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Newton, W. S.; Pelanne, C. M.; Bomberg, M. T.

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Calibration of Heat Flow Meter Apparatus Used for Quality Control of Low-Density Mineral Fiber Insulations

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

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RÉSUMÉ

Ce document fournit aux laboratoires industriels des directives pour vérifier la précision des mesures de résistance thermique. Il traite du matériel et des méthodes d'essai, ainsi que de l'élaboration d'un programme d'étalonnage du fluxmètre thermique utilisé à l'usine de la firme Manville Canada Inc., à Innisfail, pour le contrôle de la qualité des isolants en fibres minérales de faible densité. Afin d'assurer la concordance des résultats obtenus avec le fluxmètre thermique utilisé à l'usine et l'appareil à plaque chauffante employé au Conseil national de recherches du Canada, on a incorporé les éléments suivants au programme d'étalonnage : sélection, fabrication et vérification d'échantillons de référence de fibre de verre à faible densité, étalonnage de l'appareil, analyse des erreurs de mesure de l'appareil et maintien de l'étalonnage.

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CALIBRATION OF HEAT FLOW METER APPARATUS USED FOR
QUALITY CONTROL OF LOW-DENSITY MINERAL FIBER INSULATIONS

Wendy S. Newton - Manville Canada Inc., Innisfail, Alta.
C.M. Pelanne - Manville Corp. of U.S., Denver, Colorado
M. Bomberg - National Research Council of Canada
Ottawa, Ontario

ABSTRACT

This paper provides industrial laboratories with guidelines for verifying precision in thermal resistance determinations. It deals with both equipment and test procedures, and discusses the development of a calibration program for the heat flow meter (HFM) apparatus used in quality control of low-density mineral fiber insulations manufactured at the Innisfail Plant of Manville Canada Inc. To ensure agreement between the HFM apparatus used in the plant and the guarded hot plate (GHP) apparatus used by National Research Council of Canada, the following elements were incorporated in the calibration program: selection, fabrication and verification of low-density glass fiber reference systems, calibration of apparatus, error analysis of apparatus, maintenance of calibration.

INTRODUCTION

A properly calibrated heat flow meter (HFM) apparatus provides fast, accurate measurement of thermal resistance and is therefore useful for industrial quality control. The Manville Corporation has installed an HFM apparatus* in each of its insulation manufacturing plants to monitor the thermal properties of its products, thereby ensuring that the product conforms to the required standards [1]. This paper discusses a cooperative program undertaken by Manville Canada Inc. (MCI), Innisfail, Alberta, the Research and Development Center (R/D) of Manville Corporation, USA,

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* R-matic, manufactured by Dynatech R/D Company, Cambridge, Mass.

and the National Research Council of Canada (NRCC) to establish a calibration procedure and develop reference specimens for use by MCI. The calibration program contained the following elements:

1. Development of reference specimens
  - preliminary selection of candidate specimens by means of light transmission measurements,
  - determination of thermal characteristics on HFM apparatus,
  - selection of reference specimens,
  - tests of reference specimens in GHP apparatus at NRCC,
  - final tests on HFM apparatus.
2. Equipment calibration
  - calibration of the two HFM apparatuses at MCI,
  - evaluation of the characteristics of the equipment,
  - development of secondary reference specimens,
  - development of procedures to ensure consistent performance of equipment.

Prior to undertaking the calibration program the mechanical performance of each HFM apparatus was verified. Parallelism of the two surface plates was checked at various specimen thicknesses as well as the thickness measurement technique. The paper deals with the specific issue of thermal resistance measurements in an industrial quality control laboratory. The more general problems associated with the HFM apparatus are discussed elsewhere [2].

PRIMARY REFERENCE SPECIMENS

Selection of uniform specimens and closely matched pairs of specimens is imperative for success in establishing reference specimens [3]. The following criteria were defined:

1. Density and thermal resistance of pairs of specimens must match.
2. Structure and density of the specimen must match that of the material being manufactured.
3. Specimen density distribution must be uniform and free of any visible defects.

