

A History of Testing Heat Insulators at the National Institute of Standards and Technology

By

Robert R. Zarr, P.E.

**Building and Fire Research Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899**

Reprinted from ASHRAE Transactions 2001, V. 107, Pt. 2. , June 2001
NOTE: This paper is a contribution of the National Institute of Standards and
Technology and is not subject to copyright.



NIST

National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

A History of Testing Heat Insulators at the National Institute of Standards and Technology

Robert R. Zarr, P.E.

ABSTRACT

The history of testing heat insulators at the National Institute of Standards and Technology (NIST) (formerly the National Bureau of Standards) is nearly as long as the history of the institute itself. The institute's participation in building research provided an important role in the development of test methods for measuring the heat transmission of insulating and building materials. The principal measurement technique pursued in this history is the development and subsequent standardization of the guarded-hot-plate method.

INTRODUCTION

At the beginning of the 20th century, the refrigeration industry in the U.S. was becoming a commercial reality. Mechanical refrigeration technology had achieved economic viability as a means of producing ice for cooling and was on the threshold of moving into households as an everyday appliance. Advances were underway to provide mechanical “air-conditioning” for public buildings, such as theaters, department stores, and skyscrapers. In general, the early 1900s saw the evolution of a scientific approach to refrigeration through company-sponsored research and development. This early technical progress in cooling and heating for industrial processes and thermal comfort had a direct effect on the development of the test methods for thermal insulation materials at the National Bureau of Standards.¹

To assist the refrigeration industries, measurements at NBS on the thermal conductivity of heat insulators began in the early 1910s in the Heat and Thermometry Section, initially under the direction of Hobart C. Dickinson and later under Milton S. Van Dusen. These early experiments were conducted at a laboratory located along what was originally a

remote site of Connecticut Avenue in Washington DC. In 1947, the bureau formally introduced a Building Technology Division for the coordinated study of building research (NBS 1948) and, soon afterwards, the activities on thermal conductivity became an official part of the building program under the leadership of Henry E. Robinson. In what has become a valuable sustained effort, much of the work on heat insulators at NIST now resides under the auspices of the Building and Fire Research Laboratory in Gaithersburg, Md.

Many of the test methods developed at NIST for the measurement of heat transfer of solids have become accepted as standard test methods (ASTM 2000) or recognized as recommended measurement techniques (Pratt 1969; Ginnings 1970; Eckert and Goldstein 1976; Maglic 1992). In recognition of NIST's centennial anniversary (1901-2001), this paper describes the development and impact of one of these test methods—the guarded hot plate—at NIST. In particular, this paper focuses on specific historical interactions between NIST, ASHRAE (ancestral societies), and ASTM that have directly, and indirectly, affected the development and advancement of this test method. The paper also gives a brief description of other related test methods developed at NIST for the thermal conductivity of solids and thermal transmittance of compound walls.

¹. In 1901, Congress established the National Bureau of Standards (NBS) to support industry, commerce, scientific institutions, and all branches of government. In 1988, as part of the Omnibus Trade and Competitiveness Act, the name was changed to the National Institute of Standards and Technology (NIST) to reflect the agency's broader mission. For historical consistency, this narrative will use, where appropriate, NBS for events prior to 1988.

Robert R. Zarr is a mechanical engineer in the Building and Fire Research Laboratory, National Institute of Standards and Technology, Gaithersburg, Md.

EARLY DEVELOPMENTS

In 1913, the U.S. Congress, at the request of the refrigeration industry, appropriated funding for investigation of various physical and chemical properties of materials involved in the construction and operation of large-scale refrigeration machinery (Cochrane 1966). Under Hobart C. Dickinson and others, one aspect of the study included the investigation of insulating materials used in the construction of large-scale refrigeration structures. Of particular interest to the mechanical engineer was the development of usable data pertaining to heat transmission in thermal insulation needed for design purposes (Achenbach 1970). For this purpose, Dickinson developed NBS's first guarded hot plate² for an absolute method of measurement of homogenous slab-like materials (Dickinson and Van Dusen 1916).

Dickinson (1949) provides additional information in excerpts below taken from a letter to Professor Penrod at the University of Kentucky who had requested a compilation of the historical data of the hot plate at the bureau.

The first hot plate apparatus built at the National Bureau of Standards was designed and built about 1912 by the writer. At this time heat transfer tests on insulation materials were being made in a "hot box" type of apparatus, generally in the form of a cube with all the sides of similar material and construction. The results of tests by these (box) methods were in many cases expressed as thermal conductivities, although they were actually what are now known as thermal transmittance (U) values divided by the specimen thickness. ... In an attack on this problem undertaken in connection with refrigeration investigations underway at this bureau at that time, and also in an effort to devise a more direct and convenient method of test, the writer built an 8-inch hot plate with copper plates one centimeter thick. Central 4-inch squares were outlined on these plates by deep grooves, which reached very nearly to the other face of each plate. ... About 1914, Dr. M. S. Van Dusen, who had been assigned to work in this field, constructed an 8-inch guarded hot plate with a central 4-inch square metering area. ... This apparatus was found satisfactory, and another of similar construction 24 inches was built before 1916....

In 1916, Dickinson and Van Dusen published their first work in this area, a classic study identifying the causes of discordant experimental results for heat transfer through insulating materials. Over several decades, previous experimental investigations of homogeneous solid materials and engineering systems had resulted in two different, but necessary, approaches that had produced a growing discordance of test results. While the experimental physicist determined the thermal conductivity of homogeneous solids, the engineer was interested in the practical problem of the transmission of heat through systems. The net result was two distinct groups of experimental investigations with disagreements within each

². A short history of the hot plate method prior to NBS's involvement is given in the Appendix.

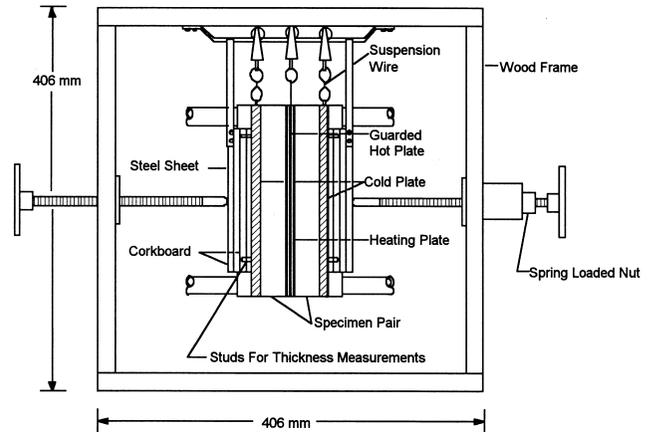


Figure 1 Schematic of NBS 200 mm guarded-hot-plate apparatus (1928 version).

approach (because of difficulties in measuring heat flow and temperature) and between each approach (because of erroneous application of results in heat transfer calculations). Dickinson and Van Dusen (1916) defined standard terms for use with both (box and plate) types of experiments and provided accurate determinations of heat flow through air spaces and 30 insulating materials.

