Thermal Conductivity, 24th and Thermal Expansion, 12th Joint Conferences. Proceedings. October 26-29, 1997, Pittsburgh, PA, Technomic Publishing Co., Inc., Lancaster, PA, Gaal, P. S.; Apostolescu, D. E.; Hurst, E. P., Editors, 662-672 pp, 1999.

Development of Thermal Insulation Standard Reference Materials Using "Good" Experimental Design

R. R. ZARR and E. S. LAGERGREN

ABSTRACT - An experimental design for the thermal characterization of Standard Reference Material 1450c, Fibrous Glass Board, is presented. The measurements have been conducted following a randomized full factorial experimental design with two variables, bulk density and temperature, using the National Institute of Standards and Technology's one-meter guarded hot plate apparatus. Certified values of thermal resistance have been established for a temperature range from 280 K to 340 K and bulk density from 150 kg/m^3 to 165 kg/m^3. Standard uncertainties for the measurements, consistent with current international guidelines, are included.

INTRODUCTION

A proper, or "good" experimental design provides the means for obtaining unambiguous results about the primary factors of interest at minimum cost. In practice, a good experimental design is more important than the statistical analysis. A good experimental design employs strategies such as randomization and blocking to reduce contaminating effects of the nuisance variables and increases the sensitivity of the experimental results. The results from a good experimental design usually lend themselves to simple graphical analyses. In particular, the experimental characterization of a Standard Reference Material (SRM) is improved by utilizing a good experimental design.

Recently, the National Institute of Standards and Technology (NIST) has developed SRM 1450c, Fibrous Glass Board [1], and SRM 1453, Expanded Polystyrene Board [2], following test plans that utilized balanced, full factorial experimental designs. These experimental designs were selected based on the underlying models for predicting the thermal conductivity as a function of bulk density and temperature, the measurement repeatability and uncertainty, as well as time

Contribution of the National Institute of Standards and Technology

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Robert R. Zarr and Eric S. Lagergren, National Institute of Standards and Technology, Gaithersburg, Maryland 20899 USA

constraints. This paper presents the experimental design and results for the thermal characterization of SRM 1450c, Fibrous Glass Board.

DESIGN OF EXPERIMENT

The objective of this study was to characterize the thermal transmission properties of a large lot of fibrous glass boards and to provide the user with a prediction equation for thermal conductivity, as well as, certified values of thermal resistance. Based on previous experience at NIST [3,4], the thermal conductivity (λ) of fibrous glass board has been shown to be a (polynomial) function of two variables: the bulk density (ρ) of the material and the mean temperature (T) of the specimen. A model for λ as a function of ρ and T was assumed to be

$$\lambda(\rho,T) = a_0 + a_1\rho + a_2T + a_3T^2 + a_4T^3. \tag{1}$$

In order to check the adequacy of Equation (1) for this experiment, a full factorial design with 3 levels for ρ and 5 levels for T was selected which required thermal conductivity measurements of 15 pairs of specimens. This design also provided the means to check the necessity of the following: a quadratic term for ρ, a fourth-order term for T, and/or a cross-product term for ρ and T in order to model the data. Table I summarizes the experimental design for characterizing SRM 1450c. A single replicate for each setting was selected due to time constraints. Completion of this design required about 2 months. All tests were conducted by the same operator.

TABLE I—FULL FACTORIAL (3 BY 5) EXPERIMENTAL DESIGN AND TEST SEQUENCE

Density Level	Temperature Level (K)				
	280	295	310	325	340
High	$\lambda_{1,1}$ (15)	$\lambda_{1,2}$ (04)	$\lambda_{1,3}$ (05)	$\lambda_{1,4}$ (14)	$\lambda_{1,5}$ (10)
Mid	$\lambda_{2,1}$ (07)	$\lambda_{2,2}$ (13)	$\lambda_{2,3}$ (09)	$\lambda_{2,4}$ (01)	$\lambda_{2,5}$ (12)
Low	$\lambda_{3,1}$ (06)	$\lambda_{3,2}$ (11)	$\lambda_{3,3}$ (02)	$\lambda_{3,4}$ (08)	$\lambda_{3,5}$ (03)

