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Standard Reference Material 1450d, Fibrous Glass Board, for Thermal Insulation Measurements

Reference

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ABSTRACT

Thermal conductivity measurements at and near room temperature are presented as the basis for certified values of thermal conductivity for Standard Reference Material 1450d, Fibrous Glass Board. The measurements have been conducted in accordance with a randomized full factorial experimental design with two variables, bulk density and temperature, using the National Institute of Standards and Technology 1016 mm line-heat-source guarded-hot-plate apparatus. The thermal conductivity of the specimens was measured over a range of bulk densities from 114 to 124 kg/m³ and mean temperatures from 280 to 340 K. Uncertainties of the measurements, consistent with current international guidelines, have been prepared. A summary of the physical properties and associated analyses of Standard Reference Material 1450d are presented.

Keywords

bulk density, certified reference material, guarded hot plate, heat flow meter, high density molded fibrous glass board, standard reference material, thermal conductivity, thermal insulation, thermal resistance

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Introduction

For 35 years, the National Institute of Standards and Technology (NIST) has issued thermal insulation Standard Reference Materials (SRMs)⁴ having certified value assignments for thermal resistance, thermal conductivity, and, most recently, bulk density. These Certified Reference Materials are provided by NIST as primary tools to assist user communities in achieving measurement quality assurance and metrological traceability. Thermal insulation SRMs, in particular, are utilized in standard test methods for the purposes of checking guarded-hot-plate apparatus [1], calibrating heat-flow-meter apparatus [2], and, when necessary, for checking or calibrating hot-box apparatus [3].

Standard Reference Material 1450 was originally issued by NIST (formerly the National Bureau of Standards⁵) in 1978 and was recently renewed in 2011 with the 1450d batch [4]. Like previous versions, SRM 1450d is a semi-rigid high-density, molded fibrous glass board consisting of fine glass fibers and a phenolic binding agent that limits the upper (test) temperature of the material to 473 K. Each unit is 26 mm thick and has lateral dimensions of 611 mm by 611 mm. The nominal thermal conductivity at 300 K is approximately 0.033 W/(m \cdot K). This paper describes the production and certification of SRM 1450d and, in particular, summarizes the material lot requirements, bulk density homogeneity and thermal conductivity batch studies.

Materials

The general requirements for the renewal lot were based on recommendations from SRM customers and members of the ASTM Sub-committee C16.30 Task Group on Reference Materials. The material consignment was processed within a 3-day period and delivered to NIST in April 2009. Table 1 summarizes the manufacturing information for the SRM 1450d material lot. The consignment included 25 molded sheets having the same nominal density as the material described in Table 1; however, the finished lateral dimensions were larger, nominally 1200 mm by 1200 mm. These large sheets were from the same production run and were utilized later as lateral guarding for the thermal conductivity measurements.

Bulk Density Homogeneity Study

The objective of the homogeneity study was to quantify the density variability of the 1450d material lot (Table 1) by 100 % sampling of the panels and, thus,

⁴ The term "Standard Reference Material" and the diamond-shaped logo which contains the term "SRM," are registered with the United States Patent and Trademark Office.

⁵ 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 a broader mission for the agency.

TABLE 1 Manufacturing data for SRM 1450d.

Attribute	Value
Bulk density	128 kg/m $^{3} \pm$ 10 %
Mold size	1245 mm by 1930 mm
Number of molded sheets	75
Number of panels per sheet	6 (die cut from molded sheet)
Number of panels	450
Nominal panel size	610 mm by 610 mm by 25.4 mm
Panel color	amber

determine bulk density rank (including upper and lower limits) and any anomalous panels (for possible exclusion). The 450 panels were randomly split into 9 groups of 50. Each group was randomly assigned to a 3-day interval in which the density of all 50 panels was determined. The sequence of events performed within the 3-day interval is shown in Table 2. The entire measurement process, which entailed several thousand mass and dimensional measurements for all 450 panels, required 30 days for completion [4].

BULK DENSITY MEASUREMENTS

The bulk density of the specimen (ρ_s), as defined in ASTM C177 [1], was determined by gravimetric and dimensional measurement procedures using Eq 1

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

where:

(1)

 $m_s =$ specimen mass, kg,

 A_s = specimen surface area, m², and

L = specimen thickness, m.