TEST METHOD OVERVIEW

In 1920, Van Dusen published a paper with additional thermal conductivity measurements of insulating materials, but more importantly, it provided detailed information on the design and theory of operation of the apparatus. Figure 1 is a diagram of a similar version of the NBS 200 mm guarded-hot-plate apparatus. Van Dusen (1920) describes the apparatus as consisting of a flat, electrically heated copper plate and two flat water-cooled cold plates³ (suspended from the framing by thin wires). The plate surfaces were treated with a high-emittance coating. Two nearly identical specimens were placed, one on each side of the center plate, between the hot and cold plates. Heat was supplied electrically at a known rate to the heated plate, and a constant temperature difference was maintained between the hot and cold plates. The temperature differences of the surfaces were measured by means of thermocouples. Additional details of the apparatus are available in Van Dusen (1920, also Adjunct of ASTM 2000a).

The guarded-hot-plate method establishes one-dimensional heat flow through a pair of specimens by reducing undesired lateral heat flows to negligible proportions. For tests involving heat flows through a pair of specimens, the alge-

³. The heated plate actually consisted of two plates enclosing a (distributed) heater grid wound around fiberboard and insulated from the metal plates by sheets of micanite. The cold plates were maintained at constant temperature by circulation of either water or brine through the plates.

braic form of Fourier's heat conduction equation for one-dimensional heat flow is:

$$Q = 2A\bar{k}\frac{(T_h - T_c)}{L} \quad (1)$$

where Q is the power to the meter area of the guarded hot plate, $2A$ is the meter area, T_h and T_c are the average hot and cold surface temperatures, respectively, and L is the average thickness measured with calipers. The term \bar{k} corresponds to the thermal conductivity at \bar{T} given by $\bar{T} = \frac{1}{2}(T_h + T_c)$. Ideally, the pair of specimens is well matched (i.e., nearly identical) so that the differences between specimens for $(T_h - T_c)$ and L are quite small.

DESIGN DATA

The long-term impact of the guarded-hot-plate technology was evident in several areas outside NBS. One of the first impacts was the outgrowth of consensus engineering design data for insulating and building materials. The guarded hot plate, originally developed by Dickinson and Van Dusen, was refined over several years, and, in 1928, Van Dusen (Van Dusen and Finck 1928) constructed a version of the apparatus that operated consistently until 1983. The thermal conductivity data obtained from this apparatus, along with the data from the earlier NBS publications, were aggregated with other data⁴ and tabulated in handbooks as design heat transmission coefficients for the engineer. These design data have served the engineering community effectively for several decades. In recent years, much of the original NBS data have been compiled in electronic format and are available on the Internet (<http://srdata.nist.gov/insulation/>) as NIST Standard Reference Database 81 (Zarr et al. 1999).

STANDARDIZATION

The singular most important impact of the guarded-hot-plate technology was the standardization of the test method in North America. This remarkable step has been extremely effective in reducing the differences among laboratories with respect to thermal testing. After many years of effort, industry, NBS, and many others produced a tentative edition of the test method in 1942. By joint action, the American Society of Heating and Ventilating Engineers (ASHVE), American Society for Testing Materials (ASTM), American Society for Refrigerating Engineers (ASRE), and the National Research Council (NRC) produced a *Standard Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot*

⁴ Thermal conductivity data were aggregated from several authorities including U.S. Bureau of Standards, University of Illinois, Armour Institute of Technology, University of Minnesota, ASHVE Research Laboratory, University of Toronto, and the Massachusetts Institute of Technology, among others (ASHVE Guide 1947).

Plate. In 1945, the method was formally adopted as a standard and designated ASTM Test Method C 177. The code set up the requirements for conducting tests and construction of a guarded-hot-plate apparatus. It is interesting to note that the standard's adjunct includes the design drawings for several apparatus, including the 1928 version of the NBS guarded-hot-plate apparatus. In a final tribute, the 1928 NBS guarded-hot-plate apparatus has recently been entrusted to the NIST Museum for historic preservation and display.

DISSEMINATION OF ASTM TEST METHOD C 177

ASTM Test Method C 177 has become the cornerstone for all thermal insulation testing in North America. Over the decades, the standard test method has achieved international acceptance as the most accurate absolute test method for the measurement of thermal conductivity of heat insulators. At NBS, Henry E. Robinson was instrumental in disseminating ASTM Test Method C 177 through laboratory intercomparison testing, development of insulation reference materials, and refinement of the measurement method.

Interlaboratory Comparison

In 1947, Robinson and colleague Thomas W. Watson extended the temperature range of Van Dusen's guarded-hot-plate apparatus. A few years later, NBS completed one of the first published laboratory intercomparisons of thermal conductivity tests on corkboard insulation (Figure 2) from -6.7°C to 54°C (20°F to 130°F) (Robinson and Watson 1951). Their paper reported data from 15 of the 20 hot plates (ranging from 200 mm to 610 mm) that were within $\pm 3\%$ of the mean value. The remaining values outside of this range demon-



Figure 2 Henry E. Robinson (1911-1972) installs a pair of corkboard specimens for interlaboratory testing (c. 1948).

strated the need for a suitable means to check the operation of apparatus from industrial and other laboratories.

Standard Reference Materials

To assist industry, a measurement program was initiated in 1958 for supplying measured samples of suitable insulating materials to the industry for calibration purposes and operational checking. By 1977, more than 300 laboratories had been served, resulting in considerable improvement in the quality of thermal conductivity data on insulating and building materials reported in technical journals and handbooks (Achenbach 1970). In 1977, the ASTM Committee C-16 on Thermal and Cryogenic Insulating Materials recommended that this program become an official part of the NIST Standard Reference Material Program (ASTM 1978). As part of this program, Siu (1980) certified the first lot of the Standard Reference Material (SRM) 1450 Series, Fibrous Glass Board. Periodic renewals of new lots of the material have been provided in subsequent years (Hust 1985a; Zarr 1997). Since 1978, NIST has developed several thermal insulation SRMs for thermal resistance (Table 1).

