1985
ANNUAL BOOK OF ASTM STANDARDS

SECTION 14

General Methods
and Instrumentation

ASTM

VOLUME 14.01

Analytical Methods — Spectroscopy; Chromatography; Temperature Measurement; Computerized Systems

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General Methods and Instrumentation

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Analytical Methods — Spectroscopy; Chromatography; Temperature Measurement; Computerized Systems

Includes standards of the following committees:

E-13 on Molecular Spectroscopy
E-14 on Mass Spectrometry
E-19 on Chromatography
E-20 on Temperature Measurement
E-23 on Resinography
E-25 on Microscopy
E-31 on Computerized Systems

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Foreword

ASTM

ASTM, founded in 1898, is a scientific and technical organization formed for "the development of standards on characteristics and performance of materials, products, systems, and services; and the promotion of related knowledge." It is the world's largest source of voluntary consensus standards.

The Society operates through 141 main technical committees with 1993 subcommittees. These committees function in prescribed fields under regulations that ensure balanced representation among producers, users, general interest, and consumer participants.

The Society currently has 29,500 active members, of whom approximately 18,500 serve as technical experts on committees, representing 87,900 units of participation.

Membership in the Society is open to all concerned with the fields in which ASTM is active. A membership application may be found at the back of this volume. Additional information may be obtained from Member, Committee, and Customer Services, ASTM, 1916 Race St., Philadelphia, PA 19103.

1985 Annual Book of ASTM Standards

The 1985 *Annual Book of ASTM Standards* consists of 66 volumes, divided among 16 sections, of which this volume is one. It contains formally approved ASTM standard classifications, guides, practices, specifications, test methods, and terminology and related material such as proposals. These terms are defined as follows in the Regulations Governing ASTM Technical Committees:

Categories:

standard—as used in ASTM, a document that has been developed and established within the consensus principles of the Society and that meets the approval requirements of ASTM procedures and regulations.

Discussion—The term "standard" serves in ASTM as an adjective in the title of documents, such as test methods or specifications, to connote specified consensus and approval. The various types of standard documents are based on the needs and usages as prescribed by the technical committees of the Society.

proposal—a document that has been approved by the sponsoring committee for publication for information and comment prior to its consideration for adoption as a standard.

Discussion—Complete balloting procedures are not required for proposals and no designation is assigned to them.

emergency standard—a document published by the Society to meet a demand for more rapid issuance of a specific standard document.

Discussion—The Executive Subcommittee of the sponsoring committee must recommend the publishing of an emergency standard and the Committee on Standards must concur in the recommendation. Emergency standards are not full consensus documents because they are not submitted to Society ballot.

Types:

The various types of ASTM documents are to provide a flexibility of form, communication, and usage for both the technical committees and the myriad users of ASTM documents. The type of ASTM document that is developed and titled is based on the technical content and intended use, not on the degree of consensus achieved. The three categories of ASTM documents (standard, emergency standard, and proposal) can be of the following forms and types:

classification—a systematic arrangement or division of materials, products, systems, or services into groups based on similar characteristics such as origin, composition, properties, or use.

guide—a series of options or instructions that do not recommend a specific course of action.
Discussion—Whereas a practice prescribes a general usage principle, a guide only suggests an approach. The purpose of a guide is to offer guidance, based on a consensus of viewpoints, but not to establish a fixed procedure. A guide is intended to increase the awareness of the user to available techniques in a given subject area and to provide information from which subsequent evaluation and standardization can be derived.

practice—a definitive procedure for performing one or more specific operations or functions that does not produce a test result. (Compare *test method*.)
Discussion—A practice is not a downgraded test method. Examples of practices include procedures for conducting interlaboratory testing programs or other statistical procedures; for writing statements on sampling or precision and accuracy; and for selection, preparation, application, inspection, necessary precautions for use or disposal, installation, maintenance, and operation of testing equipment.

specification—a precise statement of a set of requirements to be satisfied by a material, product, system, or service that indicates the procedures for determining whether each of the requirements is satisfied.
Discussion—It is desirable to express the requirements numerically in terms of appropriate units together with their limits.

terminology—a document comprising definitions of terms; descriptions of terms; explanations of symbols, abbreviations, or acronyms.

test method—a definitive procedure for the identification, measurement, and evaluation of one or more qualities, characteristics, or properties of a material, product, system, or service that produces a test result. (Compare *practice*.)

A new edition of the Book of Standards is issued annually. Each volume contains all actions approved by the Society at least six months before the issue date. New and revised standards approved by the Society between the annual appearances of any given volume are made available as separate copies. The 1985 edition of the Book of Standards comprises approximately 58,000 pages and includes over 7400 ASTM standards.

