or supplier promptly and in writing.

10. Certification

10.1 When specified in the purchase order or contract, a producer's or supplier's certification shall be furnished to the purchaser that the material was manufactured, sampled, and tested in accordance with this specification and has been found to meet the requirements. When specified in the purchase order

TABLE 3 Mass of Sieve Test Sample for Fine Lightweight Aggregates

Range of Nominal Bulk Density (Loose) of Aggregate		Weight of Test Sample, g
kg/m^3	lb/ft^3	
80–240	5–15	50
240–400	15–25	100
400–560	25–35	150
560–720	35–45	200
720–880	45–55	250
880– 1040	55–65	300
1040–1120	65–70	350

or contract, a report of the test results shall be furnished.

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Designation: C 5 – 79 (Reapproved 1997)

Standard Specification for Quicklime for Structural Purposes[1]

This standard is issued under the fixed designation C 5; 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 specification covers all classes of quicklime such as crushed lime, granular lime, ground lime, lump lime, pebble lime, and pulverized lime, used for structural purposes.

2. Referenced Documents

2.1 *ASTM Standards:*

C 25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime[2]

C 50 Practice for Sampling, Inspection, Packing, and Marking of Lime and Limestone Products[2]

C 51 Terminology Relating to Lime and Limestone (As Used by the Industry)[2]

C 110 Test Methods for Physical Testing of Quicklime, Hydrated Lime, and Limestone[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

3. Terminology

3.1 *Definitions*—Unless otherwise specified, for definitions of terms used in this standard, refer to Terminology C 51.

4. Chemical Composition

4.1 The quicklime shall conform to the following requirements as to chemical composition, calculated on a nonvolatile basis:

	Calcium Lime	Magnesium Lime
Calcium oxide, min, %	75	
Magnesium oxide, min, %		20
Calcium and magnesium oxide, min, %	95	95
Silica, alumina, and oxide of iron, max, %	5	5
Carbon dioxide, max, %:		
If sample is taken at place of manufacture	3	3
If sample is taken at any other place	10	10

5. Residue

5.1 The quicklime shall contain no more than 15 weight % of residue.

6. General Requirements

6.1 Quicklime shall be slaked and aged in accordance with the printed directions of the manufacturer. The resulting lime putty shall be stored until cool.

6.2 Lime putty prepared in accordance with X1.4.2 and adjusted to standard consistency in accordance with Test Methods C 110, shall show no pops or pits when tested in accordance with Test Methods C 110.

6.3 Lime putty prepared as above shall have a plasticity figure of not less than 200.

7. Sampling, Inspection, etc.

7.1 The sampling, inspection, rejection, retesting, packing, and marking shall be conducted in accordance with Methods C 50.

8. Test Methods

8.1 Conformance to chemical requirements shall be determined in accordance with Test Methods C 25.

8.2 Conformance to plasticity and residue requirements shall be determined in accordance with Test Methods C 110.

9. Keywords

9.1 building (structural); calcium oxide; dolomitic lime; high calcium lime; lime putty; magnesium oxide; plasticity; quicklime; residue; slaking

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[1] This specification is under the jurisdiction of ASTM Committee C-7 on Lime and is the direct responsibility of Subcommittee C07.02 on Structural Lime.

Current edition approved Nov. 30, 1979. Published January 1980. Originally published as C 5 – 13 T. Last previous edition C 5 – 59 (1974).

[2] *Annual Book of ASTM Standards*, Vol 04.01.

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

APPENDIX

(Nonmandatory Information)

X1. SLAKING AND PREPARATION OF LIME PUTTY

X1.1 *Introduction*

X1.1.1 Quicklime can never be used as such for structural purposes; it must always be slaked first. Since the method of slaking is an important factor in determining the quality of the finished product, the following directions for the preparation of lime putty are given, not as a part of the specification, but as information for the further protection of the purchaser.

X1.1.2 Different kinds of lime vary considerably in the way in which they behave with water. A little supervision over the operation of slaking will amply pay for itself by ensuring the production of the greatest possible quantity and the best possible quality of putty. To find out how to slake a new lot of lime, it is safest to try a little of it and see how it works. Since different lots of the same brand of lime vary somewhat, and since the weather conditions at the time have a decided influence, it is wise to try a sample from each lot used, whether familiar with the brand or not.

X1.2 Classification of Limes

X1.2.1 In a bucket, put two or three lumps of lime about the size of one's fist, or, in the case of granular lime, an equivalent amount. Add sufficient water to just barely cover the lime, and note how long it takes for slaking to begin. Slaking has begun when pieces split off from the lumps or when the lumps crumble. Water of the same temperature should be used for test and field practice.

X1.2.2 If slaking begins in less than 5 min, the lime is quick slaking; from 5 to 30 min, medium slaking; over 30 min, slow slaking.

X1.3 Directions for Slaking

X1.3.1 Slake quicklime in accordance with the printed directions of the manufacturer. When such directions are not provided, proceed as follows:

X1.3.2 For quick-slaking lime, always add the lime to the water, not the water to the lime. Have sufficient water at first to cover all the lime completely. Have a plentiful supply of water available for immediate use—a hose throwing a good stream, if possible. Watch the lime constantly. At the slightest appearance of escaping steam, hoe thoroughly and quickly, and add enough water to stop the steaming. Do not be afraid of using too much water with this kind of lime.

X1.3.3 For medium-slaking lime, add the water to the lime. Add enough water so that the lime is about half submerged. Hoe occasionally if steam starts to escape. Add a little water now and then if necessary to prevent the putty from becoming dry and crumbly. Be careful not to add any more water than required, and not too much at a time.

X1.3.4 For slow-slaking lime, add enough water to the lime to moisten it thoroughly. Let it stand until the reaction has started. Cautiously add more water, a little at a time, taking care that the mass is not cooled by the fresh water. Do not hoe until the slaking is practically complete. If the weather is very cold, it is preferable to use hot water, but if this is not available, the mortar box may be covered in some way to retain the heat.

X1.4 Preparation of Putty for Use

X1.4.1 After slaking, prepare putty for use as follows:

X1.4.2 *White Coat*—After slaking and aging finishing quicklime in accordance with the printed directions of the manufacturer, store the putty until cool. If no printed directions are provided by the manufacturer, prepare the putty for use as follows: After the action has ceased, run off the putty through a No. 10 (2.00-mm) sieve conforming to Specification E 11, and store for a minimum of 2 weeks.

X1.4.3 *Base Coats*—After the action has ceased, run off the putty through a No. 8 (2.36-mm) sieve conforming to Specification E 11. Add sand up to equal parts by weight, all of the hair or other fibers required, and store for a minimum of 2 weeks.

X1.4.4 *Masons' Mortar*—After the action has ceased, add part or all of the sand required, and store for a minimum of 24 h.

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WASHINGTON, D.C.

Designation: C 509 – 84

Standard Specification for
CELLULAR ELASTOMERIC PREFORMED GASKET AND SEALING MATERIAL[1]

This standard is issued under the fixed designation C 509; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification applies to those cellular elastomeric compounds of a firm grade that are manufactured in preformed shapes for use as gaskets and for use as sealing materials, in the form of compression seals or gaskets, or both, for glazing purposes.

NOTE 1—For softer cellular elastomeric materials used in secondary sealing applications, refer to Specification D 1056.

1.2 The following precautionary caveat pertains only to the test method portion, Section 11, of this specification: *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 *ASTM Standards:*

D 395 Test Methods for Rubber Property—Compression Set[2]

D 412 Test Methods for Rubber Properties in Tension[2,3]

D 746 Test Method for Brittleness Temperature of Plastics and Elastometers by Impact[3]

D 865 Test Method for Rubber Deterioration by Heating in Air (Test Tube Enclosure)

D 925 Test Methods for Rubber Property—Staining of Surfaces (Contact, Migration, and Diffusion)[2]

D 1056 Specification for Flexible Cellular Materials—Sponge or Expanded Rubber[3]

D 1149 Test Method for Rubber Deterioration—Surface Ozone Cracking in a Chamber (Flat Specimens)[2]

3. Terminology

3.1 *Definition:*

3.1.1 *cellular elastomer*—a cured elastomeric material containing cells or small voids.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *compression seal*—a type of joint seal in which weathertightness is maintained by the exertion of compressive pressure on the gasket or sealing material.

3.2.2 *gasket glazing*—a method of setting glass or panels in prepared openings, using a preformed gasket to obtain a weathertight seal.

3.2.3 *preformed gasket*—an elastomeric compound molded in the form of a continuous strip, channel, or other shape, for use in filling joints and providing weathertight seals in glazing or between building components.

3.2.4 *sealing material*—any material intended for use in providing weathertight seals in building applications.

4. Significance and Use

4.1 *Flame Propagation:*

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[1] This specification is under the jurisdiction of ASTM Committee C-24 on Building Seals and Sealants and is the direct responsibility of Subcommittee C24.72 on Compression Seal Gaskets.

Current edition approved Oct. 26, 1984. Published February 1984. Originally published as C 509 – 63 T. Last previous edition C 509 – 79.

[2] *Annual Book of ASTM Standards*, Vol 09.01.

[3] *Annual Book of ASTM Standards*, Vol 09.02.

4.1.1 This specification has two options:

4.1.1.1 *Option I*—Flame propagation test is required.

4.1.1.2 *Option II*—Flame propagation test is not required.

4.1.2 In case no option is specified, Option I will apply.

4.1.3 *The sections of this specification applicable to flame propagation should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.*

4.2 This specification has two classifications as related to ozone resistance. These are Type I and Type II, with the latter having the greater resistance to ozone. The type should be specified when making reference to this specification but in the event that the type is not specified, Type I shall apply.

NOTE 2—Type II is included in this specification for use where greater ozone resistance is required.

5. Materials and Manufacture

5.1 Cellular elastomeric materials furnished to this specification shall be manufactured from natural rubber, synthetic rubber, rubber-like materials, or mixtures of these, with added compounding ingredients of such nature and quality that, with proper curing, the finished product will comply with this specification.

5.2 The cured compounds shall be suitable for use where resistance to sunlight, weathering, oxidation, and permanent deformation under load are of prime importance.

5.3 The manufacturing process shall be such to ensure a homogeneous cellular material free of defects that may affect serviceability.

5.4 Although under this specification the manufacturer is permitted to choose constituent materials, there is no implication that the several compounds are equivalent in all physical properties. Any special characteristics other than those required by this specification, which may be needed for specific applications, shall be specified by the purchaser, since such characteristics may influence the choice of base materials and other ingredients.

6. Physical Properties

6.1 The material shall conform to the requirements prescribed in Table 1.

7. Dimensional Tolerances

7.1 Permissible variations in dimensions of the various forms shall be ±6 % unless otherwise agreed upon between the purchaser and the supplier.

8. Workmanship, Finish, and Appearance

8.1 The cellular elastomeric materials shall be manufactured and processed in a careful and workmanlike manner in accordance with the best commercial practices.

8.2 The surfaces of the finished material shall be reasonably smooth and free of excessive talc or bloom.

8.3 Unless otherwise specified, the material shall be black. When colored material is desired, it is recommended that other tests, agreed upon between the purchaser and the supplier, be conducted to ensure color stability.

9. Sampling

9.1 When possible, the completed manufactured product of a suitable section thereof shall be used for the tests specified. Representative samples of the lot being examined shall be selected at random as required.

9.2 When the finished product does not lend itself to testing or to the taking of test specimens because of complicated shape, small size, metal or fabric inserts, or other reasons, standard test strips shall be prepared. The standard extruded specimens for testing, except where a specific specimen size is defined by a particular test method, shall be 6.4 mm (¼ in.) thick by 32 mm (1¼ in.) wide in rectangular cross section. The test pieces for flame propagation tests shall be as specified in 11.8.2. All test pieces shall be made from the same compound and shall have the same apparent density and state of cure as the product they represent.

9.3 The tests for dimensional stability, ozone resistance, water absorption, and nonstaining may be made on samples from the material to be shipped or on samples representative of it. Tests for compression deflection, compression set, heat aging, flame propagation, and low-temperature brittleness may be made on standard samples previously prepared in accordance with 9.2.

10. Number of Tests and Retests

10.1 Any material that fails in one or more of the test requirements may be retested by making two additional tests for the requirements in which failure occurs. Failure in one such retest shall be cause for final rejection.

10.2 Rejected material shall be disposed of as directed by the supplier.

11. Test Methods

11.1 *Compression - Deflection* — Specification D 1056. Base calculations of compression-deflection on the original thickness of the specimens.

11.2 *Compression Set*—Test Methods D 395, Method B.

11.3 *Compression Deflection After Heat Aging:*

11.3.1 A 152-mm (6-in.) length of the finished extrusion shall be heat aged along with the specimen for Specification D 1056 and shall pass the requirements of Table 1, Footnote B.

11.3.2 Test for compression-deflection by first aging the specimen (a piece of appropriate size for the compression-deflection test, instead of the dumbbell-shaped tension specimen) in accordance with Test Method D 865, then measuring the compression-deflection value in accordance with Specification D 1056.

11.3.3 The specimen for heat aging shall be large enough to allow the taking of the appropriate number and six size of specimens as defined by Specification D 1056. The cutting of specimens for Specification D 1056 shall be done after the heat aging has been performed.

11.4 *Dimensional Stability After Heat Aging*—Determine the dimensional stability by subjecting a 150-mm (6-in.) length of the extruded shape to heat aging for 70 h at 100°C (212°F) in accordance with Test Method D 865. After aging, the changes in length and breadth dimensions of the specimen shall not exceed 4 %.

11.5 *Ozone Resistance*—Test Method D 1149. The concentration of ozone shall be 100 MPa for Type I and 300 MPa for Type II. The time of test shall be 100 h at 40°C (104°F) with a specimen as defined by 9.2 with a length of 152 mm (6 in.) and with a specimen elongation of 40 %.

11.6 *Low - Temperature Brittleness*—See Appendix X1.

11.7 *Water Absorption*—Test for water absorption by placing ten 460-mm (18-in.) strips of the elastomer, bent in a U-form, in water at 21.1°C (70°F) for a period of 24 h. Submerge only the center 406 mm (16 in.) of the specimen with the bottom of the loop 152 mm (6 in.) below the water line, and the cut ends of the specimen out of the water. After removal from the water, blot the specimens with lint-free paper before weighing. The test value is the average weight of water absorbed by the ten specimens.

11.8 *Flame Propagation*—This method determines whether or not the gasket will propagate flame, with no significance being attached to such matters as fuel contribution, rate of flame spread, smoke generation, or nature and temperature of products of combustion.

11.8.1 *Apparatus:*

11.8.1.1 The test chamber may be any enclosure that will permit normal gravity circulation of air past the specimen during burning. A hood or ventilated spray booth is recommended in order to remove any noxious products of combustion, provided the velocity of air past the specimen does not exceed 18.3 m (60 ft)/min.

NOTE 3—Air velocities greater than 18.3 m/min will have an extinguishing effect upon any flame and will present an unrealistic evaluation of the flame resistance of the material.

11.8.1.2 The burner shall be a bunsen burner with a barrel nominally 9.5 mm (0.38 in.) in diameter.

11.8.1.3 The fuel shall be ordinary fuel gas at a normal pressure.

11.8.2 The test specimen shall be 13 mm (½ in.) thick, 25 mm (1 in.) wide, and 460 mm (18 in.) long made in accordance with 9.2. It shall be free of any permanent set producing a curved section within the specimen that will not permit it to hang vertically, and it shall be free of abnormally porous sections and foreign materials.

NOTE 4—This method recognizes the fact that every flame, no matter how hot, has a kindling height above which its temperature is too low to kindle the specimen. For the specified flame, this height is considerably less than 460 mm (18 in.), so if a specimen burns above this kindling height, it does so on its own heat of combustion. Therefore it is considered to propagate flame.

11.8.3 *Procedure:*

11.8.3.1 Secure the specimen in a vertical position within the test chamber using a ring stand with a clamp positioned at the top of the specimen. Locate the specimen high enough to permit the burner to be placed beneath it. Use two wire loops to retain the position of the sample over the flame. Place one wire loop 51 mm (2 in.) from the end to be ignited, the other 127 mm (5 in.) from the end to be ignited. Fasten the wire loops to the stand holding the specimen.

11.8.3.2 Light and adjust the burner so as to produce a blue flame approximately 38 mm (1.5 in.) high.

11.8.3.3 Place the lighted burner directly below the specimen so that the tip of the inner cone of the flame just touches the lowest part of the specimen. Allow the burner to remain in this position for 5 min, then remove it.

11.8.3.4 After removal of the burner, remove loose char with a stiff brush and measure the remaining length of the specimen. Consider the unburned length to be that remaining after removal of loose char.

11.8.4 *Report*—The report should state the distance of flame propagation expressed in millimetres (or inches). The distance of flame propagation equals original length less unburned length.

11.9 *Nonstaining*—Test Methods D 925, Method B. The surface against which stain is to be tested and the acceptable degree of staining shall be specified by the purchaser.

12. Inspection

12.1 All tests and inspections shall be made at the place of manufacture prior to shipment unless otherwise specified. The supplier shall provide the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with this specification.

13. Certification

13.1 When required, the supplier shall furnish the purchaser with a certified test report giving the results of the tests required to determine conformance with all requirements specified herein.

14. Packaging and Package Marking

14.1 All material shall be properly separated according to compound, size, etc., and shall be packaged and labeled in accordance with the best commercial practice with ample protection against damage in shipment.

Table 1 Physical Requirements of Cellular Elastomeric Materials

Property	Limit	ASTM Test Method[A]
Compression-deflection, 25 % deflection limits:		
kPa	91 to 168	D 1056
psi	13 to 24	
Compression set, 22 h @ 70°C (158°F) max, %	30	D 395, Method B
Heat aging[B], 70 h @ 100°C (212°F), change in compression-deflection values:		
kPa	0 to +70	D 865 and D 1056
psi	0 to +10	
Dimensional stability, change, max %, after heat aging, 70 h @ 100°C (212°F)	4	11.4
Ozone resistance[C] at 40 % elongation, 100 h @ 40°C (104°F):		
Type I 100 MPa ozone	no cracks	D 1149
Type II 300 MPa ozone	no cracks	D 1149
Low-temperature brittleness @ −40°C (−40°F)	pass	see Appendix X1
Water absorption, max, % weight	5.0	11.7
Flame propagation:		
Option I	100 mm (4 in.) max.	11.8
Option II	no limit	
Nonstaining[D]	no migratory stain	D 925

[A] See Section 11.

[B] After heat aging, surfaces of the specimen shall be neither hard nor brittle. A 150-mm (6-in.) length of the finished extrusion shall exhibit no surface cracks when bent on itself 180°.

[C] The specimen shall exhibit no surface cracks when in the extended condition.

[D] This requirement may be waived, subject to agreement between the purchaser and the supplier.

APPENDIX

(Nonmandatory Information)

X1. TEST METHOD FOR LOW-TEMPERATURE BRITTLENESS OF RUBBER AND RUBBER-LIKE MATERIALS[4]

X1.1 Scope

X1.1.1 This test method is intended to determine the ability of compounds made from rubber or rubber-like materials to resist the effect of low temperatures that may cause them to become brittle and fracture or crack when bent. Standard specimens are exposed to specified low temperatures for definite periods after which the specimens are bent in a prescribed manner and any fracture or cracking noted. The procedure is commonly called the "Thiokol" method.

NOTE X1.1—Results obtained by this test method are influenced by the rate of flexing of the cooled specimens which can not be closely controlled in the prescribed apparatus. They are therefore of a qualitative nature and may not be closely reproducible over a range of several degrees of temperature depending on the speed of flexure. For more accurate determination of brittle temperature, and particularly in new specifications, Test Method D 746 is recommended.

X1.2 Apparatus

X1.2.1 *Cold Chamber*, of sufficient size to contain the flexing fixture when loaded with specimens, and so arranged as to permit the operation of the fixture to bend to specimens without removal from the chamber. It shall be capable of maintaining within it a uniform atmosphere of cold, dry air or a mixture of air and carbon dioxide at specified temperatures within a tolerance of ±1°C (2°F).