Laboratory Accreditation

Interestingly, the motivation for the interlaboratory comparison was actually the result of the ASHVE's (Technical Advisory Committee on Insulation) desire to update the thermal conductivity data compilations in the *Guide* (Robinson 1950). In 1946, the committee planned to update the tabulated data in the *Guide* using only thermal conductivity data obtained in compliance with the recently approved code given in ASTM C 177. The National Bureau of Standards was asked, and agreed, to cooperate with ASHVE by conducting check tests of specimens provided to participating laboratories. Initially, check measurements were provided by NBS at approximately 4.4°C, 21.1°C, and 37.8°C (40°F, 70°F, and 100°F). Later the check measurements were provided down to -6.7°C (20°F) to extend the temperature range of interest for

refrigerating engineers. As postulated by the ASHVE committee, the overall test program consisted of three objectives (Robinson 1950):

1. A participating laboratory apparatus would be "accredited" if its thermal conductivity results on the check specimens were within 3% of those determined by the National Bureau of Standards.
2. Insulation manufacturers and trade groups would be urged to have their products tested by one of the "accredited" university or commercial testing laboratories for submission of results to the ASHVE Insulation Committee for inclusion in the *Guide*.
3. It was stipulated that the name of ASHVE should not be used in advertising by any of the participating laboratories since it was planned that the society would release, in technical media, the names of the commercial and university laboratories found "accreditable" as defined in item (1) above.

The full development of the program was impeded by a bylaw of the society that forbade the approval or accreditation of commercial laboratories. There were also other logistical issues concerning the continued adherence and compliance of equipment with time. In spite of these complications, laboratory accreditation for thermal insulation materials was to re-emerge during the 1970s as part of a much larger national program.

In 1975, the U.S. Department of Commerce publicly proposed procedures for a national voluntary accreditation program and invited public response. During the review period, more than 150 respondents provided written comments and public testimony (Bryson 1981). Based on the responses, the procedures were revised and, in 1976, the U.S. Code of Federal Regulations mandated the establishment of the National Voluntary Laboratory Accreditation Program (NVLAP) to be administered by NBS. The program's purpose

TABLE 1
NIST Standard Reference Materials for Thermal Resistance

Designation	Description	Density (kg/m ³)	Thickness (mm)	Temperature (K)	Date Issued
1) SRM 1449	Fumed silica board	300 to 330	25	297	10 Jan 1989
2) SRM 1450	Fibrous glass board	100 to 180	25	255 to 330	26 May 1978
3) SRM 1450a	Fibrous glass board	60 to 140	25	255 to 330	12 Feb 1979
4) SRM 1450b(I)	Fibrous glass board	110 to 150	25	260 to 330	21 May 1982
5) SRM 1450b(II)	Fibrous glass board	110 to 150	25	100 to 330	20 May 1985
6) SRM 1450c	Fibrous glass board	150 to 165	25	280 to 340	5 Mar 1997
7) SRM 1451	Fibrous glass blanket	10 to 16	25	100 to 330	21 May 1985
8) SRM 1452	Fibrous glass blanket	10 to 16	25	100 to 330	14 Apr 1986
9) SRM 1453	Expanded polystyrene board	38 to 46	13	285 to 310	30 Dec 1996

was to evaluate the technical qualifications of public and private laboratories in specified areas of testing (Horlick and Berger 1985). The first laboratory accreditation program for thermal insulation was initiated in 1977 in response to a request from three thermal insulation trade associations. For more than 20 years, NVLAP has been accrediting thermal insulation testing laboratories with 16 thermal laboratories currently NVLAP-accredited (NIST 2000) to the requirements of ISO/IEC Guide 25 (Knab 1995).

Measurement Terminology

In the 1970s, as part of the continued application of ASTM Test Method C 177, NBS became involved in the clarification of fundamental issues of the heat-transfer properties derived from standard test methods. Carroll (NBS) and Shirtliffe (National Research Council Canada) provided technical guidance on terminology associated with heat transfer measurements of thermal insulation (ASTM 1974). In essence, the mechanisms of heat transfer in thermal insulation involve a complicated combination of gaseous and solid conduction, radiation, and sometimes convection (and possibly mass transfer). A standard test method (such as C 177), however, only empirically determines the amount of heat flow (through a specimen) from hot-to-cold surfaces under steady-state conditions (Equation 1). Thus, strictly speaking, a test result should be expressed as a thermal conductance (or resistance) for the specimen for the given test conditions. To ascribe a particular heat-transfer property to a material (as opposed to a single specimen) requires multiple measurements on several specimens of the material under different test conditions of heat flux, temperature, and thickness. Furthermore, having satisfied this requirement, the term “apparent (or effective) thermal conductivity (or resistivity)” must be used when heat-transfer mechanisms other than conduction are present.

International Efforts

During the 1980s, NBS assisted in international standardization of the test method. Under the auspices of the International Organization for Standards (ISO), Frank J. Powell (NBS), with assistance from leaders from several other laboratories, organized an international comparison for guarded hot plates on fibrous glass insulation board (Smith 1997). The purpose was to compile statistical data on the precision of the method from a global population of laboratories. The resulting data were used to establish a baseline prior to the development of the test method as a standard by ISO. Participants from Africa, Asia, Australia, Europe, and North America conducted the comparison over several years. The analysis of the data was completed by David R. Smith (NIST) and revealed that the relative standard deviation from the fitted curve, over the temperature range of 0°C to 40°C (273 K to 313 K), was 2.4% (Smith 1997).

LATER DEVELOPMENTS WITH LINE HEAT SOURCES

In 1964, Robinson presented an elegant modification of the guarded-hot-plate test method. The basic design of a line-heat-source guarded hot plate was presented to a thermal conductivity conference sponsored by the National Physical Laboratory in England. The design was reported in *Nature* (Tye 1964) as follows:

H.E. Robinson (U.S. National Bureau of Standards) discussed forms of line heat sources that could be used as heaters in apparatus for measurements at lower temperatures on insulating materials in disk and slab form. These new configurations lend themselves more readily to mathematical analysis, they are more simple to use and would appear to be able to yield more accurate results.