Prior to choosing the final reference specimens, a preliminary selection was made. A number of 610- x 610-mm pieces were cut from a batt of glass fiber insulation; the upper and lower surfaces were removed, using a horizontal bandsaw; the specimens were trimmed to a thickness slightly greater (by as much as 6 mm) than the thickness intended for testing; the pieces were pre-screened over a light box containing a series of fluorescent lights covered by white opaque plexiglas. (The box provides a very intense light over which the specimens can be observed for uniform density, with

particular attention to the central (metering) area.) Only then are density and thermal resistance determined for specimens having acceptable uniformity.

The reference specimens were selected on the basis of best match density and thermal conductivity. Values of apparent thermal conductivity and density (Fig. 1) were used to select the reference specimens. Two 25-mm thick specimens and four 76-mm thick specimens were selected. The 76-mm specimens were paired to form two 152-mm thick specimens. Six additional specimens 25 mm thick were selected for evaluating experimental errors introduced by thick, low-density specimens.

In any comparative study of equipment having different metering areas (as is the case with both the HFM and GHP), the details of specimen preparation should be decided prior to testing in order to avoid the necessity for adjustments during the test. At NRCC the specimens are placed in 25-mm wide extruded polystyrene frames; prior to testing at Innifail, therefore, the specimens should have been cut to 560 × 560 mm and placed in frames supplied by NRCC. As this was not done originally, testing at Innisfail before the specimens were placed in frames had to be disregarded.

Table 1a shows average thermal conductivity for two test series: an initial test on 610- × 610-mm specimens; one in which specimens were placed in a smaller frame by cutting 25 mm from one side, thereby displacing the metering area. There was a small difference in the thermal conductivities determined by the two

Fig. 1. Apparent thermal conductivity determined on HFM apparatus and density of the pre-selected specimens

series. To check whether this was due to specimen preparation or to malfunction of the apparatus, four other specimens originally tested at the same time were retested. The results shown in Table 1b indicate good performance of the HFM apparatus. The difference shown in Table 1a is probably associated with the shift of metering area, and the initial tests were therefore disregarded. Each of the specimens was retested three more times and the results on specimens placed in 25-mm deep frames are given in Table 2.

Table 1a. Apparent thermal conductivity* determined on HFM 1 at Manville Canada Inc. Averages for two series:
(1) initial, (2) after return from NRCC where the metering area was shifted by 25 mm in one direction

Specimen Code	Nominal Thickness mm	Initial 4 Tests λ , W/m K	After Return 3 Tests λ , W/m K	% Difference
106A	25	0.04540	0.04559	0.4
106B	25	0.04555	0.04506	1.1
305A	76	0.04800	0.04822	0.5
305B	76	0.04800	0.04793	0.1
306A	76	0.04800	0.04829	0.6
306B	76	0.04806	0.04828	0.4
305A&B	152	0.04987	0.04998	0.2
306A&B	152	0.05005	0.04991	0.3
				AVG = 0.5%

Table 1b. Repeatability of apparent thermal conductivity testing at Manville Canada Inc. during the same time without shift in metering area

Specimen Code	Nominal Thickness mm	Series (1) 4 Tests λ , W/m K	Series (2) 3 Tests λ , W/m K	% Difference
107A	25	0.04511	0.04519	0.2
107B	25	0.04514	0.04511	0.1
3½B2	89	0.04918	0.04925	0.2
6B2	152	0.05377	0.05384	0.1
				AVG = 0.1%

* Reported with four significant digits for the sake of error reduction in percentage calculations.

Table 2. Apparent thermal conductivity (λ) before and after calibration of HFM 1 at Manville Canada Inc.