NOTE X1.2—Temperatures of −40°C (−40°F) and −55°C (67°F) are commonly used.

X1.2.2 *Flexing Fixture*, consisting of two parallel plates each having a width of at least 50 mm (2 in.) so supported in guides that they may be rapidly moved from a position 63 mm (2½ in.) apart until they are separated by a distance of 25 mm (1 in.). Suitable clamping bars or devices shall be provided for holding the ends of the specimens for a distance of 6.4 mm (¼ in.) at the corresponding edge of each plate so that when mounted, the specimens form similar bent loops between the plates. A satisfactory flexing fixture is shown in Fig. X1.1.

X1.3 Test Specimens

X1.3.1 The test specimens shall conform in shape to Die C as specified in Methods D 412 and shall have a thickness of 2.032 ± 0.254 mm (0.080 ± 0.010 in.).

X1.3.2 At least two specimens from each compound shall be tested.

X1.4 Procedure

X1.4.1 Mount the test specimens in loop position between the plates of the flexing fixture with the enlarged ends spaced at least 3.2 mm (⅛ in.) apart and held in the clamps for a distance of 6.4 mm (¼ in.). With the plates in the open position separated 63 mm (2½ in.), place the fixture containing the specimens in the cold chamber and expose it for the specified period to cold, dry air or a mixture of air and carbon dioxide at the specified temperature. The standard exposure period shall be 5 h (Note X1.3). At the termination of the exposure period and while still in the cold chamber, move the plates of the flexing fixture as rapidly as possible from the 63-mm (2½-in.) distance of separation to a position where they are 25 mm (1 in.) apart. Then examine the specimens for fracture or visible cracks.

NOTE X1.3—Previously two periods were specified, 96 h for natural-rubber compounds and 5 h for synthetic-rubber compounds. It was found that 5 h is adequate for either class of compounds within the intent of this test method.

X1.5 Results

X1.5.1 When two specimens are tested and neither one fractures nor shows cracks after being tested, the compound shall be considered as having passed the brittleness test. If both specimens crack, the compound shall be considered to have failed.

X1.5.2 If only one specimen fractures or cracks, the result is inconclusive and two additional specimens shall be tested. If either one of these cracks, the compound shall then be considered to have failed.

X1.6 Report

X1.6.1 Report the following:

X1.6.1.1 The results of the test expressed as "passed" or "failed,"

X1.6.1.2 The temperature of the cold chamber,

X1.6.1.3 The duration of the exposure period,

X1.6.1.4 Identification of the material tested including description of any special treatment prior to test, and

X1.6.1.5 Data of manufacture of the material, if known, and date of test.

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[4] This test method was originally issued in 1943 under the designation D 736 which was discontinued in April 1967.

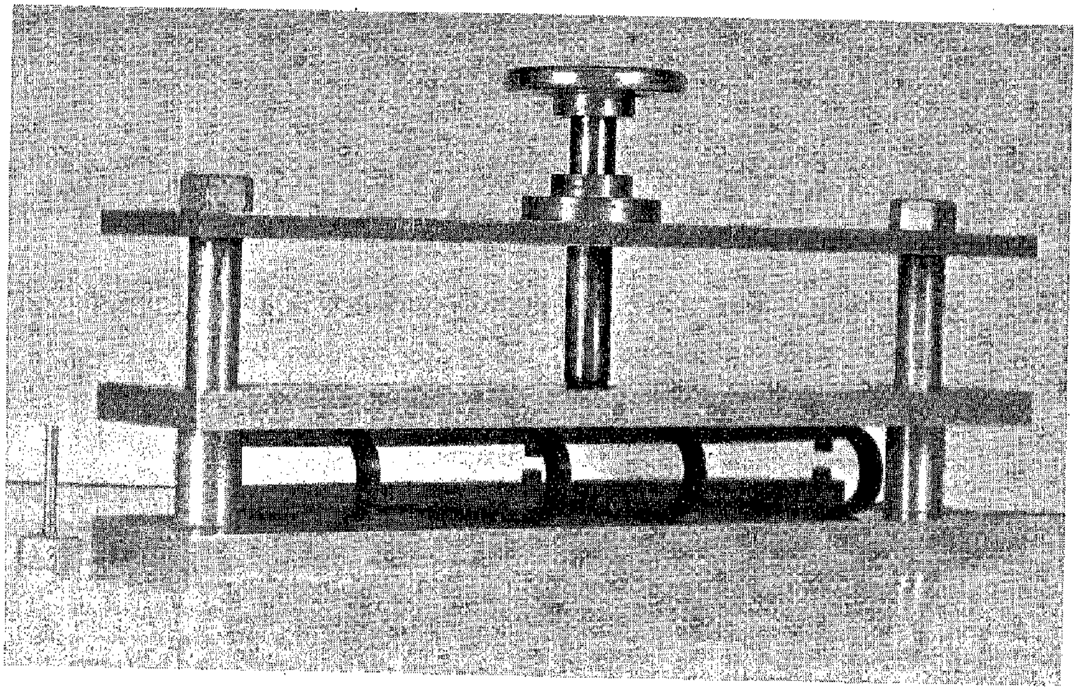

FIG. X1.1 Flexing Fixture for Low-Temperature Brittleness Test

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Standard Specification for Vermiculite Loose Fill Thermal Insulation[1]

This standard is issued under the fixed designation C 516; 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. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

$^{\epsilon 1}$ Note—Keywords were added editorially in December 1996.

1. Scope

1.1 This specification covers the composition and physical properties of expanded or exfoliated vermiculite loose fill insulation. The specification also includes the testing procedures by which the acceptability of the material may be determined. These testing procedures deal primarily with material performance in the temperature range associated with the thermal envelope of buildings; however, the commercially usable temperature range for this insulation is from −459 to 1400°F (−272 to +760°C). For specialized applications, refer to manufacturer's instructions.

1.2 The specification also covers the composition and properties of vermiculite that has been surface-treated to produce water repellency for installations where liquid moisture may be a factor.

1.3 When the installation or use of thermal insulation materials, accessories, and systems may pose safety or health problems, the manufacturer shall provide the user with appropriate current information regarding any known problems associated with the recommended use of the company's products, and shall also recommend protective measures to be employed in their safe utilization.

1.4 The following safety hazards caveat pertains only to the test methods portion, Section 9, of this specification: *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.* For specific hazard statements, see Section 12.

2. Referenced Documents

2.1 *ASTM Standards:*

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates[2]

C 168 Terminology Relating to Thermal Insulating Materials[3]

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus[3]

C 390 Criteria for Sampling and Acceptance of Preformed Thermal Insulation Lots[3]

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

C 520 Test Methods for Density of Granular Loose Fill Insulations[3]

E 84 Test Method for Surface Burning Characteristics of Building Materials[4]

E 136 Test Method for Behavior of Materials in a Vertical Tube Furnace at 750°C[4]

3. Terminology

3.1 The terms used in this specification are defined in Terminology C 168.

4. Classification

4.1 Vermiculite insulation shall be specified by two type designations and five classes, as follows:

4.1.1 *Vermiculite Loose Fill Insulation—Type I*—The product that results from the expanding or exfoliating of natural vermiculite ore by grading and heating to meet the requirements of this specification.

4.1.2 *Vermiculite Loose Fill Insulation—Type II*—Expanded vermiculite that has been surface-treated to produce water repellency and limit absorption of moisture from both liquid and vapor phase.

4.1.3 Vermiculite loose fill insulation has five grade designations established by range of particle size distribution as shown in Table 1.

5. Ordering Information

5.1 All purchase orders should designate both type and grade of insulation desired. If type designation is omitted, Type I will be furnished. The type and grade classifications in this specification differ from the classifications in earlier issues. Purchasers referencing this specification should include the date of issue.

6. Materials and Manufacture

6.1 Vermiculite is a micaceous mineral which is mined and processed to produce a high-purity concentrate. The

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[1] This specification is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.23 on Blanket and Loose Fill Insulation.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

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

[4] *Annual Book of ASTM Standards*, Vol 04.07.

TABLE 1 Density Specifications

Grade Designation	Bulk Density, lb/ft³ (kg/m³) min	Bulk Density, lb/ft³ (kg/m³) max
0—Premium	3.0 (48)	5.0 (80)
1—Large	3.7 (59)	5.5 (88)
2—Medium	4.0 (64)	6.0 (96)
3—Fine	4.5 (72)	7.0 (112)
4—Super Fine	5.5 (88)	8.0 (128)

TABLE 2 Thermal Resistance

Grade Designation	Thermal Resistance[A] for 1 in. (25.4 mm) Thickness °F·h·ft²/Btu (K·m²/W) Mean Temperature, °F (°C) −119 (−84)	40 (4)	75 (24)
0—Premium	...	...	2.3 (0.41)
1—Large	...	...	2.3 (0.41)
2—Medium	...	...	2.3 (0.41)
3—Fine	...	...	2.3 (0.41)
4—Super Fine	3.4 (0.58)	2.5 (0.43)	2.3 (0.41)

[A] The thermal values in this table are given at 1 in. (25.4 mm) of thickness with a gradient of 50°F (28°C). Test data that cover the thickness range from 1 to 6 in. show that thermal resistance per inch of thickness is essentially linear with respect to thickness. Test values do not deviate on the low side from the value in the table by more than 5 %.

concentrate, in the form of flakes of varying size and thickness weighing 55 lb/ft³ (880 kg/m³), is expanded in high-temperature furnaces to densities in the range from 3.0 to 8.0 lb/ft³ (148 to 128 kg/m³). As a naturally occurring mineral, it is classifiable as an elementary building material. It is noncombustible as determined by Test Method E 136. Material must pass combustion test criteria of Test Method E 136.

7. Physical Requirements

7.1 The physical requirements listed in this section are defined as Inspection Requirements (see Criteria C 390, 5.1.2; see also Terminology C 168). The insulation shall conform to the following requirements:

	Type I	Type II
Bulk density, lb/ft³ (kg/m³)	See Table 1	
Grading (particle size)	See Table 3	
Water properties, max g wicked in 5 min	N.A.	3

7.2 The physical properties listed in this section of the specification are defined as Qualification Requirements (see Criteria C 390, 5.1.1). The insulation shall conform to the following requirements:

Thermal resistance, °F·h·ft²/Btu (K·m²/W)	See Table 2	
Moisture absorption, max, % by weight/ 14 days	3.5	3.5
Combustibility	No flaming, glowing, or smoking	
Surface-burning characteristics (Test Method E 84):		
Flame spread, max	0	0
Smoke developed, max	0	0
Water properties, min, mL of water repelled	N.A.	175

8. Sampling

8.1 For purposes of standard tests, sampling shall be in accordance with Criteria C 390.

9. Test Methods

9.1 The physical properties, as enumerated in Section 7, shall be determined in accordance with the following methods:

9.1.1 *Bulk Density*—Test Methods C 520, Method A.

9.1.2 *Grading*—Test Method C 136, except that when a mechanical sieving device is used, the sieving time shall be 5 min and the test specimen shall be 50 g of material.

9.1.3 *Thermal Resistance*—Tests for thermal resistance may be made in accordance with Test Methods C 177 or C 518. Test at the design density. The thermal resistance of the various types shall not be lower than the values listed in Table 2, except that the average thermal resistance of any four specimens may fall up to 5 % below the value in the table. Determine the thermal resistance (*R*-value) at mean temperature of 75°F (24°C) and 40°F (4°C), at design density and in accordance with the current editions of Test Methods C 177 or C 518. Report the direction of heat flow. Thermal resistance at other mean temperatures may be determined if required.

9.1.4 *Moisture Absorption*—The test specimen shall be a sample of approximately 50 g. Loose fill the sample and level into a sample holder 9 by 9 by 5 in. (228 by 228 by 127 mm) deep.

9.1.4.1 Condition with minimum air movement across the sample surface. Condition at 50 ± 2 % relative humidity and 120°F (48.9°C) to constant weight and record. State the density of the sample conditioned to constant weight in the report of results.

9.1.4.2 Increase the relative humidity to 90 ± 2 %. Condition to constant weight by check-weighing at 24-h intervals. Determine the moisture pickup as a percent of conditioned weight.

9.1.4.3 Record percent absorption at 14 days.

9.1.4.4 *Precision and Bias*—This test establishes a typical property of vermiculite. It is an inherent characteristic. It is only intended to indicate absorption under high humidity conditions which are known to be characteristic of its usual insulation end use.

9.1.5 *Water Repellency (Type II Only)*—Determine the water repellency of Type II vermiculite insulation in accordance with the following procedure:

9.1.5.1 *Apparatus*—(*a*) Rigid plastic tube 50 mm inside diameter by 300 mm long with a 150-μm (100-mesh) screen

TABLE 3 Grading Cumulative Percent Retained

Grade Designation	U. S. Sieve No. ⅜ in. (9.5 mm)	4 (4.75 mm)	8 (2.36 mm)	16 (1.18 mm)	30 (600 μm)	50 (300 μm)	100 (150 μm)
0—Premium	30–80		80–100				
1—Large	0–10			90–100			
2—Medium		0–10	45–90		95–100		
3—Fine			0–10		90–100		
4—Super Fine				0–5		60–98	90–100

covering firmly fastened or adhered to the bottom. The tube shall be marked at 400 mL from the screen-covered end. (*b*) No. 15 rubber stopper. (*c*) 250-mL graduated cylinder. (*d*) 500-mL beaker.

9.1.5.2 *Sample Preparation*—Spoon a representative sample into the test cylinder to a level slightly above the 400-mL mark. Compact it by dropping the tube from a height of approximately 75 mm on a large rubber stopper (No. 15 recommended) for a total of ten drops. As the sample compacts to a level below the 400-mL mark, add additional material so that after the tenth drop, the level of the sample is within 3 mL of the 400-mL mark.

9.1.5.3 *Procedure*—With the tube supported in a vertical position and a beaker positioned under the tube, rapidly pour 250 mL of cold tap water onto the vermiculite. Take care while pouring, that the stream hits the middle of the surface of the bed of vermiculite and does not merely slide down the side of the test cylinder. Allow the water to drain through the bed of vermiculite for exactly 3 min. Tilt the tube at approximately 45° to drain water collected on the screen. Tilt it only momentarily for this purpose. Measure the collected water in the 250-mL graduate.

9.1.5.4 *Calculation*—Report the amount of collected water as "millilitres repelled." Report results as the mean of three independent tests.

9.1.5.5 *Precision and Bias*—The purpose of this test is to confirm that the intended degree of surface treatment has been accomplished to achieve water repellency. The specified characteristic is a minimum (no range or maximum). Quantitative values for conforming products exceeding the limits have no commercial significance.

9.1.6 *Wickability—Vermiculite Insulation (Type II Only):*

9.1.6.1 *Apparatus*—(*a*) Rigid plastic tube 50 mm in inside diameter by 300 mm long with a 150-µm (100-mesh) screen covering firmly fastened or adhered to the bottom. (*b*) 50-mm long compaction plug, fitting snugly inside the sample holder to give standard compaction to the sample. (*c*) Tubing rack capable of supporting the sample tube. (*d*) Water immersion tray.

9.1.6.2 *Sample Preparation*—Spoon a representative sample into the test cylinder to a level slightly below the top of the cylinder. Compact it with the plug until a moderate resistance pressure is felt. In no event shall compaction be more than 24 mm.

9.1.6.3 *Procedure*—Place the sample tube, screen end down, on a rack in the water immersion tray. The water level in the tray shall be 50 mm above the bottom of the tube. Allow the sample to remain in the position and allow to wick water for 5 min. Remove the tube from the tray onto a tared 75-mm watch glass so that all water that is allowed to drain, is caught by the watch glass and is included in the weight of water picked up by the sample. Determine the weight of the water picked up by the sample in grams.

9.1.6.4 *Calculation*—Report the amount of water picked up by the vermiculite sample through this wicking action as grams. Report results as a mean of three independent tests.

9.1.6.5 *Precision and Bias*—See 9.1.5.5.

10. Inspection

10.1 Inspection of the materials shall be made as agreed upon between the purchaser and the manufacturer as part of the purchase contract.

11. Packaging and Marking

11.1 *Packaging*—Unless otherwise specified, the insulation shall be packed in the manufacturer's standard commercial container. The insulation shall be packed to ensure carrier acceptance and safe delivery at destination in containers complying with the rules and regulations applicable to the mode of transportation.

11.2 *Marking*—Shipping containers shall be marked with the name of the insulation, minimum weight of container, and the name of the manufacturer. For residential insulation, a chart shall also be affixed or printed on the container listing the minimum thickness, maximum net coverage area, and minimum weight per square foot at *R* values of 11, 19, and 22. In addition to this chart, the following statements must be added: 1. "*R* means resistance to heat flow. The higher the *R*-value, the greater the insulation power. 2. To get the marked *R*-value, it is essential that this insulation be installed properly. If you do it yourself, follow the instructions carefully."

12. Health and Safety Precautions

12.1 *Preinstallation*—The insulation material should be handled and stored in accordance with manufacturer's instructions. It should be kept dry and free of extraneous materials. It is noncombustible.

12.2 *Installation:*

12.2.1 The insulating material should be poured into the spaces and cavities to be insulated in a manner that minimizes free-fall and impact. This will minimize crushing and breakdown of insulation particles and the unnecessary formation of dust.

12.2.2 Pourable loose fill insulation, which is commonly installed in confined, poorly ventilated attic spaces, may generate a buildup of airborne dust. Inhalation of dust can be injurious to health. Refer to manufacturer's instructions regarding recommended installation practices.

13. Keywords

13.1 loose fill; thermal insulation; thermal resistance; vermiculite; water repellency

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

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Document Name: ASTM C518: Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus

CFR Section(s): 46 CFR 160.174-17(f)

Standards Body: American Society for Testing and Materials

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

This standard is issued under the fixed designation C 518; 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. Consult the DoD Index of Specifications and Standards for the specific year of issue that has been adopted by the Department of Defense.

INTRODUCTION

This test method presents a comprehensive assemblage of information and requirements on the complex problem of the measurement of the thermal transmission properties of insulations using a heat flow meter apparatus. For convenience, it is organized in four major parts:

General Considerations (Sections 1 through 6)
Apparatus and Calibration (Sections 7 through 9)
Test Procedures and Report (Sections 10 through 14)
Annexes (A1 and A2)

This test method shall be used in conjunction with Practice C 1045.

Many advances have been made in thermal insulation technology, both in measurement techniques and in improved understanding of the principles of heat flow through such materials. These advances have prompted revisions in the conceptual approaches to the measurement of the thermal transmission properties, see References **(1)**, **(2)**, **(3)**, and **(4)**.[2] All users of this test method should be aware of these concepts.

1. Scope

1.1 This test method covers the measurement of steady state thermal transmission through flat slab specimens using a heat flow meter apparatus.

1.2 This is a comparative (or secondary) method of measurement since specimens of known thermal transmission properties must be used to calibrate the apparatus. Properties of the calibration specimens must be traceable to an absolute measurement method, and should be obtained from or traceable to a recognized national standards laboratory.

1.3 The test method is applicable to the measurement of thermal transmission through a wide range of specimen properties and environmental conditions. The method has been used at ambient conditions of 10 to 40°C with thicknesses up to approximately 250 mm, and with plate temperatures from −195°C to 540°C at 25 mm thickness (Refs. **(5, 6)**).

1.4 This test method may be used to characterize material properties which may or may not be representative of actual conditions of use. Other test methods such as Test Methods C 236 or C 976 should be used if needed.

1.5 To meet the requirements of this test method the thermal resistance of the sample must be greater than 0.10 $K \cdot m^2/W$ in all directions.

1.6 It is not practical in a test method of this type to try to establish details of construction and procedures to cover all contingencies that might offer difficulties to a person without pertinent technical knowledge. Thus users of this test method shall have sufficient knowledge to satisfactorily fulfill their needs. For example, knowledge of heat transfer principles, low level electrical measurements, and general test procedures is required.

1.7 Standardization of this test method is not intended to restrict in any way the future development of improved or new methods or procedures by research workers.

1.8 Since the design of a heat meter apparatus is not a simple matter, a procedure for qualifying an apparatus is given in 7.8.