MASS MEASUREMENTS

The mass measurement station consisted of: (1) digital weighing balance (32.1 kg range, 0.0001 kg resolution), (2) foot switch for manual activation, and (3) serial interface for automated data collection. As described in Table 2, each panel was

TABLE 2	Sampling plan	for bulk density	homogeneity study ^a .
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Day	Temperature, $^{\circ}C$	Time, h	Event
1	100	24	50 panels conditioned in convection oven (100°C)
2	23	4	Mass time-history for each panel
2	23	17	50 panels conditioned in ambient (24°C)
3	23	4	Dimensional measurements of each panel

^aSequence of events for 3-day interval in which the density of all fifty panels are determined.

removed individually from an oven environment of 100° C and weighed every 20 s for 180 s. By measuring the panel mass at equal time intervals and establishing a mass history, the initial mass (m_0) for each panel at time zero (t_0) was determined by linear regression, thus correcting for the small mass change with time.

DIMENSIONAL MEASUREMENTS

The dimensional measurement station consisted of: (1) granite surface plate (1.2 m by 1.8 m), (2) electronic height gage with digital readout (330 mm range or 635 mm range, 0.01 mm resolution), (3) bi-directional touch probe (3-mm diameter carbide ball contact point, 0.4 N measuring force), and (4) output cable for automated data collection. As described in Table 2, the group of panels was conditioned in air at conditions near 24°C. Each panel was placed on the granite surface plate and lateral and thickness dimensional measurements were carried out by two operators using separate height gages. The lateral dimensions, identified as length (*l*) and width (*w*), were measured by clamping the insulation panel securely in the vertical position (one edge on the granite surface plate) between an aluminum plate and a right-angle support fixture. Linear dimensions were obtained at the center lines of the panel. Preliminary tests indicated that data acquired from the two middle axes were sufficient for an accurate determination of bulk density. One panel from each group, however, was selected at random for measurements at six locations (three equally spaced locations per side of panel) as a check.

For thickness dimensions, the insulation panel was placed in the horizontal position (lateral surface on the granite surface plate) and a modest load (approximately 43 N) was applied to the top of the panel. The panel thickness was measured at four locations, with each location representing the geometric center of a 200 mm by 200 mm sub-area of the panel. Preliminary tests indicated that the data acquired from four locations were sufficient for an accurate determination of the bulk density. For each group, approximately one-half of the panels were measured at the corner sub-areas and, for the other half, at the mid-center sub-areas. One panel from each group was selected at random for measurements at all eight locations.

SUMMARY STATISTICS

Table 3 provides summary statistics across the panels for the mass (m_s) , length (l), width (w), area (A_s) , thickness (L), and bulk density (ρ_s) of the 450 panels. The values for l and w are within acceptable limits. The large range and small standard deviation for L indicate that some of the panels were either unacceptably thin or thick. The mean and standard deviation values for ρ_s are acceptable, but the mean is near the low limit requirement (Table 1).

BULK DENSITY VARIATION

Figure 1 plots the bulk density for the 450 panels in rank order. There were two panels outside the upper and lower limits equal to the grand mean plus or minus

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Statistic	<i>m_s</i> , kg	<i>I</i> , mm	<i>w</i> , mm	<i>A_s</i> , m ²	<i>L</i> , mm	$ ho_{s}$, kg/m ³
Mean	1.1466	610.92	610.85	0.37318	25.88	118.7
Std. dev.	0.0250	0.62	0.56	0.00051	0.18	2.9
Range	0.1635	2.85	3.00	0.00243	1.74	18.0
Min	1.0697	609.50	609.55	0.37195	25.25	109.8
Max	1.2331	612.35	612.55	0.37438	26.99	127.8

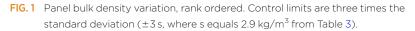
TABLE 3	Summary statistics for	SRM 1450d bulk densit	y homogeneity study.
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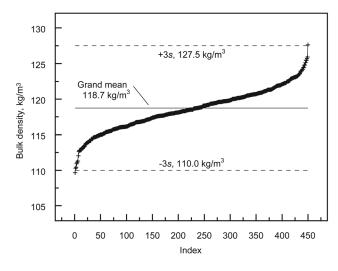
three times the standard deviation ($\pm 3s$ where *s* equals 2.9 kg/m³ from Table 3). These panels and five other panels, identified from unacceptable thickness measurements, were removed from the production lot. The excluded panels represented only 1.6 % (7 of 450) of the 1450d material lot; 443 panels, or 98.4 %, were accepted.