The design was novel. In contrast to a (conventional) guarded hot plate that used uniformly distributed heaters, line-heat-source guarded hot plates utilized circular line heat sources at precisely specified locations. By proper location of the line heat source(s), the temperature at the edge of the meter plate can be made equal to the mean temperature of the meter plate, thereby facilitating temperature measurements and thermal guarding. The benefits offered by a line-heat-source guarded hot plate included simpler methods of construction, improved accuracy, simplified mathematical analyses for calculating the mean surface temperature of the plate as well as determining the errors resulting from heat gains or losses at the edges of the specimens, and superior operation under vacuum conditions.

The essential concepts of Robinson’s design are shown schematically in a cross section of the guarded hot plate in Figure 3. The guarded hot plate is a circular plate of highly conductive metal (λ_p) and thickness m , with a guard gap at r

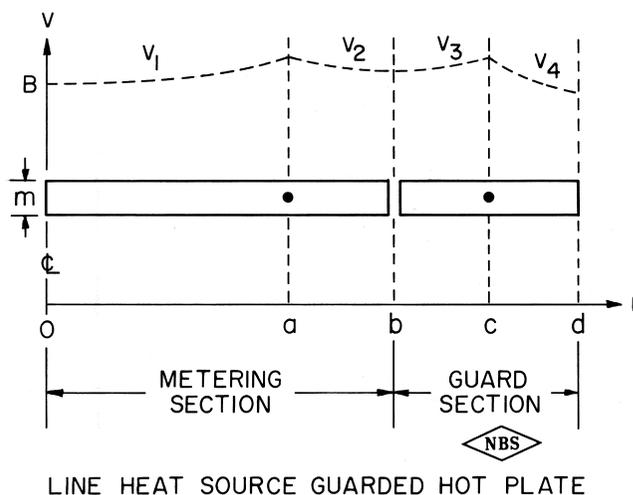


Figure 3 Schematic cross-section diagram of line-heat-source guarded hot plate (Robinson 1962).

= b dividing the meter plate and guard ring. The meter plate is heated by a single line heat source at $r = a$ and, similarly, the guard ring is heated by a single line heat source at $r = c$. The temperature profile resulting from this arrangement of heaters is indicated by dashed lines at the top of Figure 3.

Prototype Line-Heat-Source Guarded Hot Plate

After Robinson, another generation of NBS researchers continued development of the line-heat-source technology. In 1971, an in-depth analysis of the line-heat-source concept was conducted and several design options were investigated (Hahn 1971). The design, mathematical analysis, and uncertainty analysis for a prototype line-heat-source guarded hot plate were published in 1974 by M. H. Hahn, Robinson (posthumously), and D. R. Flynn (Hahn et al. 1974). Construction of the prototype apparatus was completed in 1978 (Siu and Bulik 1981). Because of the promising results from the prototype, NBS began plans for a second, larger line-heat-source guarded-hot-plate apparatus.

One-Meter Line-Heat-Source Guarded Hot Plate

In 1980, a ruling by the U.S. Federal Trade Commission concerning the labeling and advertising of home insulation dramatically accelerated the construction of this apparatus (FTC 1980). As a result of energy conservation efforts during the 1970s, there was a growing demand for thick insulations, and the Federal Trade Commission was concerned that the existing standards for insulation measurement, based on 25 mm thick specimens, were not protecting consumers' interests. The ruling required testing at "representative thickness" of labeled products. Near the end of 1980, the second line-heat-source guarded-hot-plate apparatus was completed under the efforts of Hahn, Powell, Peavy, and others (Powell and Rennex 1983). Afterwards, NBS began to provide measurements and reference artifacts of thick, fibrous-glass standards.

Further Developments

In 1996, the practice of using circular line heat sources in guarded hot plates was adopted as a standard (design) practice by the ASTM C-16 Committee on Thermal Insulation (ASTM 2000c). Currently, NIST is exploring the design of a new 500

mm guarded-hot-plate apparatus for elevated temperature that utilizes multiple line heat sources at prescribed radii.

OTHER TEST METHODS FOR SOLIDS

During the past 100 years, NIST has developed thermal-conductivity apparatus for the measurement of ceramics, refractories, loose-fill insulation, soils, and metals (Tye 1969; Ginnings 1970; Eckert and Goldstein 1976; Maglic 1992). In 1961, Robinson (1961) summarized the status of seven steady-state thermal conductivity measurement methods for solids (Figure 4, Table 2). This section briefly discusses two of

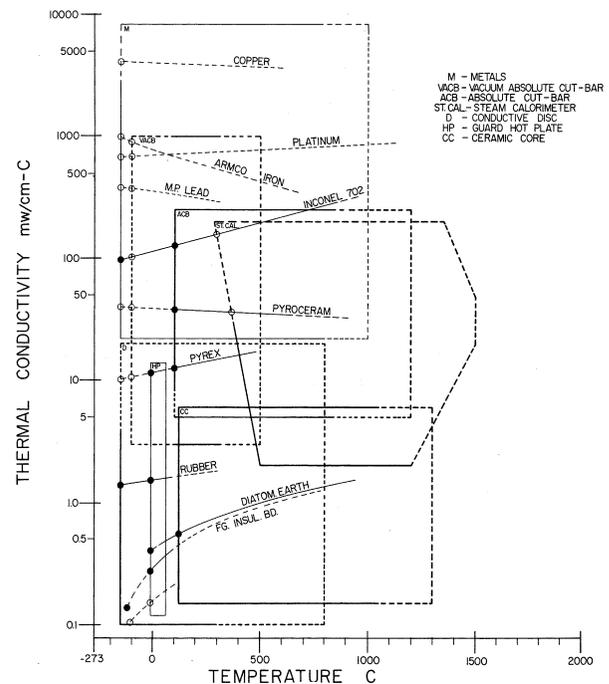


Figure 4 "Conspectus" of thermal conductivity and temperature ranges of several steady-state apparatus for solids at NBS in 1961. The NBS 200 mm guarded-hot-plate apparatus (1928 version) is denoted by symbol "HP."

