# Status of NIST Thermal Insulation Reference Materials

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## ABSTRACT

The current status of thermal insulation reference materials at the National Institute of Standards and Technology (NIST) is presented. This paper describes an ongoing thermal insulation measurement program that provides the public with Calibrated Transfer Specimens (CTS) and Standard Reference Materials (SRMs). The main distinction between these two types of reference materials is that a transfer specimen is measured individually in a guarded-hot-plate apparatus. Consequently, the value assignment and uncertainty is based on the individual measurement data for each material specimen. In contrast, a thermal insulation SRM is a batch of homogeneous material. The value assignment and uncertainty are based on the statistical characterization of a homogeneous lot of material as represented by selected samples from the lot. The paper summarizes a brief history of the thermal insulation measurement program and describes both reference materials, their current status, as well as the future outlook of the program.

## INTRODUCTION

Past interactions with different user communities have resulted in a thermal insulation program at NIST that provides two groups of reference materials—CTS and SRMs. Thermal insulation reference materials are provided to user communities for laboratory accreditation programs, measurement comparisons, equipment calibration, traceability, and internal quality-control systems, among other uses. In general, the materials are utilized by standardized test methods for the purposes of checking guarded-hot-plate apparatus, calibration of heat-flow-meter apparatus, and, occasionally, for checking or calibrating hot-box apparatus. Thermal insulation reference materials are also provided for U.S. insulation manufacturers to assist in complying with federal requirements for labeling and advertising of home insulation (hereafter, the U.S. Federal Trade Commission "R-value Rule" [1]).

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In 1983, Hust [2] summarized the status of thermal conductivity SRMs at the National Bureau of Standards (NBS)[2] that encompassed four ranges of conductivities: high, medium, low, and very low. This paper updates the fourth range—very low thermal conductivity, nominally 0.03 $W \cdot m^{-1} \cdot K^{-1}$ at 300 K, and generally measured in a guarded-hot-plate apparatus. It should be noted that thermal insulation reference materials are typically fibrous or cellular and, in either case, exhibit non-conductive mechanisms of heat transfer. As a result, the term "apparent" thermal conductivity is included by some experimentalists when describing the thermal transmission properties of heat insulators determined from standard test methods. In this paper, however, the term thermal conductivity will be used for brevity.

## HISTORICAL DEVELOPMENTS

Table 1 summarizes the historical development of NIST thermal insulation reference materials. Before 1958, customers submitted their own test specimens for measurement to NBS for an accurate determination of thermal resistance. In 1958, NBS responded to increasing requests for "thermal conductivity reference specimens" by stockpiling two materials having satisfactory characteristics of homogeneity and stability—fibrous-glass board and gum rubber [3]. From 1958 to 1978, NBS [4] provided over 300 pairs of "calibrated reference specimens" selected from four lots of fibrous-glass board (identified by year of purchase: 1958, 1959, 1961, and 1970).

In 1978, ASTM C16.30 Sub-Committee on Thermal Measurements published a position paper [5] advocating the SRM approach for thermal insulation reference materials. Based on this recommendation, NBS developed three thermal insulation SRMs: fibrous-glass board, 1450, (and ensuing lots 1450a, 1450b, and 1450c); fibrous-glass blanket, 1451 and 1452; and, fumed-silica board, 1449 (and a dimensionally smaller unit, 1459). In 1996, as a result of a request from the National Fenestration Rating Council (NFRC), NIST issued expanded polystyrene board 1453 for use in the calibration procedure for testing windows in a hot box.

