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Interlaboratory "Pilot Run" Study of Small Heat-Flow-Meter Apparatus for ASTM C 518

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ABSTRACT: Thermal conductivity measurements of a high-density glass-fiber thermal insulation material near 24°C are presented for the determination of the precision and bias of ASTM Test Method C 518. The measurements have been conducted by 13 laboratories using small (305 by 305-mm) heat-flow-meter apparatus on three specimens of high-density glass-fiber thermal insulation material that were circulated among the laboratories. Test results are analyzed using ASTM Practice E 691 and subsequently compared to measurements of the same specimens conducted in a guarded-hot-plate apparatus using ASTM Test Method C 177. The 95% repeatability and reproducibility indexes for precision have been determined to be no worse than 1.1 and 4.0%, respectively. A method for estimating bias is presented.

KEYWORDS: bias, glass fiber, guarded hot plate, heat flow meter, interlaboratory, precision, repeatability, reproducibility, thermal conductivity, thermal insulation

Nomenclature

- A Meter area normal to heat flow, m^2
- $CV\%_r$ Repeatability coefficient of variation, %
- $CV\%_R$ Reproducibility coefficient of variation, %
- E Voltage output from heat flux transducer (HFT), V
- h Between-laboratory consistency statistic
- k Within-laboratory consistency statistic
- L In-situ specimen thickness, m
- Q Heat flow rate in meter area, W
- r 95% repeatability limit
- R 95% reproducibility limit
- RH Relative humidity, %
- s_r Repeatability standard deviation
- S Calibration factor of heat-flow-meter apparatus, $W \cdot m^{-2} \cdot V^{-1}$
- S_R Reproducibility standard deviation
- T Mean temperature of specimen, °C
- T_a Ambient air temperature, °C
- x_{ij} Mean of three C 518 replicates for Laboratory j, Specimen i, $W \cdot m^{-1} \cdot K^{-1}$
- $\bar{x}_j$ Mean of C 518 thermal conductivity measurements for Laboratory j, $W \cdot m^{-1} \cdot K^{-1}$
- $\bar{x}$ Mean of all C 518 thermal conductivity measurements, $W \cdot m^{-1} \cdot K^{-1}$
- z_{ik} C 177 thermal conductivity measurement k for Specimen i, $W \cdot m^{-1} \cdot K^{-1}$
- $\bar{z}_i$ Mean of four C 177 thermal conductivity measurements for Specimen i, $W \cdot m^{-1} \cdot K^{-1}$
- $\bar{z}$ Mean of all C 177 thermal conductivity measurements, $W \cdot m^{-1} \cdot K^{-1}$
- ΔT Temperature difference across specimen, K
- λ Thermal conductivity, $W \cdot m^{-1} \cdot K^{-1}$
- $\bar{\lambda}$ Grand average of thermal conductivity measurements, $W \cdot m^{-1} \cdot K^{-1}$
- μ_{HFM} Long-run mean of C 518 thermal conductivity measurements, $W \cdot m^{-1} \cdot K^{-1}$
- μ_{GHP} Long-run mean of C 177 thermal conductivity measurements, $W \cdot m^{-1} \cdot K^{-1}$
- ρ Bulk density, $kg \cdot m^{-3}$

Subscripts

- 1,2,3 Specimen 1,2,3, respectively

Recently, a special task group under the auspices of ASTM Committee C16 on Thermal Insulation completed a "pilot run" interlaboratory study (ILS) of the ASTM Test Method for Steady-State Heat Flux Measurements and Thermal Properties by Means of the Heat Flow Meter Apparatus (C 518). The purpose of the ILS was to expand the statement for precision to include a smaller heat-flow-meter apparatus (plate size of 305 by 305 mm) that is used mainly for quality control in the manufacture of thermal insulation materials. Previous interlaboratory studies conducted by ASTM Committee C16 have focused primarily on a larger heat-flow-meter apparatus having a plate size of 610 by 610 mm.

Three specimens of a high-density glass-fiber board were circulated among 13 laboratories[3] in the United States and Canada. One specimen was provided for calibration, the other specimens for testing. All thermal conductivity measurements were requested to be carried out at the same nominal conditions of 23.9°C (75°F). Test results were analyzed using data analysis software described in the ASTM Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method (E 691). Reference measurements for calibration were provided by the National Institute of Standards and Technology (NIST) 1-m line-heat-source guarded hot plate [*1,2*]. This paper includes a description of the test plan, equipment, test data, and development of the precision and bias statements.

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[3] Initially, 14 laboratories were included. However, one laboratory was removed from the test queue at their request.

TABLE 1—*List of participating laboratories.*

Organization	Address	City, State, Zip	Supervisor
Anter Corporation	1700 Universal Road	Pittsburgh, PA 15235	Karl Coumou
Armstrong World Industries	2500 Columbia Avenue	Lancaster, PA 17603	Charles Allen
Center for Applied Engineering	10301 Ninth Street N	St. Petersburg, FL 33716	R. Gerry Miller
Dow Chemical USA	2301 Brazosport Blvd.	Freeport, TX 77541	Deb Bhattacharjee
Holometrix	25 Wiggins Avenue	Bedford, MA 01730	Troy Soos
Huntsman Chemical Corp.	501 Brunner Street	Peru, IL 61354	Doug Banko
Imi-Tech	701 Fargo Avenue	Elkgrove Village, IL 60007	Gordon Henderson
LaserComp	60 Edgemere Road	Lynnfield, MA 01940	Andrzej Brzezinski
Miles Inc.	Mobay Road	Pittsburgh, PA 15205	John L. Clemons
NIST	Route 270 and 117	Gaithersburg, MD 20899	Robert Zarr[a]
Owens Corning Canada	704 Mara Street	Point Edward, Ontario, Canada	John Scott
Pabco	1110 16 Road	Fruita, CO 81521	Thomas Whitaker
UC Industries, Inc.	137 East Avenue	Tallmadge, OH 44278	Barbara Fabian

[a] ILS Coordinator; ILS Statistician: Eric S. Lagergren (formerly with NIST, now with Nabisco).