TABLE 2
Robinson's 1961 Review of NBS Steady-State Test Methods for Solids Shown in Figure 4

Apparatus		Specimen		Uncertainty Estimate (%)
Symbol	Description	Material	Shape	
M	Metals	Solids	Bar, 2 cm to 4 cm diameter, 37 cm long	2
VACB	Vacuum Absolute Cut-Bar	Solids	Cylinders, 2.54 cm diameter, 1 cm to 10 cm long	3
ACB	Absolute Cut-Bar	Solids	Cylinders, 2.54 cm diameter, 1.27 cm long	3
SC	Steam Calorimeter	Refractory Solids	Disks, 15.3 cm diameter, 2 cm to 3 cm thick	5
D	Conductive Disk	Insulators	Disks, 30.5 cm diameter, 1 cm to 4 cm thick	2
GHP	Guarded Hot Plate (ASTM C 177)	Insulators	20.5 cm square, 0.5 cm to 3 cm thick	1
CC	Ceramic Core	Powders	Loose-fill, 2.5 L	5

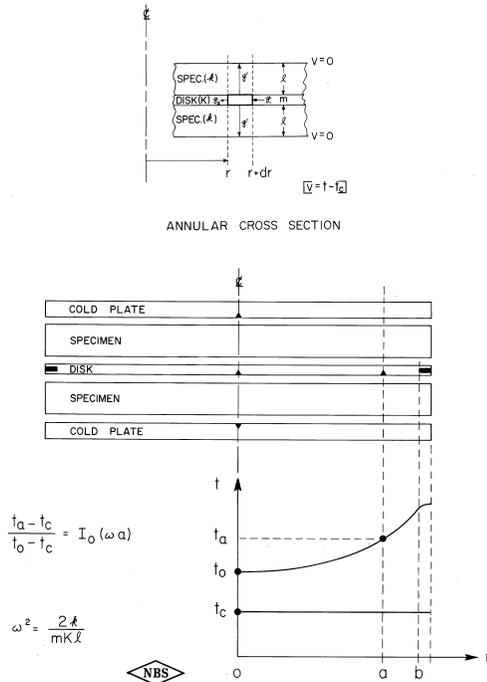


Figure 5 Schematic cross-section diagram of conductive disk (D) apparatus (Robinson 1961) for semi-rigid thermal insulation materials.

the test methods for heat insulators—the conductive disk and ceramic-core radial-heat-flow apparatus. Information on several of the other methods is available in the Bibliography.

As illustrated in Figure 4, each polygon depicts the operating range of one apparatus and is identified by a symbol (M = metals, VACB = vacuum absolute cut-bar, etc.) in the upper left corner of the polygon. Solid lines indicated established limits of operation; dashed lines indicated feasible limits. One of Robinson's goals was to utilize a battery of independent apparatus (note overlap of polygons) to define several reference materials covering an extensive range of thermal conductivities and temperatures. Van Dusen's 200 mm guarded-hot-plate apparatus (HP) covers nearly the same thermal conductivity range (but different temperature range) as the conductive disk (D) and ceramic-core (CC) apparatus (Figure 4).

Conductive Disk Apparatus

In what was a precursor to the line-source method discussed above, Robinson (1959) developed the conductive disk, a relatively simple comparative method for measuring the thermal conductivity of insulation materials. The apparatus consisted of a central disk, a pair of specimens, and two cold plates, similar in arrangement to a guarded hot plate (Figure 5). During operation, however, no direct measurements of heat input to the apparatus or heat flow were required—only measurements of temperature. The central disk was heated uniformly at the periphery by a single electric-resistance heater, and the resulting heat flowed radially inward

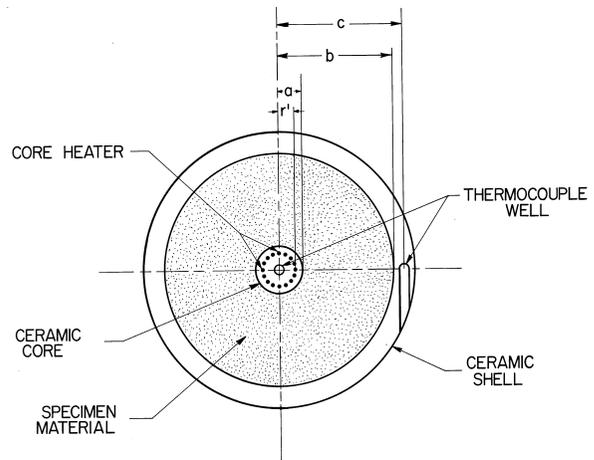


Figure 6 Horizontal cross-section of ceramic core (CC) apparatus (Flynn 1962, 1992) for granular insulation materials.

through the disk and axially through the specimens to the cold plates. By measuring the temperature at the center and suitable radius, the conductance of the specimen was determined if the temperatures of the cold plates, the thermal conductivity, and thickness of the disk were known (Robinson 1961, 1962).

Ceramic Core Apparatus

In the early 1960s, Flynn (1962, 1992) developed a ceramic-core apparatus for the measurement of thermal conductivity of granular materials from 100°C (373 K) to 1100°C (1373 K). Figure 6 is a diagram of the horizontal cross section of the essential elements of the apparatus. In contrast to the guarded-plate method discussed above, a guarded radial method promotes one-dimensional radial heat flow by minimizing undesired axial (i.e., longitudinal) heat flows. For the cylindrical geometry given in Figure 6, the algebraic form of Fourier's heat conduction equation for one-dimensional heat flow is

$$Q = 2\pi L \bar{k} \frac{-(T_a - T_b)}{\ln(b/a)} \quad (2)$$

where L is the length of the test section, T_a is the temperature at radius a , T_b is the temperature at radius b , and \bar{k} corresponds to the thermal conductivity at \bar{T} given by $\bar{T} = \frac{1}{2}(T_a + T_b)$.

CRYOGENIC AND ELEVATED TEMPERATURES

In 1951, NBS broke ground at Boulder, Colorado, to establish the Cryogenic Engineering Laboratory. As part of its mission, the laboratory studied high vacuum insulation for cryogenic applications. In the late 1950s, Kropschot (1959), Fulk (1959), and other researchers at Boulder investigated multiple-layer insulation and evacuated powders in high vacuum using several radial heat flow, boil-off calorimeters.

Later, in 1987, Dubé et al. (1987) modified a commercial boil-off calorimeter for the measurement of heat flow through flat specimens down to liquid-helium temperatures.

In 1982, Jerome G. Hust and David R. Smith modified a commercial guarded-hot-plate apparatus for stable operation at liquid-nitrogen temperatures (Smith et al. 1982). Subsequent work by Hust, Smith, and Lambert Van Poolen from 1979 to 1981 resulted in extending the certification (Table 1) of SRM 1450b, Fibrous Glass Board, and SRM 1451, Fibrous Glass Blanket, from room temperature down to -183°C (90 K) (Hust 1985a,b). In 1988, Sparks et al. (1988) used the apparatus to investigate the thermal conductivity of five cellular insulation materials from -173°C (100 K) to 27°C (300 K).