**Table 1.** Chronology of NIST Thermal Insulation Reference Materials.

| SRM or CTS Designation | Reference Material | Year Issued | Comment | Reference |
|---|---|---|---|---|
| --- | NBS Fibrous-glass board | 1958 | Superseded by 1450 | [3,4] |
| --- | NBS Gum rubber | 1958 | Discontinued in 1982 | [3,6] |
| 1450 | Fibrous-glass board | 1978 | Superseded by 1450a | [4] |
| 1450a | Fibrous-glass board | 1979 | Superseded by 1450b(I) | — |
| CTS | Low-density fibrous-glass blanket | 1981 | R-value Rule [1] | [7-9] |
| 1450b(I) | Fibrous-glass board | 1982 | Superseded by 1450b(II) | — |
| 1450b(II) | Fibrous-glass board | 1985 | Superseded by 1450c | [10,11] |
| 1451 | Fibrous-glass blanket | 1985 | Discontinued in 1997 | [11,12] |
| 1452 | Fibrous-glass blanket | 1986 | Sub-sample of 1451 | [12,13] |
| 1449 | Fumed-silica board | 1989 | Dual SRM 1459 | [14–16] |
| 1453 | Expanded polystyrene board | 1996 | In service | [17] |
| 1450c | Fibrous-glass board | 1997 | In service | [18] |

[2]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 Competiveness Act, the name was changed to the National Institute of Standards and Technology (NIST) to reflect the agency's broader mission. For historical accuracy, this paper will use, where appropriate, NBS for events prior to 1988.

## CALIBRATED TRANSFER SPECIMENS

NIST Calibrated Transfer Specimens (CTS) for thermal resistance, also known as transfer specimens, are measured directly in a NIST guarded-hot-plate apparatus. The test artifact is subsequently issued to the customer with an assigned measurement value, uncertainty, and test report. Steady-state thermal transmission properties, such as thermal resistance ($R$) or thermal conductivity ($\lambda$), are determined using Fourier's one-dimensional heat conduction equation (in algebraic form), that is:

$$Q = \lambda A \frac{(T_h - T_c)}{L} \tag{1}$$

The steady-state thermal transmission properties determined in Eq (1) and their associated uncertainties are valid only for the specimen issued to the customer and for the measured values of specimen heat flow ($Q$), meter area ($A$), plate temperatures ($T_h$, hot face and $T_c$, cold face), and in-situ test thickness ($L$). Other (influence) quantities defined in the test method, including clamping pressure, plate emittance, atmospheric pressure, and specimen conditioning temperature, among others, are specified in the NIST *Report of Test* issued to the customer.

The principal CTS reference material is a low-density (10 $kg \cdot m^{-3}$) fibrous-glass blanket (Table 1) that is issued at thicknesses of 25 mm, 75 mm, or 150 mm and provides U.S. insulation manufacturers with (thick) standards for compliance with provisions in the R-value Rule [1]. This lot of material was purchased by NBS in 1980 and is currently maintained by the NIST Building and Fire Research Laboratory (BFRL). In most cases, measurements are conducted at 297 K (23.9°C) and a temperature difference of 22 K or 28 K across the specimen using the NIST 1016 mm guarded-hot-plate apparatus [19]. At 297 K, the value assignment for the CTS fibrous-glass blanket is nominally 0.046 $W \cdot m^{-1} \cdot K^{-1}$ having expanded uncertainties ($k = 2$) of 0.0004 $W \cdot m^{-1} \cdot K^{-1}$, 0.0006 $W \cdot m^{-1} \cdot K^{-1}$, and 0.0011 $W \cdot m^{-1} \cdot K^{-1}$ at thicknesses of 25 mm, 75 mm, and 150 mm, respectively. As noted in Eq (1), values of $Q$ decrease with larger values of $L$. Consequently, the uncertainty components for $Q$ have a larger effect and, when propagated, increase the combined standard uncertainty for $\lambda$ (or $R$).

## STANDARD REFERENCE MATERIALS

A NIST Standard Reference Material® (SRM®) is a Certified Reference Material[3] (CRM) issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its

[3]The ISO *International Vocabulary of Basic and General Terms in Metrology* (VIM): 1993 [23] defines the following:

Reference Material (RM)—Material, sufficiently homogeneous and stable regarding one or more properties, used in calibration, in assignment of a value to another material, or in quality assurance.

Certified Reference Material (CRM)—Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.

characterizations and provides information regarding the appropriate use(s) of the material [20]. The certificate terminology and contents are prepared in accordance with ISO Guide 30 [21] and ISO Guide 31 [22], respectively.

