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An International Study of Guarded Hot Plate Laboratories Using Fibrous Glass and Expanded Polystyrene Reference Materials

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Abstract: 'Thermal conductivity measurements of four thermal insulation reference inaterials arc presented. The measurements were obtained from an international study of guarded-hot-plate laboratories in Canada, France, Japan, the United Kingdom, and the United States. For each reference material, the study requires five independent replicate measurements at a fixed temperature of 297.15 K, and single-point measurements at 280 K, 290 K, 300 K, 310 K, and 320 K. ***An*** important finding from the replicate analysis is the existence of a laboratory-material interaction: that is, there are laboratory-to-laboratory differences in both location and variation that change from inaterial to matenal. The major underlying source for the variability (both within- and between-laboratory) in the replicate data is discussed. The analysis of the multi–temperature (280 K to 320 K) data supports the laboratory-material interaction as exhibited in the fixed-temperature replicate data. The multi–temperature analysis also reveals an increasing ditference between laboratories as the temperature departs from 297.15 K

Keywords: certified reference matenal, guarded hot plate. interlaboratory, reference matenals, thermal insulation, thermal conductivity, SRM

Introduction

In 1996, an ASTM C-16 Workshop on thermal insulation Standard Reference Materials (SRMs) identified concerns with the transference of national reference matenals across international borders [*1*] Responding to similar concern in Europe, the National Physical Laboratory began to organize an international study of guarded-hot-plate apparatus in national standards laboratones in Canada, France, Japan, United Kingdom, and United States in 1997 The purpose of the study was to assess the measurement vanability among test results of five laboratory participants the National

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Research Council Canada (NRCC), Laboratoire National d'Essais (LNE), Japan Testing Center for Construction Materials (JTCCM), the National Physical Laboratory (NPL), and the National Institute of Standards and Technology (NIST). The study investigated one regional and three national reference materials. Ten specimens of each material were distributed to the participants by an issuing organization (or delegate laboratory).

This study requested two sets of data: 1) five replicate measurements of each specimen at 297.15 K (24 °C); and 2) individual (single-point) measurements at 280 K, 290 K, 300 K, 310 K, and 320 K. The test results were conducted in accordance with either International Standard Thermal Insulation–Determination of Steady-State Areal Thermal Resistance and Related Properties–Guarded Hot Plate Apparatus Test Method (ISO 8302) or ASTM Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded Hot Plate Apparatus, (C 177). A detailed analysis of the resulting data has been provided to the laboratory participants [2] and a summary of the results has been recently presented [3]. The present paper focuses primarily on the replicate thermal conductivity data at 297.15 K (24 °C).

Reference Materials

The reference materials were selected to test a wide -- yet manageable – variety of insulation materials from Asia, Europe, and North America. Table 1 summarizes the reference materials by designation, description, density (ρ), thickness (L), temperature range (T), source, and reference. Materials 1 through 3 were fibrous in composition, ranging from 13 kg/m^3 to 200 kg/m^3. Material 4 was a molded-beads, expanded polystyrene board (38 kg/m^3). Material 3, which is a mixture of glass and mineral oxides fibers having high-temperature capabilities, is currently undergoing an internal review process for certification. Each issuing laboratory was responsible for characterizing and distributing 10 specimens of the reference material to the laboratory participants [2]. The European Commission Institute for Reference Materials and Measurements (IRMM) agreed to provide specimens of Certified Reference Material IRMM-440 to NPL for characterization and distribution to the participants. As a side note, the NIST Standard Reference Material Program has officially designated SRM 1451 as obsolete due to historically low customer demand. (Although obsolete, SRM 1451 is available from the Building and Fire Research Laboratory at NIST.) Comparisons of the test results with predicted values of the NIST Standard Reference Materials have been presented previously [2,3].

Table 1 – *Reference Materials*

ID	Designation	Description	ρ (kg/m^3)	L (mm)	T (K)	Source and Reference
1	SRM 1451	Fibrous glass blanket	13	25	100 to 330	NIST [4]
2	IRMM-440	Resin-bonded glass fibre board	70	35	263 to 323	IRMM [5]
3	JTCCM candidate	Mineral-oxide fiberboard	200	25	---	JTCCM
4	SRM 1453	Expanded polystyrene board	38	13	285 to 310	NIST [6]

Laboratory Apparatus

Table 2 summarizes the major parameters of the guarded-hot-plate apparatus used in this study. Each laboratory determined values for their relative expanded uncertainty (U), *independently* of this study, based on international guidelines [7]. The relative expanded uncertainties reported here for a coverage factor of $k = 2$ represent a level of confidence of approximately 95% [7]. The expanded uncertainty defines an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand (λ).