Thermal Conductivity Batch Study

THERMAL CONDUCTIVITY MEASUREMENTS

The thermal conductivity measurements were determined using the NIST 1016 mm diameter guarded-hot-plate apparatus. Figure 2 shows the essential features of the guarded-hot-plate apparatus designed for operation in the double-sided mode near ambient temperature conditions. The apparatus is





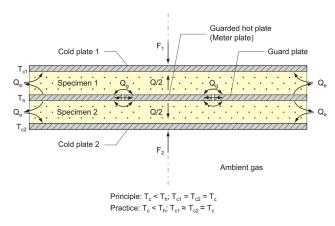


FIG. 2 Guarded-hot-plate schematic, double-sided mode of operation.

cylindrically symmetric about the axis indicated in Fig. 2, the plates are horizontal, and heat flow is vertical through the pair of specimens. The specimens, which have nearly the same density, size, and thickness, are placed on each surface of the guarded hot plate and clamped securely by the cold plates $(F_1 \text{ and } F_2)$. The guarded hot plate (T_h) and the cold plates (T_{c1}, T_{c2}) provide constant-temperature boundary conditions to the specimen surfaces. With appropriate guarding, lateral heat flows $(Q_g \text{ and } Q_e)$ are reduced to negligible proportions and, under steady-state conditions, the apparatus provides one-dimensional heat flow (Q) normal to the meter area of the specimen pair. A secondary guard is provided by an enclosed chamber that conditions the ambient air surrounding the plates to a temperature near to the mean specimen temperature (i.e., average surface temperatures of the hot and cold plates in contact with the specimens).

The experimental thermal conductivity (λ_{exp}), as defined in ASTM C1045 [6] was determined using Eq 2

$$\lambda_{\rm exp} = \frac{QL_{\rm avg}}{2A\Delta T_{\rm avg}}$$

where:

Q = time rate of one-dimensional heat flow through the apparatus metering area (A), W,

A = metering surface area (2A occurs because Q flows through two surfaces, Fig. 2), m^2 , and

 L_{avg} , m, and ΔT_{avg} , K, are defined in Eqs 3 and 4, respectively.

(3)
$$L_{\text{avg}} = (L_1 + L_2)/2$$

(4)
$$\Delta T_{\text{avg}} = (\Delta T_1 + \Delta T_2)/2 = \left[(T_h - T_c)_1 + (T_h - T_c)_2 \right]/2$$

The subscripts 1 and 2 in Eqs 3 and 4 correspond to the two specimens and respective cold plates illustrated in Fig. 2. In this study, the cold plate temperatures (T_{c1}, T_{c2}) and specimen thicknesses (L_1, L_2) are nearly the same, respectively. The thermal conductivity property determined in Eq 2 corresponds to a mean test temperature T_m given by Eq 5.

(5)
$$T_m = (T_h + T_c)/2 = \left(T_h + \frac{T_{c1} + T_{c2}}{2}\right) / 2$$

EXPERIMENTAL DESIGN

In contrast to the 100 % sampling process for bulk density, the thermal conductivity of 1450d was batch certified. The experimental design for the thermal conductivity batch study was based on an initial model that was developed from the results of the previous version SRM 1450c [5]. The initial model for thermal conductivity (λ) as a function of bulk density (ρ) and temperature (*T*) was assumed to be bilinear in ρ and *T* as shown in Eq. 6.

$$\lambda(\rho, T) = a_0 + a_1 \rho + a_2 T$$

Table 4 summarizes the experimental design for the thermal conductivity batch certification of 1450d. The design called for three levels for density qualitatively assigned as low, mid, and high and five levels for temperature from 280 to 340 K. The test sequence, shown as circled numbers ① in Table 4, was randomized to mitigate the influence of any systematic effects.

Each cell in Table 4 represents one measurement of a different pair of specimens (15 tests in total). The benefit of testing a unique pair of specimens at each combined level of temperature and density is that independent information is obtained at each such level. The experimental design given in Table 4 is balanced in the sense that an equivalent amount of information is obtained at each setting of the independent variables.

Results

Table 5 provides the measurement results for the 15 tests summarized in the experimental design (Table 4). The rows of data are arranged by T_m from 280 to 340 K and, within each level of T_m , the specimen densities (ρ_s) are ranked in ascending order. The data are partitioned in four sections: specimen bulk density (ρ_s), input quantities for Eqs 2 and 4; secondary test quantities, and (resultant) thermal conductivity (λ_{exp}). The notations "1" and "2" designate the top and bottom specimen, respectively, as illustrated in Fig. 2, and column 4 is the average of ρ_{s1} and ρ_{s2} . The "certified" density range for SRM 1450d is defined by the ultimate span for ρ (114 to 124 kg/m³).