In contrast to a NIST CTS (described above), the thermal conductivity ($\lambda$) of insulation SRMs is characterized statistically as a function of bulk density ($\rho$), mean temperature ($T_m$), and, when necessary, ambient pressure ($P$). Other (influence) quantities such as clamping pressure, plate emittance, among others, are controlled, or observed, during the measurement process. It is important to emphasize that the SRM unit issued to a customer is not measured directly in a guarded-hot-plate apparatus (as is the case for a NIST CTS). Consequently, the uncertainty statement for a thermal insulation SRM contains an additional component of uncertainty (usually small) due to the material variability within the lot. The advantage of the SRM approach is realized by characterizing a large quantity of units that are economical and available on demand.

Table 2 summarizes the thermophysical properties of the current NIST thermal insulation SRMs. Fumed-silica board (1449 and 1459) is a micro-porous material that limits the mean-free-path length of nitrogen and oxygen molecules resulting in a very low apparent thermal conductivity (Table 2). Although certified thermal transmission values are currently available only at 297 K (Table 2), the material is entirely inorganic and, thus, potentially useful for high-temperature conditions. Fibrous-glass blanket (1452) is similar to the low-density fibrous-glass blanket CTS, described above, but has a slightly higher bulk density (14 kg·m$^{-3}$ compared to 10 kg·m$^{-3}$ for CTS).

**Table 2.** Thermophysical Properties of Current Thermal Insulation SRMS.

| SRM | Description | $\lambda$ at 300 K (W·m$^{-1}$·K$^{-1}$) | $\rho_{nominal}$ (kg·m$^{-3}$) | Unit Size (mm) | $T_m$ (K) |
|---|---|---|---|---|---|
| 1449 | Fumed-silica board* | 0.02 | 320 | 25.4×600×600 | 297 |
| 1450c | Fibrous-glass board | 0.03 | 160 | 25.4×610×610 | 280 to 340 |
| 1452 | Fibrous-glass blanket | 0.04 | 14 | 25.4×600×600 | 100 to 330 |
| 1453 | Expanded polystyrene board | 0.03 | 40 | 13.4×660×930 | 281 to 313 |
| 1459 | Fumed-silica board* (see 1449) | 0.02 | 320 | 25.4×300×300 | 297 |

*The certified pressure range for 1449 and 1459 is 97 kPa to 102.0 kPa (barometric site conditions at Gaithersburg, Maryland).

The statistical characterizations of recently issued SRMs 1453 and 1450c (Table 1) were developed in collaboration with the NIST Statistical Engineering Division [24]. Considering SRM 1450c as a case in point, the underlying statistical model was assumed to be

$$\lambda(\rho,T_m) = a_0 + a_1\rho + a_2T_m + a_3T_m^2 + a_4T_m^3 \qquad (2)$$

where the $T_m^3$ term is included to account for any significant effects due to radiation heat transfer. To verify the adequacy of Eq (2), a full factorial experimental design with 3 levels of $\rho$ and 5 levels of $T$ was selected. This design also provides a means for checking the necessity of additional terms (i.e., $\rho^2$, $T_m^4$, and/or a $\rho T_m$ cross product).

In order to select matched pairs of test specimens, the bulk density ($\rho$) of each board in the material lot was determined and was ranked by $\rho$ (from lowest to highest). Three pairs of specimens (low-, mid-, and high-$\rho$) were selected and a different pair of specimens was tested in a random sequence at each level of $T_m$. The advantage of testing a unique pair of specimens at each level of $T_m$ is that completely new and independent information is obtained. In contrast, testing the same pair of specimens sequentially at each level of $T_m$ introduces some bias from the previous measurement.

Regression analyses of the data for 1450c were carried out using the NIST computer program Dataplot™ [25]. The final prediction model was

$$\hat{\lambda} = -7.7663 \times 10^{-3} + 5.6153 \times 10^{-5} \rho + 1.0859 \times 10^{-4} T_m \quad (3)$$

The deviations of the fit, that is, the predicted values minus the data, were plotted as a function of $T_m$. There was no improvement noted in the deviations by inclusion of the higher order temperature terms and, thus, a linear fit in temperature was deemed sufficient for the temperature range of 280 K to 330 K.