1.9 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

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

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

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

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

C 168 Terminology Relating to Thermal Insulating Materials[3]

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties By Means of the Guarded-Hot-Plate Apparatus[3]

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

C 519 Test Method for Density of Fibrous Loose-Fill Building Insulations[3]

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

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

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

C 1132 Practice for Calibration of the Heat Flow Meter Apparatus[3]

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

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

3. Terminology

3.1 *Definitions*—The terms and symbols are as defined in Definitions C 168 with exceptions included as appropriate.

3.2 *Symbols and Units*—The symbols used in this test method have the following significance:

λ = thermal conductivity, W/m·K,
C = thermal conductance, W/m^2·K,
R = thermal resistance, m^2·K/W,
q = heat flux, W/m^2,
Q = heat flow, W,
A = area measured on a selected isothermal surface, m^2,
L = thickness of specimen measured along a path normal to isothermal surfaces, m,
T_m = mean temperature, K,
ΔT = temperature difference, K,
ρ = density of the material as tested, kg/m^3,
S = sensitivity of heat flow transducer (W/m^2)/V,
E = heat flow transducer output, V,
T_h = temperature of hot plate surface, K, and
T_c = temperature of cold plate surface, K.

4. Summary of Test Method

4.1 The heat flow meter apparatus establishes steady state unidirectional heat flux through a test specimen between two parallel plates at constant but different temperatures. By appropriate calibration of the heat flux transducer(s) with calibration standards and by measurement of the plate temperatures and plate separation, Fourier's law of heat conduction is used to calculate thermal conductivity, thermal resistance, or resistivity.

4.2 The accurate use of the test method is limited by the capability of the apparatus to reproduce unidirectional constant heat flux density in the specimens, and by the precision in the measurement of temperature, thickness, EMF produced by heat flux transducer, etc.

5. Significance and Use

5.1 This test method provides a rapid means of determining thermal properties with high precision and as such is useful in research and quality control work.

5.2 The thermal transmission properties of specimens of a given material or product may: (*1*) vary due to variability of the composition of the material; (*2*) be affected by moisture or other conditions; (*3*) change with time; (*4*) change with mean temperature and temperature difference, and (*5*) depend upon the prior thermal history. It must be recognized, therefore, that the selection of typical values of thermal transmission properties representative of a material in a particular application should be based on a consideration of these factors and will not necessarily apply without modification to all service conditions. As an example, this test method provides that the thermal properties shall be obtained on specimens that do not contain any free moisture although in service such conditions may not be realized. Even more basic is the dependence of the thermal properties on variables such as mean temperature and temperature difference. These dependencies should be measured or the test made at conditions typical of use.

5.3 Special care must be taken in the measurement procedure for specimens exhibiting appreciable inhomogeneities, anisotropies, rigidity, or especially high or low resistance to heat flow (see Practice C 1045). Special considerations are necessary when the measurements are conducted at either high or low temperatures, in a vacuum or in hazardous gases.

5.4 The determination of the accuracy of the method for any given test is a function of the apparatus design, of the related instrumentation and of the type of specimens under test (see Section 9), but this method is capable of determining thermal transmission properties within ±2 % of those determined by Test Method C 177 when the ambient temperature is near the mean temperature of the test (T (ambient) = T (mean) ±1°C), and in the range of 10 to 40°C.

5.4.1 Where certification of measurement by this test method is required, the apparatus shall have the capabilities required in 7.8 and one of the following procedures must be followed:

5.4.1.1 The testing laboratory apparatus shall be calibrated within 24 h before or after the certification test using calibration standards that have been issued or whose values have been established by a recognized National Standards Laboratory not more than five years prior to the certification date. The average of two calibrations shall be used as the calibration factor and the specimen(s) certified with this average value. When the change in calibration factor is greater than one percent, the standard specimen shall be retested and a new average calculated. If the change in calibration factor is still greater than 1 % the apparatus must be calibrated using the procedure in Section 8.

5.4.1.2 Where both the short and long term stabilities of the apparatus have been proven to be better than 1 % of the reading, see 8.4, the apparatus may be calibrated at less frequent intervals, not exceeding 30 days. The specimens so tested cannot be certified until after the calibration following the test and then only if the change in calibration factor from

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

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

the previous calibration test is less than 1 %. When the change in calibration is greater than one percent, test results from this interval shall be considered void and the tests repeated in accordance with 5.4.1.1.

5.4.2 The precision (repeatability) of measurements made by the heat flow meter apparatus calibrated as in 5.4.1 are normally much better than ±1 % of the mean value. This precision is required to identify changes in calibration and is desirable in quality control applications.

6. Limitations to Test Method

6.1 *Limitations Due to Apparatus:*

6.1.1 The apparatus shall not be used at temperatures, thicknesses, or resistances, other than those within the range of the calibration, unless it can be shown that there is no difference in accuracy.

6.1.2 The apparatus must be capable of maintaining at least a 10°C temperature difference across the specimen for the duration of the test, unless a smaller ΔT is a requirement of a particular test.

6.2 *Limitations Due to the Specimens:*

6.2.1 The specimens under test may also limit the use of the test method and these limitations are outlined in Practice C 1045.

7. Apparatus

7.1 The construction details given in this section should be understood by the user of this test method. While it is mandatory that these details be carefully followed when constructing an apparatus, it behooves the user to verify that the equipment was built as specified. Serious errors of measurement may result from this oversight.

7.2 *General:*

7.2.1 The general features of a heat flow meter apparatus with the specimen or the specimens installed are discussed in Annex A1. A heat flow meter apparatus consists of two isothermal plate assemblies, one or more heat flux transducers, equipment to measure and control temperature, thickness, and the output of the heat flux transducers and equipment to control the environmental conditions when needed. Each configuration will yield equivalent results if used within the limitations stated in this test method. There are distinct advantages for each method in practice and these are discussed in Annex A1.

7.2.2 The working surfaces of the plates and the heat flux transducer (that is, the surfaces making contact with the specimens) shall be painted or otherwise treated to have a total hemispherical emittance of greater than 0.84 at operating temperatures (Note 1).

NOTE 1—Hard anodizing of aluminum produces a surface with a total hemispherical emittance of approximately 0.85. Several paints are available which when applied as directed, produce a total hemispherical emittance of approximately 0.86.

7.3 *Plate Assemblies, Hot and Cold*—The two plate assemblies provide isothermal surfaces in contact with either side of the test specimen. The assemblies consist of heat source or heat sink, a high conductivity surface, means to measure surface temperature, and means of support. A heat flux transducer may be attached to one, both or neither plate assembly, depending upon the design. In all cases the area defined by the dimensions of the heat flux transducer is called the metering area and the remainder of the plate is the guard area.

7.3.1 The portion of each plate assembly in contact with the heat flux transducer, or if a heat flux transducer is not mounted on the plate assembly, the portion in contact with the sample, shall consist of a high conductivity material, usually metal, known as the isothermal plate. The isothermal plate shall be supported so as to minimize temperature gradients across its surface. For a 2 % total uncertainty, deviations from isothermal conditions over the plate surface of no more than ±0.5 K shall be allowed.

7.3.2 A means shall be provided to maintain the temperature of the isothermal plates at the desired level. Examples are fluid baths, electrical heaters and combinations of these (see Refs (**7, 8, 9**)).

7.3.3 If a heat flux transducer is mounted at the midplane of the specimen then means shall be provided to determine the average temperature of the transducer in order to apply temperature corrections to the calibration, except when test temperatures are equal to those used in calibration, in which case no correction is required.

7.3.4 Means shall be provided to measure the temperature of the surfaces of the heat flux transducer or plates in contact with the specimen.

7.3.5 The surface plates shall be sufficiently rigid to maintain flatness and parallelism to within one-tenth the accuracy required of the final result. For measurements on non-rigid materials and an allowable total uncertainty of ±2 %, a flatness and parallelism of 0.2 % of the maximum linear dimension of the heating unit is thus required (Note 2). One recommended way to check this is to generate a L-map over the metering area. This is discussed fully in Ref (**10**).

NOTE 2—The planeness of the surface can be checked with a straightedge, of a length greater than the width or diameter of the unit, held against the surface and viewed with a light behind the straightedge. Departures as small as 25 μm are readily visible, and larger departures can be measured using shimstock or thin paper.

7.3.5.1 The parallelism of the plates must be maintained for several reasons. In most cases it is the plate separation which is measured in order to determine specimen thickness. Furthermore the plate parallelism is important in maintaining consistent surface contact with specimens in repeat testing (such as calibration), and is required to maintain a uniform temperature difference across the specimen(s).

7.3.6 The rigidity, flatness, and parallelism of the plates may impede the testing of rigid specimens where it is not possible to obtain good surface contact. In such cases the use of a thin sheet of suitable homogeneous material may be interposed between the specimen and the plate surfaces. This thin sheet should have a low thermal resistance relative to the specimen. The resistance of the thin sheet should be determined using a Test Method C 177 apparatus. The resistance of the composite sandwich (sheet-rigid specimen-sheet) is then determined, and the value of the sheet resistance (determined in a separate test) subtracted from the total resistance measured.

7.4 *Temperature Measuring and Control Systems:*

7.4.1 Any proven device possessing adequate accuracy may be used for measurement and control of the temperatures in the apparatus.

7.4.2 Surface temperatures are often measured by permanently mounting temperature sensors, such as thermocouples, RTD's or thermistors, in grooves or flush with the working surfaces on both plates.

7.4.3 Many existing systems utilize a minimum of two temperature sensors to determine the average temperature of each isothermal surface. However, to determine the actual surface temperature variations under typical operating conditions, the temperature must be sampled at considerably more than two points. This is especially true when the specimen surface temperature itself must be measured or when the specimen is highly inhomogeneous. Therefore, no strict specification is given as to the number of temperature sensors that must be used for each surface. However, the user must report the uncertainty of the temperature measurement, including the component, due to temperature nonuniformity across the surface. This determination is probably best done with a multijunction thermocouple in ad-hoc experiments and not part of every measurement. (See Ref **(10)**.) In some cases where temperature mapping of the plate surfaces has indicated high uniformity under all conditions of use, one thermal sensor per surface has been satisfactorily used.

7.4.3.1 Special precautions should be taken to ensure that the temperature sensors are thermally anchored to the surface to be measured and that the temperature gradients along the wires leading to the sensors are minimized. If sensors on opposing surfaces are connected differentially, they must be electrically insulated from the plates with a resistance of 1MΩ or greater **(11, 12)**.

7.4.3.2 Thermocouples mounted in the surfaces of the plates should normally be made of wire not larger than 0.51 mm in diameter (No. 24 B & S gage). Thermocouples placed against or set into the surfaces of the specimens should be made of wire not larger than 0.25 mm in diameter (No. 30 B & S gage). For information concerning sensitivity and accuracy of thermocouples in the cryogenic temperature range, and installation, see Refs **(6)** and **(13)**.

7.4.3.3 Temperature sensors should be calibrated to an accuracy equivalent to that for thermocouples conforming to Tables E 230. The temperature measuring system shall have an equivalent accuracy. The precision of the temperature measuring system may need to be better than this to detect the effect of drift on the results as discussed in 7.9. Tables E 230 lists both "standard" and "special" limits on the accuracy of thermocouples. The accuracy required by a heat flow meter apparatus can best be determined by carrying out an error analysis (see Section 9), and then calibrating the temperature sensors to the degree required.

7.5 *Heat Flux Transducer*—The heat flux transducer is a device including both the metering and guard areas which measures heat flux by sensing the temperature difference across a core of appropriate thermal resistance and thermal stability. The metering area usually consists of a number of differentially connected temperature sensors placed on each face of the core and surface sheets to protect the assembly. The guard area is usually constructed of the same material as the core. A properly designed transducer will have a sensitivity that is essentially independent of the specimens' thermal properties **(14, 15)**.

7.5.1 *Types of Heat Flux Transducers*—The type of heat flux transducer described in this test method is called a gradient type. This type consists of a slab of material, the "core," across which the temperature gradient is measured, normally with a thermopile.

7.5.2 *Temperature Difference Detector*—The temperature difference across the core material shall be measured with a sensitive stable temperature detector. The main transducer surfaces are assumed to be isothermal, so that heat flow will be normal to them. Precautions must be taken to limit the effect of heat flow through the leads on the output of the temperature difference detector. Often the heat flux transducer is also instrumented to measure one of the surface temperatures of the specimen(s).

7.5.3 *Surface Sheets*—To prevent damage to the temperature difference detector that will affect its calibration, both surfaces shall be covered with a layer of material as thin as is compatible with protection from thermal shunting of the temperature difference detector wires. The working surfaces of the heat flux transducer shall be smoothly finished to conform to the desired geometric shape to within the limits of 7.3.

7.6 *Plate Separation, Specimen Thickness*—A means shall be provided to determine the average separation between the heating and cooling plate surfaces during operation. Rigid specimens generally act as the spacers themselves and plate separation is determined by their thickness at operating temperature. In this case a small constant force is generally applied to hold the plates against the specimen. It is unlikely that a pressure greater than 2.5 kPa (approximately 50 lbf/ft^2) will be required. For easily compressible specimens, small stops interposed between the corners of the hot and cold plates, or some other positive means shall be used to limit the compression of the specimens, (Note 3). Provision must be made for checking the linearity of any thickness measuring system.

NOTE 3—Because of the changes of specimen thickness possible as a result of temperature or compression by the plates, it is recommended that specimen thickness be measured in the apparatus, at the existing test temperature and compression conditions whenever possible.

7.7 *Edge Insulation*—Heat loss from the outer edges of the heat flow meter apparatus and specimens shall be restricted by edge insulation or by governing the surrounding air temperature or by both methods. The three different configurations differ in their sensitivity to edge heat losses as is discussed in Annex A1 **(2, 4, 16)**.

7.7.1 For all three configurations, the sensitivity to edge heat losses is strongly related to the sensitivity of the transducer to temperature differences along its main surfaces and therefore only experimental checks while changing environmental conditions can confirm, for each operating condition, the magnitude of the effect of edge heat losses on measured heat flux. This error should be smaller than 0.5 %. An approximate guideline is to maintain power lost through the edges below 5 % of the power flowing through the specimen.

7.8 *Measuring System Requirements for Certification*—When the apparatus is used for certification testing, the measuring system shall have the following capabilities:

7.8.1 The accuracy of the measurement of the temperature difference across the specimens shall be within ±0.5 %. The accuracy of the measurement of the output from the heat flux transducer shall be within ±0.6 %.

7.8.2 A voltage sensitivity of better than 0.2 % of the minimum output (from the transducer) to be measured.

7.8.3 Sufficient linearity so that it contributes less than 0.2 % error at all outputs.

7.8.4 Sufficient input impedance so that it contributes less than 0.1 % error for all readings. One megohm has been found adequate for many apparatuses.

7.8.5 Sufficient stability so that it contributes less than 0.2 % error during the period between calibrations, or 30 days, whichever is greater.

7.8.6 Adequate noise immunity so that less than 0.2 % rms noise occurs in the readings.

7.8.7 Where direct readout equipment is used, adequate provision shall be made for calibration of the electronic circuitry, independent of the remainder of the apparatus, and shall contribute no more than 0.2 % error, for each variable.

7.9 *Proven Performance*—Any heat flow meter apparatus that is new or has been modified must be tested for the following characteristics and corrections shall be made where a change of greater than one percent occurs in the transducer output due to the effects of 7.9.1 to 7.9 over the range of operation and are recommended for changes of 0.3 % over the range of operation.

7.9.1 *Zero Offset*—If there is any output from the transducer for zero heat flux, this may be due to:

7.9.1.1 Electrical contact resistance on a transducer with low output: improve connections to eliminate the problem as this type of output may be temperature dependent.

7.9.1.2 Also check grounding circuits because such a signal may be due to AC pickup in the leads from the transducer. If after checking 7.9.1.1 and 7.9.1.2 there is still a zero off-set, it may be possible to correct for this by assuring that the calibration curve of output versus heat flux is linear over the range of operating conditions.

7.9.2 Sensitivity to warm or cold plate temperature non-uniformity. Check for temperature non-uniformity under all operating conditions.

7.9.3 Drift in the transducer due to material aging or delamination. This can be used to determine the required calibration frequency.

7.9.4 Temperature coefficient of the transducer sensitivity, as this depends on the type of temperature detectors used in the temperature difference detector (thermocouple materials used in the thermopile) and the type of material used for the transducer core. If it is found that the sensitivity is temperature dependent, a curve of sensitivity versus temperature shall be developed and used to correct measurement data.

7.9.5 Heat flow down the leads. One possible way to check for this is to move one's hand across the surface of the transducer between the metering area to the point where the leads exit the plate assembly, while observing the transducer output. In a well designed plate or transducer assembly there should be no observable output from the transducer except in the metering area.

7.9.6 Effect of the thermal conductivity of the specimen on the sensitivity of the transducer. A "thermal shorting" effect between elements caused by low thermal resistance between the sensors of the thermopile or a funneling of heat through the sensors can change the sensitivity of the meter. This can best be tested by running specimens with known widely different thermal conductivities.

7.9.7 Effect of loading pressure on the transducer sensitivity, if the transducer core is flexible.

7.9.8 The test results obtained by this method can only be assured if the limitations of the apparatus are known. To establish the limitations, one must prove the performance by comparing the results with materials of similar thermal properties previously tested on a guarded hot plate apparatus as those to be evaluated.

7.9.8.1 A single point of reference may lead to serious errors. It is best to select a range of transfer standards having known thermal transmission properties which cover the range of values to be tested, in both resistance and thickness.

7.9.8.2 If the apparatus is to be used at thicknesses greater than that of the available reference materials, a series of calibration measurements must be performed to insure that the equipment does not introduce additional errors which may be due to lateral heat losses or gains brought about by insufficient guarding (**4, 17**).

7.10 *Environmental Control*—In many applications, it is desirable to control the environment surrounding the test specimen to reduce edge heat losses and it is especially important if the mean test temperature must be below the ambient temperature, in order to avoid condensation on the cold plate.

7.10.1 A cabinet or enclosure surrounding the heat flow meter assembly to maintain the ambient temperature at the mean temperature of the specimen may also be used as a means to maintain the dew point temperature at least 5 K lower than the temperature of the cold plates, that will prevent condensation and moisture pickup by the specimen. This is required when conducting tests at mean temperatures substantially lower than the laboratory air temperature.

7.10.2 Any environmental control system employed in conjunction with a heat flow meter apparatus shall be capable of maintaining its set point condition within ±1°C in temperature, and ±2 % in humidity.

8. Calibration

8.1 The calibration of a heat flow meter apparatus is a very critical operation. Since lateral heat losses or gains of heat are not automatically controlled or eliminated, but only lessened by increasing the size of the guard area and edge insulation, there is no guarantee that the heat losses or gains are negligible under all testing conditions. To ensure that the equipment is performing properly with specimens of different thermal resistances, the apparatus must be calibrated with materials having similar characteristics as the materials to be evaluated.

8.2 See Practice C 1132.

9. Error Analysis

9.1 A complete error analysis is complex. However, it is mandated for compliance with this test method. The performance of this analysis involves consideration of:

9.1.1 Estimates of errors in each individual measurement procedure and propagation of these errors to the final result,

9.1.2 Measurements to determine apparatus sensitivity to intentional deviations from normal operations, and

9.1.3 Measurements on reference materials and participation in round-robin programs.

9.1.4 For a more complete discussion of this error analysis the reader is directed to Test Method C 177, and Ref **(10)**.

9.2 To illustrate a procedure of error analysis estimation, consider the operational definition of thermal conductivity:

$$\lambda = Q \cdot L / A \cdot \Delta T$$

The uncertainties in Q, A, L, and ΔT (δQ, δA, δL, and $\delta \Delta T$) can be used to form the uncertainty $S\lambda$ by the usual error propagation formula where the total uncertainty is calculated from the square root of the sums of the squares of the individual standard deviations.

$$S\lambda^2 = (\delta Q/Q)^2 + (\delta A/A)^2 + (\delta L/L)^2 + (\delta \Delta T/\Delta T)^2$$

This equation assumes that the errors in Q, A, etc., are random and independent of each other since the covariance terms are omitted. This is valid here if different instrumentation is used for measurements on each of the variables.