NBS Boulder also developed a high-temperature guarded hot plate for operation between 27°C (300 K) and 477°C (750 K) (Hust et al. 1990). The apparatus was used to develop high-temperature data for fumed silica board (Table 1) as a “candidate” SRM at elevated temperatures (Smith and Hust 1988).

BOX METHODS

The history of testing heat insulators at NIST would be incomplete without including a discussion on the development of the box methods for heat transmission measurements of compound walls. A complete narrative, however, merits an entire paper in itself and, regrettably, only cursory coverage of the subject is given here. In the two decades prior to 1935, Van Dusen and Finck developed a variety of calorimeters, hot boxes, and heat-flow-meter methods for determining the heat flow through walls, roofs, and floors (Van Dusen and Finck 1931). In 1937, in connection with the low-cost housing research program initiated at the bureau, R. S. Dill envisioned a guarded hot box to determine the thermal resistance values of wall assemblies. Robinson built and developed the apparatus and, in collaboration with Watson, measured the heat transmission coefficients of about 120 walls. Several of the results were published in the *Building Materials and Structures Reports* with the structural properties of the wall specimens (Achenbach 1970). Using a rotatable guarded-hot-box apparatus, NBS determined the insulating values of reflective and nonreflective airspaces (Robinson et al. 1954) and the results became generally available in the *1956 Guide*. In 1954 the guarded-hot-box method and apparatus designed at NBS was used as the basis for ASTM Standard C 236 (ASTM 2000b). In 1979, Achenbach (1979) proposed the construction of a large calibrated hot box for measuring the heat, air, and moisture transfer of room-sized (3.0 m by 4.5 m) exterior wall specimens under a range of simulated climatic conditions. The calibrated hot box was constructed and subsequently utilized for testing superinsulated wood frame and innovative masonry walls (Zarr et al. 1986; Burch et al. 1989, respectively).

SUMMARY AND CONCLUSIONS

For nearly 90 years, the National Institute of Standards and Technology (formerly the National Bureau of Standards)

has successfully developed and utilized the guarded-hot-plate method to determine steady-state thermal transmission properties of heat insulators. The original apparatus and subsequent versions have been important for the development of aggregate tabulated design data for the thermal transmission properties of heat insulators. In recent years, the apparatus at NIST has been put to use in the development of reference artifacts and standard reference materials for thermal resistance.

The 1928 version of the NBS guarded-hot-plate apparatus has served as one of the foundations for standardization as an ASTM Test Method C 177. Over the years, NIST has continually sought to improve and refine the method by extending the operating temperature range, conducting interlaboratory comparisons, improving the heat transfer analyses, and developing new heating arrangements that included line-source heaters embedded within the plates to improve the accuracy of the apparatus. These line-heat-source techniques for guarded hot plates have been developed as ASTM Standard Practice C 1043.

Historical interactions between ASHRAE, ASTM, and NIST during the past 90 years have played a major role in the direction taken in the development and advancement in testing heat insulators in North America. Indeed, the first exchange near the turn of the last century proved directly responsible for the development of the guarded-hot-plate apparatus at NIST. A later exchange resulted in one of the first interlaboratory comparisons of guarded-hot-plate apparatus, demonstrating the need for thermal insulation reference materials. If the past is indicative of the future, further interactions between ASHRAE, ASTM, and NIST should continue to benefit industry and stimulate further development of the guarded-hot-plate apparatus for testing heat insulators.

ACKNOWLEDGMENTS

The author appreciates the many conversations with Daniel R. Flynn (retired, NBS), Jeffrey Horlick (NVLAP/NIST), Dr. David L. McElroy (retired, Oak Ridge National Laboratory), John R. Mumaw (Owens Corning), Frank J. Powell (retired, NBS), Thomas W. Watson (retired, NBS), Dr. David R. Smith (NIST), and Ron Tye (retired, National Physical Laboratory) in preparing this paper. Many of the thermal insulation SRMs described herein were developed with support from the Department of Energy and Oak Ridge National Laboratory.

REFERENCES

- Achenbach, P. R. 1970. Building research at the national bureau of standards. *Building Science Series* (0): 11.
- Achenbach, P. R. 1981. Design of a calibrated hot-box for measuring the heat, air, and moisture transfer of composite building walls. *Thermal performance of the exterior envelopes of buildings, Proceedings*, (1): 308-324.
- ASTM. 1974. What property do we measure. *ASTM STP 544*, R. P. Tye, ed., pp. 5-12.

