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Analysis of Uncertainties in Calibration of a Heat Flow Meter Apparatus

by M. Bomberg, C.M. Pelanne and W.S. Newton

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RÉSUMÉ

Cette communication analyse les principales sources d'incertitude dans les mesures en laboratoire de la résistance thermique à l'aide d'un fluxmètre thermique et ce, pour une vaste gamme de conditions d'essai. Elle traite en termes généraux des erreurs d'étalonnage, indique les techniques pour assurer la précision de l'instrument, analyse la sensibilité des résultats à certains changements dans les conditions d'essai et décrit deux méthodes permettant d'établir des normes de correspondance pour la comparaison des mesures absolues et relatives.

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ANALYSIS OF UNCERTAINTIES IN CALIBRATION
OF A HEAT FLOW METER APPARATUS

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ABSTRACT

This paper reviews the main sources of uncertainty in laboratory measurement of thermal resistance with a heat flow meter apparatus over a wide range of testing conditions. It deals with calibration uncertainties in general terms, indicates techniques for controlling instrument precision, analyses the sensitivity of results to some changes in testing conditions, and describes two procedures for establishing transfer standards that permit comparison of absolute and relative measurements.

INTRODUCTION

Errors in measurements with the heat flow meter (HFM) apparatus arise out of uncertainties in electrical and thickness measurements, as well as out of deviations from one-dimensional heat flow and uncertainty in the HFM calibration factor. Bomberg and Solvason [1] have compared various calibration techniques and concluded that the HFM apparatus calibration using transfer standards is the preferred technique. The use of transfer standards (or calibrated specimens) requires, however, that guarded hot plate (GHP) tests be performed on a variety of materials. If the HFM apparatus is to be used for quality control in manufacturing of thermal insulation, the transfer standard should be made of the same material [2], and this may introduce a significant increase in the uncertainties of both HFM and GHP test results.

Errors associated with GHP equipment have already been discussed [3], but the effect of the characteristics of the material under test have not. Precision of the GHP apparatus was shown to be strongly dependent on uniformity of heat flow across the gap between the metering and guard ring areas; and only homogeneous, uniform, "ideal" specimens could be used for experimental determination of the uncertainties in GHP testing.

Ideally, calibration of the heat flow sensor (HFS) should not vary with specimen thickness. Thin, uniform specimens could then be used to utilize, as the calibration base, the high level of precision and accuracy possible with the GHP apparatus. Changes in the design of the heat flow meter apparatus to reduce the effects of lateral heat flow on the calibration of the heat flow transducer have been described [1]. The new design makes the calibration coefficient less dependent on the characteristics of the material being tested and produces the same calibration curve for both thick, low-density specimens and thin, high-density ones. The range of conditions under which materials may be tested accurately is thus enlarged. These aspects will be discussed in the present paper and in another [4] that describes the verification of heat flow meters used for quality control in the production of low-density mineral fiber insulation. Both papers are based on a cooperative study carried out by the Division of Building Research, National Research Council of Canada (NRCC), the Research and Development Division of Manville Corporation, USA, and the Quality Control Group in the Innisfail Plant of Manville Canada Inc.

This paper reviews a selection of materials and test conditions used for calibration and certain aspects of HFM apparatus performance (i.e., measurements of temperature and specimen thickness), and discusses three techniques suitable for examining uncertainties due to testing procedure. All utilize air layers, layered low-density glass fiber specimens, and adjustment of ambient temperature to study the lateral heat flow effect. The importance of material structure in the development of a transfer standard is stressed.

Test Conditions for Calibration of HFM Apparatus

As the precision and accuracy of an HFM test depends on the construction of the apparatus as well as the testing procedure, both factors must be considered in selecting a range of test conditions. At NRCC a 600- x 600-mm HFM apparatus is used to test low-density thermal insulations up to 150 mm thick (or up to 200 mm with somewhat lower test precision). The working range is between 6 and 180 mm, corresponding to a thermal resistance range of 0.2 to 7 m²K/W.

Material acceptance standards usually require thermal testing at a mean temperature of $24 \pm 1^\circ\text{C}$, with a temperature difference of 22 deg (Canada), or 27 deg (USA). To establish the dependence of thermal resistance on mean temperature, tests are normally conducted over a range of mean temperatures from 0 to 50 deg C.