Test Plan

The 13 participating laboratories, their ILS supervisors, the ILS coordinator, and ILS statistician are shown in Table 1. A test protocol based on procedures in C 518 was drafted and, after discussion, approved by all participants. In keeping with the ILS objective, the apparatus dimensions were limited specifically to a plate size of 305 by 305 mm (12 by 12 in.). Three specimens (hereafter, 1, 2, and 3) of high-density glass-fiber thermal insulation material, nominally 25 by 305 by 305 mm (1 by 12 by 12 in.), were circulated (Table 2) among the laboratories. Although E 691 recommends that an ILS should include at least three materials covering a range of different test levels because of time limitations, only a single material was circulated; hence, the reason for considering this study a "pilot run." The task group decided to investigate other materials in future interlaboratory studies. As it was, circulating the three specimens among all the participants (Table 1) required nearly one year.

The ILS test protocol required that all participants conduct thermal conductivity measurements[4] at the same nominal test conditions, that is, a mean specimen temperature of 23.9°C (75°F) and a temperature difference no less than 22.2 K (72°F). Each laboratory was required to calibrate its apparatus with Specimen 1 before conducting measurements with Specimens 2 and 3. Replicate measurements of Specimens 2 and 3 were conducted such that the operator removed the specimen from the apparatus at the completion of each test and reconditioned the specimen under similar ambient conditions. The actual test sequence was left to the operator. All tests were to be conducted by the same operator and completed in the shortest time possible (two-week limit per laboratory). Test data from participants were reported using the "official" data sheets shown in the Appendix.

The basic design for the ILS was kept simple in order to minimize secondary effects on estimates of precision. However, the task group was concerned that the specimens could be over-compressed as the ILS progressed; therefore, Specimens 1 and 3 were fitted with spacer stops to investigate the effect of compression, if any. The spacer stops were Bakelite,[5] 9.5 mm in diameter, placed 25.4 mm (1 in.) from the corners of Specimens 1 and 3 (Table 2).

[4] SI units are used throughout the paper. The original units are included in parentheses.

[5] "Bakelite," a trademark of Union Carbide.

TABLE 2—*Specimens of glass-fiber board.*

No.	Nominal ρ, kg·m^{-3}	Purpose	Spacers	Replicates
1	115	Calibration	4	1
2	141	Measurement	None	3
3	142	Measurement	4	3

Test Method C 518

Test Method C 518 covers the determination of steady-state thermal transmission properties through flat slab specimens using a heat-flow-meter apparatus. In principle, the method can be used for a variety of materials, but was limited to a glass-fiber thermal insulation material for purposes of the ILS. The measurement procedure is based on a one-dimensional form of Fourier's heat conduction equation for steady-state heat flow through a flat slab.

$$Q = \lambda A \frac{\Delta T}{L} \tag{1}$$

The thermal transmission properties of thermal insulations determined from standard test methods typically include several mechanisms of heat transfer, including conduction, radiation, and possibly convection. For this reason, ASTM Committee C16 includes the adjective "apparent" when describing thermal conductivity of thermal insulation. In this paper, however, the term thermal conductivity shall be used for brevity.

The heat-flow-meter apparatus is a comparative device and requires a reference material with known thermal properties, preferably traceable to a national standards laboratory, for calibration. Calibration of the heat-flow-meter apparatus is accomplished by using a specimen having known thermal transmission properties, in this case thermal conductivity (λ). For a single calibration specimen, the calibration factor of the apparatus was determined as follows.

$$S = \frac{\lambda \Delta T}{LE} \tag{2}$$

Several different models of commercial heat-flow-meter apparatus were utilized by the participating ILS laboratories. With the exception of one apparatus, all apparatus manufacturers claimed

that their equipment conformed to the apparatus specifications provided in C 518. The data from the apparatus in question were subsequently included in this ILS because no significant deviations or inconsistencies were noted in the data. However, this may not be the case for this particular apparatus for other materials.

Test Method C 177

ASTM Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus (C 177) covers the determination of steady-state thermal transmission properties through flat slab specimens using a guarded-hot-plate apparatus, and, like C 518, the measurement procedure is based on Eq 1. However, in this case, the guarded hot plate apparatus is considered an absolute method for the determination of steady-state thermal transmission properties. For the ILS, NIST provided a calibration equation for λ for Specimen 1 based on 24 thermal conductivity measurements of the three specimens using the NIST 1-m guarded-hot-plate apparatus [*1,2*]. The thermal conductivity measurements were conducted using the experimental design given in Table 3.

The thermal conductivity data were fit to a regression equation having a final form of

$$\lambda = 0.025287 + 3.0226 \times 10^{-5}\,\rho + 1.3238 \times 10^{-4} T \quad (3)$$

Equation 3 was later reformatted and simplified for Specimen 1 (ρ = 115 kg/m^3) as shown in Eq 4. The participating laboratories used the inch-pound (IP) version for calibration of their apparatus.

$$\begin{aligned} &\text{SI units:} \quad \lambda = 0.031935 + 1.3238 \times 10^{-3}\,(T - 23.9) \\ &\text{IP units:} \quad \lambda = 0.22142 + 5.0993 \times 10^{-3}\,(T - 75) \end{aligned} \quad (4)$$

TABLE 3—*Experimental design for C 177 measurements.*

Specimen No.	Nominal ρ, kg·m^{-3}	Replicate Measurements 21.1°C	23.9°C	26.7°C
1	115	2	4	2
2	141	2	4	2
3	142	2	4	2

Results

The test data reported by the laboratory participants were tabulated and examined graphically for consistency. Figure 1 plots the following parameters versus laboratory, where the laboratory number has been *randomly* ordered: (1) λ; (2) *T;* (3) ΔT; (4) *E;* (5) *L;* (6) *A;* (7) heat flow up (1) or down (2); (8) dry-air purge off (0) or on (1); (9) T_a during test; (10) RH during test; (11) test time; (12) specimen mass; (13) T_a during specimen conditioning; (14) RH during specimen conditioning; and (15) conditioning time. Test data for Specimens 2 and 3 are represented by the open square and diamond data points, respectively. Note that some laboratories (for whatever reason) did not report test data for a particular parameter. Summary statistics for the above parameters, where applicable, are given in Table 4. The subscripts 2, 3 refer to Specimens 2 and 3, respectively.