9.2.1 An alternative approach, which generally leads to a larger calculated uncertainty, is to sum the upper limits of the individual uncertainties **(10)**.

9.2.2 Care must be taken to properly evaluate all of the uncertainties in the variables Q, A, etc. For example, the obvious sources of error in Q are those causing extraneous transverse heat flow along leads and deviations from one dimensional heat flow. However, an often neglected but important quasi-heat leak is that caused by a temperature drift of the transducer itself, or in the heater, or both.

9.2.2.1 This can readily be estimated from the heat capacity of the transducer assembly and the drift detection limit of the measurement system. The error in ΔT, $\delta \Delta T$, can be caused by calibration errors and measurement errors but also by incorrect placement, incorrect thermal anchoring, and disturbances introduced by the thermocouple itself.

9.3 Numerous experiments can be performed to determine the sensitivity of the measurement results to deviations from normal operating conditions. This sensitivity combined with the probable deviation under normal operating conditions can be used to estimate the error from this source. As one example, the effect of imperfect guard balance control can be determined by purposely offsetting the guard (if this is possible) by a sufficient ΔT in both directions and measuring the differences in the measured output.

9.3.1 Care should be taken to not use such large offsets that nonlinear effects occur in the specimens. These results combined with the probable value of the offset during normal operation yield the error due to imperfect guard balance.

9.4 Finally, measurements should be performed on accepted reference materials, if available, to qualify the apparatus. Care should be taken here to ensure that the reference materials have characteristics similar to the specimens to be measured and that the uncertainties of the standards themselves are known.

9.5 The total estimated imprecision can be listed in a table of errors such as shown in Table 1. This table is shown as an example only and does not represent any one particular heat flow meter apparatus since the errors will be specific to each apparatus.

10. Test Procedures

10.1 *Foreword on Testing Procedures*—The simplicity of this test procedure may lead one to overlook very important factors which may affect the results. To ensure accurate measurement, the operator must be fully instructed in the operation of the equipment. Furthermore, the equipment must be properly calibrated with reference materials having similar heat transfer characteristics. It is also necessary that the specimen be properly prepared for the evaluation.

10.2 *Sampling and Preparation of Specimens:*

10.2.1 *Test Specimens*—One or two-piece specimens may be used, depending on the configuration selected for the test. Where two pieces are used, they shall be selected from the same sample to be essentially identical in construction, thickness, and density. For loose fill materials, the method specified in the material specification or in Practice C 687 shall be used to produce a specimen or specimens of the desired density.

10.2.2 *Selection of Specimens*—The specimen or specimens shall be of such size as to cover the heat flux transducer, cold plate and warm plate surfaces and shall either be of the actual thickness to be applied, or of sufficient thickness to give a true average representation of the material to be tested.

10.3 *Specimen Conditioning*—Details of the specimen selection and conditioning are preferably specified in the material specification. Where such specifications are not given, the specimen preparation shall be conducted in accordance with the requirement that materials shall not be exposed to temperatures that will change the specimens in an irreversible manner. Typically the material specifications call for sample conditioning at 22°C (72°F) and 50 % R.H. for a

TABLE 1 Estimates of Random and Systematic Errors at Room Temperature

Variable	Absolute Variations		Percent Variations	
	Random	Systematic	Random	Systematic
Mean Temperature, T	0.02 K	0.2 K	0.007	0.07
ΔT	0.01 K	0.2 K	0.04	0.40
ΔL	0	0.1 mm	0	0.40
Transducer area	0	50 mm²	0	0.10
Heat flux				
drift	1 mW	1 mW	0.2	0.2
lateral	1 mW	2 mW	0.2	0.4
calibration	0	2 mW	0	0.2
Temperature				
drift	0.05 K	0.05 K	0.01	0.01
calibration	0	0.1 K	0	0.4
λ	0.2 mW/m·K	0.3 mW/m·K	0.4	0.6

period of time until less than a 1 % weight change is observed.

10.4 *Measurements on Specimens:*

10.4.1 *Blanket and Batt Type Materials*—When specified, the test thickness of blankets and batt type materials shall be determined before testing in accordance with Test Methods C 167, provided that good contact is maintained between the specimen and the isothermal plates.

10.5 *Limitations on Specimen Thickness:*

10.5.1 *General*—The combined thickness of the specimen or specimens, the heat flux transducer and any damping material, which in total equals the distance between the cold and hot plates, must be restricted in order to limit the effect of edge losses on the measurement of heat flux. The edge losses are affected by the edge insulation and the ambient temperature, so the requirements on both the these parameters must be met as well.

10.5.2 *Maximum Spacing Between Hot and Cold Plates*—The maximum allowable distance between the hot and cold plates during the test, is related to the dimensions of the heat flux transducer, the heat metering area, the width of the non-metering plate, the construction of the heat meter apparatus, and the properties of the specimen. No suitable theoretical analysis is available to predict the maximum allowable thickness of specimens. It is possible to use the results of an analysis for a similarly sized guarded hot plate as a guide **(8, 18)**.

11. Procedure of Measurement

11.1 *Temperature Difference and Gradient*—For any test, make the temperature difference across the specimen not less than 10 K. For specimens that are expected to have a large thermal resistance, a larger temperature difference in the specimen is recommended. The actual temperature difference or gradient is best specified in the material specifications or by agreement of the parties concerned.

11.2 *Edge Insulation*—Wrap the edges of the specimens with the required thermal insulation to reduce edge heat losses to an acceptable level (see 7.7).

11.3 *Settling Time and Measurement Interval*—Check for thermal equilibrium by observing and if possible recording, the mean temperature and the emf output of the heat flux transducer, the mean temperature of the specimens, and the temperature drop across the specimen. Make observations at time intervals of at least 10 minutes unless experience has shown that a shorter time interval may be used until five successive observations yield values of thermal conductance which fall with one-half percent of their mean value. For high density specimens ($\rho > 40$ Kg/m^3) or for low conductance specimens ($C < 0.05$ W/K·m^2) the time between readings may have to be increased to 30 minutes or longer **(19)**. If the five readings show a unidirectionally increasing or decreasing trend the data is suspect and additional sets of readings shall be taken.

12. Calculation

12.1 *Density and Change in Mass*—When required, calculate the density of the dry specimen as tested, ρ, the loss of mass due to conditioning of the material, and the mass regain of the specimen during test.

12.1.1 *Density of Batt and Blanket Specimens*—It has been found that in characterizing the thermal performance of low density materials it is important to measure the mass of the specimens in contact with the heat flux transducer metering area. For these materials, the area of the specimen directly measured must be cut out and its mass determined after testing. This procedure is desirable for all specimens.

12.2 *Thermal Properties for One Specimen*—When only one specimen is used, calculate the thermal conductance of the specimens as follows:

$$C = S \cdot E / \Delta T$$

and where applicable, calculate the thermal conductivity, as follows:

$$\lambda = S \cdot E \cdot (L / \Delta T)$$

12.3 *Thermal Properties for Two Specimens*—When two specimens are used, calculate the total thermal conductance, *C*, as follows:

$$C = S \cdot E / (\Delta T_1 + \Delta T_2)$$

The λ factor, that is, the average thermal conductivity of the specimen is calculated as follows:

$$\lambda_{\text{ave}} = \frac{S \cdot E}{2} \frac{L_1 + L_2}{\Delta T_1 + \Delta T_2}$$

where the subscripts refer to the two specimens.

12.4 Other derived thermal transmission properties may be calculated but only under the provisions given in Practice C 1045.

12.5 *Thermal Properties for Two Transducers*—All pertinent specifications of 12.2 apply on this configuration, provided S·E will be replaced by $(S' \cdot E' + S'' \cdot E'')/2$, where the superscripts ′ and ″ refer to the first and second heat flux transducer, respectively.

13. Precision and Bias

13.1 The precision and bias of this test method cannot be stated quantitatively for all apparatus variations and test conditions. However, it is mandated that for compliance with this test method, any result must be accompanied with its uncertainty. Further details on doing this are given in Section 9.

13.2 It should be noted that the only statistically valid procedure to determine uncertainty is to force all sources of systematic bias to be exhibited as random errors and then uncertainty can be directly estimated from imprecision. However, this is impractical since it involves variation of all measurement variables (Global set) and cost constraints make this impossible. For example, this implies the repetition of an experiment with differently calibrated thermocouples, different sizes and configurations of the apparatus, different operators, different environment, differently calibrated instrumentation, etc. For any one given apparatus a careful error analysis as outlined in Section 9 will, in most cases, show up any major difficulties, which may need correcting in order to improve the measurement accuracy of the heat flow meter apparatus.

13.3 A series of three round robins was conducted between 1976 and 1983, as reported by Hust and Pelanne **(21)**, and employed low density fiberglass specimens from 2.54 to 10.2 cm thick with densities ranging from 10 to 33 kg/m^3. A total of twelve laboratories was involved in these studies. The imprecision, at the two standard deviation level, was found to vary from 1.92 % to 3.54 % between 2.54 cm and 10.2 cm.

13.4 Inter-laboratory testing carried out between nine laboratories under the National Voluntary Laboratory Accreditation Program is currently showing an imprecision of 2.12 % at the two standard deviation level based on testing of similar but not identical specimens.

13.5 A round robin, conducted in 1987, was reported by Adams and Hust with eleven participating laboratories testing a fiberglass blanket and several types of loose-fill insulations (22). The blanket insulation had an imprecision of 3.7 % at the two standard deviation level. The loose-fill imprecision was found to be >10 % for different materials at the two standard deviation level. It has been suggested that the principal cause for the significant differences observed was the various sample preparation techniques used by the various laboratories.

14. Report

14.1 The report of the results of each test shall include the following information with all data to be reported in both SI and inch-pound units unless specified otherwise:

14.1.1 Name and any other pertinent identification of the material including a physical description.

14.1.2 Description of the specimen and its relationship to the sample, including a brief history of the specimen, if known.

14.1.3 Thickness of the specimen as received and as tested. In a two piece specimen configuration this is the thickness of one piece only.

14.1.4 Method and environment used for conditioning, if used.

14.1.5 Density of the conditioned specimen as tested, kg/m^3.

14.1.6 Mass loss of the specimen during conditioning and testing, in percentage of conditioned mass, if measured.

14.1.7 Mass regain of the specimen during test, in percentage of conditioned mass, if measured.

14.1.8 Average temperature gradient in the specimen during test as computed from the temperatures of the hot and cold surfaces, K/m.

14.1.9 Mean temperature of the test, K or °C.

14.1.10 Heat flux per unit area through the specimen, W/m^2.

14.1.11 Thermal conductance, W/m^2·K.

14.1.12 Orientation and position of the heat meter apparatus during test (vertical, horizontal, etc.) and whether the meter was against the hot or cold surface of the specimen and whether the edges of the specimen(s) were sealed or open to the ambient.

14.1.13 Duration of the measurement portion of the test, min or h.

14.1.14 For loose-fill materials report the sample preparation followed.

14.1.15 Date of the test, the date of the last heat meter calibration, and the type or types of materials used.

14.1.16 For certification testing only, the specimens used in calibration must be identified as to the type, thermal resistance, date of specimen certification, source of certification, expiration date of calibration and the certification test number.

14.1.17 Statement of Compliance or, where circumstances or requirements preclude complete compliance with the procedures of the test, agreed exceptions. A suggested wording is: "This test conformed with all requirements of ASTM C 518 – ____ with the exception of (a complete list of exceptions follows)."

14.1.18 For direct reading apparatus, the results of the calibration of electronic circuitry and equipment, or a statement of compliance including date, and a statement of compliance on linearity requirements.

14.1.19 Estimated or calculated uncertainty in reported values.

15. Keywords

15.1 thermal insulation; thermal resistance; thermal conductivity; test method; heat flux; instrument verification; test procedures; error analysis; heat flow meter apparatus

ANNEXES

(Mandatory Information)

TABLE A1.1 Limits of Error for Thermocouples

NOTE 1—Limits of error in this table apply to new thermocouple wire, normally in the size range 0.25 to 3 mm in diameter (No. 30 to No. 8 Awg) and used to temperatures not exceeding the recommended limits of Table 2. If used at higher temperatures these limits of error may not apply.

NOTE 2—Limits of error apply to new wire as delivered to the user *and do not allow for calibration drift during use.* The magnitude of such changes depends on such factors as wire size, temperature, time of exposure, and environment.

NOTE 2—NOTE 3—Where limits of error are given in percent, the percentage applies to the temperature being measured when expressed in degrees Celsius. To determine the limit of error in degrees Fahrenheit multiply the limit of error in degrees Celsius by 9/5.

Thermo-couple Type	Temperature Range		Limits of Error-Reference Junction 0°C (32°F)			
			Standard		Special	
	°C	°F	°C (whichever is greater)	°F	°C (whichever is greater	°F
T	0 to 350	32 to 700	±1 or ±0.75 %	Note 3	±0.5 or 0.4 %	Note 3
J	0 to 750	32 to 1400	±2.2 or ±0.75 %		±1.1 or 0.4 %	
E	0 to 900	32 to 1600	±1.7 or ±0.5 %		±1 or ±0.4 %	
K	0 to 1250	32 to 2300	±2.2 or ±0.75 %		±1.1 or ±0.4 %	
R or S	0 to 1450	32 to 2700	±1.5 or ±0.25 %		±0.6 or ±0.1 %	
B	870 to 1700	1600 to 3100	±0.5 %		—	
T[A]	−200 to 0	−328 to 32	±1 or ±1.5 %		[B]	
E[A]	−200 to 0	−328 to 32	±1.7 or ±1 %		[B]	
K[A]	−200 to 0	−328 to 32	±2.2 or ±2 %		[B]	

[A] Thermocouples and thermocouple materials are normally supplied to meet the limits of error specified in the table for temperatures above 0°C. The same materials, however, may not fall within the sub-zero limits of error given in the second section of the table. If materials are required to meet the sub-zero limits, the purchase order must so state. Selection of materials usually will be required.

[B] Little information is available to justify establishing special limits of error for sub-zero temperatures. Limited experience suggests the following limits for Types E and T thermocouples:

Type E −200 to 0°C ±1°C or ±0.5 % (whichever is greater)

Type T −200 to 0°C ±0.5°C or ±0.8 % (whichever is greater)

These limits are given only as a guide for discussion between purchaser and supplier.

Due to the characteristics of the materials, sub-zero limits of error for Type J thermocouples and special sub-zero limits for Type K thermocouples are not listed.

A.2 Response of Heat Flux Transducers

A2.1 *Configurations*—The general features of a heat flow meter apparatus with the specimen or the specimens installed are shown in Fig. A2.1. A heat flow meter apparatus consists of two isothermal plate assemblies, one or more heat flux transducers, equipment to measure and control temperature, thickness and the output of the heat flux transducers and equipment to control the environmental conditions when needed. Configuration a is called single transducer, one face; the heat flux transducer may be placed against either plate. The Configuration b is called two transducer, both faces. The Configuration c is called single transducer, mid-plane; in this case, the specimens should be halves of the same specimen (**20**). Each configuration will yield equivalent results if used within the limitations stated in this method. For a particular heat flux transducer, the test configuration, that has the fastest response, that is shortest settling time, is best determined by experiment. Four specimens of different materials, such as urethane, ceramic fiber board, a rubber, and a high conductivity, low-thermal capacity material, each of which is conditioned to the mean test-temperature, should be tested in each configuration. A study of these will allow either the selection of the proper configuration for each type of material, or the selection of a reasonable configuration for all types.

A2.1.1 In order to predict settling times for all types of specimens, each of the above specimens must be retested after being conditioned to temperatures both below and above the mean temperature of the test.

A2.2 *Time Responses of Heat Flux Transducers:*

A2.2.1 *High Thermal Resistance Transducer*—A transducer with a high thermal resistance is generally used in Configuration a. When the specimens are preconditioned to the mean temperature of the test and when the plates are capable of both heating and cooling the specimen, the time response of a high resistance transducer will be more rapid than a comparable guarded hot plate apparatus. However, if the heat flux transducer has appreciable mass the response will not be rapid.

A2.2.1.1 The temperature drop across a high thermal resistance transducer may be greater than desirable, that is, one percent of the total, when used in Configuration c, and thus its use in this configuration is not recommended.

A2.2.1.2 Configuration b can be used to obtain very rapid response times if both plates are capable of heating and cooling and if the outputs of both transducers are summed, (**15**). Configuration b is always faster than Configuration a with the same transducers and specimens and can be comparable to Configuration c if a low thermal resistance transducer is employed.

A2.2.2 *Low Thermal Resistance Transducers*—The low-thermal resistance, gradient-type heat flux transducer is better suited to the Configuration c. The temperature drop across the low-resistance transducer is small enough so that the two specimens can be considered as halves of a single specimen. When the specimens are first conditioned to the mean temperature of the test and when the specimens are identical, the response is sufficiently rapid to be used for quality control work.

TABLE A1.2a Conversion Factors (Thermochemical)

NOTE—Conversion factors for thermal resistivity and thermal conductance can be found by using these tables in the reverse direction.

Thermal Conductivity[A]

	$W \cdot m^{-1} \cdot K^{-1}$ ([B])	$W \cdot cm^{-1} \cdot K^{-1}$	$cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}$	$kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}$	$Btu \cdot h^{-1} \cdot ft^{-1} \cdot °F^{-1}$	$Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$
1 $W \cdot m^{-1} \cdot K^{-1}$ =	1.000	1.000×10^{-2}	2.390×10^{-3}	0.8604	0.5782	6.938
1 $W \cdot cm^{-1} \cdot K^{-1}$ =	100.0	1.000	0.2390	86.04	57.82	693.8
1 $cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}$ =	418.4	4.184	1.000	360.0	241.9	2903
1 $kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}$ =	1.162	1.162×10^{-2}	2.778×10^{-3}	1.000	0.6720	8.064
1 $Btu \cdot h^{-1} \cdot ft^{-1} \cdot °F^{-1}$ =	1.730	1.730×10^{-2}	4.134×10^{-3}	1.488	1.000	12.00
1 $Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$ =	0.1441	1.441×10^{-3}	3.445×10^{-4}	0.1240	8.333×10^{-2}	1.000

Thermal Resistance[A]

	$K \cdot m^2 \cdot W^{-1}$ ([B])	$K \cdot cm^2 \cdot W^{-1}$	$K \cdot cm^2 \cdot s \cdot cal^{-1}$	$K \cdot m^2 \cdot kg\text{-}cal^{-1}$	$°F \cdot ft^2 \cdot h \cdot Btu^{-1}$
1 $K \cdot m^2 \cdot W^{-1}$ =	1.000	1.000×10^{4}	4.184×10^{4}	1.162	5.674
1 $K \cdot cm^2 \cdot W^{-1}$ =	1.000×10^{-4}	1.000	4.184	1.162×10^{-4}	5.674×10^{-4}
1 $K \cdot cm^2 \cdot s \cdot cal^{-1}$ =	2.390×10^{-5}	0.2390	1.000	2.778×10^{-5}	1.356×10^{-4}
1 $K \cdot m^2 \cdot h \cdot kg\text{-}cal^{-1}$ =	0.8604	8.604×10^{3}	3.600×10^{4}	1.000	4.882
1 $°F \cdot ft^2 \cdot h \cdot Btu^{-1}$ =	0.1762	1.762×10^{3}	7.373×10^{3}	0.2048	1.000

[A] Units are given in terms of (1) the absolute joule per second or watt, (2) the calorie (thermochemical) = 4.184 J, or the British thermal unit (thermochemical) = 1054.35 J.

[B] This is the SI (International System of Units) unit.

TABLE A1.2b Conversion Factors (International Table)

NOTE—Conversion factors for thermal resistivity and thermal conductance can be found by using these tables in the reverse direction.