Transfer Standard and Reference Materials

A change in testing conditions, even one so small that it may not be observed by the operator, may produce significant deviations in measured results, depending on the nature of the tested material. Although quality control can select a reference specimen of the same material as that being controlled, an independent testing laboratory must have several reference specimens. At NRCC the following materials are used to establish transfer standards because of their uniformity, time stability, and handling qualities: expanded polystyrene (preferably high density) or, alternatively, air-filled extruded polystyrene; medium or high-density glass fiber, e.g., standard reference materials (SRM) 1450 from the National Bureau of Standards [5]. In addition, NRCC has a set of layered, low-density glass fiber transfer standards to verify testing of materials with a significant fraction of radiative heat transport.

Apparatus Construction

The accuracy of an HFM depends on several factors related to the construction of the apparatus: for example, flatness and parallelism of the hot and cold plates (these factors are described in ASTM C518 [2]).

Temperature Measurement

The following characteristics related to temperature measurement must be checked:

- (1) temperature uniformity - uniformity or differences in temperatures measured over the surface of the plates,
- (2) temperature stability with time, and
- (3) repeatability of temperature measurements.

Temperature uniformity and stability can be determined by adjusting both HFM plates to the same temperature and placing them in contact with each other. The standard deviation for each thermocouple recording will indicate stability, while the range between minimum and maximum measured surface temperatures will indicate temperature uniformity.

The precision of measurement of a temperature difference depends on the temperature difference used in the test. For

example, 0.1 C deg means an uncertainty of 1% for a 10-deg temperature difference but an uncertainty of only 0.5% for the usual 22- to 27-deg difference. A good HFM apparatus has, for example, temperature uniformity better than $\pm 0.15^{\circ}\text{C}$, temperature stability better than $\pm 0.02^{\circ}\text{C}$, and repeatability precision (with readings on DVM) of 1.0%.

Thickness Measurement

If the HFM apparatus is provided with a thickness indicator, the zero position and linearity of the thickness gauge should be checked as well as the parallelism of the two plates enclosing the specimen [4]. At NRCC, specimen thickness is calculated as the average of the distance between plates measured on each side of the apparatus. This procedure includes a check of parallelism as well as an averaging of a number of measurements.

Additional consideration is given to all high precision measurements (such as calibration), namely, comparison of specimen thickness measured inside and outside the HFM apparatus. The two thicknesses are permitted to differ by as much as $\pm 0.2\%$. If the thickness measured in the apparatus differs by more than this, the test is excluded from the calibration series; if the difference is less than 0.1%, no correction is made; if the difference is between 0.1 and 0.2%, an appropriate correction is applied.

UNCERTAINTIES RELATING TO TESTING PROCEDURE

A laboratory routine for testing must be established, including methods of preparing specimens (drying or conditioning): measure density and thickness of specimen outside and inside the apparatus, estimate time to reach the steady-state condition; record, calculate and report temperatures and heat flux. Then select a material known for uniformity and time stability to establish "a standard repeatability precision," i.e. repeatability obtained in standard testing conditions on the reference material. This basic measure of repeatability precision (two standard deviations on minimum nine test results) may then be compared to the repeatability precision obtained for different materials or changing test conditions, for example, when testing in the range of mean temperature. These concepts may be illustrated by the following: standard repeatability precision related to 90% probability level, i.e., two standard deviations, is approximately 0.2% for high-density glass fiber specimens 344-6 (Table 1) on the 600-mm GHP and for specimens 290-48 on the 450-mm GHP.

Repeatability precision determined on low-density glass fiber (LDGF) specimens, code 354-79, is in the same range as that for the HDGF specimens; for LDGF specimens, code 354-68, repeatability

Table 1. Repeatability Precision of 450- and 600-mm GHP on 26-mm thick Glass Fiber Specimens

Specimen		Equipment Size, mm	λ Mean Value* W/m K	Standard Deviation %
Code	Density kg/m ³			
344 - 6	91 $\pm$ 2	600	0.03174	0.10
290 - 48	80 $\pm$ 1	450	0.03136	0.10
354 - 79	8.7 $\pm$ 0.1	450	0.04817	0.09
354 - 68	10.6 $\pm$ 0.1	450	0.04416	0.26

* Last digit shown for calculation purposes only

precision is almost three times poorer. It appears that repeatability precision of the GHP apparatus depends on the specimens selected for testing. From tests on the 600- and 450-mm GHP's shown in Table 1 repeatability precision of the instruments on a 95% probability level appears to be 0.2%. The lower precision apparent in some of the tests must be attributed to the effects of specimen preparation and conditioning, and perhaps to some other material variable.