Purpose and Use of ASTM Standards

An ASTM standard represents a common viewpoint of those parties concerned with its provisions, namely, producers, users, consumers, and general interest groups. It is intended to aid industry, government agencies, and the general public. The use of an ASTM standard is purely voluntary. It is recognized that, for certain work or in certain regions, ASTM standard specifications may be either more or less restrictive than needed. The existence of an ASTM standard does not preclude anyone from manufacturing, marketing, or purchasing products, or using products, processes, or procedures not conforming to the standard. Because ASTM standards are subject to periodic review and revision, those who use them are cautioned to obtain the latest revision.

Consideration of Comments on ASTM Standards

An ASTM standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of any standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Using the Annual Book of ASTM Standards

The standards are assembled in each volume in alphanumeric sequence of their ASTM designation numbers except for Volumes 11.01, 11.02, and 05.04, which are assembled by subject matter. Volume 06.03 is assembled first by committee, then in alphanumeric sequence. Each volume has a table of contents, listing the standards in alphanumeric sequence by ASTM designation; and a list by subjects, categorizing the standards according to subject. A subject index of the standards in each volume appears at the back of each volume.

Availability of Individual Standards

Each ASTM standard is available as a separate copy from ASTM. Special quantity prices and discounts for members can be obtained from Sales Service. When ordering, provide the ASTM standard designation and year of issue, title, quantity desired, and shipping instructions.

Obsolete Editions

This new edition of the *Annual Book of ASTM Standards* makes last year's edition obsolete. Each volume of the *Annual Book of ASTM Standards* is published annually because of additions of new standards and significant revisions in existing standards. On the average, about 30 % of each volume is new or revised. For practical purposes, therefore, it is not wise to use obsolete volumes. However, for teaching purposes, these outdated volumes might be useful.

Precautionary Caveat

In January 1983, the Board of Directors approved the inclusion of the following precautionary caveat:

> *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.*

Inclusion of the caveat is required in test methods, specifications (where test methods are detailed other than by reference), practices, and guides. Implementation of the caveat will be phased in as new, revised, and reapproved standards are approved by the Society.

Disclaimer

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in these standards. Users of these standards are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

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A complete Subject Index begins on p. 977

Listed below are those standards included in this book and those standards that appeared previously that have been discontinued within the past five years. Since the standards in this book are arranged in alphanumeric sequence, no page numbers are given in this contents.

In the serial designations prefixed to the following titles, the number following the dash indicates the year of adoption as standard or, in the case of revision, the year of last revision. Thus, standards adopted or revised during the year 1985 have as their final number, 85. A letter following this number indicates more than one revision during that year, that is 85a indicates the second revision in 1985, 85b the third revision, etc. Standards that have been reapproved without change are indicated by the year of last reapproval in parentheses as part of the designation number, for example, (1985). A superscript epsilon indicates an editorial change since the last revision or reapproval—$\epsilon 1$ for the first change, $\epsilon 2$ for the second change, etc.

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§ Approved for use by agencies of the Department of Defense and, if indicated on the standard, replaces corresponding Federal or Military document.

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‡ Although this standard has been officially withdrawn from Society approval, a brief description is included for information only.

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Designation: C 177 – 76$^{\epsilon 2}$

An American National Standard

Standard Test Method for STEADY-STATE THERMAL TRANSMISSION PROPERTIES BY MEANS OF THE GUARDED HOT PLATE[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.

This method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

$^{\epsilon 1}$ NOTE—New Note 3 was editorially added in August 1978.
$^{\epsilon 2}$ NOTE—Tables 2a and 2b were editorially corrected in August 1981.

1. Scope

1.1 This method covers the determination of the steady-state thermal transmission properties of insulating specimens using a guarded hot plate (Note 1), within the limits set by 1.4, 1.5, and 1.6. For purposes of certification this method is limited to specimens with thermal resistances greater than 0.017 $K \cdot m^2 \cdot W^{-1}$ (Note 2) in all directions.

NOTE 1—Frequently it is desirable to determine the thermal transmission properties of some materials in the forms in which they are used, such as molded pipe coverings, etc., or the thermal transmittance of a composite wall construction or part thereof. For such purposes, ASTM Test Method C 335, for Thermal Conductivity of Pipe Insulation,[2] and ASTM Test Method C 236, for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box,[2] are recommended.

NOTE 2—Special techniques may be required for measuring surface temperatures with specimens having thermal resistances less than 0.1 $K \cdot m^2 \cdot W^{-1}$.

NOTE 3—The accuracy of measurement on specimens of low-density thermal insulation by this method may be difficult to verify and may require an extensive analysis of the equipment and/or a performance check using calibration standards having heat transmission characteristics and thickness similar to the test specimens.