The effect of moisture content is minimized by appropriate conditioning of the material prior to thermal tests as described in the *Instruction for Use* section in each SRM certificate. In the case of 1453, the material is a closed-cell plastic foam and is conditioned at ambient conditions for several days, if necessary, until stable mass is obtained. For other SRM materials (Table 2), the certificate specifies a conditioning interval of 24 h at 368 K ± 5 K prior to testing.

## STATUS

Figure 1 summarizes the number of measurements for thermal insulation CTS issued to the public from 1981 to 2006 (cross-hatched bars). The majority of measurements were issued in 1981, immediately after the reference material was made available to the public. In subsequent years, the number of measurements has averaged less than 10 per year with the exception of 1995 when several organizations requested renewals. Interestingly, as observed in Figure 1, a small number of organizations have purchased renewal specimens while other organizations have submitted their own materials for measurement as transfer specimens (open bars)[4].

The data in Figure 1 indicate a pattern reminiscent of the period prior to 1958 in which customers submitted their own test specimens for measurement to NBS (described above). The proportion of industry materials submitted may increase in the future because the CTS lot, which has an indefinite shelf life (when stored in an indoor environment), was manufactured nearly 30 years ago using production technologies that have changed considerably. There is, however, a sufficient stockpile of CTS specimens to maintain the current rate of issue for about 5 years.

[4]Although the R-value Rule contains no specific requirements to renew standards, manufacturers must ensure that their advertised R-values, yielded from past testing, are consistent with the R-values of material actually produced and sold to consumers, within tolerances specified by the Rule [1].

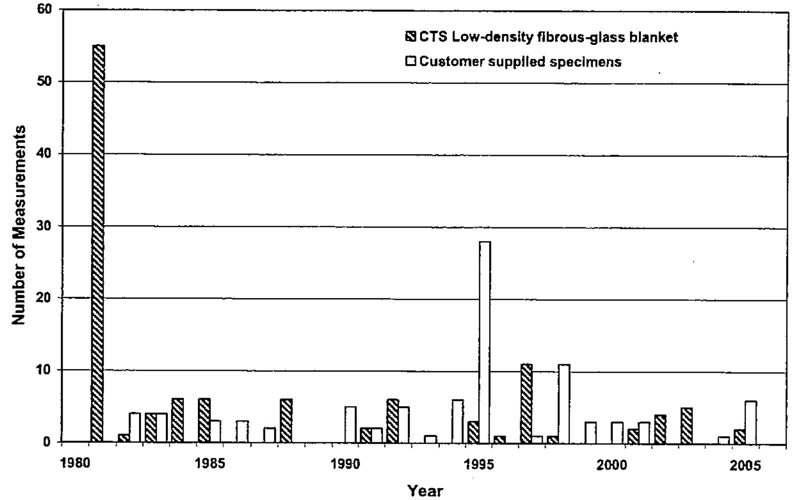


*Figure 1. Measurements of Thermal insulation CTS and Transfer Specimens: 1980 to 2006.*

Figure 2 summarizes the number of units of current thermal insulation SRMs (Table 2) issued to the public from 1988 to 2007. The majority of the specimens issued are primarily 1450c (Fibrous-glass board, 25 mm) and 1453 (Expanded polystyrene board, 13 mm). Only a few units of 1449, 1459 (Fumed-silica board), and 1452 (Fibrous-glass