The summary statistics in Table 4 as well as Fig. 1 revealed several interesting trends. The data for λ_2 and λ_3 were nearly identical, indicating little difference due to the absence or presence of the rigid spacer stops. In contrast, however, the standard deviation for L_3 (spacer stops) was nearly 2 times the standard deviation for L_2 (no spacer stops). The variabilities in the mass measurements for Specimens 2 and 3 were quite precise, less than 0.5% (Table 4, Fig. 1). For the test equipment, values of T ranged from 22.8 to 25.1°C (73 to 77°F), indicating that the temperature control was less precise for some laboratories. The ΔT across the specimens, which is generally fixed for a particular laboratory, ranged from 21.9 to 28.3 K (71 to 83°F); HFT output, from 0.390 to 3.394 mV; and, *A,* from 25.81 to 232.3 cm^2. The majority of the apparatus was operated with the direction of heat flow down and the dry air purge off (Fig. 1). The environmental conditions of the laboratory and the time for testing and conditioning varied widely (Table 4, Fig. 1).

As a check of these secondary factors, the thermal conductivity was plotted versus each factor shown in Fig. 1. Figure 2 shows a multiplot of λ versus thermal conductivity versus (1) *T;* (2) ΔT; (3) *E;* (4) *L;* (5) *A;* (6) heat flow up (1) or down (2); (7) dry-air purge off (0) or on (1);(8) T_a during test; (9) RH during test; (10) test time; (11) specimen mass; (12) T_a during specimen conditioning; (13) RH during specimen conditioning; and (14) conditioning time. No systematic trends were noted in the plots, and the data were subsequently analyzed with the "ASTM Interlaboratory Data Analysis Software" provided in the Adjunct for E 691.

TABLE 4—*Summary statistics for test data.*

Test Parameter	Units	Average	Standard Deviation	Minimum	Maximum
λ_2	W·m^{-1}·K^{-1}	0.0329	0.0005	0.0320	0.0336
λ_3	W·m^{-1}·K^{-1}	0.0330	0.0004	0.0321	0.0336
T	°C	23.9	0.5	22.8	25.1
ΔT	K	25.1	2.6	21.9	28.3
E	mV	1.228	0.539	0.390	3.394
L_2	mm	25.84	0.17	25.58	26.20
L_3	mm	25.76	0.31	24.89	26.09
A	cm^2	111.98	59.00	25.81	232.26
T_a lab	°C	22.1	1.4	19.4	24.4
RH lab	%	46	12	14	70
Test time	h	3.6	6.2	0.6	25.1
m_2	g	338.0	1.1	335.7	340.6
m_3	g	351.9	1.1	349.3	354.3
T_a condition	°C	22.5	1.0	20.0	24.4
RH condition	%	44	9	18	53
Time condition	h	36.3	44.1	2.0	194.4