The above design is balanced in the sense that an equivalent amount of information is obtained at each setting of the independent variables. If, on the other hand, either extra information had been obtained at some of the settings, or worse, critical information omitted at one setting, the design would be unbalanced and the resulting statistical analysis would suffer. It is also important to emphasize that each cell in Table I represents a measurement of a different pair of specimens. The advantage of testing a unique pair of specimens at each level of temperature is that completely new and independent information is obtained at each level of temperature. Otherwise, the data would not be independent because data from subsequent observations would contain some contamination (bias) from the previous observation. The introduction of error in any experiment is inevitable, but it is preferable that the error be random rather than systematic.

As with any experiment, other minor parameters considered to be "nuisance" variables were either fixed at specified levels during testing in the guarded hot plate apparatus or, in some cases, merely recorded. Fixed parameters included the following: (1) the temperature difference across the specimen (ΔT) and direction of heat flow; (2) the ambient air temperature (T_a) of the apparatus chamber; and, (3) the specimen thickness (L). Parameters that were recorded included the clamping load applied to the specimens, the ambient air pressure (P_a), and the relative humidity (RH) which, although low, varied with the chamber ambient air temperature (T_a).

EXPERIMENTAL

SPECIMENS

In 1996, NIST procured 130 boards of high-density, fibrous glass thermal insulation from a commercial manufacturer. The nominal dimensions of the boards were 1220 mm × 1220 mm × 25 mm thick, and the nominal density was 160 kg/m^3. The insulation was manufactured by molding glass-fiber "pelts" and binder to produce a semi-rigid board. The glass fibers were an alkali-alkaline alumino-borosilicate glass, bound with a phenyl-formaldehyde binder, and oriented such that the lengths were essentially parallel to the board faces. Test specimens were selected to minimize the possibility of the user having to extrapolate outside the bulk density range given in the SRM 1450c Certificate. Therefore, in accordance with the experimental design in Table I, 30 boards (15 pairs) were selected from the lot: 5 pairs having the lowest density; 5 pairs about the median density; and, 5 pairs having the highest density. A single specimen, 1016 mm in diameter, was cut from the center of each board using a sharp knife and metal template. Prior to thermal conductivity measurements, a 406-mm diameter cylinder corresponding to the meter area of the apparatus was cut in order to rank the specimens by their meter-area bulk densities.

THERMAL CONDUCTIVITY MEASUREMENTS

A schematic of the NIST one-meter line-heat-source guarded-hot-plate apparatus is shown in Figure 1. The apparatus has been described previously [5,6] and its operation is summarized briefly here. Two specimens having nearly the same density, size, and thickness are placed on the two sides of the guarded hot plate and clamped securely by the circular cold plates. Ideally, the guarded hot plate and cold plates provide constant-temperature boundary conditions to the surfaces of the specimens. With proper guarding in the lateral direction, the apparatus is designed to provide one-dimensional heat flow (Q) through the meter area of the pair of specimens. Additional guarding was provided by a temperature-controlled environmental chamber. The ambient temperature within the chamber was maintained at the same value as the mean temperature (T) of the hot and cold plates.

Data for Q and the plate temperatures (T_h, T_c) were collected every two minutes and thermal equilibrium for the apparatus was attained when the plate temperatures were in a state of statistical control within 0.05 K of their target temperatures and Q