During a test, the input temperature estimates, T_h , T_{c1} , and T_{c2} , were maintained within 0.01 K, or less, of their respective set-point temperatures. The estimates for Q/2 ranged from 3.9 W to 4.8 W for T_m at 280 and 340 K, respectively. However, for a given level of T_m the variation of Q/2 due to changes in ρ_s was much smaller. The

	Temperature Level, K						
Density Level	280	295	310	325	340		
Low	1 obs.ª ①	1 obs. @	1 obs.9	1 obs. 🚯	1 obs.5		
Mid	1 obs.	1 obs. 🕲	1 obs.3	1 obs. 🚯	1 obs.®		
High	1 obs. ④	1 obs. 🚇	1 obs.@	1 obs. 🕕	1 obs.@		

 TABLE 4
 Experimental design for SRM 1450d batch study.

T

^aObservation; circled number represents random sequence.

estimates for *A* have been corrected for thermal expansion effects of the meter-plate radius [4] and the estimates for the in situ test thickness *L* were determined by averaging the digital outputs of eight linear position transducers (four for each cold plate) [4]. The estimates for λ_{exp} include an extra digit for display purposes.

During a test, the secondary (influence) quantities (T_a , p_a , RH, and f) were either controlled or only recorded. The chamber air temperature (T_a) was controlled to be the same temperature as T_m (within 0.1 K, or less) and the chamber air pressure (p_a) varied with the site barometric conditions (from 97.5 to 101.3 kPa). The chamber relative humidity (RH) was maintained less than 10 % RH by a dry-air purge and varied with T_a . The clamping pressure (f) was determined by averaging the loading force (F) applied by each cold plate divided by the surface area of the cold plate, which was corrected for thermal expansion effects. Table 6 provides summary statistics for test quantities given in Table 5 that were fixed at one value across all tests. The temperature difference (ΔT) of 25.0 K was based on ASTM C1058 [7], standard practice for selecting test temperatures.

Discussion

DATA SCREENING-GRAPHICAL ANALYSIS

Figures 3 and 4 plot values of λ_{exp} from Table 5 as a function of the design model (Eq 6) input variables ρ_s and T_m , respectively. For Fig. 3, the individual data points are plotted as filled circle symbols corresponding to T_m levels of 280, 295, 310, 325, and 340 K. The error bars represent expanded uncertainties (described later) of 0.86 %. For Fig. 4, the individual data points are plotted as filled circle, square, and triangle symbols (without error bars for clarity) corresponding to the three main levels selected for bulk density.

DATA EVALUATION—CHARACTERIZATION

The data in Fig. 3 strongly suggest that, in the range of ρ_s and T_m covered for the 450 panels comprising the current SRM:

- (1) λ_{exp} is insensitive to bulk density for the range studied (in contrast with previous 1450 version materials), and
- (2) the dependence of λ_{exp} on mean temperature is strongly linear.