- ASTM. 1978. Reference materials for insulation measurement comparisons. *ASTM STP 660*, R. P. Tye, ed., pp. 7-29.
- ASTM. 2000a. C 177-97, Standard test method for steady-state heat flux measurements and thermal transmission properties by means of the guarded-hot-plate apparatus. *Annual Book of ASTM Standards*, (04.06).
- ASTM. 2000b. C 236-89, Standard test method for steady-state thermal performance of building assemblies by means of a guarded hot box. *Annual Book of ASTM Standards*, (04.06).
- ASTM. 2000c. C 1043-97, Standard practice for guarded-hot-plate design using circular line-heat sources. *Annual Book of ASTM Standards*, (04.06).
- Bryson, J. O. 1981. Thermal insulation laboratory accreditation program. *Thermal performance of the exterior envelopes of buildings, Proceedings*, (1): 269-282.
- Burch, D. M., B. A. Licitra, D. F. Ebberts, and R. R. Zarr. 1989. Thermal resistance measurements and calculations of an insulated concrete block wall. *ASHRAE Transactions* (95): 398-404.
- Cochrane, R. C. 1966. *Measures for progress: A history of the national bureau of standards*. National Bureau of Standards, U.S. Department of Commerce, p. 130.
- Dickinson, H. C., and M. S. Van Dusen. 1916. The testing of thermal insulators. *The American Society of Refrigeration Engineers* (3): 5-25.
- Dickinson, H. C. 1949. Letter to Professor E. B. Penrod at the University of Kentucky, July 12. NIST Museum.
- Dubé, W. P., L. L. Sparks, and A. J. Slifka. 1988. NBS boil-off calorimeter for measuring thermal conductivity of insulating materials. *Advances in cryogenic materials* (34): 67-73.
- Eckert, E. R. G., and R. J. Goldstein. 1976. *Measurements in heat transfer*. Washington: Hemisphere Publishing.
- Flynn, D. R. 1962. A radial-flow apparatus for determining the thermal conductivity of loose-fill insulations to high temperatures. *Bureau of Standards Journal of Research* (67C): 129-137.
- Flynn, D. R. 1992. Thermal conductivity of loose-fill materials by a radial-heat-flow method. *Compendium of thermophysical property measurement methods: Volume 2, Recommended measurement techniques and practices*. K. D. Maglic, A. Cezairliyan, and V. E. Peletsky, eds., pp. 33-75. New York: Plenum Press.
- FTC. 1980. *Trade regulation rule: Labeling and advertising of home insulation: 16 CFR part 460*. Federal Register, Vol. 45 (203).
- Fulk, M. M. 1959. Evacuated powder insulation for low temperatures. *Progress in cryogenics* (1): 65-84.
- Ginnings, D. C. 1970. *Precision measurement and calibration: Selected NBS papers on heat*. *NBS Special Publication 300*, (6).
- Hahn, M. H. 1971. The line source guarded hot plate for measuring the thermal conductivity of building and insulating materials. Ph.D. dissertation, Catholic University of America.
- Hahn, M. H., H. E. Robinson, and D. R. Flynn. 1974. Robinson line-heat-source guarded hot plate apparatus. *ASTM STP 544*, R. P. Tye, ed., pp. 167-192.
- Hechler, F. G., and A. J. Wood. 1928. Review of methods of testing insulating and building materials for heat transmission. *The American Institute of Refrigeration* (17): 151-178.
- Horlick, J., and H. W. Berger. 1985. NVLAP and the Thermal insulation proficiency testing program. *Journal of Thermal Insulation* (8): 278-297.
- Hust, J. G. 1985a. Glass fiberboard SRM for thermal resistance. *NBS Special Publication 260-98*.
- Hust, J. G. 1985b. Glass fiberblanket SRM for thermal resistance. *NBS Special Publication 260-103*.
- Hust, J. G., B. J. Filla, J. A. Hurley, and D. R. Smith. 1990. An automated high-temperature guarded high-temperature guarded-hot-plate apparatus for measuring apparent thermal conductivity of insulations between 300 and 750 K. *ASTM STP 1030*, D. L. McElroy and J. F. Kimpflen, eds., pp. 710-722.
- Knab, L. I. 1995. National Voluntary Laboratory Accreditation Program: Thermal Insulation Materials. *NIST Handbook* 150-15.
- Kropschot, R. H. 1959. Cryogenic insulation. *ASHRAE Journal*, September, pp. 48-54.
- Maglic, K. D., A. Cezairliyan, and V. E. Peletsky. 1992. *Compendium of thermophysical property measurement methods: Volume 2, Recommended measurement techniques and practices*. New York: Plenum Press.
- NBS. 1948. *Annual report of the national bureau of standards 1947*. U.S. Government Printing Office, pp. XIII-XIV.
- NIST. 2000. Directory: National voluntary laboratory accreditation program. *NIST Special Publication 810*.
- Péclet, E. 1863. *Traité de la chaleur*. Paris (1st edition, 1863; 4th edition, 1878).
- Poensgen, R. 1912. Ein technisches verfahren sur ermittlung der wärmeleitfähigkeit plattenförmiger stoffe. *zeitschrift des vereines deutscher ingenieure* (56): 1653-1658.
- Powell, F. J. and B. G. Rennex. 1983. The NBS line-heat-source guarded hot plate for thick materials. *Thermal performance of the exterior envelopes of buildings, Proceedings*, (2): 657-672.
- Pratt, A. W. 1969. Heat transmission in low conductivity materials. *Thermal conductivity*, R. P. Tye, ed. Vol. 1, Academic Press, London, pp. 301-405.
- Randolph, C. P. 1912. The thermal resistivity of insulating materials. *American Electrochemical Society* (21): 545-555.
- Robinson, H. E. 1950. Agreement of thermal conductivity determinations made by several testing laboratories. *Refrigerating Engineering* 58(10): 988-989, 1018-1020.

- Robinson, H. E., and T. W. Watson. 1951. Interlaboratory comparison of thermal conductivity determinations with guarded hot plates. *ASTM STP 119*, pp. 36-44.
- Robinson, H. E., F. J. Powlitch, and R. S. Dill. 1954. The thermal insulating value of airspaces. *Housing research paper No. 32*, U.S. Government Printing Office.
- Robinson, H. E., D. R. Flynn, and T. W. Watson. 1959. Measurements of the thermal conductivity of several explosives by the conductive disc method. *NBS Report 6527*.
- Robinson, H. E. 1961. Current NBS steady-state thermal conductivity measurement methods. *Conference on thermal conductivity methods*, Battelle Memorial Institute, Columbus, Ohio, (1): 11-27.
- Robinson, H. E. 1962. The conductive-disk method of measuring the thermal conductivity of insulations. *International Institute of Refrigeration*, August 20-29, pp. 43-50.
- Siu, M. C. I. 1980. Fibrous glass board as a standard reference material for thermal resistance measurement systems. *ASTM STP 718*, D. L. McElroy and R. P. Tye, eds., pp. 343-360.
- Siu, M. C. I. and C. Bulik. 1981. National bureau of standards line-heat-source guarded-hot-plate apparatus. *Review of scientific instruments* 52: 1709-1716.
- Smith, D. R., J. G. Hust, and L. J. Van Poolen. 1982. A guarded-hot-plate apparatus for measuring effective thermal conductivity of insulations between 80 K and 360 K. *NBSIR 81-1657*.
- Smith, D. R., and J. G. Hust. 1988. Microporous fumed-silica insulation board as a candidate standard reference material of thermal resistance. *NISTIR 88-3901*.
- Smith, D. R. 1997. Thermal conductivity of fibrous glass board: an international intercomparison for guarded hot plates and heat flow meters. *NIST Technical Note 1391*.
- Sparks, L. L., W. P. Dubé, and A. J. Slifka. 1988. Thermal conductivity of selected foams and systems from 100 to 300 K. *NBSIR 88-3086*.
- Tye, R. P. 1964. Thermal conductivity. *Nature* (204): 636.
- Tye, R. P. 1969. *Thermal conductivity*, London: Academic Press.
- Van Dusen, M. S. 1920. The thermal conductivity of heat insulators. *The American Society of Heating and Ventilating Engineers* (26): 385-414; also *The American Society of Refrigerating Engineers* (7): 202-231.
- Van Dusen, M. S. and J. L. Finck. 1928. Heat transfer through insulating materials. *The American Institute of Refrigeration* (17): 137-150.
- Van Dusen, M. S. and J. L. Finck. 1931a. Heat transfer through building walls. *Bureau of Standards Journal of Research* (6): 493-522.
- Van Dusen, M. S. and J. L. Finck. 1931b. The refrigerator cabinet: new heat flow studies. *Refrigerating Engineering* (22): 310-313, 385-387, 406.
- Worthing, A. G. and D. Halliday. 1948. *Heat*. New York: John Wiley & Sons, Inc., pp. 181-182.
- Zarr, R. R., D. M. Burch, T. K. Faison, and C. E. Arnold. 1986. Thermal resistance measurements of well-insulated and superinsulated residential walls using a calibrated hot box. *ASHRAE Transactions* (92): 604-619.
- Zarr, R. R. 1997. Glass fiberboard, SRM 1450c, for thermal resistance from 280 K to 340 K. *NIST Special Publication 260-130*.
- Zarr, R. R., G. R. Dalton, and S. M. Fioravante. 1999. Development of a NIST standard reference database for thermal conductivity of building materials. *Thermal Conductivity (25) - Thermal Expansion* (13), pp. 259-265.