Thermal Conductivity[A]

	$W \cdot m^{-1} \cdot K^{-1}$ ([B])	$W \cdot cm^{-1} \cdot K^{-1}$	$cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}$	$kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}$	$Btu \cdot h^{-1} \cdot ft^{-1} \cdot °F^{-1}$	$Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$
1 $W \cdot m^{-1} \cdot K^{-1}$ =	1.000	1.000×10^{-2}	2.388×10^{-3}	0.8598	0.5778	6.933
1 $W \cdot cm^{-1} \cdot K^{-1}$ =	100.0	1.000	0.2388	85.98	57.78	693.3
1 $cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}$ =	418.7	4.187	1.000	360.0	241.9	2903.
1 $kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}$ =	1.163	1.163×10^{-2}	2.778×10^{-3}	1.000	0.6720	8.064
1 $Btu \cdot h^{-1} \cdot ft^{-1} \cdot °F^{-1}$ =	1.731	1.731×10^{-2}	4.134×10^{-3}	1.488	1.000	12.00
1 $Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$ =	0.1442	1.442×10^{-3}	3.445×10^{-4}	0.1240	8.333×10^{-2}	1.000

Thermal Resistance[A]

	$K \cdot m^2 \cdot W^{-1}$ ([B])	$K \cdot cm^2 \cdot W^{-1}$	$K \cdot cm^2 \cdot s \cdot cal^{-1}$	$K \cdot m^2 \cdot h \cdot kg\text{-}cal^{-1}$	$°F \cdot ft^2 \cdot h \cdot Btu^{-1}$
1 $K \cdot m^2 \cdot W^{-1}$ =	1.000	1.000×10^{4}	4.187×10^{4}	1.163	5.678
1 $K \cdot cm^2 \cdot W^{-1}$ =	1.000×10^{-4}	1.000	4.187	1.163×10^{-4}	5.678×10^{-4}
1 $K \cdot cm^2 \cdot s \cdot cal^{-1}$ =	2.388×10^{-5}	0.2388	1.000	2.778×10^{-5}	1.356×10^{-4}
1 $K \cdot m^2 \cdot h \cdot kg\text{-}cal^{-1}$ =	0.8598	8.598×10^{3}	3.600×10^{4}	1.000	4.882
1 $°F \cdot ft^2 \cdot h \cdot Btu^{-1}$ =	0.1761	1.761×10^{3}	7.373×10^{3}	0.2048	1.000

[A] Units are given in terms of (1) the absolute joule per second or watt, (2) the calorie (International Table) = 4.1868 J, or the British thermal unit (International Table) = 1055.06 J. These tables are meant for use in Canada and Great Britain.

[B] This is the SI (International System of Units) unit.

A2.2.2.1 Where half thicknesses of the normal specimen can be used, it can be more rapid than Configuration a, especially when each of the specimens is first conditioned to the mean temperature at which it will operate in the test. The use of the low-resistance transducer in Configuration a is ideal if Plate 1 can provide both heating and cooling, but unless the temperature of Plate 1 is extremely well controlled, a damping layer will be required between the transducer and the plate. The advantage of the fast response time is then partially lost, but the response time is likely rapid enough in any event. The transducer can be mounted permanently in this configuration.

A2.3 *Sensitivity of Configurations to Edge Losses:*

A2.3.1 The Configuration a is similar to the guarded hot plate apparatus regarding edge heat losses through the specimen. The edge heat losses in the transducer may be much more significant than those in the guarded hot plate apparatus because they may produce errors due to the temperature non-uniformity on the side in contact with the specimen.

A2.3.2 The Configuration b is the most insensitive to edge conditions if the average of the readings of the two meters is assumed to be the measured heat flux per unit area through the specimen. If the plates were perfectly uniform in temperature, if the two transducers were exactly equal in the layout of the thermopile junctions, and if the specimen had thermal conductivity independent of temperature, this configuration would be insensitive to edge conditions. However, even under these ideal conditions the use of Configuration b does not eliminate edge losses but only reduces the apparatus sensitivity to variations in the boundary temperatures. Also, the settling time is short if the average of the two readings is used.

A2.3.3 Configuration c is very sensitive to edge heat losses on the heat flux transducer as the power that flows through the edges is supplied, not by a heavy nearly isothermal metal plate, but by the specimens, so that their surface temperatures may not be uniform. If the transducer is sensitive to temperature differences along its main surfaces, edge heat

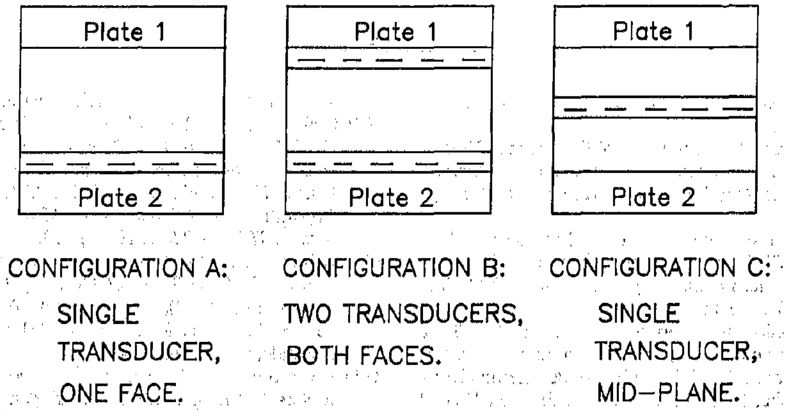

FIG. A2.1 Typical Heat Flow Meter Apparatus Configurations

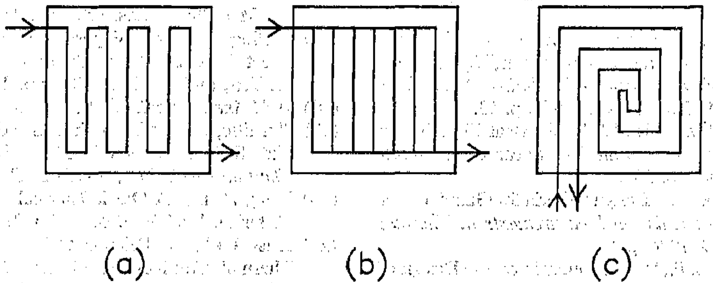

FIG. A2.2 Some Layouts of the Liquid Paths in Heating or Cooling Plates

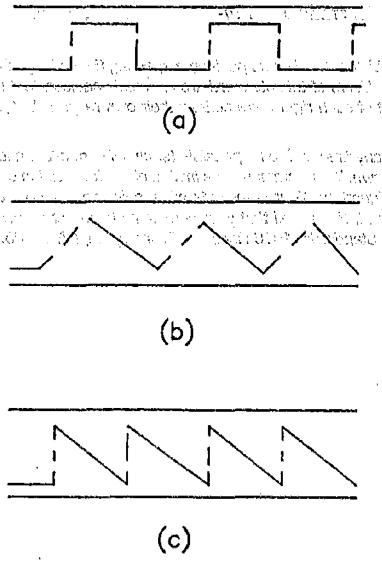

FIG. A2.3 Schematic Designs of Transducers

losses may now create serious errors. Edge heat losses within the specimens are similar to those in the guarded hot plate when the surrounding temperature is that of the hot or cold plate.

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CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
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By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

* * *

Document Name: ASTM C549: Standard Specification for Perlite Loose Fill Insulation

CFR Section(s): 10 CFR 440 Appendix A

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: C 549 – 81 (Reapproved 1995)$^{\epsilon 1}$

Standard Specification for Perlite Loose Fill Insulation[1]

This standard is issued under the fixed designation C 549; 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.

ϵ^1 Note—Keywords were added editorially in May 1995.

1. Scope

1.1 This specification covers the composition and physical properties of expanded perlite loose fill insulation. The specification also includes the testing procedures by which the acceptability of the material may be determined. These testing procedures deal primarily with material performance in the temperature range associated with the thermal envelope of buildings; however, the commercially usable temperature range for this insulation is from − 459 to 1400°F (1 to 1033 K). For specialized applications, refer to the manufacturer's instructions.

1.2 The specification also covers the composition and properties of perlite that has been surface-treated to produce water repellency and dust suppression for installations where liquid moisture and dust may be a factor.

1.3 *When the installation or use of thermal insulation materials, accessories and systems, may pose safety or health problems, the manufacturer shall provide the user appropriate current information regarding any known problems associated with the recommended use of the company's products, and shall also recommend protective measures to be employed in their safe utilization. The user shall establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.* For additional precautionary statements, see Section 11.

1.4 The following precautionary caveat pertains to the test methods portion, Section 8, of this specification. *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates[2]

C 168 Terminology Relating to Thermal Insulating Materials[3]

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded Hot Plate Apparatus[3]

C 390 Criteria for Sampling and Acceptance of Preformed Thermal Insulation Lots[3]

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

C 520 Test Methods for Density of Granular Loose Fill Insulations[3]

E 84 Test Method for Surface Burning Characteristics of Building Materials[4]

E 136 Test Method for Behavior of Materials in a Vertical Tube Furnace at 750°C[4]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[5]

2.2 *Federal Specification:*

HH-I-515D Insulation Thermal (Loose Fill for Pneumatic or Poured Application) Cellulosic or Wood Fiber[6]

3. Classification

3.1 Perlite loose fill insulation shall be specified by four type designations as follows:

3.1.1 *Type I*—The product that results from the expanding of natural perlite ore by grading and heating to meet the requirements of this specification.

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[1] This specification is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.23 on Blanket and Loose Fill Insulation.

Current edition approved Oct. 30, 1981. Published February 1982. Originally published as C 549 – 64 T. Last previous edition C 549 – 73 (1979).

[2] *Annual Book of ASTM Standards*, Vol 04.02.

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

[4] *Annual Book of ASTM Standards*, Vol 04.07.

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

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

3.1.2 *Type II*—Expanded perlite that has been surface-treated to produce water repellency and limited absorption of moisture from both liquid and vapor phase.

3.1.3 *Type III*—Expanded perlite that has been surface-treated to limit the amount of dust generated during application.

3.1.4 *Type IV*—Expanded perlite that has been surface-treated to produce water repellency and to limit the dust generated during application.

4. Ordering Information

4.1 All purchase orders should designate the type of insulation desired. The type and grade classification in this specification differ from the classification in earlier issues. Purchasers referencing this specification should include the date of issue.

5. Materials and Manufacture

5.1 Perlite is a generic term for a naturally occurring siliceous mineral which is mined, crushed, sieved and dried to produce a crude perlite ore. The crude ore, in the form of particles of varying size, weighing 60 to 75 lb/ft^3 (960 to 1200 kg/m^3), is expanded in high-temperature furnaces to densities in the range from 2.0 to 11.0 lb/ft^3 (32 to 176 kg/m^3). As a naturally occurring mineral, it is classified as an elementary building material. It is noncombustible as determined by Test Method C 136.

6. Physical Requirements

6.1 The physical requirements listed in this section are defined as Inspection Requirements (see Criteria C 390). The insulation shall conform to the following requirements:

		All Types
Bulk density, lb/ft^3(kg/m^3)		2–11 (32–176)
Grading (particle size):		
Cumulative percentage retained, maximum (by weight), Sieve No. 4 (4.75 mm), %		5
	Types I and III	Types II and IV only
Water repellency, min mL of water repelled	N.A.	175
Small-scale fire test	no sparking or ignition	

6.2 The physical requirements listed in this section are defined as Qualification Requirements (see Criteria C 390). The insulation shall conform to the following requirements:

		All Types
Thermal resistance, h·ft^2·°F/Btu (m^2·K/W)		See Table 1
Moisture absorption, weight %/14 days, max, %		1.0
	Types I and III	Types II and IV
Wickability, max g wicked in 5 min	N.A.	1.0
Combustibility (by Test Method E 136)	noncombustible	N.A.[A]
Surface burning characteristics:		
Flame spread, max	0	25
Smoke developed, max	0	50
Dust suppression:		
Weight of collected material, max, mg	N.A.	85

[A] Federal Standards for attic floor insulation require conformance to two non-ASTM tests for combustibility: Critical Radiant Flux (HH-I-515D, 4.8.7) and Smoldering Combustion (HH-I-515D, 4.8.8). Types III and IV perlite insulation, when tested for Critical Radiant Flux, showed no ignition or flame front advance at a flux of 1.07 W/cm^2. When tested for Smoldering Combustion, Types III and IV perlite insulation showed no flaming combustion and 0 % weight loss.

7. Sampling

7.1 For purposes of standard tests, sampling shall be in accordance with Criteria C 390.

8. Test Methods

8.1 *Bulk Density*—Test Methods C 520, Method A.

8.2 *Grading*—Test Method C 136, except that when a mechanical sieving device is used, the sieving time shall be 5 min and the test sample shall be 50 g of material.

8.3 *Water Repellency* (Types II and IV only):

8.3.1 *Apparatus*:

8.3.1.1 *Rigid Plastic Tube*, 50 mm inside diameter by 300 mm long with a 150-μm (100-mesh) screen covering firmly fastened or adhered to the bottom. The tube shall be marked at 400 mL from the screen-covered end.

8.3.1.2 *Rubber Stopper*, No. 15.

8.3.1.3 *Graduated Cylinder*, 250-mL.

8.3.1.4 *Beaker*, 500-mL.

8.3.2 *Sample Preparation*:

8.3.2.1 Spoon a representative sample into the test cylinder to a level slightly above the 400-mL mark.

8.3.2.2 Compact it by dropping the tube from a height of approximately 75 mm on a large rubber stopper (No. 15 recommended) for a total of ten drops. As the sample compacts to a level below the 400-mL mark, add additional material so that after the tenth drop the level of the sample is within 3 mL of the 400-mL mark.

8.3.3 *Procedure*:

8.3.3.1 With the tube supported in a vertical position and a beaker positioned under the tube, pour 250 mL of cold tap water rapidly onto the perlite. Take care while pouring, that the stream hits the middle of the surface of the bed of perlite and does not merely slide down the side of the test cylinder.

8.3.3.2 Allow the water to drain through the bed of perlite for exactly 3 min. Tilt the tube at approximately 45° to drain water collected on the screen. Tilt it only momentarily for this purpose.

8.3.3.3 Measure the collected water in the 250-mL graduate.

8.3.4 *Calculations*:

8.3.4.1 Report the amount of collected water in millilitres as "millilitres repelled."

8.3.4.2 Report results as the mean of three independent tests.

8.3.5 *Precision and Bias*—The purpose of this test is to confirm that the intended degree of surface treatment has been accomplished to achieve water repellency. The specified characteristic is a minimum (no range or maximum). Quantitative values for conforming products exceeding the limit have no commercial significance.

8.4 *Small-Scale Fire Test*:

8.4.1 *Scope*—The purpose of this quality control test is to confirm that excessive coating or organic material has not been added during processing.

8.4.2 *Significance*—This test is intended as a means to determine the resistance of the material to heat and flame under controlled conditions and should not be used to describe or appraise the fire hazard or fire risk under actual conditions.

TABLE 1 Thermal Resistance

Density, lb/ft^3(kg/m^3)	All Types		
	Thermal Resistance for 1-In. (0.0254-m) Thickness, h·ft^2·°F/Btu (m^2·K/W)[A]		
	Mean Temperature,° F (°C)		
	40 (4)	75 (24)	105 (41)
2.0–4.1 (32.0–65.6)	4.3–3.9 (0.78–0.69)	3.7–3.3 (0.65–0.58)	3.7–3.2 (0.65–0.56)
4.1–7.4 (65.6–118.4)	3.9–3.3 (0.69–0.58)	3.3–2.8 (0.58–0.49)	3.2–2.7 (0.56–0.47)
7.4–11.0 (118.4–176.0)	3.3–2.9 (0.58–0.51)	2.8–2.4 (0.49–0.42)	2.7–2.4 (0.47–0.42)

[A] The thermal values in this table are given at 1 in. (0.0254 m) of thickness with a gradient of 50°F (10°C). Test data that cover the thickness range from 1 to 6 in. (0.0254 to 0.1524 m) show that thermal resistance per inch of thickness is essentially linear with respect to thickness. Test values do not deviate on the low side from the value in the table by more than 5 %.

8.4.3 *Procedure*—Hold a small sample (such as 10 to 20 gr) of the perlite insulation in a suitable fixture (such as 6 by 6 by 2-in. deep wire mesh basket constructed from (150 μm 100-mesh) stainless steel woven wire cloth) and positioned in direct contact with the flame of a bunsen burner for about 20 s. Sparking or ignition indicates failure to conform to the requirements of 6.1

8.4.4 *Precision and Bias*—No statement is made about either the precision or the accuracy of the small-scale fire test for determining resistance of the perlite insulation to heat and flame since the result merely states whether there is conformance to the criteria for success specified in the procedure.

8.5 *Thermal Resistance*—Tests of thermal resistance may be made in accordance with Test Method C 177 or C 518. Testing shall be done at the design density. The thermal resistance of the various types shall not be lower than the values listed in Table 1, except that the average thermal resistance of any four specimens measured in accordance with 8.5, may fall up to 5 % below the value in the table. The thermal resistance (*R*-value) shall be determined at mean temperature of 75°F (24°C) and 40°F (4°C) at design density and in accordance with the current editions of Test Methods C 177 and C 518. Report the direction of heat flow. Thermal resistance at other mean temperatures may be determined if required.

8.6 *Moisture Absorption*—The test specimen shall be a sample of approximately 50 g. Loose fill and level the sample into a sample holder 9 by 9 by 5 in. (228 by 228 by 127 mm) deep.

8.6.1 Precondition in accordance with Test Method C 177 or C 518. Conditioning shall be with minimum air movement across the sample surface. Condition at 50 ± 2 % relative humidity and 120°F (49°C) to constant weight and record. State the density of the sample conditioned to constant weight in the report of results.

8.6.2 Increase the relative humidity to 90 ± 2 %. Condition to constant weight by check-weighing at 24-h intervals. Determine the moisture pickup as a percent of conditioned weight.

8.6.3 Record percent absorption at 14 days.

8.6.4 *Precision and Bias*—This test establishes a typical property of perlite. It is an inherent characteristic. It is only intended to indicate absorption under high humidity conditions which are known to be characteristic of its usual insulation end use.

8.7 *Wickability* (Types II and IV only):

8.7.1 *Apparatus*:

8.7.1.1 *Rigid Plastic Tube*, 50 mm inside diameter by 300 mm long with a 150-μm (100-mesh) screen covering firmly fastened or adhered to the bottom.

8.7.1.2 *Compaction Plug*, 50 mm long, fitting snugly inside the sample holder to give standard compaction to the sample.

8.7.1.3 *Tubing Rack*, capable of supporting the sample tube.

8.7.1.4 *Water Immersion Tray*.

8.7.2 *Sample Preparation*:

8.7.2.1 Spoon a representative sample into the test cylinder to a level slightly below the top of the cylinder.

8.7.2.2 Compact it with the plug until a moderate resistance pressure is felt. In no event insert the compaction plug into the sample holding tube more than 24 mm.

8.7.3 *Procedure*:

8.7.3.1 Place the sample tube screen end down, on a rack in the water immersion tray. The water level in the tray will be 50 mm above the bottom of the tube.

8.7.3.2 Sample shall be in this position and allowed to wick water for 5 min.

8.7.3.3 Remove the tube from the tray onto a tared 75-mm watch glass so that all water that is allowed to drain is caught by the watch glass and is included in the weight of water picked up by the sample.

8.7.3.4 Determine the weight of the water picked up by the sample in grams.

8.7.4 *Calculations*:

8.7.4.1 Report the amount of water picked up by the perlite sample through this wicking action as grams.

8.7.4.2 Report results as the mean of three independent tests.

8.7.5 *Precision and Bias*—The purpose of this test is to ascertain the resistance of the material to wicking action of water. The specified characteristic is a maximum (no range or minimum). Quantitative values for conforming products below the limit have no commercial significance.

8.8 *Combustibility* of Types I and II perlite insulation may be determined in accordance with Test Method E 136. Combustibility of Types III and IV insulation may be determined in accordance with tests on Critical Radiant Flux and Smoldering Combustion as specified in Federal Specification HH-I-515D.

8.9 *Surface Burning Characteristics* of perlite insulation may be determined in accordance with Test Method E 84.

8.10 *Dust Suppression* (Types III and IV only):

8.10.1 *Apparatus*:

8.10.1.1 *Glass Plate*, 16½ by 16½ by ¼ in. (420 by 420 by 6.4 mm).