Table 2 *Laboratory Guarded-Hot-Plate Apparatus*

Parameter	JTCCM	LNE	NIST	NPL	NRCC
ID	1	2	3	4	5
Plate, mm	300×300	610×610	1016 ϕ	610×610	610×610
Meter plate, mm	150×150	300×300	406.4 ϕ	305.2×305.2	250×250
Plate emittance	0.9	0.86 ± 0.05	0.89	>0.9	0.89
Edge guarding	Condition air	[1]	Condition air	[2]	Glass-fiber
Type of heater	Distributed	Distributed	Line source	Distributed	Distributed
Temperature sensor	Type T	Type K	PRT[3]	Type E	Type T
Operation mode	2-sided	2-sided	2-sided	1-sided	2-sided
U ($k = 2$) (%)	Not reported	1.5	1.5 (IRMM-440) 1.0 (others)	1.2	1.0

[1] Edge insulation, temperature controlled peripheral guard and additional outer edge insulation
[2] Linear temperature gradient edge guard and 100 mm expanded polystyrene
[3] Platinum resistance thermometer

Test Protocol

Under steady-state conditions, measurements of thermal conductivity[2] (λ) for the pair of specimens are determined using the following equation:

$$Q = \lambda\, 2A \frac{\Delta T}{L} \tag{1}$$

where Q is the heat flow (W) through the meter area of the specimens; $2A$ is the meter area normal to direction of heat flow (m^2); ΔT (K) is the temperature difference across the specimen hot (T_h) and cold surfaces (T_c); and, L (m) is the in-situ thickness of the pair of specimens. Values of λ are reported at the mean temperature, $T_m = (T_h + T_c)/2$.

For a single-sided mode of operation (Table 2), a single specimen is placed between the hot and cold plates of the apparatus. The other specimen is replaced with an auxiliary piece of insulation. The auxiliary guard plate is maintained at the same temperature as

[2] The thermal transmission properties of heat insulation 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.

the hot plate. For determining λ in the single-sided case, Eq 2 is modified slightly by taking a meter area *(A)* coefficient of unity.

Each participant was requested to conduct five replicate measurements for each pair of specimens at 297.15 K (24 °C) and a temperature difference of 20 K (100 observations). The operator was requested to remove the specimens from the apparatus after each measurement and re-install the specimens after sufficient conditioning. After completion of the replicate measurements, thermal conductivity measurements were conducted for each material at 280 K, 290 K, 300 K, 310 K, and 320 K and a temperature difference of 20 K (100 observations). The multi-temperature tests were conducted in random order; however, the specimens were not removed from the apparatus between temperature settings.

Except for SRM 1451, the materials were tested at thicknesses determined by each laboratory with the only provision that the clamping pressure exerted on the specimens by the measuring equipment was limited from 1000 Pa to 2000 Pa. For SRM 1451, the test thickness was limited to 25.4 mm by utilizing spacer stops placed at the perimeter of the specimen to prevent over-compression of the material during testing. The use of spacer stops for the other materials (for example, limiting plate movement due to specimen creep, if any) was left to the operator's discretion. The test data were recorded in SI units on "official" data forms and returned to MST for analysis.

Fixed Temperature (297.15 K) Replicate Data

Figure 1 plots the measurements of λ (297.15 K) versus laboratory (identified[3] in Table 2) for each of the four materials (Table 1). For each laboratory, the replicate observations are offset along the x-axis to assess trends in the run-sequence of an individual laboratory. For laboratories 2, 3, 4, and 5, the data points include symmetric error bars representing the respective laboratory's estimate of expanded uncertainty (*U*) for λ (Table 2). The major conclusions from Figure 1 are as follows:

1) For materials 1, 2, and 3, the laboratories differed in average response.
2) In contrast, for material 4, the average laboratory responses were essentially the same.
3) Fer materials 1 and 2, laboratory 1 had a significantly high average response.
4) For materials 1, 2, and 3, laboratory 2 was consistently higher than laboratory 3.
5) For material 3, laboratory 4 was significantly low.
6) The differences between the five laboratories changed from material to material – that is, there is a laboratory-material interaction.