1.2 This is a primary method for measuring the thermal transmission properties of specimens, as only measurements of length, electrical power, and temperature difference are required.

1.3 Considerable latitude is given to the designer in this standard. And since the design of a guarded hot plate apparatus is not a simple matter, a procedure for qualifying an apparatus is given in 3.5.

1.4 The specimens must meet the following conditions if the thermal *resistance* and thermal *conductance* of the *specimen* are to be determined by this method (Note 4):

1.4.1 The portion of the specimen over the test area must be typical of the whole specimen in every aspect.

1.4.2 The remainder of the specimen must not, on average, distort the heat flow in that part of the specimen adjacent to the metering area (Note 5).

1.4.3 The specimen must be free of low thermal resistance paths that create thermal short circuits between the test surfaces.

1.4.4 The heat flux through the specimens must be directly proportional to the temperature difference across the specimens.

NOTE 4—Further discussion on the definition of these limitations can be found in "What Property Do We Measure?" *Heat Transmission Measurements in Thermal Insulations, ASTM STP 544*, Am. Soc. Testing Mats., 1974.

NOTE 5—Appendix X1 describes tests that can help ascertain whether conditions of 1.4 are satisfied. For the purposes of this method, differences in the measurements of less than 2 % may be considered insignificant, and the requirements fulfilled.

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

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

1.5 The specimen(s) must meet one of the following requirements, in addition to 1.4, if the average thermal resistivity and average thermal conductivity of the *specimen* are to be determined by this method:

1.5.1 The specimen must be homogeneous, *or*

1.5.2 The specimen must not be layered, and the net direction of heat flow must not be altered by any inhomogeneities.

NOTE 6—Appendix X1 gives one test for checking the condition described in 1.5.2.

1.6 The following requirements, in addition to 1.4 and 1.5, must be met if the values of thermal *resistivity* and thermal *conductivity* measured for the specimen are to be considered valid for the *material:*

1.6.1 The material must be homogeneous.

1.6.2 The thickness of the specimen must be greater than that for which the apparent thermal resistivity of the material does not change by more than 2 % with further increases in thickness (Note 7).

1.6.3 Adequate sampling must be performed to ensure that the measurements are representative of the whole material.

NOTE 7—Appendix X1 gives one test for checking the condition described in 1.6.2. The material standard normally specifies the sampling procedure required for the material.

1.7 Two different types of guarded hot plate apparatus are described. They are similar in principle but differ enough in construction to warrant separate descriptions for each in regard to design. The low-temperature guarded hot plate, which has metal surface plates and a definite guard gap (see 5.3), is generally used for measurements where the temperature of the cold plate is not below 21 K and the temperature of the heating unit is not above 500 K. It is described in Section 5. The high-temperature guarded hot plate, which may or may not have metal surface plates and may or may not have a definite guard gap, is ordinarily used for measurements where the heating unit temperature is greater than 550 K but less than 1350 K. It is usually made of a cast or otherwise formed refractory material which is electrically insulating at the highest temperature of operation. Metal surface plates may be used. They are recommended because they provide a more uniform temperature distribution on the surfaces of the plate. The high-temperature design is described in Section 6. If compliance with this method is to be reported, all measurements made with heating-unit temperatures below 550 K shall be performed using a heating unit having metal surface plates and a definite guard gap. In all other respects, the method is the same for both types of apparatus. It is intended, in presenting these descriptions, to indicate the essential elements and details that experience has shown to be necessary or important for reliable measurements by this method.

NOTE 8—For the convenience of new workers in the field, detailed drawings and descriptions for the construction, and some phases of the operation, of typical hot plate apparatuses complying with the requirements of this method have been made available.[3] Two of these hot plate apparatuses, known as the National Bureau of Standards plate and the National Research Council plate, are metal-surfaced plates; one is a high-temperature plate.

1.6 The test method is organized as follows:

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[3] Detailed information on all of these hot plate apparatuses is available at a nominal cost from ASTM, 1916 Race St., Philadelphia, PA 19103. Request Adjunct No. 12-301770-00.

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2. Applicable Documents

2.1 *ASTM Standards:*

C 167 Test Methods for Thickness and Density of Blanket- or Batt-Type Thermal Insulating Materials[2]

C 168 Definitions of Terms Relating to Thermal Insulating Materials[2]

C 236 Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box[2]

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

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

2.2 *Adjunct:*

Descriptions of Three Guarded Hot Plates[3]

3. Significance

3.1 The thermal transmission properties of a specimen of material *(1)* may vary due to variability of the composition of the material or samples of it, *(2)* may be affected by moisture or other conditions, *(3)* may change with time or high temperatures, *(4)* may change with specimen thickness, *(5)* may change with temperature difference across the specimen, and *(6)* may change with mean temperature. It must be recognized, therefore, that the selection of a typical value of thermal transmission properties representative of a material, or of a particular application, shall be based on a consideration of these factors and an adequate amount of test information.