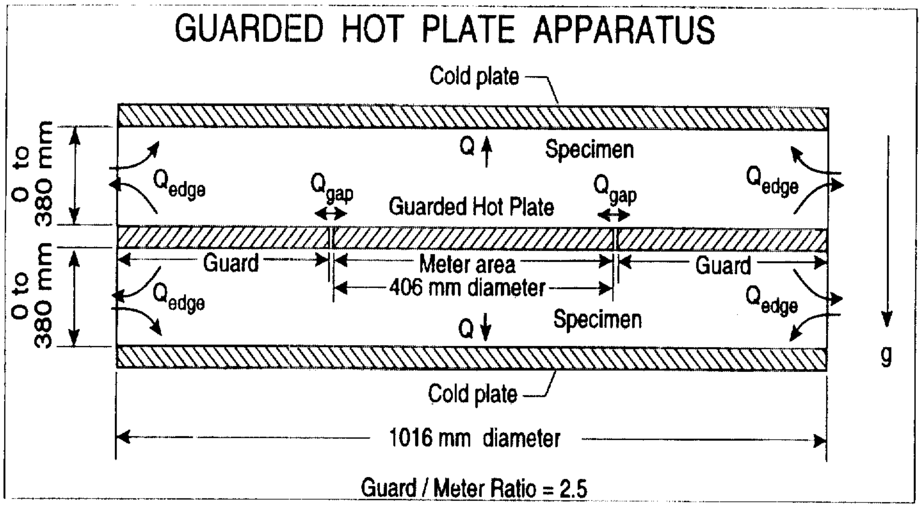

Figure 1. Schematic of NIST one-meter guarded hot plate apparatus.

was also "in control". This means that values for the plate temperatures and Q fluctuated randomly about a fixed level and the variation in fluctuations was also fixed. Steady-state data were collected for four hours and averaged for the interval. Measurements of (apparent)[1] thermal conductivity (λ) for the pair of specimens were determined in accordance with ASTM Test Method C 177 [7] using the following equation:

$$Q = \lambda A \frac{\Delta T}{L}, \tag{2}$$

where Q is the heat flow through the meter area of the specimens [W]; A is the meter area normal to direction of heat flow [m^2]; ΔT is the temperature difference across specimens [K]; and, L is the in-situ thickness of the pair of specimens [m]. Values of λ were reported at the mean temperature (T) of the hot and cold plates, $T = \frac{1}{2}(T_h + T_c)$.

In order to minimize the effect of moisture, the specimens were conditioned in an oven at 90 °C for a minimum of 16 h prior to testing. During the test, dry air was continuously injected into the environmental chamber, decreasing the relative humidity to 15 percent or less, depending on the ambient dry bulb temperature (T_a). At the conclusion of each test, the specimen masses were measured. The maximum regain in mass was found to be no more than 0.5 percent. The effect of the meter-area incision was examined with a separate series of thermal conductivity tests. A pair of specimens was initially tested in the guarded-hot-plate apparatus before the meter area was cut. After cutting the meter area, the pair of specimens was retested in the

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[1] The thermal transmission properties of heat insulators determined from standard test methods typically include several mechanisms of heat transfer, including conduction, radiation, and possibly convection. For that reason, some experimentalists will include the adjective "apparent" when describing thermal conductivity of thermal insulation. However, for brevity, the term thermal conductivity will be used in this paper.

guarded-hot-plate apparatus at the same conditions. The difference in the initial and final thermal conductivities was quite small, less than 0.05 percent, and was subsequently neglected.

BULK DENSITY MEASUREMENTS

The bulk densities (ρ) of the 406-mm-diameter meter area were determined in accordance with ASTM Test Method C 177 [7] by dividing the mass (m) of the cylinder by its corresponding volume (V), i.e., $\rho = m/V$. The mass was obtained by using a precision balance having a sensitivity of 0.1 g. The diameters of the cylinders were measured at two locations using a steel rule having a resolution of 0.5 mm and the thickness for the 406-mm diameter was averaged from five measurements taken on a granite flat table with a precision caliper, 0.1 mm resolution.

UNCERTAINTY IN MEASUREMENTS

The measurement uncertainties for thermal conductivity, mean temperature, and bulk density were derived in accordance with current ISO guidelines [8,9] and described previously [1]. The standard uncertainties (i.e., coverage factor, $k = 1$) for the thermal conductivity, mean temperature, and bulk density were 0.00020 W/(m·K), 0.034 K, and 0.72 kg/m^3, respectively. These standard uncertainties were included in the combined standard uncertainty for predicted values of thermal conductivity.