[3] While planning this study, the laboratory participants decided that the international user communities would derive maximum benefit by open presentation of the data; hence, the data are *not* presented anonymously.

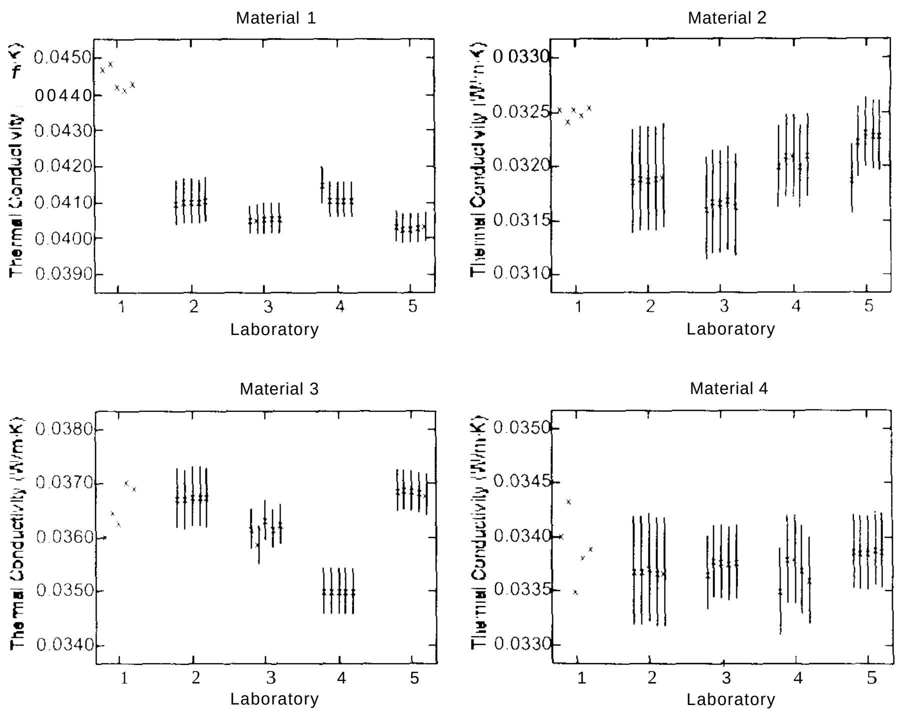

Figure 1 - *Replicate data (297.15 K) versus laboratory. Error bars equal laboratory expanded uncertainty (Table 2).*

Summary Statistics

The statistical treatment of interlaboratory data typically involves determining location and variation parameters based on an assumed underlying model for the data. For the fixed temperature (207.15 K) replicate data, there are two primary factors: laboratory (5 levels) and reference material (4 levels). Thus, the underlying model for these data is assumed to have the following form:

$$y = a_{ij} + \varepsilon \qquad (2)$$

where y is the response variable λ, a_{ij} is a constant for laboratory i and material j, and ε is error. The effect of temperature as a primary factor, from 280 K to 320 K, is discussed later.

Table 3 summarizes the mean values (location) and standard deviations (variation) for the replicate data (100 observations). Each entry represents the local (5 observations) mean ($\bar{\lambda}$) or standard deviation (SD(λ)), respectively, for a particular laboratory. The last column provides the respective grand or "pooled" statistic (25 observations) for each laboratory (across all materials). The last row in each table provides the respective grand or "pooled" statistic (25 observations) for each material (across all laboratories).

Table 3a – *Means for Replicates (297.15 K)*

Lab	Material 1 $\bar{\lambda}$ (W/m K)	Material 2 $\bar{\lambda}$ (Wim K)	Material 3 $\bar{\lambda}$ (W/m K)	Material 4 $\bar{\lambda}$ (W/m K)	Lab Average (W/m K)
1	0.04448	0.03251	0.03655	0.03391	0.03686
2	0.04104	0.03189	0.03675	0.03369	0.03584
3	0.04055	0.03166	0.03616	0.03375	0.03553
4	0.04118	0.03206	0.03500	0.03368	0.03548
5	0.04032	0.03220	0.03686	0.03387	0.03581
Average	0.04151	0.03206	0.03626	0.03378	0.03591