Effect of Lateral Heat Flow

In testing to determine the significance of lateral heat flow, the same temperature is maintained on both plates and ambient air temperature is constant but much lower. Heat loss from the edge of the specimen can be assumed symmetrical, half from the "hot" plate and half from the "cold" plate. Output from the heat flow transducer, corrected for any small temperature difference across the specimen, will give the edge loss associated with the temperature difference between the two plates and ambient air.

As an example, Table 2 shows the results of a series of lateral heat flow tests conducted at NRCC on a particular apparatus* using 95- and 190-mm thick polystyrene specimens. It is evident that the temperature difference between the temperature-controlled enclosure surrounding the specimen (the sleeve) and the HFM plates causes a measurable output from the transducer (error). It is also evident that the use of edge insulation reduces this error.

* Manufactured by Foundation Electronic Instruments, Ottawa, Canada.

Table 2. Lateral Heat Flow

EPS Spec. Thickness, mm	25-mm Edge Insulation	Temperature, degree C			Output, mV	
		Sleeve	Spec. Edge	Plates*	Meas.	Corrected for ΔT of Plates
190.35	No	-	29.9	36.0	34	37
190.52	Yes	26.9	30.1	36.1	21	16
95.12	Yes	28.0	32.9	36.5	-14	-11

* Both plates maintained at the same temperature

If the lateral heat flow component is less than 0.5% of the average output of the heat flow meter (i.e., 3-5 μV in Table 2), edge insulation should not be required when testing thin specimens. A difference of up to 2 C deg between sleeve temperature and mean specimen temperature may be accepted if the specimen thickness does not exceed 40 mm. Specimens thicker than 60 mm should, however, be tested with edge insulation. For those thicker than 90 mm, the difference between sleeve and mean specimen temperatures should be limited to 1 C deg.

Use of Air Layers

Air layers can be used as test specimens to verify the accuracy of the HFM apparatus when convection of air is eliminated and the thermal conductivity of still air is either known or determined from test results for at least two levels of plate emittance and two or three air layer thicknesses. The latter approach was proposed by Klarsfeld [6] as an extrapolative procedure in which thermal conductivity of still air (λ_g) can be determined. This procedure consists of extrapolation to zero thickness for the following:

- (1) apparent thermal conductivity* of an air layer at a given mean air temperature for each value of boundary emittance,
- (2) apparent thermal conductivity* of an air layer and known boundary emittance for a given value of mean air temperature.

Figure 1 shows NRCC data for apparent thermal conductivities of three air layers tested with and without aluminum foils (on both

* For brevity, "apparent" is dropped in subsequent discussions. Thermal conductivity of an air layer or of low-density thermal insulations means, however, apparent thermal conductivity.

Fig. 1. Thermal conductivities of an air layer, with and without aluminum foil, extrapolated to determine still-air conductivity. Measured in 600-mm GHP apparatus

sides of the air layer), at the same mean temperature, and with a temperature difference of 28 C deg across the air gap [7]. Extrapolating the data to zero thickness, one obtains a still-air conductivity of 0.0247 W/m K at a mean temperature of 24°C. It is somewhat lower than the 0.0254 W/m K shown for air at 297 K by Raznjevic [8] or the 0.0252 W/m K quoted in the Smithsonian Physical Tables [9]. The level still-air conductivity can then be used to calculate plate emittance using techniques described by Hager [10] and Pelanne [11].

In addition to the data shown in Fig. 1, eight other tests on a 10-mm air gap over a mean temperature range of 0 to 45°C yielded the following equation:

$$\lambda_a = 0.0652 + 0.0061 T_m$$

Data generated from it can be compared with values in Refs. 8 and 9, making this method useful for verifying the temperature coefficient of the calibration curve.

Use of Layered, Low-density, Glass Fiber Specimens

A technique used at NRCC to fabricate low-density transfer standards ranging in thickness from 50 to 250 mm is slightly different from that described in Ref. 4. Using a light table [12], thirty 12-mm thick, low-density glass fiber specimens were selected as most uniform among one hundred and thirty. Their densities varied between 8.2 and 10.0 kg/m³. To reduce the number of tests to be performed, pairs of specimens, each separated by a paper septum to intercept radiative heat transfer, were tested at one time. The results of the thermal conductivity tests are shown in Fig. 2. Some of the specimens were recombined in new pairs and nine pairs were finally selected. The results of thermal conductivity tests on a glass fiber stack, with and without paper septa, are shown in Fig. 3.