3.2 Apparatus constructed and operated in accordance with this method are capable of measuring thermal properties accurate to within ±2 % when the ambient temperature is near the mean temperature of the test. With adequate precautions in the design of the apparatus, and after extensive checking and cross referencing of measurements with other similar apparatus, an accuracy of about ±5 % should be obtainable anywhere in the full operating range of an apparatus. Such accuracy is normally easier to attain using separate apparatus for the extremes in the range. The precision and reproducibility of measurements made by the apparatus are normally much better than 1 %. This precision is required to identify errors in the method and is desirable in quality control applications.

3.3 Because of the requirements as to test conditions prescribed by this method it shall be recognized that the thermal properties obtained will not necessarily apply without modification to all service conditions. As an example, this 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 dependence of the thermal properties on variables such as mean temperature and temperature difference. These dependencies should be measured or the tests

[4] *Annual Book of ASTM Standards*, Vol 14.01.

made at conditions typical of use.

3.4 It is not practical in a method of this type to try to establish details of construction and procedures to cover all contingencies that might offer difficulties to a person without technical knowledge concerning theory of heat flow, temperature measurements, and general testing practices. Standardization of this method is not intended to restrict in any way the future development by research workers of new or improved methods or improved procedures. Requirements for qualifying an apparatus are outlined in 3.5.

3.5 When a new or modified design is evolved, tests shall be made on at least two sets of material of known thermal stability, which have been calibrated at a nationally recognized laboratory. Tests shall be made for each specimen at two mean temperatures typical of the operating range. All tests shall be conducted within 90 days of calibration, where possible. Any differences in results should be carefully studied to determine why they arise and how they can be removed. Appropriate action should be taken. Only after a successful comparison shall materials be certified with the apparatus. No further checking is necessary, though periodic checks are recommended.

3.6 The thermal transmission properties of many materials depend upon the prior thermal history. Care must be exercised when testing a single specimen at a number of conditions to perform the tests in a sequence that limits such effects on the results.

3.7 If results are to be reported as having been obtained by this method, then all pertinent requirements prescribed by this method shall be met. Where such conditions are not met, the phrase "All requirements of Method C 177 have been met with the exception of . . ." shall be added and a complete list of the exceptions included.

4. Terminology

NOTE 9—As Definitions C 168 is under revision, the definitions and symbols given here should be used.

4.1 *Definitions:*

4.1.1 *thermal resistance, R*—the temperature difference required to produce a unit of heat flux through the specimens under steady-state conditions. For a flat slab, it is calculated as follows:

$$R = \frac{A(T_1 - T_2)}{Q} = \frac{1}{\Gamma} = \frac{D}{\lambda}$$

4.1.2 *thermal conductance, Γ*—under steady-state conditions, the heat flux required to produce a unit temperature difference; the reciprocal of the thermal resistance of the specimen. For a flat slab, it is calculated as follows:

$$\Gamma = \frac{Q}{A(T_1 - T_2)} = \frac{1}{R} = \frac{\lambda}{D}$$

4.1.3 *thermal conductivity, λ*—under steady-state conditions, the heat flux per unit temperature gradient in the direction perpendicular to an isothermal surface. For thin specimens or low-density materials this definition must be applied with caution. Thermal conductivity of a material can be defined only where several conditions are met (see 1.6): the thermal resistance of specimens of a material must be sufficiently independent of the area of the specimen, of where the specimen is selected in the sample, of the temperature difference across the specimen, and, for a flat slab specimen, the thermal resistance must be proportional to the thickness. The latter can be demonstrated by plotting the thermal resistance of a number of specimens of the material against specimen thickness. The line through the point must increase linearly with thickness from zero thermal resistance at zero thickness. When this condition is met, the thermal conductivity can be determined as the inverse of the slope of the straight line and the thermal conductivity can be calculated as follows:

$$\lambda = \frac{Q \times D}{A(T_1 - T_2)} = \frac{D}{R}$$

The above requirement assumes that the heat transfer within the specimen is independent of thickness, and temperature difference. It recognizes the existence of a minimum thickness and maximum temperature difference for which thermal conductivity can be defined. For the purposes of this method, a 2 % dependence will be considered maximum for each.

4.1.4 *thermal resistivity, r*—under steady-state conditions, the temperature gradient, in the direction perpendicular to the isothermal surface, per unit heat flux; the reciprocal of