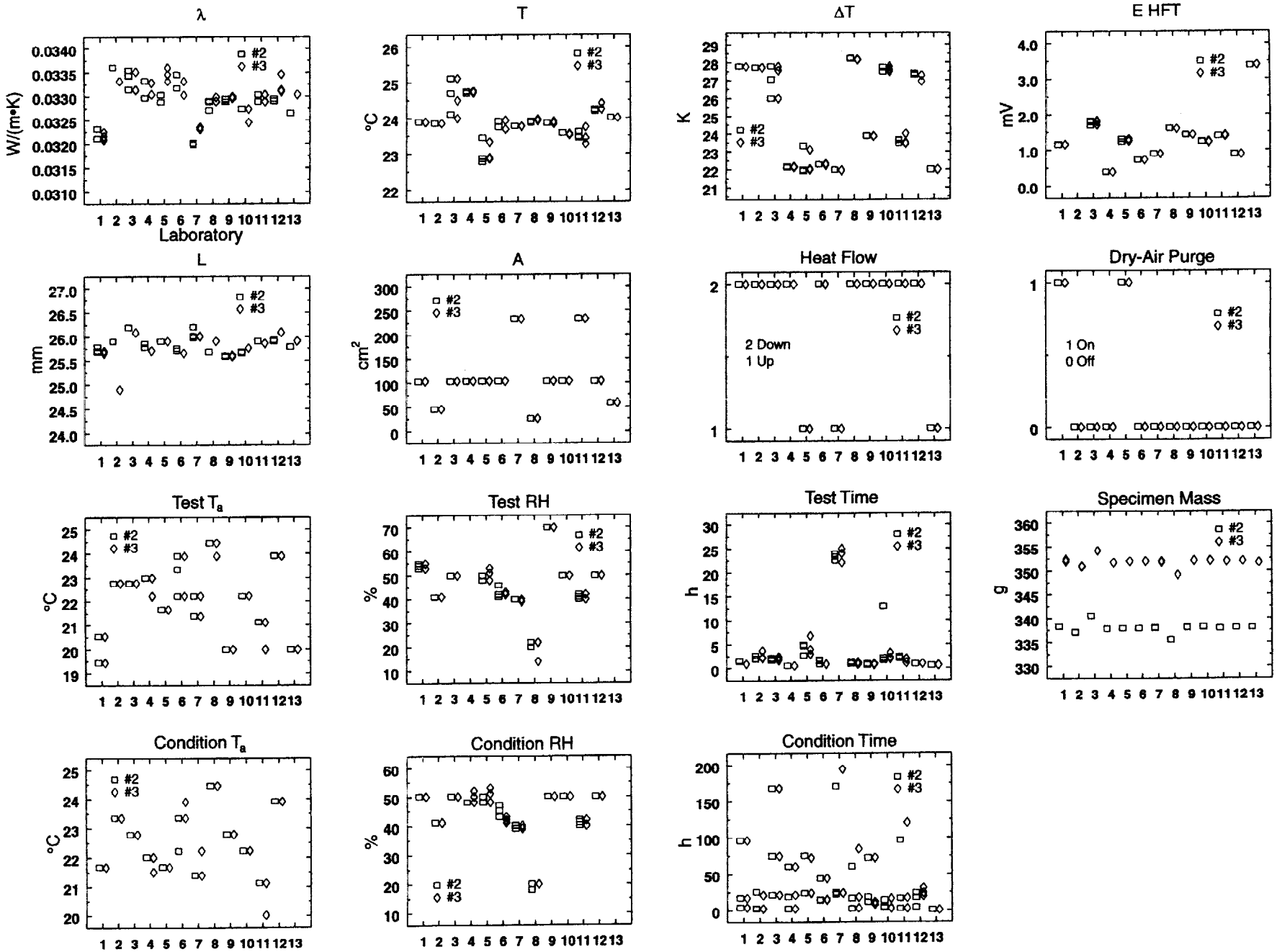

FIG. 1—*Multiplot of test parameters versus laboratory.*

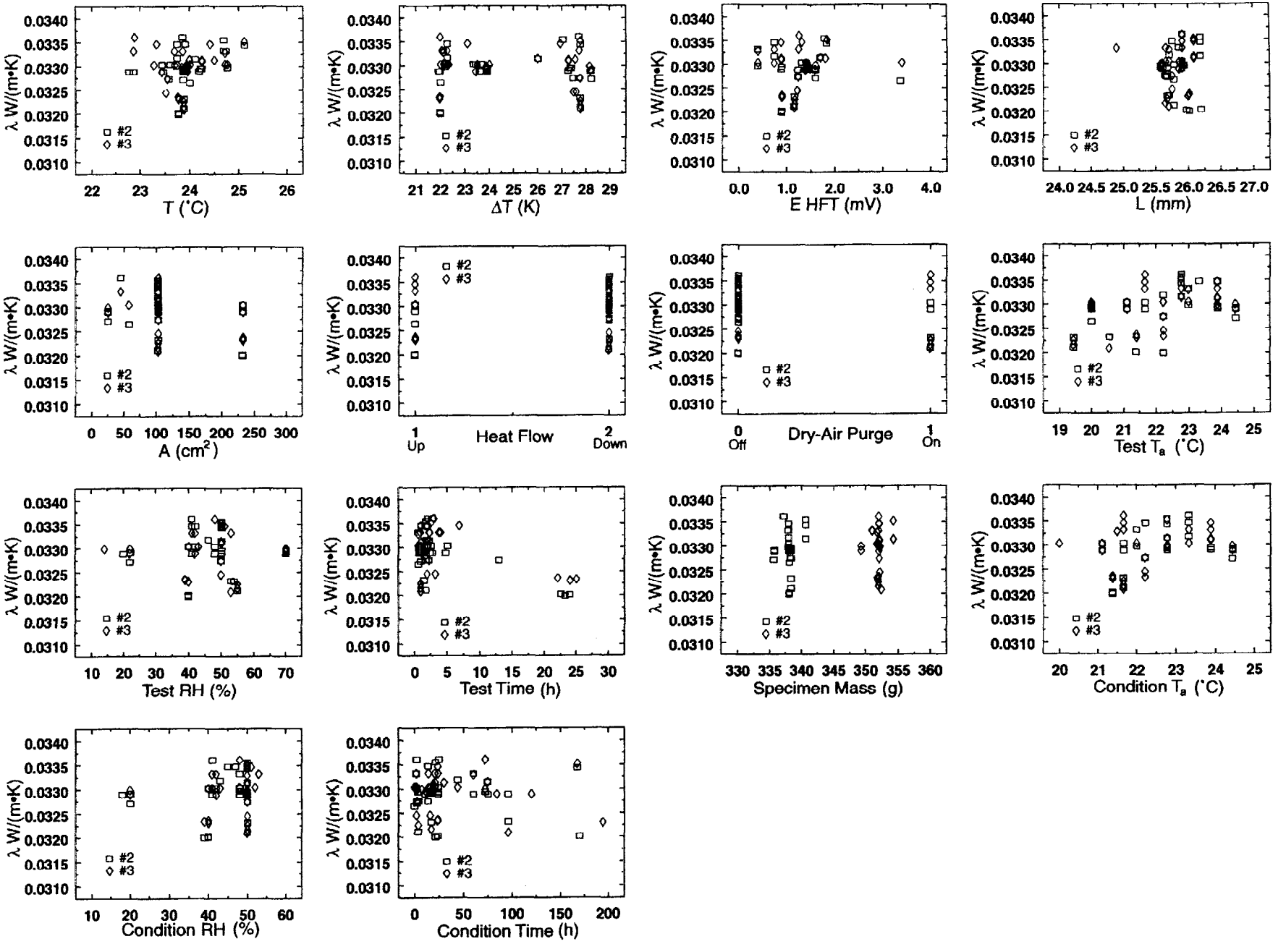

FIG. 2—*Multiplot of λ versus test parameters.*

Table 5 summarizes the thermal conductivity data (rounded to three significant digits) for Specimens 2 and 3 for 12 of the laboratories. Only the laboratories that followed the test protocol and conducted three independent replicates for Specimens 2 and 3 have been included. Laboratory 13 (Fig. 1) did not conduct replicates as instructed in the test protocol, and, therefore, their data were subsequently removed from further analysis.