		$ ho_{\rm s}$, kg/m ³			Inp	out Quantities f	or Eqs <mark>2, 4</mark> , an	d 5			Secondary Qu	uantities		
<i>Т_т</i> , К	1	2	Avg.	<i>Т_h</i> , К	<i>Т_с</i> 1, К	<i>Т</i> _{с2} , К	Q/2, W	<i>A</i> , m ²	L _{avg} , mm	<i>T_a,</i> K	<i>p_a,</i> kPa	RH, %	<i>f</i> , Pa	λ_{exp}^{a} , W/(m · K)
280	112.8	114.3	113.5	292.50	267.50	267.50	3.895	0.12980	25.93	280.0	99.3	8	477	0.03112
280	118.3	118.7	118.5	292.50	267.50	267.51	3.894	0.12980	25.80	280.0	99.6	8	457	0.03096
280	121.0	121.4	121.2	292.50	267.50	267.49	3.924	0.12980	25.56	280.0	100.6	9	478	0.03090
295	113.5	115.2	114.3	307.50	282.50	282.50	4.050	0.12989	26.02	295.0	100.5	4	460	0.03245
295	118.9	119.1	119.0	307.50	282.50	282.50	4.086	0.12989	25.82	295.0	100.6	4	461	0.03249
295	121.1	121.8	121.5	307.50	282.50	282.50	4.105	0.12989	25.80	295.0	100.2	3	472	0.03261
310	116.0	115.6	115.8	322.50	297.50	297.50	4.240	0.12998	26.13	310.0	100.1	2	484	0.03409
310	118.5	118.9	118.7	322.50	297.50	297.50	4.324	0.12998	25.76	310.0	98.6	2	506	0.03428
310	122.0	122.8	122.4	322.50	297.51	297.50	4.317	0.12998	25.76	310.0	99.7	2	488	0.03422
325	115.2	116.1	115.7	337.50	312.50	312.50	4.494	0.13007	25.85	325.0	100.5	1	495	0.03572
325	118.8	118.9	118.9	337.50	312.50	312.50	4.499	0.13007	26.00	325.0	101.3	1	491	0.03597
325	122.5	123.9	123.2	337.50	312.50	312.50	4.549	0.13007	25.73	325.0	97.5	1	501	0.03599
340	115.3	116.4	115.8	352.50	327.49	327.50	4.701	0.13016	25.98	340.0	99.7	1	466	0.03752
340	118.6	119.7	119.1	352.50	327.50	327.50	4.747	0.13016	25.78	340.0	101.3	1	478	0.03760
340	123.3	124.3	123.8	352.50	327.50	327.49	4.760	0.13016	25.85	340.0	99.1	<1	432	0.03782

TABLE 5 Thermal conductivity measurement data (sorted by T_m and ρ_s)

^aExtra digit included for display purposes.

	Δ <i>Τ</i> , Κ	L _{avg} , mm	p _a , kPa	<i>f</i> , Pa
Grand mean	25.001	25.85	99.9	476
Std. dev.	0.003	0.14	1.0	19

 TABLE 6
 Summary statistics for fixed primary and secondary quantities.

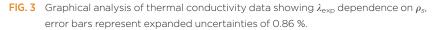
The first assertion is borne out by linear least squares fits to the 3-point horizontal profiles of Fig. 3. Table 7 summarizes the slopes of the lines shown in Fig. 3 and their corresponding *t*-values, which are defined as the ratio of the slope and the standard error of the slope estimate. For the three middle profiles (Fig. 3), the slope of the fitted line is statistically indistinguishable from zero. The slopes of the extreme profiles are opposite in sign.

A fit of the no-intercept line model $\lambda_{exp} = a_2 T_m$ yields excellent fit statistics (*R*-square = 0.997) and is visually an excellent fit as well (Fig. 4). The residual standard deviation for the fit is 0.00012 W/(m · K). Table 8 summarizes the regression statistics for the no-intercept linear fit to temperature.

For comparison and further model validation, other models were fit including:

- (1) bilinear in ρ and *T*, with and without constant term, and
- (2) quadratic and cubic in *T*, with and without constant term.

For all the models assayed, one or more fitted coefficients were non-significant, with the exception of the pure third power model (T^3) with no constant, linear, or



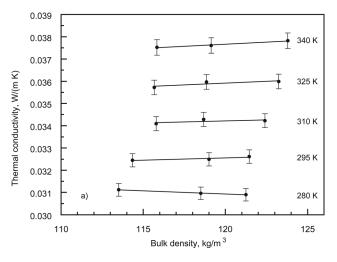
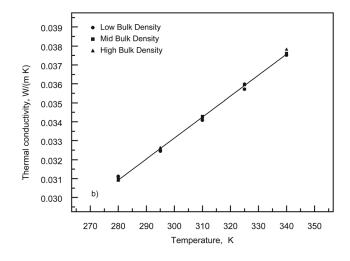


FIG. 4 Graphical analysis of thermal conductivity data showing λ_{exp} dependence on T_m (without error bars for clarity).



quadratic term. Both visually (superimposed and residual plots) and statistically (magnitudes of residual standard errors, fitted coefficients, standard errors of coefficients, and associated *t*-values), all other models, including T^3 , were found to be inferior to the simplest first power (linear) model in *T* with no constant term.