Table 3b – *Standard Deviations* ***for*** *Replicates (297.15 K)*

Lab	Material 1 SD (λ) (W/m K)	Material 2 SD (λ) (W/m K)	Material 3 SD (λ) (W/m K)	Material 4 SD (λ) (W/m K)	Pooled SD (W/m K)
1	0.00032	0.00005	0.00043	0.00030	0.00031
2	0.00003	0.00002	0.00002	0.00002	0.00002
3	0.00002	0.00004	0.00017	0.00005	0.00009
4	0.00018	0.00005	0.00000	0.00013	0.00011
5	0.00003	0.00018	0.00004	0.00001	0.00009
Pooled SD	0.00016	0.00009	0.00021	0.00015	0.00016

The last column in Table 3a reveals that the average of laboratory 1 across all four materials is consistently higher than the other laboratories. On the average across all four materials, laboratories 2 and 5, and 3 and 4, are closely paired and each pair of laboratories differs by about 0.8%. The last column in Table 3b reveals that laboratory 1 **is** consistently noisy across all four materials. Laboratories 3, 4, and 5 exhibit similar levels of variability while laboratory 2 is extremely precise (by nearly a factor of ***5*** in comparison to the other three laboratories) across all four materials.

Treatment **of** Anomalous Data

The results from Figure 1 and Table 3 reveal that the test results for materials 1 and 3 from laboratories 1 and 4, respectively, **are** significantly different than the other laboratories. In general, the treatment of anomalous (or outlying) data can be handled either by retaining, correcting, or deleting the data. Obviously, none of these options are completely satisfactory; however, the third option (deletion) is acceptable when a physical cause can be identified to explain the behavior of the **data.** For interlaboratory studies, it is extremely helpful (and inevitably necessary) for the laboratories in question to present their own explanations for the behavior of the test results. To their credit, laboratories 1 and 4 did provide explanations for their anomalous data.

After submission of their test data, laboratory 1 reported that the surface temperatures for determinations of **specimen** ***AT*** were measured using 0.2-mm-diameter thermocouples placed directly on the surface of the specimen with adhesive tape. In contrast. the other laboratories utilized temperature sensors permanently mounted in the heating and cooling surfaces.[4] It is surmised that much of the variability observed in Figure 1 could be attnbuted to the technique of affiing thermocouples to the specimen surface An early comparison of guarded hot plates [8] noted that discrepancies could result between conductivity values obtained using temperatures from plate surfaces and those measured using surface thermocouples. These data for laboratory 1 and material 1 were considered sufficiently different from the others to warrant rejection *as* an outlying observation and were omitted in further analyses of the replicate data.

For inaterial 3, laboratory 4 reported values of λ that are 3.5% below the grand mean for inaterial 3. In the comment section of their official test report form, laboratory 4 reported that, "this inaterial had completely delaminated on arrival so that the test specimen consisted of two pieces which were always aligned in the *same* orientation with respect to each other whilst tcsting." Unfortunately, although laboratory 4 made a notable effort to test material 3, the sp:cimens received by laboratory 4 were physically different than those received by the other laboratories. Since no other laboratories reported similar capenences. this set of data for inaterial 3 was considered sufficiently different from the other specimens to warrant rejection as an outlying observation and was omitted in further analyses of the replicate data.

Laboratory-to-Laboratory Differences

Ideally, interlaboratory studies are designed to investigate within- and between-laboratory variability of the primary factors by minimizing the effects of secondary laboratory factors. Thus, the resulting variability in the test data may then be attributed to unavoidable random error; present in every experiment. In actuality, however, lab-to-lab differences reflect a confusing mixture of random and systematic errors. **As** noted above. the presence of relatively large lab-to-lab differences offer easier targets for identifying plausible physical explanations. Unfortunately, as lab-to-lab differences approach some minimum level of engineering significance, separating the random and system effects becomes difficult, if not impossible. An underutilized technique for examining lab-to-lab differences is the cause-and-etfect chart.