8.10.1.2 *Clear Methacrylate Sheet Box*, open bottom, 15½ by 15½ by 19⅝ in. (400 by 400 by 500 mm) high with a hole in the center of the top 2¹⁄₁₆ in. (52.4 mm) in diameter.

8.10.1.3 *Plastic Tube*, 2 in. outside diameter by 1¾ in. inside diameter (50 by 45 mm), 17¾ in. long (450 mm), with a rubber washer such that the washer snugly fits the outside diameter of the plastic tube and has a larger outside diameter than the hole in the box.

8.10.1.4 *Beaker*, 2000-mL.

8.10.1.5 *Laboratory Funnel* with a minimum nozzle inside diameter of 1 in. (25.4 mm).

8.10.1.6 *Graduated Cylinder*, 100-mL.

8.10.1.7 *Small Brush.*

8.10.1.8 *Scale* capable of measuring perlite material to 0.001 g.

8.10.2 *Sample Preparation*—Spoon a representative sample into the graduated cylinder to the 1000-mL level.

8.10.3 *Procedure*:

8.10.3.1 Place the glass plate flat on a suitable work surface.

8.10.3.2 Place the 2000-mL beaker in the exact center of the glass plate.

8.10.3.3 Place the plastic box on the glass plate such that the box is centered on the plate and hole in top of the box is centered directly over the 2000-mL beaker.

8.10.3.4 Insert the plastic pipe into the hole in the top of the box such that it protrudes down into the box such that the clear vertical distance from the bottom of the tube to the top of the 2000-mL beaker is 1³⁄₁₆ in. (30 mm). The tube is supported in the vertical position by the rubber washer.

8.10.3.5 Place the laboratory funnel into the plastic tube.

8.10.3.6 Pour 1000 mL of test material into the funnel such that it empties of all material in 10 s. It is the intent to have a uniform stream of material falling into the box for 10 s.

8.10.3.7 After all visible material has settled onto the glass plate, carefully remove the plastic box and gently sweep the material that has collected on the glass plate into a pile, remove it, and determine its weight.

8.10.4 *Calculations*:

8.10.4.1 Report the amount of test material in milligrams as "milligrams collected."

8.10.4.2 Report results as the average of five or more independent tests with no single reading over 100 mg with separate samples taken from the same source.

8.10.5 *Precision and Bias*—The single-specimen, single-operator, single-day precision is a standard deviation of ±0.02 g (1S) maximum for the dust-suppressed material over a range from 0.01 to 0.08 g as defined in Practice E 177.

9. Inspection

9.1 Inspection of the materials shall be made as agreed upon by the purchaser and the manufacturer as part of the purchase contract.

10. Packaging and Marking

10.1 *Packaging*—Unless otherwise specified, the insulation shall be packed in the manufacturer's standard commercial container. The insulation shall be packed to ensure carrier acceptance and safe delivery at destination in containers complying with the rules and regulations applicable to the mode of transportation.

10.2 *Marking*—Shipping containers shall be marked with the name of the insulation, minimum weight of container, and the name of the manufacturer. For residential insulation, a chart shall also be affixed or printed on the container listing the minimum thickness, maximum net coverage area, and maximum weight per square foot at minimum *R* values of 11, 19, and 22. In addition to this chart, the following statements must be added: 1. "*R* means resistance to heat flow. The higher the *R*-value, the greater the insulating power." 2. "To get the marked *R*-value, it is essential that this insulation be installed properly. If you do it yourself, follow the instructions carefully."

11. Health and Safety Precautions

11.1 *Preinstallation*—The insulation material should be handled and stored in accordance with manufacturer's instructions. It should be kept dry and free of extraneous materials.

11.2 *Installation*:

11.2.1 The insulating material should be poured into the spaces and cavities to be insulated in a manner that minimizes free-fall and impact. This will minimize crushing and breakdown of insulation particles and unnecessary formation of dust.

11.2.2 Perlite loose fill insulation that may be installed in confined, poorly ventilated attic spaces may generate a buildup of airborne nuisance dust. Avoid creating this nuisance dust. The use of respiratory and eye protection may be necessary in some applications. Refer to manufacturer's instructions regarding recommended installation practices.

12. Keywords

12.1 dust suppression; insulation; loose fill; perlite; water repellency

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

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WASHINGTON, D.C.

Designation: C 564 – 70 (Reapproved 1982)

Endorsed by the
Cast Iron Soil Pipe Institute

Standard Specification for
RUBBER GASKETS FOR CAST IRON SOIL PIPE AND FITTINGS[1]

This standard is issued under the fixed designation C 564; 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 specification has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This specification covers performed rubber gaskets used to seal joints in cast iron soil pipe and fittings.

1.2 The values stated in inch-pound units are to be regarded as the standard.

2. Materials and Manufacture

2.1 Gaskets shall be made of a properly vulcanized virgin rubber compound containing no scrap or reclaim.

2.2 If a splice occurs in the gasket, the strength of the splice shall be such that the gasket will withstand the stretch test described in 7.9 with no visible separation or peeling.

3. Physical Requirements

3.1 Sample gaskets selected as specified in Section 6 shall conform to the requirements for physical properties listed in Table 1 when tested in accordance with the methods specified in Section 7.

4. Dimensions and Permissible Variations

4.1 Gaskets shall conform to the dimensions specified by the manufacturer of the pipe in which they are to be used.

4.2 All cross-sectional dimensions shall have an RMA Class 3 tolerance as shown in the appendix, and all diametral dimensions shall have a tolerance of ± 1 percent unless otherwise mutually agreed by the pipe manufacturer and the gasket supplier.

5. Workmanship

5.1 The surface of the gasket shall be smooth and free of pitting, cracks, blisters, air marks, and any other imperfections that will affect its behavior in service. The body of the gasket shall be free of porosity and air pockets.

5.2 Neither the flash thickness nor the flash extension shall exceed $\frac{1}{32}$ in. (1 mm), at any point on the ring.

5.3 The offset, or failure of the mold to register accurately, shall not exceed $\frac{1}{64}$ in. (0.5 mm).

6. Sampling

6.1 For the stretch test specified in 7.9, sample gaskets shall be selected at random from each shipment of gaskets, in accordance with Table 2. For each of the other tests, gaskets shall be selected at random as required by the method of test specified.

7. Test Methods

7.1 *Hardness*—Test for hardness in accordance with ASTM Method D 2240, Test for Rubber Property—Durometer Hardness.[2] ASTM Method D 1415, Test for Rubber Property—International Hardness,[2] shall be used as the referee method. Make hardness measurements on specimens prepared in accordance with 7.2. However, hardness readings for guidance purposes may be taken directly on the gasket, recognizing that these may vary slightly from those taken on the dumb-bell specimens.

7.2 *Elongation and Tensile Strength*—Test for elongation and tensile strength in accordance with ASTM Method D 412, Tests for Rubber Properties in Tension[3] Cut standard

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[1] This specification is under the jurisdiction of ASTM Committee C-24 on Building Seals and Sealants.

Current edition effective May 29, 1970. Originally issued 1965. Replaces C 564 – 68.

[2] *Annual Book of ASTM Standards*, Vol 09.01.

[3] *Annual Book of ASTM Standards*, Vols 09.01 and 09.02.

ASTM Type C dumbbell specimens conforming to Fig. 1 of Method D 412 from sections of the gasket for this test. To obtain a uniform thickness, these sections may be buffed prior to cutting into dumbbell specimens, so as to produce a finely ground surface without cuts or burns.

7.3 *Tear Strength*—Test for tear strength in accordance with ASTM Method D 624, Test for Rubber Property—Tear Resistance,[2] using Die C.

7.4 *Compression Set*—Test for compression set in accordance with ASTM Methods D 395, Tests for Rubber Property—Compression Set,[2] using Method B. Oven age specimens 22 h at 158 ± 2 F (70 ± 1 C). Where plied specimens are necessary, the results shall comply with the requirements of Table 1.

7.5 *Heat Aging*—Test for effects of heat aging in accordance with ASTM Method D 573, Test for Rubber Deterioration in an Air Oven.[2] Prepare specimens in accordance with 7.2, and age for 96 h at 158 ± 2 F (70 ± 1 C). Hardness measurements shall be made as specified in 7.1.

7.6 *Water Absorption*—Test for weight increase due to water absorption in accordance with ASTM Method D 471, Test for Rubber Property—Effect of Liquids.[2] If a 1-in. (25.4-mm) specimen cannot be cut from the sample gasket, use the greatest width obtainable. Immerse the test specimen in distilled water at 158 ± 2 F (70 ± 1 C) for 7 days.

7.7 *Ozone Resistance*—Test for ozone resistance in accordance with ASTM Method D 1149, Test for Rubber Deterioration- Surface Ozone Cracking in a Chamber (Flat Specimens),[2] using specimens and procedure specified under Method B. The ozone concentration shall be 150 parts/100,000,000 of air by volume. Age specimens 100 h at 104 F (40 C). Use a two-power hand glass to examine the gasket for cracks.

7.8 *Oil Immersion*—Test for volume decrease due to oil absorption in accordance with Method D 471. If a 1-in (25.4-mm) specimen cannot be cut from the sample gasket, the greatest width obtainable shall be used. The test specimen shall be immersed in ASTM Oil No. 3 for 70 h at 212 ± 2 F (100 ± 1 C).

7.9 *Stretch Test for Spliced Gaskets*—Stretch gaskets until the circumference is increased 50 percent, then visually inspect for defects as described in 2.2 and 5.1. The number of gaskets to be examined and the maximum number of defective gaskets for acceptance of the lot is shown in Table 2.

8. Certification

8.1 Upon request of the purchaser, the manufacturer shall be prepared to certify that his product conforms to the requirements of this specification.

9. Marking

9.1 Mark each gasket with clearly legible letters not exceeding ¼ in. (6.35 mm) in height. These markings shall include the gasket manufacturer's name or symbol, the pipe size and class, the year of manufacture, and the ASTM specification number.

TABLE 1 Physical Requirements of Gaskets

Property	Requirements			ASTM Test Method
Hardness (nominal durometer ±5) as specified by the pipe manufacturer	50	60	70	D 2240
Elongation, min, percent	350	300	250	D 412
Tensile strength, min, psi	1500	1500	1500	D 412
(MPa)	(10)	(10)	(10)	
Tear strength, min, lbf/in.	150	150	150	D 624
(N/cm)	(268)	(268)	(268)	
Compression set, max, percent	25	25	25	D 395
Heat aging, 96 h at 158 ± 2 F (70 ± 1 C):				D 573
Hardness increase, max, durometer points	10	10	10	. . .
Loss in tensile strength, max, percent	15	15	15	. . .
Loss in elongation, max, percent	20	20	20	. . .
Water absorption:				D 471
Weight increase, max, percent	20	20	20	. . .
Ozone resistance	no cracks	no cracks	no cracks	D 1149
Oil immersion:				
Volume increase, max, percent	80	80	80	D 471

TABLE 2 Sampling Plan for Stretch Test for Visual Inspection

Number of Gaskets in Shipment	Number of Gaskets in Sample	Maximum Number of Defectives for Acceptance
Up to 800	75	4
801 to 3200	150	8
3201 to 8000	225	11
8001 to 22 000	300	14

APPENDIX

X1. RUBBER MANUFACTURERS ASSOCIATION, INC. TOLERANCES

TABLE X1 RMA Class 3 Dimensional Tolerances (Commercial Tolerances)

Size, in.	Fixed[A]	Closure[B,C]
0 to 0.499	±0.010	±0.015
0.500 to 0.999	±0.010	±0.018
1.000 to 1.999	±0.015	±0.020
2.000 to 2.999	±0.020	±0.025
3.000 to 3.999	±0.025	±0.030
4.000 to 4.999	±0.030	±0.035
5.000 to 7.999	±0.035	±0.050
Greater than 8.000	multiply by	0.0050

[A] Fixed dimensions are those which are parallel to the mold parting line or major mold sections and which are not affected by flash thickness variations. Tolerances apply individually to each fixed dimension according to its own size.

[B] Closure dimensions are those vertical to the mold parting line or parting lines of major sections and are affected by flash thickness variation.

[C] The tolerance on closure dimensions is that tolerance for the largest closure dimension. This tolerance is then applied to all other closure dimensions.

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CERTIFICATE

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Document Name: ASTM D1217: Standard Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer

CFR Section(s): 40 CFR 75, Appendix D

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 1217 – 93 (Reapproved 1998)

Standard Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[1]

This standard is issued under the fixed designation D 1217; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the density of pure hydrocarbons or petroleum distillates boiling between 90 and 110°C that can be handled in a normal fashion as a liquid at the specified test temperatures of 20 and 25°C.

1.2 This test method provides a calculation procedure for conversion of density to relative density (specific gravity).

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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 1, Note 2, and Note 3.

2. Referenced Documents

2.1 *ASTM Standards:*

E 1 Specification for ASTM Thermometers[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *density*—the weight in vacuo, (that is, the mass) of a unit volume of the material at any given temperature.

3.1.2 *relative density (specific gravity)*—the ratio of the mass (weight in vacuo) of a given volume of material at a temperature, t_1, to the mass of an equal volume of water at a reference temperature, t_2; or it is the ratio of the density of the material at t_1 to the density of water at t_2. When the reference temperature is 4.00°C, the temperature at which the relative density of water is unity, relative density (specific gravity) and density are numerically equal.

4. Summary of Test Method

4.1 The liquid sample is introduced into a pycnometer, equilibrated to the desired temperature, and weighed. The relative density (specific gravity) or density is then calculated from this weight and the previously determined weight of water that is required to fill the pycnometer at the same temperature, both weights being corrected for the buoyancy of air.

5. Significance and Use

5.1 Density is a fundamental physical property which can be used in conjunction with other properties to characterize pure hydrocarbons and their mixtures.

5.2 This test method was originally developed for the determination of the density of the ASTM Knock Test Reference Fuels *n*-heptane and *iso*octane, with an accuracy of 0.00003 g/mL. Although it is no longer employed extensively for this purpose, this test method is useful whenever accurate densities of pure hydrocarbons or petroleum fractions with boiling points between 90 and 110°C are required.

6. Apparatus

6.1 *Pycnometer*, Bingham-type,[3] conforming to the dimensions given in Fig. 1, constructed of borosilicate glass and having a total weight not exceeding 30 g.

6.2 *Constant-Temperature Bath*, provided with suitable pycnometer holders or clips and means for maintaining temperatures constant to ±0.01°C in the desired range.

6.3 *Bath Thermometer*, graduated in 0.1°C subdivisions and standardized for the ice point and the range of use to the nearest 0.01°C. ASTM Saybolt Viscosity Thermometer 17C as prescribed in Specification E 1, designed for tests at 21.1°C and 25°C, is recommended. A standardized platinum resistance thermometer may also be used, and offers the best means for observing minute temperature changes in the bath. Whichever means are available, it must be realized that for most hydrocarbons the density coefficient is about 0.0008 units/°C, and therefore an error of ±0.013°C would cause an error of ±0.00001 in density.

6.4 *Hypodermic Syringe*, 30-mL capacity, of chemically resistant glass, equipped with a 152-mm (6-in.) needle made of stainless steel tubing as shown in Fig. 2.

6.5 *Draw-Off Needle*, made of stainless steel tubing as shown in Fig. 2.

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[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.04on Hydrocarbon Analysis.

Current edition approved Feb. 15, 1993. Published May 1993. Originally published as D 1217 – 52 T. Last previous edition D 1217 – 91.

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

[3] Pycnometer available from Reliance Glass Co., 220 Gateway Rd., Bensenville, IL 60106-0825, has been found satisfactory.

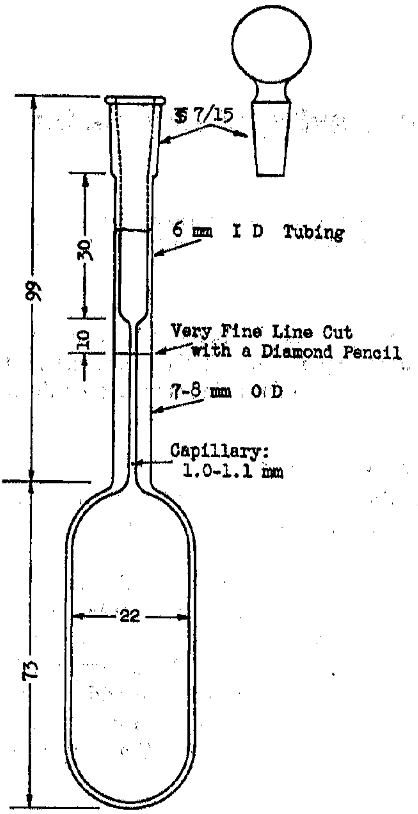


FIG. 1 Bingham-Type Pycnometer, 25 mL

6.6 *Solvent-Cleaning Assembly*, as shown in Fig. 3.

6.7 *Chromic Acid Cleaning Apparatus*, similar to that shown in Fig. 4.

6.8 *Balance*, capable of reproducing weighings within 0.1 mg. Mechanical balances should have sensitivity which causes the pointer to be deflected 2 or 3 scale divisions per 1 mg when carrying a load of 30 g or less on each pan. The balance should be located in a room shielded from drafts and fumes and in which the temperature changes between related weighings (empty and filled pycnometer) do not cause a significant change in the ratio of the balance arms. Otherwise weighings shall be made by the method of substitution, in which the calibrated weights and pycnometer are alternately weighed on the same balance pan. The same balance shall be used for all related weighings.

6.9 *Weights*, whose relative values are known to the nearest 0.05 mg or better. The same set of weights shall be used for the calibration of the pycnometer and the determination of densities.

7. Reagents and Materials

7.1 *Acetone*—(**Warning**—See Note 1).

NOTE 1—**Warning:** Extremely flammable. Use adequate ventilation.

7.2 *Isopentane*—(**Warning**—See Note 2).

NOTE 2—**Warning:** Extremely flammable. Avoid buildup of vapors and remove all sources of ignition, especially non-explosion proof electrical apparatus.

7.3 *Chromic Acid (Potassium Dichromate/Conc. Sulfuric Acid)*—(**Warning**—See Note 3).

NOTE 3—**Warning:** Causes severe burns. A recognized carcinogen. Do not get in eyes, or on skin or clothing.

8. Preparation of Apparatus

8.1 Thoroughly clean the pycnometer with hot chromic acid cleaning solution by means of the assembly shown in Fig. 4 (**Warning**—See Note 3). Chromic acid solution is the most effective cleaning agent. However, surfactant cleaning fluids have also been used successfully. Mount the apparatus firmly and connect the trap to the vacuum. Warm the necessary amount of cleaning acid in the beaker, place the pycnometer on the ground joint, and evacuate by opening the stopcock to vacuum. Fill the pycnometer with acid by turning the stopcock, repeat several times or remove the filled pycnometer, and allow it to stand for several hours at 50 to 60°C. Remove the acid from the pycnometer by evacuation, empty the acid from the trap, and flush the pycnometer with water. Cleaning should be made in this manner whenever the pycnometer is to be calibrated or whenever liquid fails to drain cleanly from the walls of the pycnometer or its capillary. Ordinarily, the pycnometer may be cleaned between determinations by washing with a suitable solvent, rinsing with pure, dry acetone, followed by isopentane, and vacuum drying.

8.2 Transfer the pycnometer to the cleaner assembly shown in Fig. 3, with vacuum line and trap attached to the side tube as indicated. Place the pycnometer on the cleaner with the upper hypodermic needle extending upward into the pycnometer, and press the edge of the ground joint on the rubber stopper until the vacuum holds it in place. Draw out all the liquid or sample. Immerse the lower end of the hypodermic tube in a suitable solvent and draw 20 to 25 mL through the pycnometer. Leaving the pycnometer in place, draw air through it until it is dry. Clean the hypodermic syringe with the same apparatus.