FINAL MODEL

Equation 7, derived by least squares linear regression of λ on *T*, gives the final certification model for SRM 1450d.

$$\hat{\lambda} = (1.10489 \times 10^{-4}) \times T_m$$

Figure 5 plots the deviations $(\lambda_{exp} - \hat{\lambda})/\hat{\lambda}$ (in %) versus T_m by bulk density (filled circle, square, and triangle symbols corresponding to low-, mid-, and high- levels of bulk density, respectively). The deviations for mid- and high-bulk densities are randomly and interchangeably scattered (i.e., no discernible pattern) around zero. The

<i>Т_т</i> , К	Slope, $(W \cdot m^2)/(K \cdot kg)$	<i>t</i> -value
280	-2.939×10^{-5}	-12.1
295	2.114×10^{-5}	2.0
310	1.850×10^{-5}	0.8
325	3.314×10^{-5}	1.6
340	3.741×10^{-5}	6.4

TABLE 7 Summary of linear profiles for λ_{exp} versus ρ_s (Fig. 3).

TABLE 8 Summary of regression statistics for λ_{exp} versus T_m (Fig. 4).

Regression coefficient Slope, W/(m · K ²)		s of slope, W/(m \cdot K ²)	t-value
a ₂	1.10489×10^{-4}	1.010 × 10 ⁻⁷	1094

low-bulk densities, however, are biased low (about -0.5 %), with the exception of one low-temperature point. The same pattern is visible on close inspection in Fig. 4. The bias magnitude, however, is dominated by the experimental uncertainty of ± 0.86 %.

UNCERTAINTY BUDGET

For the multiplicative expression of Eq 2, the relative combined standard uncertainty in λ_{exp} can be expressed as the relative uncertainties associated with each factor combined in quadrature.

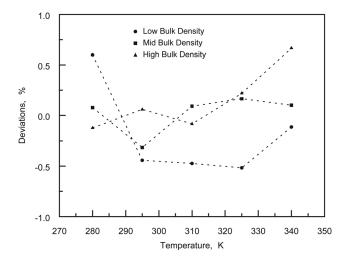
(8)
$$u_{c,rel}(\lambda_{exp}) = \frac{u_c(\lambda_{exp})}{\lambda_{exp}} = \sqrt{\left(\frac{u(Q)}{Q}\right)^2 + \left(\frac{u(\Delta T)}{\Delta T}\right)^2 + \left(\frac{u(L)}{L}\right)^2 + \left(\frac{u(A)}{A}\right)^2}$$

The standard uncertainties and input quantities used in Eq 8 are derived in Ref. [4]

(9)
$$u_{c,rel}(\lambda_{exp}) = \sqrt{\left(\frac{0.0074}{4.736}\right)^2 + \left(\frac{0.077}{25.001}\right)^2 + \left(\frac{0.065}{25.85}\right)^2 + \left(\frac{0.000043}{0.13016}\right)^2}$$

(10)
$$u_{c,rel}(\lambda_{exp}) = \sqrt{(0.00156)^2 + (0.00308)^2 + (0.00251)^2 + (0.00033)^2} = 0.0043$$

FIG. 5 Graphical analysis of deviations (in %) for the fit given in Eq 7.



The comparative contribution for the first term in Eq 10 is 13.3 %; for the second, 51.9 %; for the third, 34.2 %; and, for the fourth, 0.6 %. The major contributors to the uncertainty for SRM 1450d are the empirical determinations for specimen temperature difference (ΔT) and thickness (L_{avg}). These findings are consistent with results from previous NIST guarded-hot-plate analyses [5].

The relative expanded uncertainty, U_{rel} , for a coverage factor of k equals 2 is given in Eq 11.

(11)
$$U_{\rm rel}(\lambda_{\rm exp}) = 2u_{c,\rm rel}(\lambda_{\rm exp}) = 0.0086$$

Expressed as a % (×100), $U_{\rm rel}$ is equal to 0.86 %, which has been rounded to 1 % on the 1450d certificate for ease of use.

Summary

Thermal conductivity measurements of Standard Reference Material 1450d, Fibrous Glass Board, have been conducted using the NIST 1016 mm line-heatsource guarded-hot-plate apparatus following a full factorial experimental design with two variables, bulk density, and temperature. The thermal conductivity measurements were conducted over ranges of bulk densities from 114 to 124 kg/m^3 and mean temperatures from 280 to 340 K. Analysis of the measurements revealed that the thermal conductivity of the material lot was insensitive to bulk density for the range exhibited by the 1450d material and strongly linearly dependent on temperature. The final model for the predicted thermal conductivity of 1450d was found to be a first power (linear) model in *T* with no constant term. The expanded uncertainty for thermal conductivity was prepared consistent with current international guidelines and was determined to be 0.86 %, which has been rounded to 1 % on the 1450d certificate for ease of use.

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