Specimen Code	Average of 6 HFM Tests λ , W/m K	St. Deviation of λ on HFM, %	GHP at NRCC W/m K	λ on Recalibrated HFM
106A	0.04564	0.14	0.04611	0.04638
106B	0.04513	0.14		0.04585
305A	0.04823	0.20	0.04787	0.04799
305B	0.04800	0.27		0.04776
306A	0.04832	0.16	0.04790	0.04793
306B	0.04823	0.15		0.04786
305A,B	0.04996	0.32	0.04934	0.04939
306A,B	0.04986	0.47		0.04929

CALIBRATION OF HEAT FLOW METER APPARATUS

All the tests were performed according to ASTM C518 "Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter." The apparatus was operated at a mean temperature of 24°C (75°F), with the hot surface plate at 37.8 ±0.6°C (100 ±1°F) and the cold surface plate at 10.1 ±6°C (50 ±1°F). Room temperature was maintained between 22°C and 26°C, and relative humidity of the ambient air was below 40%. No condensation on the cold plate was expected under such conditions.

After the thermal resistance values had been determined on GHP for each calibrated specimen, the calibration coefficient was calculated from the following equation:

$$C = \frac{T_h - T_c}{e} \frac{1}{R}$$

where C = heat flow meter calibration coefficient for a given temperature and specimen thickness, in units of heat flux per millivolt
 T_h, T_c = hot and cold surface temperatures, in degrees
e = output of heat flow meter, in millivolts
R = thermal resistance measured on the GHP apparatus at the same thickness and mean temperature, with a known confidence interval.

Fig. 2. Calibration coefficient (British units) versus specimen thickness. Note that calibration curves for the two heat flow meters are different, indicating need for calibration covering the full range of thicknesses for which equipment is to be used

Calculation of the calibration coefficient for HFM apparatus No. 1 was based on the test values obtained during six tests (Table 2). For HFM No. 2, each specimen was tested at least twice. The average calibration coefficients calculated for each apparatus and thickness are shown on Fig. 2. Calibration coefficients are relatively constant between 25 and 75 mm and increase for thicknesses greater than 75 mm. This thickness effect appears to be significant, particularly for HFM No. 2. Table 2 compares the average values of the six HFM tests with the results obtained in the GHP apparatus at NRCC on matched pairs of specimens.

For the comparison, values of the apparent thermal conductivity, λ_a , are used. They are calculated from thermal resistance R , using the following equation:

$$\lambda_a = \frac{L}{R}$$

where L = thickness of specimen.

Either a single or a variable calibration coefficient may be used for a specimen thickness range of 25 to 125 mm. Table 3 shows percentage differences for apparent thermal conductivity values

determined with either a variable or single coefficient. The results suggest that a calibration coefficient varying with specimen thickness is preferred.

EVALUATION OF MEASUREMENT ERRORS

Radiation-absorbing membranes or septa were used to assess HFM apparatus measurement errors [4]. Radiation-absorbing barriers or septa (Kraft paper 0.06-0.08 kg/m²) with an emittance similar to that of the plates of the HFM apparatus ($\epsilon = 0.95$) were placed between 25-mm thick specimens of similar density and thermal conductivity to make up a specimen 152 mm thick. In effect, the septa absorb and re-emit radiant energy, thereby simulating a series of 25-mm thick specimens placed in the HFM apparatus. By comparing the results for single 25-mm thick specimens with those for the combined thicknesses of septa (Table 4), the apparatus error can be approximated.

One assumes that all test results with septa 25 mm apart should be equal to those obtained for a 25-mm thick specimen. Although this may not be strictly correct, it is applicable to low-density glass fiber products [5]. The difference between thermal conductivity measured for the insulation stack with septa and thermal conductivity measured for 25-mm thick specimen may be called "apparatus error." "Effect of thickness" of the material is obtained from test results without septa, corrected for instrument error.