Figure 7 categorizes 19 secondary factors that could affect the test result of an individual laboratory. The inajor categories of variation examined in this study include: 1) procedure: 2) specimen; 3) equipment; and, 4) measurement, among others. Here, procedure refers to a particular technique utilized by a laboratory. For example, the technique utilized to determine the ΔT across the test material. Specimen refers, in this case, to the effect of **bulk** density within a matenal. Other matenal effects, although desirable, were not investigated in this study. Equipment covers the component differences noted in Table 2. and measurement covers all properties measured in-situ in

[4] Temperature sensors such as thermocouples are typically installed in grooves cut in the surfaces of the plates For laboratory 3, a platinum resistance thermometer is actually installed in the guard-gap on the perimeter of the meter plate in accordance with ASTM Practice for Guarded-Hot-Plate Design Using Circular Line-Heat Sources practice (C 1043)

the guarded-hot-plate apparatus for the determination of λ. Obviously, **this** list is not all-inclusive – the effects associated with operator and environment are not considered.

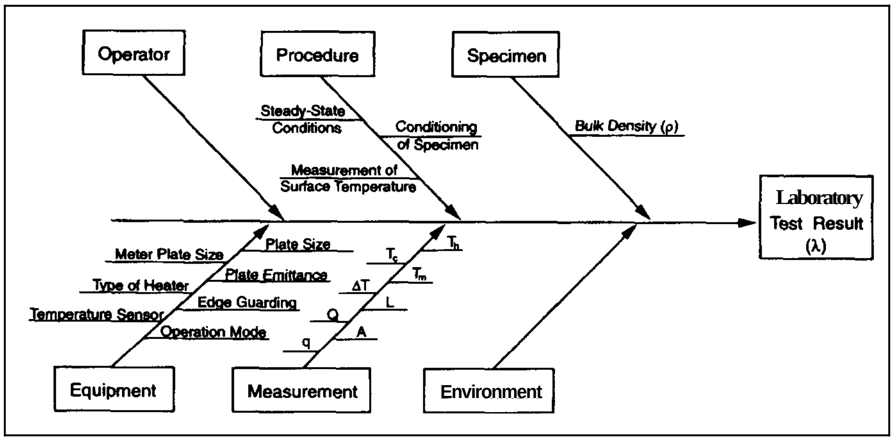

Figure *2* – ***Cause-and-effect chart for secondary factors.***

***An* analysis** of variance **(ANOVA)** for h is useful in determining whether there are factor effects on h. Specifically, values of the **ANOVA** cumulative probability near 100% are indications of factor significance. Significance, however, does not necessarily imply causation – especially given the fact that many correlations exist among the factors themselves. For example, if T_h is significant and/or T_c is significant, then **it** is not surprising that T_m and/or ΔT would also be significant.

Table **4** **summarizes** whether a factor is statistically significant. The term ***FCDF*** (F-cumulative distribution function) is the percent point of the F-distribution [9]; only ***FCDF*** values above 95% are considered significant (i.e., at the **5%** level). It is important to note that values of ***FCDF*** are based on the assumption that the variances of the treatments' are constant across treatments – **this** is decidedly ***not*** the case for many analyses. ***An*** advantage of the **ANOVA** analysis is that it is applicable to both **types** of data: quantitative (numeric) or qualitative (categorical).

From Table **4,** the single most important conclusion is **that,** for material **4,** the primary factor laboratory is ***not*** statistically significant. This is not the case for materials 1, 2, and **3** – there is statistically significant difference across the five laboratories. Further examination of Table **4** above indicates that many of the 19 (secondary) laboratory factors **are** significant. Finding the root significant factor(s) is done by using **results** from Table **4** in conjunction with engineering judgment (and possibly additional tests) by the participating laboratories.

The nearly homogeneous behavior of the laboratories for material **4** is noteworthy. One possible explanation is material composition. Material ***4*** **is** a molded-beads, expanded polystyrene board [6]; the three others **are** (essentially) fibrous glass and

[5] A treatment is a particular combination of levels of the factors involved in an experiment.

binder, having nominal densities ranging from 13 kg/m^3 to 200 kg/m^3 (Table 1). The cellular nature of polystyrene board, consisting primarily of small spheres, would have different anisotropic properties and specimen/plate contact characteristics than the fibrous materials. Another possible explanation is that the relatively thin specimen (13 mm) would have less effect on edge heat losses, if present.