Summary statistics for the ILS were determined by using the "ASTM Interlaboratory Data Analysis Software" provided in the Adjunct for E 691. Table 6 gives summary statistics for Specimens 2 and 3, including the grand average ($\bar{\lambda}$), repeatability standard deviation (s_r), coefficient of variation (CV%$_r$), reproducibility standard deviation (S_R), and coefficient of variation (CV%$_R$). Note that the summary statistics are slightly different than the values given in Table 4 because test data from Laboratory 13 were omitted from the final analysis.

Not surprisingly, the repeatability standard deviation was less (by about a factor of 4) than the reproducibility standard deviation for both specimens. As alluded to above in Table 4, the data indicated that the effect of the Bakelite spacers, although slightly less for the case with spacers, was negligible for test results of this ILS.

For ASTM test methods, the preferred index of precision for repeatability and reproducibility is the 95% limit on the difference between two test results. This means that, in a larger context, if one considers 100 pairs of test results from laboratories similar to this study, approximately 95 can be expected to differ in absolute value by less than $1.960 \times \sqrt{2} \times$ the standard deviation (E 177). The 95% repeatability and reproducibility limits (r and R, respectively) are defined in Eq 5 (E 691).

$$r = 2.8\ s_r \text{ or } r = 2.8\ \text{CV}\%_r$$
$$R = 2.8\ S_R \text{ or } R = 2.8\ \text{CV}\%_R \quad (5)$$

Table 7 summarizes the 95% repeatability and reproducibility limits for Specimens 2 and 3, respectively.

TABLE 5—*Thermal conductivity data ($W \cdot m^{-1} \cdot K^{-1}$).*

Laboratory No.	Specimen 2 Replicate			Specimen 3 Replicate		
	1	2	3	1	2	3
1	0.0323	0.0323	0.0321	0.0321	0.0322	0.0322
2	0.0336	0.0336	0.0336	0.0333	0.0333	0.0333
3	0.0334	0.0331	0.0335	0.0335	0.0331	0.0331
4	0.0333	0.0333	0.0330	0.0333	0.0330	0.0330
5	0.0329	0.0329	0.0330	0.0336	0.0333	0.0335
6	0.0332	0.0335	0.0335	0.0330	0.0333	0.0330
7	0.0320	0.0320	0.0320	0.0323	0.0324	0.0323
8	0.0329	0.0329	0.0327	0.0329	0.0330	0.0330
9	0.0329	0.0329	0.0329	0.0330	0.0330	0.0330
10	0.0327	0.0327	0.0327	0.0325	0.0327	0.0325
11	0.0329	0.0330	0.0330	0.0329	0.0330	0.0330
12	0.0329	0.0329	0.0329	0.0335	0.0331	0.0331

Discussion

Values for the consistency statistics h (between-laboratory) and k (within-laboratory) were computed using the interlaboratory analysis software for E 691. Critical values of h depend on the number of laboratories, and critical values of k depend on the number of laboratories and number of replicates per laboratory per specimen (E 691). For 12 laboratories and 3 replicates per laboratory per specimen, the critical values of h and k were 2.38 and 2.14, respectively. Critical values of h and k were calculated using values of Student's t at the 0.5% two-tailed significance level, that is, t_α where $\alpha = 0.025$.

Figure 3 plots h-values for each specimen by laboratory. The values for each laboratory are either positive or negative, which is typical. For Specimen 2, h-values ranged from −2.02 to +1.49; for Specimen 3, from −1.96 to +1.25. None of the h-values exceeded the critical value of 2.38. Opposite signs for different specimens (in this case, the same material) measured by one laboratory would indicate that the laboratory was most likely experiencing some measurement problems. For Laboratory 9, the extremely small difference in sign for Specimens 2 and 3 probably does not represent a "true" difference.

Figure 4 plots k-values for each specimen by laboratory. Here, large or small values are important. For Specimen 2, the k-values ranged from 0.00 to 1.84; for Specimen 3, from 0.00 to 1.70. None of the k-values exceeded the critical value of 2.14. The extremely low k-values for Laboratories 2 and 10 (Specimen 2) are questionable because the level of variability was essentially zero (see also Table 5). This could be caused by insensitive equipment, replicate measurements that were not truly independent, or some other problem. Laboratories that had large within-laboratory variability relative to the other laboratories included 3, 4, 6, 10, and 12. The reasons for the large k-values are unknown.

It is interesting to compare the results from this analysis to a previous interlaboratory study involving similar equipment but different materials. In 1991, a two-stage interlaboratory study was published for rigid urethane-modified polyisocyanurate foam board stock [3] at thicknesses of 25, 50, and 75 mm. Measurements were conducted at a mean temperature of 23.9°C (75°F). In the first stage, 50-mm-thick specimens were measured by 16 laboratories, and, in the second stage, 25-mm-thick and 75-mm-thick specimens were measured by 15 laboratories. Furthermore, in the second stage, the participants investigated the presence of a thermal break in the foil laminate corresponding to the meter area of the heat-flow-meter apparatus. In general, the study determined that there was no statistically significant difference in the data for specimens with and with-

TABLE 7—*95% precision indexes.*

Specimen No.	$\bar{\lambda}$, $W \cdot m^{-1} \cdot K^{-1}$	r, $W \cdot m^{-1} \cdot K^{-1}$	R, $W \cdot m^{-1} \cdot K^{-1}$	r, %	R, %
2	0.03293	0.00032	0.00130	1.0	4.0
3	0.03296	0.00036	0.00117	1.1	3.5

TABLE 6—*Summary precision statistics.*

Specimen No.	$\bar{\lambda}$, $W \cdot m^{-1} \cdot K^{-1}$	s_r, $W \cdot m^{-1} \cdot K^{-1}$	S_R, $W \cdot m^{-1} \cdot K^{-1}$	CV%$_r$, %	CV%$_R$, %
2	0.03293	0.00011	0.00047	0.3	1.4
3	0.03296	0.00013	0.00042	0.4	1.3