9. Calibration of Pycnometer

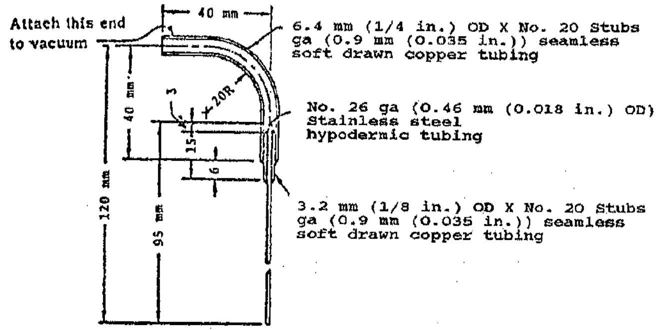
9.1 Proceeding as directed in Section 10, determine the weight of freshly-boiled and cooled distilled water (distilled from alkaline permanganate through a tin condenser) held by the pycnometer when equilibrated to volume at the bath temperature to be used in the determination. Repeat until at least three values agree to ±0.2 mg.

10. Procedure

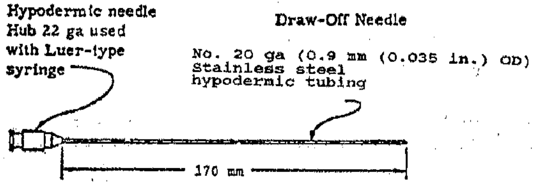
10.1 Using another 25-mL pycnometer as a tare (Note 4), weigh the clean, dry pycnometer to 0.1 mg and record the weight.

NOTE 4—It is convenient to use the lightest of a set of pycnometers as a tare. For best results the treatment and environment of both pycnometer and tare should be identical for some time prior to weighing.

10.2 Cool the sample to 5 to 10°C below the test temperature, and fill the clean 30-mL hypodermic syringe. Transfer the sample to the pycnometer through the filling needle; avoid trapping air bubbles (Note 2) in the bulb or capillary of the pycnometer. If any are present, draw them into the syringe where possible. Also remove with the syringe or draw-off



Draw-Off Needle



Filling Needle
To be used with a 30 ml Yale B-D Lok-Syringe
Becton-Dickinson and Co., Rutherford, N.J.

FIG. 2 Accessories for Bingham-Type Pycnometer

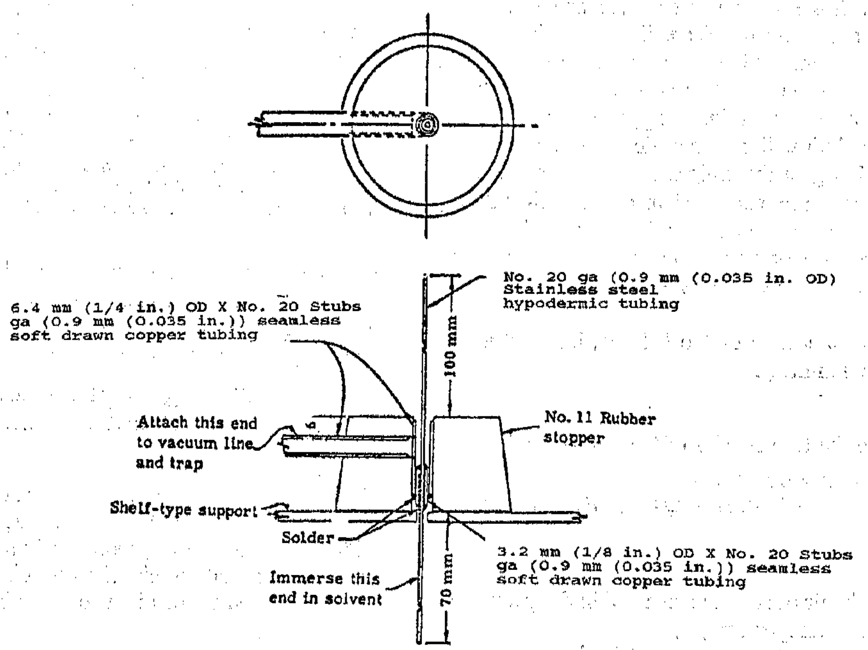


FIG. 3 Cleaner Assembly for Bingham-Type Pycnometer

needle any liquid above the calibration mark in the capillary or overflow reservoir. Dry the remainder with a cotton fiber pipe cleaner or cotton swab which has been dampened slightly with acetone.

NOTE 5—For work of highest accuracy on pure compounds, dissolved air may be removed from the sample by repeated freezing and remelting of the sample under vacuum in the pycnometer.

10.3 Close the pycnometer with the glass stopper and immerse it to a point above the calibration mark in the constant-temperature bath adjusted to a constancy of ±0.01°C at the desired temperature. Periodically, or before the liquid expands into the overflow chamber, remove the stopper, raise the pycnometer sufficiently to expose the calibration mark to view, and readjust the liquid level to the mark by withdrawing liquid through the steel draw-off needle until expansion has stopped, indicating that the liquid has reached the temperature of the thermostat. Do not allow the liquid to expand more than 10 mm above the calibration mark at any time, to minimize errors caused by faulty drainage. Allow the contents to equilibrate an additional 10 min and draw the level down exactly to

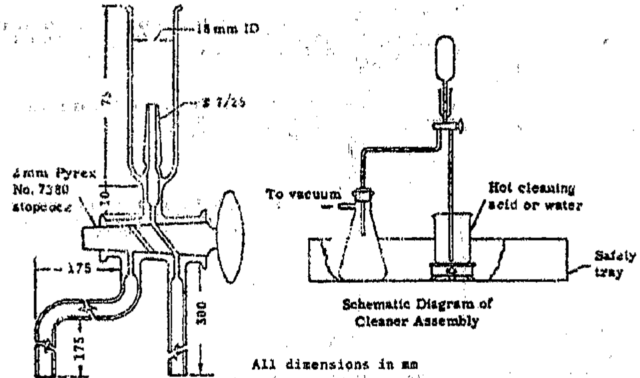


FIG. 4 All-Glass Pycnometer Cleaner Assembly for Use with Hot Chromic Acid Cleaning Solution

the calibration line, avoiding parallax and using a magnifier, if necessary, to obtain good visibility. Remove any liquid adhering to the walls above the calibration mark, with the draw-off needle or pipe cleaner, depending upon the volatility of the sample. Portions in the overflow bulb may be removed with a cotton swab moistened with acetone.

10.4 Replace the glass stopper, remove the pycnometer from the bath, wash the outside surface with acetone, and dry thoroughly with a chemically clean, lint-free, slightly damp cloth. Place the pycnometer in or near the balance case for 20 min and weigh to the nearest 0.1 mg. In atmospheres of low humidity (60 % or lower), drying the pycnometer by rubbing with a dry cotton cloth will induce static charges equivalent to a loss of about 1 mg in the weight of the pycnometer. This charge need not be completely dissipated in less than 30 min. The use of about 0.1-mg radium bromide- or polonium-coated foil in the balance case, or maintaining the relative humidity at 60 % or higher, aids in reducing weighing difficulties due to static charges.

10.5 Record temperature of the balance, barometric pressure, and relative humidity.

11. Calculation

11.1 Calculate the true density of the sample as follows:

$$\text{Density, g/mL at °C} = W_s(1 + (d_a/d_s) - (d_a/d_{wt}))d_w/W_w(1 + (d_a/d_w) - (d_a/d_{wt})) \quad (1)$$

where:

W_s = weight in air of sample contained in the pycnometer at the test temperature, g,

W_w = weight in air of the water contained in the pycnometer at the calibration temperature, g,

d_w = density of water at the calibration temperature, as obtained from Table 1,

d_a = density of air in balance case at the time of weighing, as calculated from 10.3,

d_{wt} = density of weights used in weighing the sample and water (brass = 10.4 g/mL, stainless steel = 7.75 g/mL), and

d_s = approximate density of sample, or

$$(W_s \times d)/W_w \quad (2)$$

11.2 The equation assumes that the weighings of the pycnometer empty and filled are made in such a short time interval that the air density has not changed. If significant change should occur, the calculated apparent weight of the sample, W_s, in this equation, must be corrected for the difference in air buoyancy exerted on the pycnometer as follows:

TABLE 1 Density of Water[A]

Temperature,° C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0	0.999840	21	0.997991	40	0.992212
3	0.999964	22	0.997769	45	0.990208
4	0.999972	23	0.997537	50	0.988030
5	0.999964	24	0.997295	55	0.985688
10	0.999699	25	0.997043	60	0.983191
15	0.999099	26	0.996782	65	0.980546
15.56	0.999012	27	0.996511	70	0.977759
16	0.998943	28	0.996231	75	0.974837
17	0.998774	29	0.995943	80	0.971785
18	0.998595	30	0.995645	85	0.968606
19	0.998404	35	0.994029	90	0.965305
20	0.998203	37.78	0.993042	100	0.958345

[A] Densities conforming to the International Temperature Scale 1990 (ITS 90) were extracted from Appendix G, *Standard Methods for Analysis of Petroleum and Related Products 1991*, Institute of Petroleum, London.

$$W_s = W^2{}_{PS} - W'_p(1 + (d'_a/2.2) - (d'_a/d_{wt}))/(1 + (d_a{}^2/2.2) - (d_a{}^2/d_{wt})) \quad (3)$$

where:

$W^2{}_{PS}$ = weight of pycnometer and contained sample under second or final air density,

W'_P = weight of pycnometer in air of first density,

d'_a = density of air when weighing empty pycnometer,

$d_a{}^2$ = density of air when weighing filled pycnometer, and

d_{wt} and 2.2 = density of weights and borosilicate glass, respectively.

Likewise, if the pycnometer, empty and filled with water for calibration, is weighed under different air densities a similar correction for different air buoyancies shall be applied.

11.3 Calculate the relative density (specific gravity) of the sample by dividing the density as obtained in 11.1 by the relative density of water at the reference temperature obtained from Table 1.

11.4 Calculate the density of air in the balance room as follows:

Air density (d_a), g/mL

$$= [(B - 0.3783\ Hp)(0.000465)]/(273 + t) \quad (4)$$

where:

B = barometric pressure, mm Hg, corrected to 0°C,
H = relative humidity, decimal fraction,
p = vapor pressure of water at temperature t, mm Hg, and
t = room temperature, °C.

NOTE 6—If this test method is to be used frequently, a considerable amount of calculation can be avoided by use of a gas density balance to determine the air density. Weigh a sealed 250-mL glass bulb at several different air densities and plot the weight against the air density. To determine the air density at some later time, weigh the bulb and read the air density from the point on the curve corresponding to the weight.

11.5 To calculate the density or relative density (specific gravity) at any test temperature, t, other than the calibration temperature, t_c(to correct for the cubical coefficient of thermal expansion of borosilicate glass), divide the value obtained in 10.1 or 10.2 by the following expression:

$$1 + 9.6 \times 10^{-6}\,(t - t_c) \quad (5)$$

12. Report

12.1 In reporting density, give the test temperature and the units (for example, density, 20°C = x.xxxxx g/mL). In reporting relative density (specific gravity), give both the test temperature and the reference temperature, but no units (for example, relative density (specific gravity), 20/4°C = x.xxxxx). Carry all calculations to one digit beyond the last significant figure, but report the final result to the fifth decimal place (0.00001).

13. Precision and Bias

13.1 *Precision*—Results, using the 25-mL Bingham-type pycnometer, should not differ from the mean by more than the following amounts:

Repeatability One Operator and Apparatus	**Reproducibility** Different Operators and Apparatus
0.00002	0.00003

NOTE 7—The precision for this method was not obtained in accordance with RR:D02-1007.

13.2 *Bias*—The difference of results from the established values when compared to pure reference materials is not expected to be more than ±0.00003 g/mL. Specific bias has not been established by cooperative testing.

14. Keywords

14.1 Density; pycnometer; relative density; specific gravity

CERTIFICATE

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By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

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Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 1246 – 95 (Reapproved 1999)

Standard Test Method for Bromide Ion in Water[1]

This standard is issued under the fixed designation D 1246; 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 is applicable to the measurement of bromide ion in water, ground water, and drinking water.

1.2 Samples containing 0.5 mg/L to 1000 mg/L of bromide may be analyzed by this test method. The concentration range may be extended by the dilution of an appropriate aliquot.

1.3 The precision and bias statements were determined on natural and ground waters. It is the responsibility of the analyst to assure the validity of this test method for untested matrices.

1.4 A titrimetric and two colorimetric test methods for iodide and bromide were discontinued. Refer to Appendix X1 for historical information.

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1066 Practice for Sampling Steam [2]
D 1129 Terminology Relating to Water [2]
D 1193 Specification for Reagent Water [2]
D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water [2]
D 3370 Practices for Sampling Water from Closed Conduits [2]
D 4127 Terminology Used with Ion-Selective Electrodes [2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminologies D 1129 and D 4127.

4. Summary of Test Method

4.1 Bromide ion is measured potentiometrically using a bromide ion-selective electrode in conjunction with a single-junction, sleeve-type reference electrode. Potentials are read on a pH meter having an expanded millivolt scale capable of being read to the nearest 0.1 mV, or a selective ion meter having a direct concentration scale for bromide. For less precise work, a pH meter having a millivolt scale capable of being read to the nearest 1.0 mV is adequate, however, no supporting data are available.

4.2 The electrodes are calibrated in known bromide solutions and the concentrations of unknowns are determined in solutions with the same background. In most cases, addition of an ionic strength adjustor to both standards and samples is sufficient to maintain constant background ionic strength. For samples above 0.1 *M* in ionic strength, prepare standard solutions similar to the sample composition.

5. Significance and Use

5.1 By analysis for bromide in water, wastewater, and brackish waters, it is possible to evaluate the origin of the water, its potential as a source of bromide, and its condition with regard to pollution.

6. Interferences

6.1 Strongly reducing solutions and solutions containing ions which form insoluble silver salts may coat the electrode membrane. These may be removed by polishing the membrane surface. Sulfide ion and cyanide ion both poison the electrode, and should be removed (see Section 11).

6.2 Halide ions form complexes with some metals. Since the electrode responds only to free bromide ions, the presence of any complexing agents lowers the measured concentrations. Concentrations of free metal ions causing a 10 % error of a bromide concentration of 8.1 mg/L are bismuth $^{+3}$, 80 mg/L; cadmium $^{+2}$, 100 mg/L; lead $^{+2}$, 1600 mg/L; tin $^{+2}$, 2400 mg/L; and thallium $^{+3}$, 4 mg/L.

6.3 Chloride ion and hydroxide ion do not interfere when present in the concentrations of up to 400 and 30 000 times the bromide concentration, respectively. There will be no interference from ammonia when present in concentrations twice that of bromide, nor from thiosulfate 20 times as concentrated as bromide. Iodide is an interference at a concentration ratio as low as 2×10^{-4}. Mercury should be absent from samples.

7. Apparatus

7.1 *pH Meter*, capable of reading to 0.1 mV, or a selective-ion meter.

7.2 *Bromide Ion-Selective Electrode.*

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[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved Dec. 10, 1995. Published February 1996. Originally published as D 1246 – 52. Last previous edition D 1246 – 88 (1995) $^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

7.3 *Sleeve-Type Single-Junction Reference Electrode*, filled with manufacturer's filling solution.

7.4 *Mixer*, magnetic, with a TFE fluorocarbon-coated stirring bar.

7.5 *Laboratory Glassware.*

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. [3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*— Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193, Type I. Other reagent water types may be used, provided it is first ascertained that the water is of sufficiently high purity to permit its use without adversely affecting the bias and precision of the test method. Type II water was specified at the time of round robin testing of this test method.

8.3 *Ionic Strength Adjustor* (42.5 g/100 mL $NaNO_3$)—In a 100-mL volumetric flask, dissolve 42.5 g of sodium nitrate ($NaNO_3$) in water and dilute to volume.

8.4 *Nickel Sulfate Solution, 1 M*—In a 100 mL volumetric flask dissolve 26.3 g of nickel sulfate hexahydrate ($NiSO_4 \cdot 6H_2O$) in water and dilute to volume.

8.5 *Sodium Bromide Standard Solution, 1000 mg/L*—In a 1 L volumetric flask dissolve 1.288 g dried sodium bromide in water and dilute to volume.

8.6 *Sodium Bromide Standard Solutions*, (100, 10, and 1 mg/L)—Dilute 1 volume of the 1000 mg/L bromide standard with 9 volumes of water to prepare the 100 mg/L standard. By further 1+9 serial dilutions, prepare the 10 and 1 mg/L standards.

9. Sampling

9.1 Collect the samples in accordance with Practice D 1066 and Practices D 3370, as applicable.

10. Calibration and Standardization

10.1 To 100 mL of the 1, 10, 100, and 1000 mg/L standards add 2 mL of the ionic strength adjustor (ISA). If any samples require treatment for interferences, prepare standards with the same background.

10.2 Connect and fill electrodes in accordance with manufacturer's instructions.

10.3 Transfer the 1 mg/L standard-ISA mixture to a 150 mL beaker and stir gently using the magnetic mixer. Immerse the electrodes in the solution and wait 2 min for the potential to stabilize. Record the value.

10.4 Rinse electrodes thoroughly and repeat for the 10, 100, and 1000 mg/L standard-ISA mixtures. Wait 2 min and record the potential.

10.5 The calibration curve is generated by plotting on semilogarithmic graph paper, the potential observed versus the concentration of the standard used. Note that volume corrections are incorporated into the calibration, so that samples analyzed according to Section 11 of this test method can be read directly.

11. Procedure

11.1 To any sample containing sulfide or cyanide ion, add 0.1 mL nickel sulfate solution to 100 mL sample.

NOTE 1—This concentration of nickel sulfate will react with 58 mg/L sulfide and 117 mg/L cyanide.

11.2 Transfer 100 mL sample to a 150 mL beaker and add 2 mL ionic strength adjustor. Stir thoroughly for 1 min using the magnetic mixer.

11.3 Immerse the electrodes in the sample-ISA mixture and wait 2 min for the potential to stabilize. Record the value.

11.4 Read bromide concentration of the sample, in mg/L, directly from the calibration curve. Note that volume corrections are incorporated into the calibration.

12. Precision and Bias [4]

12.1 *Precision*—The overall and single-operator precision of this test method may be expressed as follows:

12.1.1 *For Reagent Water:*

$$S_T = 0.077X + 1.10, \text{ coefficient of correlation} = 1.0$$
$$S_O = 0.067X + 0.79, \text{ coefficient of correlation} = 1.0$$

12.1.2 *For Water Matrices:*

$$S_T = 0.064X + 0.84, \text{ coefficient of correlation} = 1.0$$
$$S_O = 0.049X + 0.09, \text{ coefficient of correlation} = 1.0$$

where:

S_T = overall precision, mg/L,
S_O = single-operator precision, mg/L, and
X = concentration of bromide determined.

12.2 The selected water matrices included natural waters, ground waters, and tap water. These data on precision and bias may not apply to waters of other matrices.

12.3 *Bias*—Recoveries of known amounts of bromide from reagent water and selected water matrices were as shown in Table 1.

12.4 This information is derived from round-robin testing, in which five laboratories, including eight operators, participated. Of the eight sets of data ranked, as described in Practice D 2777, none was rejected. One operator submitted reagent water data only. One outlier data point within each set was also rejected. Four sample levels were run on three days and blanks were obtained for the water used.

12.5 This section on precision and bias conforms to Practice D 2777 – 77, which was in place at the time of collaborative testing. Under the allowances made in 1.5 of Practice

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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[4] Supporting data for this test method have been filed at ASTM Headquarters. Request Research Report RR:D19-1078.

TABLE 1 Recoveries of Known Amounts of Bromide from Reagent Water and Selected Water Matrices

	Amount added, mg/L	Amount found, mg/L	±Bias, %	Statistically Significant (95 % Confidence Level)
Reagent Water	0.65	0.77	+ 18.5	yes
	1.00	1.19	+ 19.0	no
	92.7	96.4	+ 3.99	no
	864	854	− 1.16	no
Water	0.65	0.80	+ 23.1	yes
	1.00	1.21	+ 21.5	yes
	92.7	95.6	+ 3.2	no
	864	836	− 3.3	yes

D 2777 – 86, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D-19 test methods.