BIBLIOGRAPHY

- Finck, J. L. 1930. Mechanism of heat flow in fibrous materials. *Bureau of Standards Journal of Research* (5): 973-984.
- Flynn, D. R., and D. A. Didion. 1961. A steam calorimeter apparatus for refractories. *Conference on thermal conductivity methods*, Columbus, Ohio: Battelle Memorial Institute, (1): 133-150.
- Flynn, D. R. and M. E. O'Hagan. 1967. Measurements of the thermal conductivity and electrical resistivity of platinum from 100 to 900 °C. *Bureau of Standards Journal of Research* (71C): 255-284.
- Hust, J. G. and P. J. Giarratano. 1975a. Thermal conductivity and electrical resistivity standard reference materials: Austenitic stainless steel, SRM's 735 and 798 from 4 to 1200 K. *NBS Special Publication 260-46*.
- Hust, J. G. and P. J. Giarratano. 1975b. Thermal conductivity and electrical resistivity standard reference materials: electrolytic iron SRM's 734 and 797 from 4 to 1000 K. *NBS Special Publication 260-50*.
- Hust, J. G. and P. J. Giarratano. 1975c. Thermal conductivity and electrical resistivity standard reference materials: Tungsten SRM's 730 and 799 from 4 to 3000 K. *NBS Special Publication 260-52*.
- Powell, R. L., W. M. Rogers, and D. O. Coffin. 1957. Apparatus for measurement of thermal conductivity of solids at low temperatures. *Bureau of Standards Journal of Research* (59): 349-355.
- Shelton, S. M. and W. H. Swanger. 1933. Thermal conductivity of irons and steels and some other metals in the temperature range 0 to 600 C. *American Society for Steel Treating* (21): 1061-1078.
- Shelton, S. M. 1934. Thermal conductivity of some irons and steels over the temperature range 100 to 500 C. *Bureau of Standards Journal of Research* (12): 441-450.
- Van Dusen, M. S. 1922. A simple apparatus for comparing the thermal conductivity of metals and very thin specimens of poor conductors. *Journal of Optical Society of America and Review of Scientific Instruments*, (6): 739-743.
- Van Dusen, M. S. 1930. Note on the theory of heat conduction. *Bureau of Standards Journal of Research* (4): 753-756.

- Van Dusen, M. S. 1931. Note on applications of data on the thermal conductivity of metals. *Symposium on effect of temperature on the properties of metals*, pp. 545-549.
- Van Dusen, M. S. 1934. Apparatus for measuring thermal conductivity of metals up to 600 C. *Bureau of Standards Journal of Research* (12): 429-440.
- Watson, T. W. and H. E. Robinson. 1961. Thermal conductivity of some commercial iron-nickel alloys. *ASME Journal of Heat Transfer* (83C): 403-408.
- Watson, T. W., D. R. Flynn, and H. E. Robinson. 1967. Thermal conductivity and electrical resistivity of armco iron. *Bureau of Standards Journal of Research* (71C): 285-291.
- Watson, T. W. and D. R. Flynn. 1968. Thermal conductivity of four heat-resistant alloys. *Metallurgical Society of AIME* (242): 844-846.

APPENDIX – NOTE ON THE ORIGIN OF THE HOT PLATE METHOD

The systematic investigations by the French physicist Péclet (circa 1860) are recognized as the first experiments of heat transmission of “poor conductors of heat” (Péclet 1863). Using rectangular plates insulated around the edges, one surface heated by steam and the other exposed to the free air of a constant temperature chamber, Péclet determined the thermal conductivity of various solid materials such as marble, limestone, brick, wood, and cork, among others (Hechler and Wood 1928). Among the earliest experiments approaching the modern method, involving a pair of specimens of poor conductors, was reported by Lees in England in 1898 (Worthing and Halliday 1948). Circular specimens were clamped between three circular plates of copper forming a

cylindrical apparatus. Heat was produced from the electrically heated center plate and conducted through the specimens to the copper plates. Richard Poensgen in Germany improved upon Lee’s method by the addition of guard rings provided for the central plate. The improvement over Lee’s design consisted largely in the greater certainty of unidirectional heat flow, particularly for specimens of such thickness that lateral heat losses might not be disregarded (Worthing and Halliday 1948). Thus, Poensgen is generally recognized for developing the first truly guarded hot-plate apparatus (Poensgen 1912).

It is interesting to note that Dickinson’s biographical papers mentioned that, while traveling in Europe, he learned that Richard Poensgen in Germany had independently developed and been using a guarded hot plate for thermal conductivity measurements since 1910. In a letter, Dickinson (1949) writes,

The writer and Dr. Van Dusen have the impression, separately, that the 1912 hot-plate apparatus was the first built in this country to their knowledge. Possibly some type of hot-plate apparatus had been built in Germany before 1912, but so far as is known the work at the Bureau was done without acquaintance with such a development.

To be accurate, there was also other related work ongoing in industry in the United States prior to the institute’s involvement. An early review by Hechler and Wood (1928) cites a 1910 report by C. L. Norton to Armstrong Cork Company on insulation tests describing several test methods, including a small flat plate, electrically heated but without a guard annulus. Using a cylindrical apparatus with an unguarded electrically heated copper plate, Randolph (1912) measured the thermal resistivity of several insulating materials.