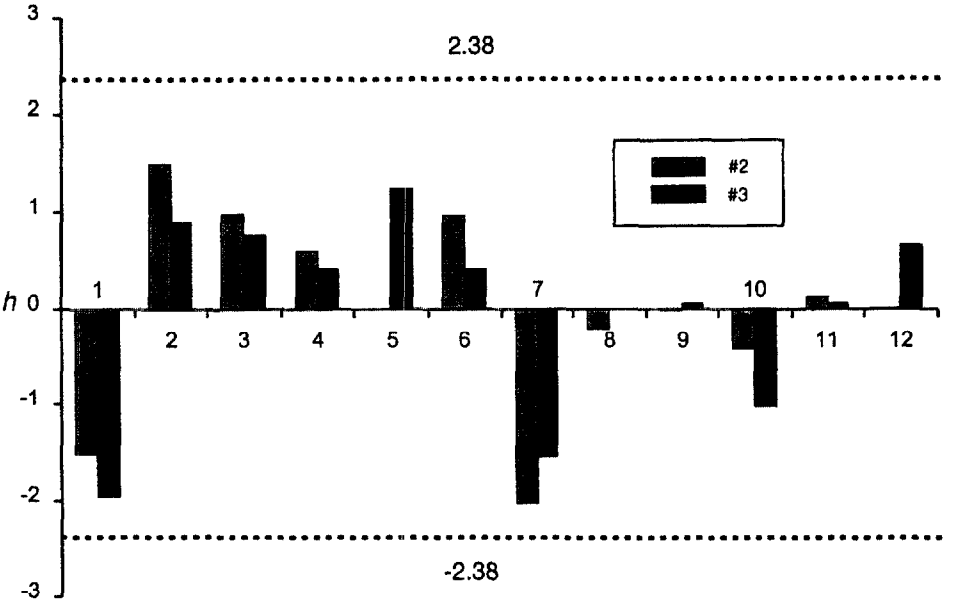

FIG. 3—h-*values by laboratory.*

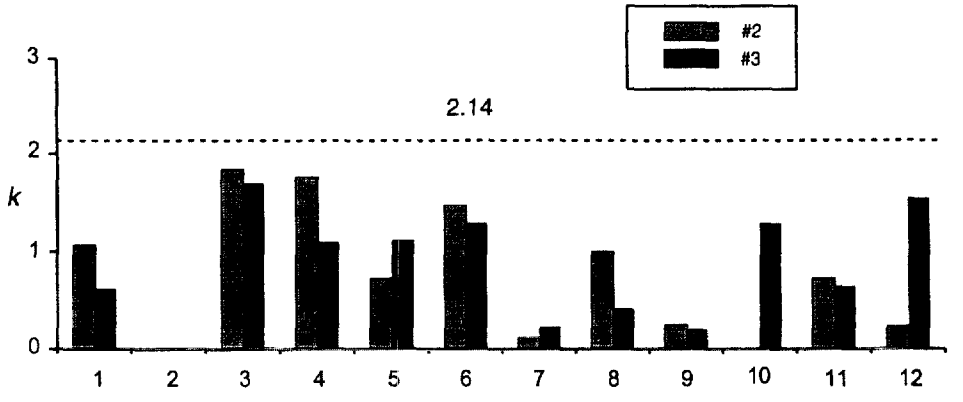

FIG. 4—k-*values by laboratory.*

out a thermal break. It is interesting to note, however, that at thicknesses 25, 50, and 75 mm and for specimens without a thermal break, the 95% repeatability limits were 6.7, 7.2, and 11.2% [*3*]. Similarly, the 95% reproducibility limits were 8.4, 11.7, and 20.2% [*3*]. The reported levels of imprecision are much greater than the levels reported here and would suggest that further investigations are needed to determine the reasons for the different levels of imprecision for different materials and different specimen thicknesses.

TABLE 8—*NIST GHP thermal conductivity measurements.*

Replicate No.	Mean *T*, °C	Specimen 2, $W \cdot m^{-1} \cdot K^{-1}$	Mean *T*, °C	Specimen 3, $W \cdot m^{-1} \cdot K^{-1}$
1	23.89	0.03258	23.88	0.03287
2	23.89	0.03263	23.88	0.03288
3	23.89	0.03265	23.89	0.03286
4	23.89	0.03267	23.89	0.03288

Bias

Unfortunately, E 691 provides guidance in determining estimates *only* for precision in a test method. A definition for bias and example statements are described in ASTM Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods (E 177). Practice E 177 states that

> "the bias of the test method, for a specific material, may be calculated by comparing the average of all the (ILS) test results obtained for that material with the accepted reference value". . . . In determining the bias, the effect of the imprecision is averaged out by taking the average of a very large set of (30 or more) test results."

In view of the last statement, it is our contention that it is necessary to include the level of imprecision when determining the bias for C 518 as explained below.

"Accepted reference values" were obtained by conducting thermal conductivity measurements using the NIST 1-m guarded-hot-plate apparatus [*1,2*] for Specimens 2 and 3 in accordance with C 177, Table 8. Figure 5 compares the C 177 measurements with the C 518 mean value of each laboratory for Specimens 2 and 3.