13. Keywords

13.1 bromide; ISE; ion-selective electrode

APPENDIX

(Nonmandatory Information)

X1. RATIONALE FOR DISCONTINUATION OF TEST METHODS

X1.1 *Colorimetric and Titrimetric Test Methods for Iodide and Bromide*

X1.1.1 These test methods were discontinued in 1988, and the title of the standard was changed because no iodide test methods remained in the standard. These test methods may be found in their entirety in the *1988 Annual Book of ASTM Standards*, Vol 11.01. Additional relevant data may be found in Research Report RR:D-19-57, which is filed at ASTM Headquarters.

X1.1.2 *Former Test Method A*—Colorimetric for Iodide

X1.1.2.1 This test method covers the colorimetric determination of iodide in water when concentrations up to 100 µg/L are present.

X1.1.2.2 This test method is based on the catalytic effect of traces of iodide on the rate of oxidation of arsenious ion by ceric ion in acid solution. In the presence of a small amount of iodide, the reaction follows first-order reaction rate kinetics, and, at any given temperature and reaction time, the extent of reduction of ceric ion is directly proportional to iodide concentration. Since solutions of ceric ions are yellow and those of cerous ions colorless, the extent of the reaction may be determined photometrically.

X1.1.3 *Former Test Method B*—Colorimetric for Bromide

X1.1.3.1 This test method covers colorimetric determination of bromide in water when concentrations equivalent to no more than 100 µg/L are present.

X1.1.3.2 This test method is based on the catalytic effect of traces of bromide ion on the rate of oxidation of iodine to iodate by potassium permanganate in sulfuric acid solution. Under controlled conditions of pH, temperature, and concentration of reactants, and for a given reaction time, the concentration of unreacted iodine is inversely proportional to the concentration of bromide. To determine bromide, the reaction is stopped after a given time by extraction of the unreacted iodine with carbon tetrachloride and measuring the color intensity of the exact solution at 515 nm.

X1.1.4 *Former Test Method C*—Titrimetric, for Iodide and Bromide

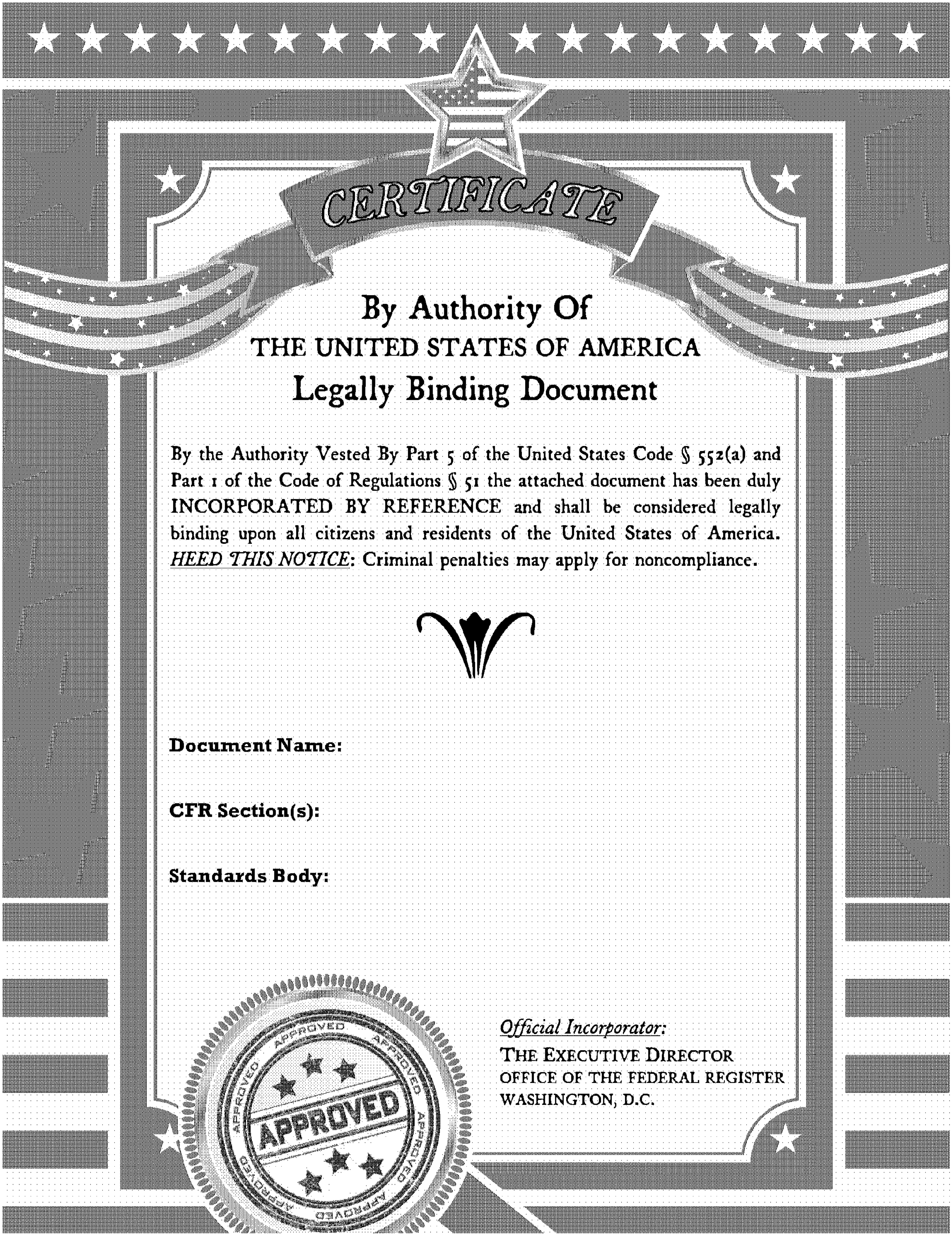
X1.1.4.1 This test method is recommended for samples containing appreciable amounts of iodide or bromide, or both, at concentrations greater than 5 mg/L.

X1.1.4.2 Any iodide in the sample is oxidized with bromine to the iodate in a buffered solution, the excess bromine being destroyed subsequently. Iodine equivalent to the iodate is liberated from potassium iodide and titrated with sodium thiosulfate.

X1.1.4.3 Iodide and bromide occurring together are oxidized to iodate and bromate, respectively, with hypochlorite. Iodine equivalent to the combined reaction products is liberated and measured after destroying the excess hypochlorite.

X1.1.4.4 The bromide content of the sample is calculated by difference between the iodide and combined iodide and bromide determinations.

X1.1.5 These test methods were discontinued because there were insufficient laboratories interested in participating in a collaborative study to obtain the necessary precision and bias data as required by Practice D 2777.



CERTIFICATE

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Document Name:

CFR Section(s):

Standards Body:

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 1253 – 86 (Reapproved 1996)

Standard Test Method for Residual Chlorine in Water[1]

This standard is issued under the fixed designation D 1253; 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 test method has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the determination of residual chlorine in water by direct amperometric titration.

1.2 Within the constraints specified in Section 6, this test method is not subject to commonly encountered interferences and is applicable to most waters. Some waters, however, can exert an iodine demand, usually because of organic material, making less iodine available for measurement by this test method. Thus, it is possible to obtain falsely low chlorine readings, even though the test method is working properly, without the user's knowledge.

1.3 Precision data for this test method were obtained on estuary, inland main stem river, fresh lake, open ocean, and fresh cooling tower blowdown water. Bias data could not be determined because of the instability of solutions of chlorine in water. It is the user's responsibility to ensure the validity of the test method for untested types of water.

1.4 In the testing by which this standard was validated, the direct and back starch-iodide titrations and the amperometric back titration, formerly part of this standard, were found to be unworkable and were discontinued in 1986. Historical information is presented in Appendix X1.

NOTE 1—Orthotolidine test methods have been omitted because of poor precision and accuracy.

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]
D 1193 Specification for Reagent Water[2]
D 3370 Practices for Sampling Water from Closed Conduits[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *combined residual chlorine, n*—residual consisting of chlorine combined with ammonia nitrogen or nitrogenous compounds.

3.2.2 *free available chlorine residual, n*—residual consisting of hypochlorite ions, hypochlorous acid, or a combination thereof.

3.2.3 *total residual chlorine (chlorine residual), n*—the amount of available chlorine-induced oxidants present in water at any specified period, subsequent to the addition of chlorine.

NOTE 2—Chlorine present as chloride is neither included in these terms nor determined by this test method.

NOTE 3—Bromine, bromine combined with ammonia or nitrogenous compounds, and chlorine dioxide are not distinguished by this test method from the corresponding chlorine compounds.

4. Summary of Test Method

4.1 This is an amperometric titration test method utilizing phenylarsine oxide as the titrant. When the titrator cell is immersed in a sample containing chlorine, current is generated. As phenylarsine oxide is added, the chlorine is reduced and the generation of current ceases. When chlorine is present as a chloramine, potassium iodide is added, releasing iodine, which is titrated in a similar manner. The iodine content is calculated in terms of free chlorine.

5. Significance and Use

5.1 Chlorine is used to destroy or deactivate a variety of unwanted chemicals and microorganisms in water and wastewater.

5.2 An uncontrolled excess of chlorine in water, whether free available or combined, can adversely affect the subsequent use of the water.

6. Interferences

6.1 This test method is not subject to interferences from temperature, color, or turbidity of sample.

6.2 Values of pH above 8.0 interfere by slowing the reaction rate. Buffering the sample to pH 7.0 or less eliminates the interference.

6.3 Erratic behavior of the apparatus in the presence of cupric ions has been reported.

6.4 Cuprous and silver ions tend to poison the electrode of the titrator.

6.5 Nitrogen trichloride and some N-chloro compounds are often present as products of the chlorination of wastewaters and will titrate partially as free available chlorine and partially as combined residual chlorine. This error can be avoided only in the determination of total residual chlorine.

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[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water, and is the direct responsibility of Subcommittee D 19.05 on Inorganic Constituents in Water.

Current edition approved Feb. 28, 1986. Published April 1986. Originally published as D 1253 – 53. Last previous edition D 1253 – 76.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

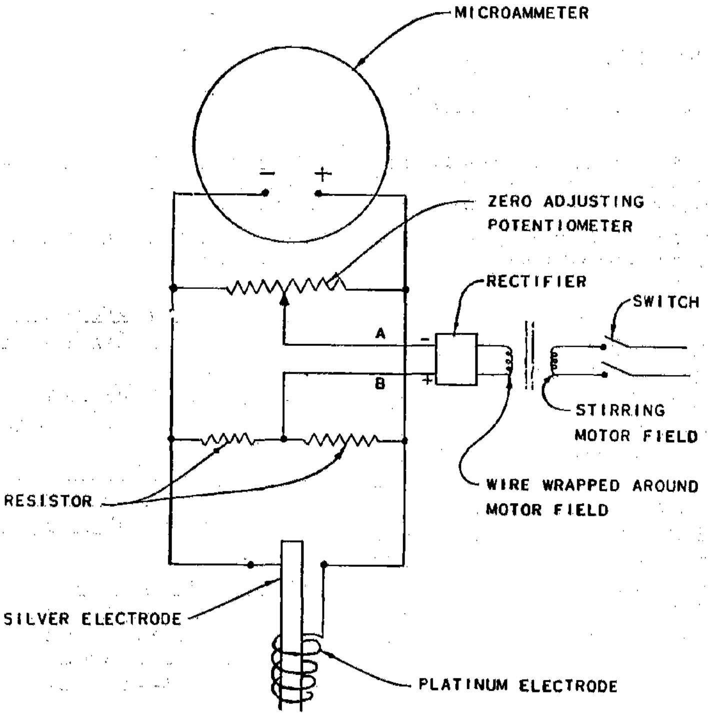

FIG. 1 Wiring Diagram of Amperometric Titrator

6.6 Exposure to high concentrations of free available chlorine causes a film-type polarization that reverses very slowly. This can be avoided by diluting the sample with water to less than 10 mg/L of free available chlorine.

6.7 If chlorine dioxide is present, an unknown portion titrates as free available chlorine. Total chlorine dioxide titrates as total residual chlorine.

6.8 Depending upon final pH, chlorination of waters containing ammonia or nitrogenous organic compounds can produce high concentrations of dichloramine. This compound produces four to five times as much current as monochloramine. The current produced by as little as 5 mg/L of dichloramine can cause the microammeter pointer to read offscale even at the end point in the titration of free available chlorine. This may be overcome by use of an opposing voltage in the apparatus' circuitry. The instrument's manufacturer should be consulted in this regard.

7. Apparatus

7.1 *Amperometric Titration Apparatus*[3,4]—Refer to Fig. 1.

NOTE 4—When the titrator has been out of service for a day or more, check the electrode for sensitivity by noting the rapidity of the pointer deflection. If the pointer responds slowly after the addition of KI solution, add a small amount of biiodate. If it responds slowly to free available chlorine, sensitize it by adding chlorine.

7.2 *Glassware*—Condition with water containing at least 10 mg/L of residual chlorine for at least 2 h prior to use and then rinse thoroughly.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type III, further treated to be free of chlorine demand. A suggested method for preparation of chlorine demand-free water is to add approximately 20 mg/L of available chlorine to Type III water, let it stand for about a week in darkness, and then expose it to sunlight until no chlorine remains.

8.3 *pH 4.0 Buffer Solution*—Dissolve 243 g of sodium acetate trihydrate and 480 g of glacial acetic acid in water and dilute to 1 L.

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[3] *Water and Sewage Works*, May 1949, p. 171, and *Journal American Water Works Association*, Vol 34, 1942, pp. 1227–1240.

[4] Amperometric titrators are available commercially from most laboratory supply houses.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.4 *pH 7.0 Buffer Solution*—Dissolve 25.4 g of monobasic potassium phosphate and 86 g of dibasic sodium phosphate in water and dilute to 1 L.

8.5 *Biiodate, Solution Standard* (0.0282*N*)—Dissolve 0.9163 g of potassium biiodate in water and dilute to 1 L in a volumetric flask. Store in an amber glass-stoppered bottle.

8.6 *Phenylarsine Oxide, Solution Standard* (0.00564 *N*)—Dissolve 0.8 g of phenylarsine oxide in 150 mL of sodium hydroxide solution (12 g/L). After settling, decant 110 mL of this solution, add 800 mL of water, and bring to a pH of 9.0 by adding hydrochloric acid (1 + 1). This should require about 2 mL of HCl (1 + 1). Continue acidification with HCl (1 + 1) until a pH of 6 to 7 is reached, as indicated by a glass-electrode system; then dilute to a total volume of 1L. Standardize to 0.00564 *N* against 0.0282 *N* biiodate solution using the titrator (7.1) as the end-point indicator. Add 1 mL of chloroform for preservation.

8.7 *Potassium Iodide Solution* (50 g/L)—Dissolve 50 g of KI in water and dilute to 1 L. Add 1 g of sodium bicarbonate to stabilize the solution. Store in an amber bottle and avoid direct exposure to sunlight.

9. Sampling

9.1 Collect the sample in accordance with Practices D 3370. Take care that the sample is representative and keep it away from direct sunlight prior to analysis.

9.2 All tests should be made as soon as possible after collection of the sample (not more than 5 min) because the residual chlorine may diminish with time, due to the chlorine demand of the sample. Where time of contact is important, the elapsed time between the addition of chlorine and the determination of chlorine should be taken into account.

10. Procedure

10.1 For residual chlorine concentrations of 2.0 mg/L or less, use a 200-mL sample. For greater concentrations, use a 100-mL sample. It is preferable that the size of the sample be such that not more than 2 mL of titrant will be required to complete the titration.

10.2 *Determination of Total Residual Chlorine:*

10.2.1 Add 1 mL of KI solution to a 200-mL sample and immediately add 1 mL of pH 4.0 buffer solution.

10.2.2 Immerse the electrodes in the sample and start the stirrer. Adjust the microammeter pointer of the potentiometer to the right or high current side of the scale so the pointer can deflect counterclockwise during the analysis.

10.2.3 Titrate using standard phenylarsine oxide solution, adding the titrant in small increments, and noting the deflection of the microammeter pointer. Plot the progress of the titration on linear graph paper with current on the vertical axis and titrant volume on the horizontal axis. Add a small volume of titrant, wait a few seconds, and plot the current-volume point on the graph.

10.2.4 Readjust the potentiometer several times during the titration, if necessary, to bring the pointer back on scale.

10.2.5 Continue the analysis by determining at least three points spread over the downward sloping titration curve and at least three points after the equivalence or end point. The latter points will indicate practically no change in current. Points just before the end point shall be disregarded in its determination. The millilitres of titrant at the end point defined by the intersection of the two linear sections of the titration curve should be recorded.

10.3 *Determination of Free Available Chlorine Residual:*

10.3.1 Add 1 mL of pH 7.0 buffer solution to a 200-mL sample.

10.3.2 Repeat the phenylarsine oxide titration beginning with 10.2.2.

10.3.3 Note a rapid deflection of the pointer for each increment of titrant indicates the presence of free available chlorine. Slight counterclockwise movements of the pointer after addition of individual drops of titrant is a drift effect and does not indicate the presence of free available chlorine.

10.4 *Determination of Combined Available Chlorine Residual:*

10.4.1 Complete the titration for the determination of free available chlorine residual as in 10.3.

10.4.2 To the same sample, add 1 mL of KI solution and 1 mL of pH 4.0 buffer solution and repeat the titration as in 10.2.

11. Calculation

11.1 Calculate the various types of chlorine residual, in milligrams per litre, as follows:

$$\text{Chlorine residual, mg/L} = 200\ A/V$$

where:

A = phenylarsine oxide solution (0.00564 *N*) required for the titration of 10.2, 10.3, or 10.4, depending on the specific type of chlorine residual determined, mL, and

V = sample used, mL.

12. Precision and Bias[6]

12.1 The overall precision (S_t) and the single operator precision (S_o) of this test method for free available chlorine (FAC) and for total residual chlorine (TRC) were determined by eight or nine qualified cooperators each with analysis equipment and reagents at each of five sites. Each site constituted a different chlorinated cooling water matrix: estuary, inland main stem river, fresh lake, open ocean, and fresh cooling tower blowdown. Each site water was chlorinated up to nine levels. Samples were collected simultaneously and analyzed within 5 min of collection by all eight or nine cooperators. Duplicate sampling and analysis runs were made at each level.

12.2 The S_t and S_o for FAC was found to vary linearly with the mean concentration of FAC, X, in mg/L, over the range for X from 0.0 to 1.0.

12.2.1 For the pooled results from all of the matrices tested:

$$S_t = 0.025 + 0.199\,X\ (n = 37,\ r = 0.848)$$
$$S_o = 0.008 + 0.081\,X\ (n = 35,\ r = 0.638)$$

where:

n = number of runs, and

r = correlation coefficients.

12.3 The S_t and S_o for TRC was found to vary linearly

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[6] Supporting data are available from ASTM Headquarters. Request RR: D19-1124.

with the mean concentration of TRC, Y, in mg/L, over the range for Y from 0.0 to 3.5.

12.3.1 For the pooled results from all of the matrices tested:

$$S_t = 0.022 + 0.098\ Y\ (n = 39,\ r = 0.865)$$
$$S_o = 0.012 + 0.024\ Y\ (n = 38,\ r = 0.695)$$

12.4 The bias of the test method could not be determined since the instability of solutions of chlorine in water does not permit the determination of an acceptable true value for TRC and FAC in the samples.

13. Keywords

13.1 amperometric; analysis; chlorine; water

APPENDIX

(Nonmandatory Information)

X1. RATIONALE FOR DISCONTINUATION OF TEST METHODS

X1.1 *Direct and Back Starch-Iodide Titrations and Amperometric Back Titration:*

X1.1.1 These two test methods were discontinued in 1986. These test methods may be found in *1985 Annual Book of ASTM Standards*, Vol 11.01. These test methods were originally issued in 1953.

X1.1.2 These test methods are biiodate solutions as titrating agents. Attempts to include these test methods in the round-robin testing were not successful because the reaction rate of the biiodate solution with phenylarsine oxide was slow and inconsistent. The little data obtained were widely varied, nonreproducible, and were not relatable to the values being tested.

X1.1.3 Field experience indicates that both test methods can work if iodine solution is used in place of biiodate solution as the titrating agent. Validation of these test methods through round-robin testing, however, has not been carried out.