Layered specimens are particularly useful in examining the effect of specimen thickness [13,14] on apparent thermal conductivity measured for a given set of test conditions. The instrument error has been estimated (another approximative model has also been presented [15]) by assuming that all test results with radiation-absorbing barriers (paper septa) placed 25 mm apart should be equal to the results obtained on 25-mm thick specimens alone [4]. Although such an approximation appears to be acceptable for mineral fiber insulations, it has not been proved for low-density cellular plastics.

Fig. 2. Thermal conductivities of two specimens separated by paper septum. Result of preliminary series used for specimen selection

Fig. 3. Thermal conductivity of glass fiber stack with and without paper septa. Eighteen layers, each 12.5 mm, were tested

Fig. 4. Thermal conductivity of polystyrene stack with and without paper septa. Twelve layers, each 12.5 mm, were tested

For comparison, Fig. 4 shows the results of two series of tests on the same batch of expanded polystyrene, density 15-16 kg/m³, with and without paper septa. The curve of thermal conductivity versus specimen thickness shows a different character from that of Fig. 3, implying a somewhat different mechanism of radiative heat transport. Layered low-density glass fiber insulation appears, therefore, to be more suitable for verification of the HFM apparatus [16,17] than low-density expanded polystyrene insulation, although radiative heat transport is a significant fraction of the total heat transfer for both types of insulation.

Importance of Material Structure

The foregoing discussion of repeatability (reproducibility) precision illustrates the importance of test specimens of uniform material structure. Some low-density glass fiber specimens (e.g., 354-79, Table 1) were shown to have the same precision of repeatability as the high-density glass fiber standard reference material (SRM) produced by the National Bureau of Standards [5], but much lower precision was achieved with other specimens. Use of thick specimens of low-density thermal insulation for calibration purposes usually led to lower precision than could be achieved with high-density, standard reference material [18]. As the precision of thermal conductivity determination is inversely proportional to specimen thickness, an alternative approach would be to fabricate thick specimens from thin layers already tested separately. This can only be done, however, if the material has a high degree of structural uniformity.

Specimens of almost identical density and thermal conductivity were selected at the Innisfail plant [4]. Specimens with thermal conductivity very close to that calculated as a function of density from the model [15] were used for the NRCC tests shown in Fig. 3. In both cases, however, the specimens were chosen with a light table to ensure structure uniformity.

Figure 5 illustrates the importance of structure uniformity on precision, showing the range of measured thermal conductivities as a function of specimen thickness for two sets of specimens of different uniformity:

Set 1 contains six 25-mm thick low-density glass fiber specimens of good uniformity;

Set 2 contains six 25-mm thick specimens of poor uniformity selected as the extremes from the same sample as Set 1.

The variability in thermal conductivity was about 1% for a density variation of 9% in Set 1; a range of 5% in thermal conductivity was observed for a 15% range in density in Set 2. Figure 5 also shows

Fig. 5. Range of thermal conductivities as a function of specimen thickness for low-density glass fiber
 Set 1 - six 25-mm thick, uniform specimens
 Set 2 - six 25-mm thick, non-uniform specimens

that a different curve of thermal conductivity as a function of thickness is obtained for each of the sets, although a large and statistically significant number of tests were performed.

One must remember that properties of a material vary in all three directions and that the manner in which specimens are selected for comparative tests may have a decisive effect on the studied dependence. In previous work [4] specimens with the same thermal conductivity and density but different thickness were selected. The relation between thermal conductivity and density of specimens was also maintained in the final selection of the matched pairs in the present research (Fig. 2). It was not, however, observed in selecting Sets 1 and 2 of Fig. 5.

These results are not surprising at all if one also remembers that for a given fiber diameter the relation between thermal

conductivity and glass fiber specimen density* describes material structure [15]. Comparison of Figs. 3 and 5 shows that in studying functional dependence one must use specimens with identical structure.

CONCLUSIONS

Calibration of a heat flow meter apparatus used for quality control of manufactured products can be extended to a wide range of testing conditions. Air layers and layered, low-density glass fiber transfer standards have been very useful in calibrating and verifying HFM apparatus precision and accuracy.

Lateral heat flow tests performed at the extreme thickness to be tested and determination of the mean temperature dependence of the calibration coefficient are also helpful in establishing the range of uncertainties caused by changes in test conditions.

This and previous NRCC studies [1,3,4] suggest that with proper calibration and careful verification of its errors, the HFM apparatus can replace the GHP apparatus in many industrial and commercial testing programs.

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* Only if a material is uniform will the specimen density be identical to that of the metering section.

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