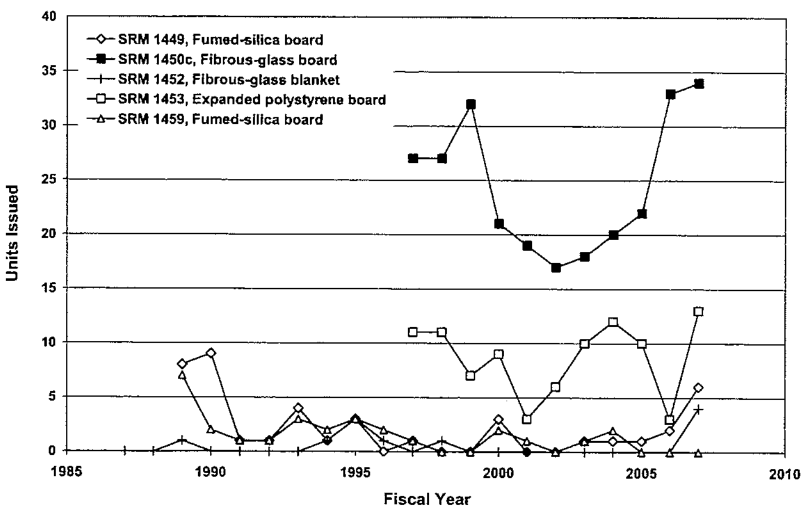


*Figure 2. Thermal insulation NIST SRMs issued: 1988 to 2007.*

blanket) are currently issued each year (typically less than 5 units). In the case of 1452 and discontinued 1451 (both having bulk densities of 14 $kg{\cdot}m^{-3}$), most customers prefer to purchase the low-density fibrous-glass blanket CTS (10 $kg{\cdot}m^{-3}$) described above.

For 1449 and 1459 (Fumed-silica board), the original objective, as defined in the ASTM C16.30 Sub-Committee on Thermal Measurements position paper [5], was to satisfy a two-fold need. The material has a very low thermal conductivity (Table 2), similar in value to newly-manufactured refrigerant-blown plastic foams, and is entirely inorganic in composition and, thus, suitable for high-temperature applications (up to 650°C [15,26-27]). Perhaps the demand for 1449 and 1459 would increase if a high-temperature characterization was developed (as originally proposed [5]). However, it should be noted that a recently formed ASTM C16.30 Reference Material Task Group is considering other candidate materials for high temperature.

## FUTURE ACTIVITIES

Currently, there is an enormous effort underway at NIST to implement a uniform and consistent Quality System for NIST laboratories that provide measurement services to the public, specifically calibration services and SRMs. For calibration services, laboratories must develop their Quality System in accordance with International Standard ISO/IEC 17025 [28] and, for SRMs, in accordance with International Standards ISO/IEC 17025 [28] and Guide 34 [29]. Full implementation requires the development and subsequent assessment of Quality Manuals and Quality Systems for NIST laboratories involved in Calibration Services or the technical development of SRMs. In the future, the results of this activity will affect the development of all new thermal insulation SRMs, as well as the calibration services provided for thermal insulation CTS and transfer specimens.

The development of a new thermal insulation SRM is only undertaken upon the receipt of a formal request from an outside organization. As alluded to above, a recently formed ASTM C16.30 Reference Material Task Group has begun the process of ultimately recommending candidate materials for development as a high-temperature thermal insulation SRM. At present, the task group has prepared and distributed a questionnaire to members of Committee C16 on Thermal Insulation and other interested ASTM committees for input. Results from the questionnaire and subsequent study will be summarized in a future publication. In preparation for these activities, NIST has designed and fabricated a 500 mm guarded-hot-plate apparatus for use in controlled atmospheres over a temperature range of 90 K to 900 K [30–32].

## SUMMARY

NIST (formerly NBS) has officially issued thermal insulation reference materials to the public from 1958 to the present. These reference materials were first issued as thermal conductivity transfer specimens. Later, at the recommendation of the ASTM C16.30 Sub-committee on Thermal Measurement, SRMs were also issued. The main difference between these two types of reference materials is that a Transfer Specimen is measured individually in a NIST guarded-hot-plate apparatus. The value assignment and uncertainty are determined from the measurement data. Thermal insulation SRMs are prepared from a lot of homogeneous material and, thus, the value assignment and uncertainty are based on the statistical characterization of a homogeneous lot of material as represented by selected samples from the lot.

Current thermal insulation SRMs, with the exception of SRM 1452 (Fibrous-glass blanket), cover temperatures at or near 297 K (Table 2). An ASTM C16.30 task group is examining potential candidates that may be suitable for subsequent development by NIST as high-temperature reference materials. With regards to Transfer Specimens, NIST has issued low-density, fibrous-glass blanket CTS beginning in 1981 (at thicknesses of 25 mm, 75 mm, and 150 mm) to assist U.S. insulation manufacturers in complying with federal requirements for labeling and advertising of home insulation. This lot of material, however, was manufactured nearly 30 years ago. The potential replacement of this lot of material will be an important issue for future discussion and research.

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