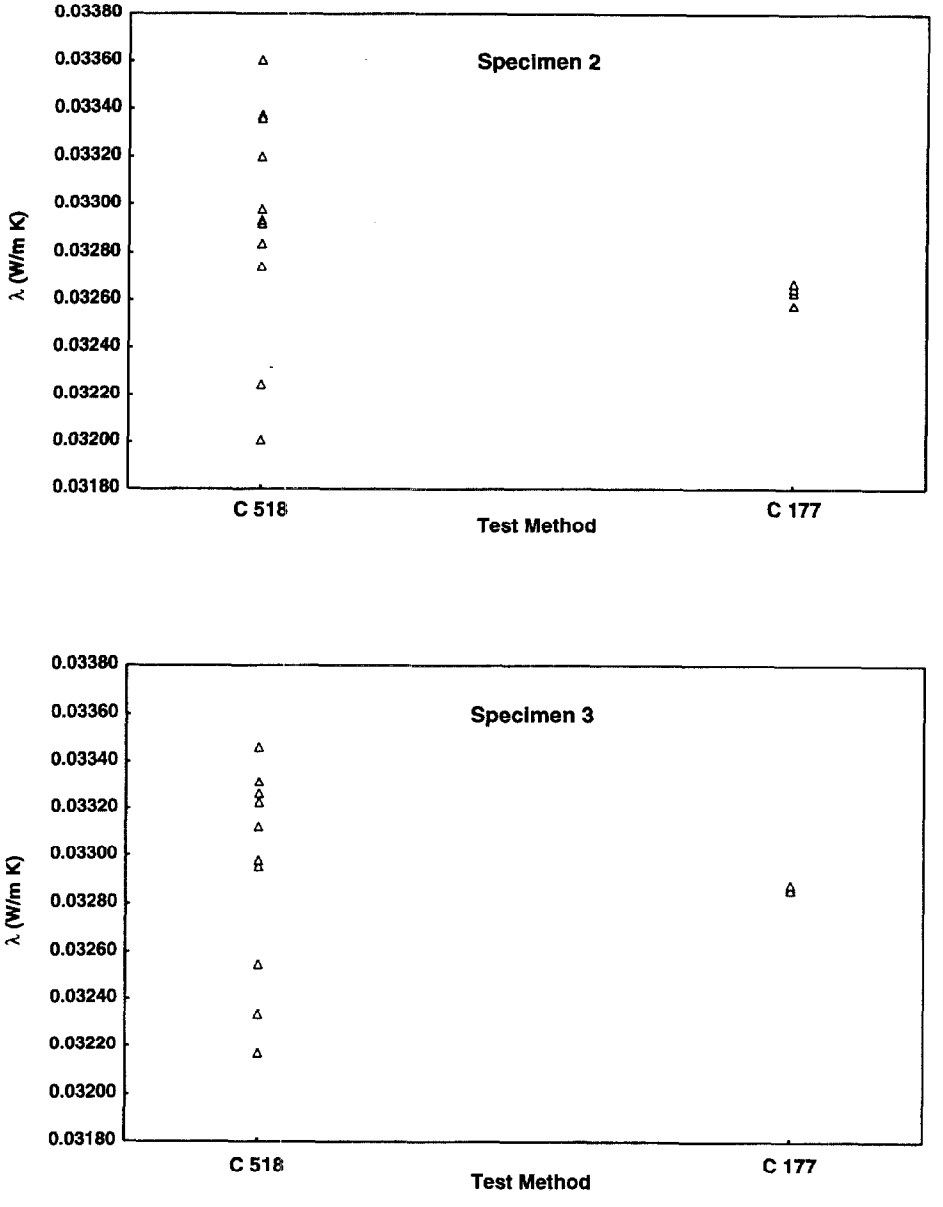

FIG. 5—*Comparison of C 518 laboratory means and C 177 measurements.*

TABLE 9—*Precision indexes.*

Materials	Average $\bar{\lambda}$, $W \cdot m^{-1} \cdot K^{-1}$	s_r, $W \cdot m^{-1} \cdot K^{-1}$	S_R, $W \cdot m^{-1} \cdot K^{-1}$	r, $W \cdot m^{-1} \cdot K^{-1}$	R, $W \cdot m^{-1} \cdot K^{-1}$	r, %	R, %
Glass-fiber board-no spacers	0.03293	0.00011	0.00047	0.00032	0.00130	1.0	4.0
Glass-fiber board-spacers	0.03296	0.00013	0.00042	0.00036	0.00117	1.1	3.5

Assuming the NIST 1-m guarded hot plate apparatus (C 177) measures the true thermal conductivity, the true bias in C 518 is $\mu_{HFM} - \mu_{GHP}$. In actuality, however, one can only estimate true bias. Therefore, a 95% confidence interval is constructed for the true bias. If the interval does *not* contain the value zero, we *would* conclude that the C 518 measurements are biased. If the interval contains zero, we *could* conclude that the C 518 measurements are not biased. Note the difference in the last two sentences. In the first case, $\mu_{HFM} - \mu_{GHP} = 0$ is not contained in the interval; therefore, we can conclude that the measurements are biased (stronger case). In the second case, $\mu_{HFM} - \mu_{GHP} = 0$ is contained in the interval; however, it is only one of many plausible values for $\mu_{HFM} - \mu_{GHP} = 0$. Hence, the confidence interval may contain values of engineering significance. In our opinion, other analyses, such as an engineering assessment of uncertainties, may be needed to assess bias.

An estimate of bias for C 518 was obtained as follows. Let x_{ij} be the mean of the HFM measurements for Laboratory j (=1,. . ., n_x = 12) on Specimen i (=2,3) calculated from the data in Table 5. Let z_{ik} be measurement k (=1,. . ., n_z = 4) on Specimen i (2,3) given in Table 8. Let n_s = 2 be the total number of specimens. A 95% confidence interval for (true) bias in the HFM test method averaged

over specimens is

$$\bar{x} - \bar{z} \pm t_{0.975,\nu} \sqrt{\frac{s_x^2}{n_x} + \frac{s_z^2}{n_s n_z}} \tag{6}$$

where

$$s_x^2 = \frac{\sum_j^{n_x} (\bar{x}_j - \bar{x})^2}{n_x - 1} \qquad s_z^2 = \frac{\sum_i^{n_x} \sum_k^{n_z} (\bar{z}_{ik} - \bar{z}_i)^2}{n_s(n_z - 1)}$$

$$\nu = \frac{(s_x^2/n_x + s_z^2/(n_s n_z))^2}{\frac{(s_x^2/n_x)^2}{n_x - 1} + \frac{(s_z^2/(n_s n_z))^2}{n_s(n_z - 1)}}$$

and where ν is Welch-Satterthwaite's effective degrees of freedom [*4*] and $t_{0.975,\nu}$ is the 97.5 percentile of the t-distribution with ν degrees of freedom.