RESULTS

The fifteen pairs of specimens were tested in the NIST one-meter guarded-hot-plate apparatus following the test sequence given in Table I, which randomized both independent variables, T and ρ. Table II summarizes the experimental test conditions and measured thermal conductivity (λ) for each pair of specimens. Note that an extra digit is provided for λ. The average meter-area bulk density was computed for each pair of specimens at the conclusion of each test.

Several parameters in Table II indicate the "average" value for the top and bottom specimen, e.g., bulk density (ρ), thickness (L), clamping load, etc. The average thickness (L) was determined from in-situ measurements of the top and bottom plate separation. The grand average of the test thicknesses was 25.33 ± 0.22 mm (one standard deviation, 1s). The grand average of the clamping loads was 475 ± 217 N (1s) which varied from test to test (Table II) due to the thermal expansion and contraction of the specimens and apparatus. The maximum clamping pressure of 1 kPa (Table II) was well below the established compression limit [1].

TABLE II—THERMAL CONDUCTIVITY MEASUREMENTS OF SRM 1450c

Test	T (K)	ρ_{avg} (kg/m^3)	L_{avg} (mm)	Load* (N)	T_a (K)	P_a (kPa)	RH (%)	T_h (K)	$T_{c,avg}$ (K)	λ (W/(m·K))
1	325	159.2	25.07	648	325.2	100.48	<5	335.15	315.15	0.03679
2	310	151.5	25.88	458	310.2	100.90	<5	320.15	300.15	0.03439
3	340	154.8	25.39	850	340.2	100.63	<5	350.15	330.15	0.03777
4	295	163.2	25.15	359	295.2	101.34	<10	305.15	285.15	0.03340
5	310	162.6	25.27	321	310.2	101.14	<10	320.15	300.15	0.03499
6	280	155.8	25.51	72	280.2	100.40	15	290.15	270.15	0.03143
7	280	158.5	25.16	430	280.1	100.46	14	290.15	270.15	0.03166
8	325	149.4	25.09	626	325.2	100.10	<5	335.15	315.16	0.03597
9	310	160.1	25.29	479	310.2	101.60	<5	320.15	300.15	0.03534
10	340	164.9	25.35	770	340.2	101.24	<5	350.15	330.15	0.03828
11	295	156.1	25.42	200	295.2	100.87	<10	305.15	285.15	0.03297
12	340	156.5	25.01	701	340.2	100.69	<5	350.15	330.15	0.03798
13	295	157.1	25.39	308	295.2	100.67	<10	305.15	285.14	0.03272
14	325	161.5	25.42	543	325.2	101.03	<5	335.15	315.16	0.03648
15	280	166.0	25.55	352	280.2	100.39	14	290.15	270.15	0.03199

*Plate Surface Area = 0.811 m^2.

DISCUSSION

MULTIPLE VARIABLE REGRESSION ANALYSIS

The thermal conductivity (λ) as a function of specimen bulk density (ρ) and mean temperature (T) is shown in Figures 2a and 2b, respectively. Both plots show a positive correlation for λ and the independent variables. That is, λ increased with increasing levels of ρ or T, although the change with respect to ρ was small (Figure 2a) in comparison to the effect of T (Figure 2b). There were, however, small inconsistencies in the data. As noted in Figure 2a, the change in λ was not monotonic for some temperature levels.

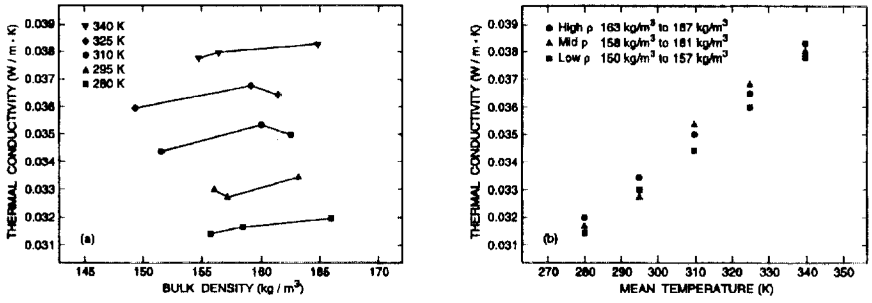

Figure 2. Thermal conductivity as a function of (a) bulk density and (b) mean temperature.