Table **4** -- *Is a Factor Statistically Significant? (FCDF > 95 %? Yes/No)*

Laboratory Factors	Material 1	Material 2	Material 3	Material 4
0) Laboratow (primary)	Y	Y	Y	N
1) Steady-state conditions	Y	Y	Y	N
2) Conditioning of specimen	Incomplete	Incomplete	Incomplete	Incomplete
3) Measurement technique for surface temperatures	Y	Y	N	Y
4) Bulk density (ρ)	Y	Y	Y	N
5) Plate size	Y	Y	N	N
6) Meter plate size	Y	Y	Y	N
7) Plate emittance	Y	Y	Y	N
8) Type of heater	N	Y	N	N
9) Edge guarding	Y	N	Y	N
10) Temperature sensor	N	Y	Y	Y
11) Operation mode	N	N	Y	N
12) T_h	Y	N	Y	Y
13) T_c	Y	N	Y	Y
14) T_m	Y	N	Y	Y
15) ΔT	Y	Y	Y	Y
16) L	N	Y	Y	N
17) Q	Y	Y	Y	N
18) A	Y	Y	Y	N
19) q	Y	Y	Y	N

Laboratory Equivalence

Two sets of laboratory data (matenal 1, laboratory 1 and matenal 3 laboratory **4)** have been identified that are sufficiently different to warrant rejection as outlying observations based physical causes. Excluding these 10 observations, laboratory *relative* means and the grand *relutive* standard deviations are re-computed and summarized in Table 5.

The laboratory relative standard deviation represents the relative variation of **data** about the local laboratory mean. **A** low value represents a "tight" or quiet laboratory; correspondingly, a high value for the relative standard deviation represents a "noisy" laboratory. From Table 5, laboratory 2 is tight for all four matenals. In some cases, as noted in Table 5, the laboratory variation is high (above 1%) or marginally high (approaching 0.5%). With regards to laboratory variation, ISO 8302 specifies a reproducibility[6] limit of better than 1% for independent replicate measurements near room temperam. With the exception of one set of data (material 3, laboratory 1), the laboratory standard deviations are all less than 1% (Table 5).

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[6] **ASTM** defines this quantity as repeatability

Table 5 – ***Relative Means and Standard Deviations for Replicates (297.15 K) Excluding Outlying Data (Material 1-Lab1 and Material 3-Lab 4)***

Lab	Material 1		Material 2		Material 3		Material 4	
	Mean *(Yo)*	**SD** *(Yo)*	Mean (Yo)	SD (%)	Mean (%)	**SD** *(Yo)*	Mean (%)	**SD** (%)
1	----	----	1.4	0.16	−0.1	1.19[1]	0.39	0.89[1]
2	0.7	0.07	−0.6	0.06	0.5	0.04	−0.26	0.05
3	−0.5	0.04	−1.3	0.11	−1.1	0.47[2]	−0.09	0.13
4	1.0	0.43[2]	0.0	0.17	----	----	−0.30	0.39[2]
5	− 1.1	0.06	0.4	0.56[1]	0.8	0.11	0.27	0.04
Grand	----	0.91	----	0.95	----	0.95	----	0.49
Range	1.8	----	2.7	----	1.9	----	0.69	----
Half-Range	± 0.9	----	± 1.4	----	± 1.0	----	± 0.35	----

[1]High; [2]Marginally high

The laboratory relative mean represents the relative differences of the laboratory mean from consensus values (i.e., the grand mean) for each material. As observed earlier in Figure 1, the differences for many of the laboratories in Table 5 change sign from material to material. It is important to note that the laboratory relative means represent relative, differences currently utilized in key comparisons as part of the international Mutual Recognition Agreement [*10*]. From Table 5, the ranges of laboratory mans for materials 1, 2, 3, and 4 are 1.8%, 2.7%, 1.9%, and 0.69%, respectively. The corresponding half-ranges (last row of Table 5) for materials 1, 2, 3, and 4 are ± 0.9%, ± 1.4%, ± 1.0%, and ± 0.35%, respectively.

Arc the relative differences among laboratories at 297.15 K significant? The answer depends on the uncertainty metric considered, and there are several metrics that can be used for comparison, including:

1) An international comparison of a large population (nearly 50) of international guarded-hot-plate laboratories from Africa, Asia, Australia, Europe and North America [*11*];
2) C 177 imprecision statements;
3) ISO 8302 uncertainty statements;
4) NIST **SRMs** 1451 and 1453 uncertainty limits;
5) The minimum difference (A) accepted as significant from an engineering perspective;
6) Individual laboratory expanded uncertainties as reported in Table 2; and,
7) Laboratory statistical significance, **ANOVA,** 95% as reported in Table 4.