From Table 5, we compute $\bar{x} = 0.032942$ W·m^{-1}·K^{-1} and $s_x = 0.000412$ W·m^{-1}·K^{-1} and from Table 8, $\bar{z} = 0.032753$ W·m^{-1}·K^{-1} and $s_z = 0.000029$ W·m^{-1}·K^{-1}, and so $\nu = 11.2$. Thus, using Eq 6, the 95% confidence interval for true bias is

$$0.000190 \text{ W·m}^{-1}\text{·K}^{-1} \pm 0.000262 \text{ W·m}^{-1}\text{·K}^{-1},$$

$$\text{or } (-0.000072, +0.000452) \text{ W·m}^{-1}\text{·K}^{-1}$$

Since the interval contains zero, we would *not* conclude that C 518 is biased. In other words, it is plausible that $\mu_{HFM} - \mu_{GHP} = 0$. However, it is also plausible that $\mu_{HFM} - \mu_{GHP}$ could equal any other value in the interval, for example $\mu_{HFM} - \mu_{GHP} = +0.00045$ W·m^{-1}·K^{-1}. A valid question could then be asked: Is a bias of this magnitude considered to be of engineering significance? The answer is not trivial but the following explanation is offered. For the specimens measured, the relative difference for a bias of magnitude +0.00045 W·m^{-1}·K^{-1} is +1.4%. The current accepted uncertainty for C 518 is, at best, ±2% within the measurements provided by C 177. Therefore, from an engineering point of view, we would also conclude that C 518 is *not* biased for this particular set of measurements.

Conclusions

A precision statement for C 518 has been prepared in accordance with E 691. Precision indexes, characterized by repeatability, s_r, r, and reproducibility, s_R, R, are summarized in Table 9. This precision statement is provisional because an insufficient number of materials were involved. Within five years, additional data will be obtained and processed that do meet the requirements of E 691.

A bias statement for C 518 has been prepared following example statements suggested in E 177. Bias, characterized by the difference between the test results above and "accepted reference values" prepared from C 177 for the same specimens, has been determined to be statistically insignificant at the $\alpha = 5\%$ level (95% confidence interval) for these materials.

References

[*1*] Powell, F. J. and Rennex, B. G., "The NBS Line-Heat-Source Guarded Hot Plate for Thick Materials," ASHRAE SP 38, 1983, pp. 657–672.

[*2*] Zarr, R. R. and Hahn, M. H., "Line-Heat-Source Guarded-Hot-Plate Apparatus," Adjunct ASTM Designation C 1043, Request PCN No. 12-310430-61.

[*3*] Smith, S. A., Lynch J. J., Moore, M. L., and Galbraith, C. J., "A Round Robin Testing Program to Estimate the Precision of ASTM C 518 for Measuring the *k*-Factor of Rigid Foam Product," *Journal of Thermal Insulation*, Vol. 14, January 1991, pp. 184–194.

[*4*] ANSI, "U.S. Guide to the Expression of Uncertainty in Measurement," ANSI/NCSL Z540-2-1997, pp. 59–66.

Appendix

TEST DATA SHEET - ASTM C 518 ILS (1993)
** CALIBRATION, ROUND #1 **

Laboratory _____ Date _____

ILS Supervisor/Phone _____

	CAL #1
1. Specimen	
2. Conditioning	
a. Temperature (°F)	_____
b. Humidity (% RH)	_____
c. Duration (hrs)	_____
3. Date/Time in Apparatus	_____
4. Date/Time out of Apparatus	_____
5. Initial Mass (lb)	_____
6. Final Mass (lb)	_____
7. Bulk Density (lb/ft^3) (Optional)	_____
8. Test Thickness of specimen (in.)	_____
8a. From C 518 apparatus? (yes/no)	_____
8b. Independent method? (yes/no) If yes, specify in 19.	_____
9. Hot Face (°F)	_____
10. Cold Face (°F)	_____
11. HFT Output (mV)	_____
12. λ (NIST) ($Btu \cdot in/h \cdot ft^2 \cdot °F$)	_____
13. S ($(Btu/h \cdot ft^2)/mV$)	_____
14. Laboratory Temp. (°F)	_____
15. Laboratory Humidity (% RH)	_____
16. Was dry-air purge used? (yes/no)	_____
17. Heat flow direction (up/down)	_____
18. Meter area (in. by in.)	_____

19. Observation/Remarks _____

Send to: R. Zarr, NIST, Building 226, Room B-320, Gaithersburg, MD 20899

Appendix

TEST DATA SHEET - ASTM C 518 ILS (1993)
** MEASUREMENTS, ROUND #1 **

Laboratory _____ Date _____

ILS Supervisor/Phone _____

	1	2	3
1. Specimen	MES #2	MES #2	MES #2
2. Conditioning			
a. Temperature (°F)			
b. Humidity (% RH)			
c. Duration (hrs)			
3. Date/Time in Apparatus			
4. Date/Time out of Apparatus			
5. Initial Mass (lb)			
6. Final Mass (lb)			
7. Bulk Density (lb/ft^3) (Optional)			
8. Test Thickness of specimen (in.)			
8a. From C 518 apparatus? (yes/no)			
8b. Independent method? (yes/no) If yes, specify in 19.			
9. Hot Face (°F)			
10. Cold Face (°F)			
11. HFT Output (mV)			
12. S from "CAL#1" ($(Btu/h{\cdot}ft^2)/mV$)			
13. λ ($Btu{\cdot}in/h{\cdot}ft^{2}{\cdot}°F$)			
14. Laboratory Temp. (°F)			
15. Laboratory Humidity (% RH)			
16. Was dry-air purge used? (yes/no)			
17. Heat flow direction (up/down)			
18. Meter area (in. by in.)			

19. Observation/Remarks _____

Send to: R. Zarr, NIST, Building 226, Room B-320, Gaithersburg, MD 20899