For example, at 325 K the value of λ for the highest density specimen was lower than the value of λ for the mid-density specimen. These inconsistencies were also present in Figure 2b where, for a given temperature, the data points were not necessarily arranged from the lowest to highest density. The cause of these inconsistencies was unknown but most likely was due to between-specimen variability such as localized density variations [1].

The data for the bulk density, mean temperature, and corresponding value of thermal conductivity were fit to the $\lambda(\rho,T)$ model, Equation (1), by a multiple variable regression analysis [10]. Higher order temperature terms were not statistically significant, and so a final form, linear in ρ and T, was acceptable. The final model is

$$\hat{\lambda} = -7.7663\times10^{-3} + 5.6153\times10^{-5}\rho + 1.0859\times10^{-4}T, \tag{3}$$

where ρ is in units of [kg/m^3] and T in [K]. The last digit of each coefficient is provided to reduce rounding errors. It is interesting to note that the prediction models for all previous lots of the SRM 1450 Series included a nonlinear term for temperature [3,4]. Previous lots were characterized using different guarded-hot-plate apparatus, a larger temperature range, particularly SRM 1450b [4], and different experimental designs from that of the current study. In this study, higher-order temperature terms in the model did not improve the results of the curve fit. The authors acknowledge that, for a larger temperature range, the above model is probably unacceptable. For this reason, the authors strongly advise against extrapolation of the model beyond the temperature range of this study, 280 K to 340 K.

The residual standard deviation for the above fit was 0.000205 W/(m·K) which was quite small. The adequacy of the fit was further examined by plotting the individual deviations (δ) from the model as defined by

$$\delta = \lambda - \hat{\lambda}. \tag{4}$$

Individual deviations versus ρ and T are shown in Figures 3a and 3b, respectively. The data in Figures 3a and 3b do not indicate any trends in the deviations, signifying a satisfactory fit. The majority of the deviations were within ± 0.00025 W/(m·K) of the measured values. The relative standard deviation multiplied by 2 for the fitted model was 1.1 percent. For comparison, the relative standard deviation multiplied by 2 for the fitted model of SRM 1450b was 1.5 percent [4].

CERTIFIED VALUES OF THERMAL RESISTANCE

Using Equation (3) to compute predicted values of thermal conductivity ($\hat{\lambda}$), certified values of thermal resistance ($\hat{R}$) of SRM 1450c were calculated for a (hypothetical) 25.4-mm-thick specimen with the following equation:

$$\hat{R} = \frac{L}{\hat{\lambda}}. \tag{5}$$

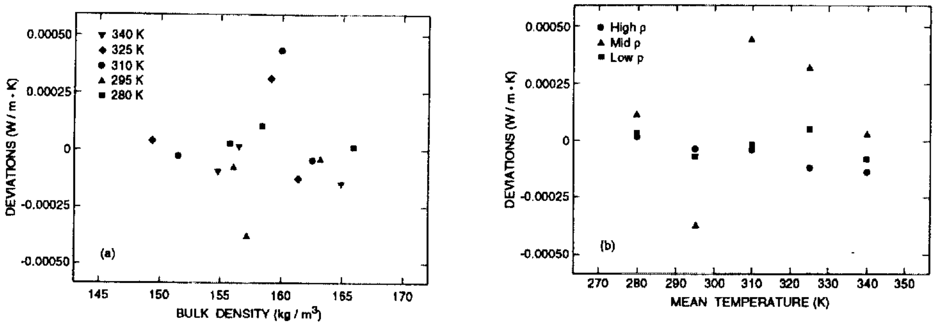

Figure 3. Scatter plots for deviations versus (a) bulk density and (b) mean temperature

The value of 25.4 mm for L was selected to be consistent with the SRM 1450 Series. Certified values of $\hat{R}$ and expanded uncertainties (described in the next section) are given in Table III for bulk density and mean temperature ranging from 150 kg/m^3 to165 kg/m^3, and 280 K to 340 K, respectively.