The first metric is from a *study* that was intended to determine the worldwide state-ofthe-art in guarded hot plate measurements prior to the development of ISO standards [*11*]. Participants measured the thermal conductivity of fibrous glass board at mean temperatures of 283 K, 297 K, and a third temperature within the range from 273 K to 313 K. The results indicated that the relative standard deviation of the data from the fitted curve is 2.4%, although several data points deviated from the curve by more than 5% and some by more than 10% [*11*]. The metrics **for 2)** to 4) are well known and summarized in Table 6.

The participants have agreed to accept 1.5% as the minimum engineering significance difference (Δ) for the above comparison of national standards laboratories. In other words, for national standards laboratories, any difference less than 1.5% from the consensus mean is considered insignificant from an engineering perspective.

Table 6 summarizes the responses (yes or no) by matenal for the seven different uncertainty inetrics and their corresponding estimate (in parentheses) at the two standard-deviation level. Note that only for inatenal 4 are the laboratories considered equivalent for all the uncertainty metrics. For the other materials. however. the laboratories are considered equivalent with respect to the minimum engineering ditt'erence of 1.5% (as well as the first four uncertainty metrics). For the individual labontory expanded uncertainty (at k 2) metric. the laboratones are not equivalent for inaterials 1, 2, and 3. Particular combinations of laboratones. however, are equivalent as shown in Table *6* and these combinations change for materials 1. 2, and 3.

Table 6 *Are the Laboratories Equivalent at 297.* **IS** *K? (Yes/No)*

Uncertainty Metrics (2 x Standard Deviation)	Material 1 (± 0.9%)	Material 2 (± 1.4%)	Material 3 (± 1.0%)	Material 4 (± *0.35%"*)
1) International GHP Study (4.8%) [*11*]	Y	Y	Y	Y
2) ASTM C 177 (2% to 5%)	Y	Y	Y	Y
3) ISO 8302 (2% to 5%)	Y	Y	Y	Y
4) SRMs 1451 (3%) and 1453 (1.3%)	Y	---	---	Y
5) Minimum Engineering Significance Δ (1.5%)	Y	Y	Y	Y
6) Laboratory Uncertainty (1.0% to 1.5%)	N:(2,4)(3,5)	N:(2,3)(4,5)	N:(1,2,5)(3)	Y
7) Statistical Significance **(ANOVA,** 95%)	N	N	N	Y

Multi-Temperature Data (280 K to 320 K)

for the multi-temperature data, there are three pninary factors: laboratory (*5* levels), reference inaterial (4 levels), and temperature (5 levels). Although the single data-point at each temperature precludes a rigorous statistical analysis, the analyses are driven by the same central theme considered for the fixed-temperature replicate data: How do the laboratories behave across the four materials'! In particular, what are the location and vanation estimates for each inaterial? Examination of these questions is provided by a linear regression analysis of the multi-temperature data using the following model:

$$\hat{\lambda} = b_0 + b_1 T_m \tag{3}$$

where $\hat{\lambda}$ is the predicted value for Eq 3 based on least-squares estimates for b_0 and b_1.

Figure 3 plots the relative deviations from the fitted curve for each data pomt. **As** observed with the replicate data, the principal conclusion from Figure 3 is that the behavior of the laboratones does, in fact, change from material to material. For the four plots, the location and variation of each set of laboratory data changes from material to material. Further examination of the slopes reveals that there is a change in slope for several laboratories (most notably for laboratories 1, 2, and *5). A* final conclusion of Figure 3 is that the relative deviations among the laboratories **are** affected substantially **as** the mean temperature decreases from room-temperature conditions. This conclusion is less evident if data from laboratory 1 **are** omitted,