The certified values of $\hat{R}$ in Table III are restricted to the measured ranges of bulk density, mean temperature, thickness, and thermal conductivity presented herein. Extrapolation outside these ranges is not recommended. The range for thickness (L) is 24.9 mm to 25.6 mm, based on the variation of two times the standard deviation for the thickness data in Table II. In cases where the user may need to extrapolate outside the recommended temperature range, the temperature limits for the material are required. The upper temperature of use for SRM 1450c is limited to the decomposition

TABLE III—CERTIFIED VALUES OF THERMAL RESISTANCE AND EXPANDED UNCERTAINTIES (IN m^2·K/W), 25.4 mm THICK SPECIMEN FOR SRM 1450c

Temperature (K)	Bulk Density (kg/m³)			
	150	155	160	165
280	0.818 ± 0.013	0.810 ± 0.013	0.803 ± 0.013	0.796 ± 0.012
285	0.804 ± 0.013	0.797 ± 0.012	0.790 ± 0.012	0.783 ± 0.012
290	0.790 ± 0.012	0.783 ± 0.012	0.777 ± 0.012	0.770 ± 0.012
295	0.777 ± 0.012	0.770 ± 0.012	0.764 ± 0.011	0.757 ± 0.011
300	0.764 ± 0.011	0.758 ± 0.011	0.752 ± 0.011	0.745 ± 0.011
305	0.752 ± 0.011	0.746 ± 0.011	0.740 ± 0.011	0.734 ± 0.011
310	0.740 ± 0.011	0.734 ± 0.011	0.728 ± 0.010	0.722 ± 0.010
315	0.729 ± 0.010	0.723 ± 0.010	0.717 ± 0.010	0.711 ± 0.010
320	0.717 ± 0.010	0.712 ± 0.010	0.706 ± 0.010	0.701 ± 0.010
325	0.707 ± 0.010	0.701 ± 0.010	0.696 ± 0.009	0.690 ± 0.009
330	0.696 ± 0.009	0.691 ± 0.009	0.686 ± 0.009	0.680 ± 0.009
335	0.686 ± 0.009	0.681 ± 0.009	0.676 ± 0.009	0.671 ± 0.009
340	0.676 ± 0.009	0.671 ± 0.009	0.666 ± 0.009	0.661 ± 0.009

point of the phenolic binder, approximately 473 K (200 °C) [1]. A lower temperature limit for SRM 1450c has not been established, however there is no known lower limit, in principle.

UNCERTAINTY FOR PREDICTED VALUES

The expanded uncertainties for predicted values of thermal conductivity ($\hat{\lambda}$) and certified values of thermal resistance ($\hat{R}$) were derived in accordance with current ISO guidelines [8,9] and described in Reference [1]. The expanded uncertainty, $U(\hat{\lambda})$, for $\hat{\lambda}$ was obtained by multiplying the combined standard uncertainty for $\hat{\lambda}$, $u_c(\hat{\lambda})$, by a coverage factor of $k = 2$:

$$U(\hat{\lambda}) = ku_c(\hat{\lambda}). \tag{6}$$

The combined standard uncertainty for $u_c(\hat{\lambda})$ was determined from the following: (1) the standard uncertainty for the regression analysis for $\hat{\lambda}$ in Equation (3); (2) the standard uncertainty for the measurement of λ; and, (3) the standard uncertainties for the measurements of ρ and T. The conservative estimate for the standard uncertainty for the regression analysis in Equation (3) was 0.00014 W/(m·K) [1] which was computed at 150 kg/m^3 and 280 K. The standard uncertainties for the measured values of λ, ρ, and T were 0.00020 W/(m·K), 0.72 kg/m^3, and 0.034 K, respectively [1]. The standard uncertainties for ρ and T were propagated in Equation (3) to yield a standard uncertainty of 0.00004 W/(m·K). These standard uncertainties for $\hat{\lambda}$ were combined in quadrature to yield a combined standard uncertainty of 0.00025 W/(m·K) ($k = 1$). The corresponding expanded uncertainty, $U(\hat{\lambda})$, was 0.00050 W/(m·K) ($k = 2$). This estimate does not include any estimates for uncertainties introduced by the user or long-term drifts in the material.

The expanded uncertainties, U, (k =2) for certified values of $\hat{R}$ in Table III were based on the following equation:

$$U(\hat{R}) = ku_c(\hat{R}) = k\sqrt{c_{\hat{\lambda}}^2 u_c^2(\hat{\lambda})}, \tag{7}$$

where the sensitivity coefficient $c_{\hat{\lambda}} = -(0.0254/\hat{\lambda}^2)$ and $u_c(\hat{\lambda}) = 0.00025$ W/(m·K) ($k = 1$). Note that the sensitivity coefficient $c_{\hat{\lambda}}$ varies with $\hat{\lambda}$ and, therefore, the standard uncertainties for $\hat{R}$ also vary, as noted in Table III. Consequently, the values of expanded uncertainty quoted in Table III are valid only for the given hypothetical thickness of 0.0254 m (1 in.). The maximum expanded uncertainty for $\hat{R}$ in Table III is ± 0.013 m^2·K/W ($k = 2$) at 150 kg/m^3 and 280 K, which, in relative terms, is ± 1.6 percent. This value of relative expanded uncertainty compares quite well to the previous published uncertainty values of ± 2 percent for SRM 1450b [4].

SUMMARY AND CONCLUSIONS

Certified values of thermal resistance for Standard Reference Material 1450c, Fibrous Glass Board, have been derived for bulk density and mean temperature ranging from 150 kg/m^3 to 165 kg/m^3, and 280 K to 340 K, respectively. Thermal conductivity measurements were performed following a randomized full factorial experimental design with these two variables, bulk density and mean temperature, using the National Institute of Standards and Technology's one-meter line-heat-source guarded hot plate apparatus. A model dependent on these two parameters has been developed that describes the thermal conductivity over the range of the parameters. Expanded uncertainties, consistent in format with current international guidelines, have been prepared for predicted values of thermal conductivity and certified values of thermal resistance.

REFERENCES

1. Zarr, R.R. "Glass Fiberboard, SRM 1450c, for Thermal Resistance from 280 K to 340 K," *NIST Special Publication 260-130*, April 1997.

2. Zarr, R.R., "Expanded Polystyrene Board as a Standard Reference Material for Thermal Resistance Measurement Systems," *Insulation Materials: Testing and Applications: Third Volume, ASTM 1320*, R.S. Graves and R.R. Zarr, Eds., American Society for Testing and Materials, West Conshohocken, PA 1997, pp. 91-105.

3. Siu, M.C.I. "Fibrous Glass Board as a Standard Reference Material for Thermal Resistance Measurement Systems," *Thermal Insulation Performance, ASTM STP 718*, D.L. McElroy and R.P. Tye, Eds., American Society for Testing and Materials, Philadelphia, 1980, pp. 343-360.

4. Hust, J.G. "Standard Reference Materials: Glass Fiberboard SRM for Thermal Resistance," *NBS Special Publication 260-98*, August 1985.

5. Powell, F.J. and B.G. Rennex. "The NBS Line-Heat-Source Guarded Hot Plate for Thick Materials," *Thermal Performance of the Exterior Envelopes of Buildings - II, ASHRAE SP 38*, 1983, pp. 657-672.

6. Zarr, R.R. and M.H. Hahn. "Line Heat Source Guarded Hot Plate Apparatus: Adjunct ASTM Practice C 1043," *Annual Book of ASTM Standards, Vol. 04.06.*

7. ASTM. "C 177 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, Vol. 04.06.*

8. ISO. *Guide to the Expression of Uncertainty in Measurement*, International Organization for Standardization, Geneva, Switzerland, 1993.

9. Taylor, B.N. and C.E. Kuyatt. "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," *NIST Technical Note 1297*, 1994.

10. Neter, J., Wasserman, W. And M. Kutner. *Applied Linear Statistical Models*, Irwin, Boston 1990.