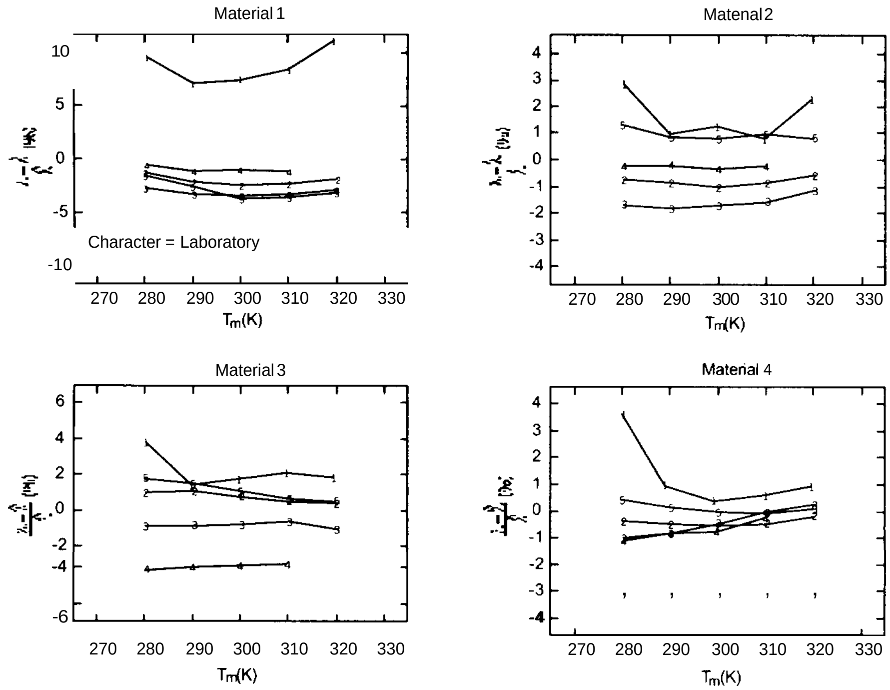

Figure 3 – ***Multi-plot of relative deviations versus mean temperature.***

Conclusions

This international comparison investigated the variability in thermal conductivity results among guarded hot plate laboratories in Canada, France, Japan, the United Kingdom, and the United States using four regional/national reference materials. The reference materials were SRM 1451 (fibrous-glass blanket), IRMM-440 (resin-bonded **glass** fibre board), JTCCM "candidate" mineral-oxide fiberboard, and SRM 1453 (expanded polystyrene board). The collaboration assessed the effects of two primary factors – laboratory and material – for five replicate measurements at 297.15 K (24 °C), and included a **third primary** factor – temperature – for single-point measurements at 280 K, 290 K, 300 K, **3** 10 K, and 320 K.

The thermal conductivity test **data** (Figures 1 and 3) indicate that there is a laboratory-to-laboratory difference for each of the materials, except SRM 1453. **As** expected, there is a material-to-material difference – material 1 (SRM 1451) was the highest thermal conductivity; material 2 (IRMM-440) was the lowest. **This** material-to-material difference was greater than the laboratory-to-laboratory difference. Ranking the matenals by variability (all data included) yields the following order (lowest to highest): material 4 (SRM 1453), material 2 (IRMM-440); material 3 (JTCCM "candidate"); and, material 1 (SRM 1451). The results of the multi-temperature (280 K to 320 K) data were consistent with the results observed for the fixed-temperature (297.I5 K) replicate data. In addition, the results indicated that disagreement among the laboratories tended to increase as mean temperatures decreases from 297.I5 K.

Two of the replicate data sets (at 297.15 K) were identified as anomalous and later excluded after the laboratories in question identified physical causes for the behavior of their data. After exclusion of the anomalous data, the half ranges for matenals 1, 2, 3, and 4 were ± 0.9%, ± 1.4%, ± 1.0%, and ± 0.35%, respectively. These laboratory-to-laboratory differences are considered small by many different uncertainty metrics. including ISO 8302 uncertainty statements, C 177 precision indices. and NIST SRM uncertainty statements, among others. For this comparison, the laboratory participants have accepted a minimum engineering significance difference of 1.5% from the consensus mean for national standards laboratories. In other words, laboratory differences less than 1.5% from the consensus mean arc currently considered insignificant based on an engineering perspective.

One of the most plausible factors affecting the test data was procedural in nature. In particular, a significant difference in average value and vanation was experienced by one laboratory that affixed temperature sensors directly to the specimen surface rather than using permanent sensors affixed to the apparatus plates. The approach of adhering fine-diameter temperature sensors to the specimen surface appears to have contributed to measurement differences and may be an *unintended* extension of the test procedures specified in ISO 8302 and C 177. Further measurements comparing different techniques for determining the temperature difference across a test specimen would be extremely useful. With regard to ISO 8302 and C 177, the appropriate sections on determination of the temperature difference should be reexamined for clarity and revised if necessary.

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