It is the purpose of this section to show the error associated with each apparatus, and it is reasonable to assume that each has a systematic bias that increases with increase in specimen thickness. Figure 3 shows the variation in error with test thickness. The

Table 3. Estimate of error in thermal conductivity with four calibration coefficients

Specimen			Error		
Code	Thickness mm	Apparatus Number	Variable Coeff.	Coeff. Average	Coeff. Median
106A	25	1	0	0.3	0.4
		2	0	0.6	0.8
305A	76	1	-0.3	0.4	0.6
		2	0.1	0.8	1.1
305A,B	152	1	0	-0.9	-0.8
		2	0.1	-1.3	-1.1

Fig. 3. Apparatus error and effect of thickness versus specimen thickness for both apparatuses. The differences are within the expected uncertainty limits of the HFM apparatus

Table 4. Apparent thermal conductivity, λ , of low-density glass fiber specimens tested with and without paper septa, HFM 1 and HFM 2

Specimen Code mm	From 25 mm Testing λ , W/m K	Apparatus 1				Apparatus 2			
		λ Measured, W/m K		Error %	EOT** %	λ Measured, W/m K		Error %	EOT** %
		Septa	No Septa			Septa	No Septa		
50 AB* CD EF	0.0430	0.0430	0.0439	0.20	2.12	0.0428	0.0436	0.57	1.82
	0.0430	0.0430	0.0438	0.13	1.81	0.0429	0.0437	0.24	1.95
	0.0428	0.0427	0.0436	0.27	2.02	0.0432	0.0434	0.47	1.92
				0.20	1.98			0.43	1.90
76 ABC DEF	0.0431	0.0432	0.0444	0.23	2.88	0.0433	0.0443	0.43	2.43
	0.0428	0.0427	0.0441	0.17	3.16	0.0427	0.0438	0.17	2.43
				0.20	0.30			0.30	2.43
102 ABCD CDEF	0.0430	0.0433	0.0448	0.77	3.35	0.0433	0.0446	0.63	3.00
	0.0429	0.0432	0.0448	0.67	2.86	0.0429	0.0441	0.00	2.83
				0.72	3.10				2.92
152 ABCDEF CDEFAB EFABCD	0.0430	0.0438	0.0454	2.01	3.63	0.0434	0.0449	1.07	3.37
	0.0430	0.0438	0.0452	1.94	3.39	0.0435	0.0452	1.23	3.87
	0.0430	0.0439	0.0458	2.25	4.43	0.0437	0.0451	1.80	3.07
				2.07	3.83			1.37	3.44

* Last letter denotes specimen adjacent to the hot side

** EOT - Effect of thickness for the particular low-density glass fiber product

measurements show excellent agreement for the two apparatuses. Apparatus error, which is small up to 76 mm (3 in.), increases significantly beyond this thickness. The experiments were performed with and without paper septa, and the results were calculated using the variable coefficients shown in Fig. 2.

For practical purposes a reasonable range of uncertainty should be assigned to the results obtained at different thicknesses. The tolerances should take into account any measurement uncertainty and variation in specimen structure as well as factors related to the testing procedure, for example, specimen preparation and conditioning. Uncertainty values will increase as a function of thickness. The following uncertainty ranges were deemed appropriate for the HFM's reported: for 25 mm, $\pm 0.5\%$; for 76 mm, $\pm 0.8\%$; and for 152 mm, $\pm 1.57\%$.

MAINTENANCE OF CALIBRATION

Calibration of an apparatus must be monitored on a continuous basis. Secondary reference specimens were therefore selected using the same criteria except that they were not paired. Each secondary reference specimen was tested a minimum of eight times and control limits were developed. These were later established once more, based on values obtained over a period of time and taking into account the variability due to different operators.

Any one of the secondary reference specimens may be used for control purposes. If a test result is outside the control limits, another secondary reference specimen should be checked before any calibration adjustment is considered. Only if such tests confirm that the results fall beyond the control limits and the cause of the discrepancy is not immediately obvious (for example, an error in thickness determination), new calibration coefficients must be developed based on the primary reference specimens.

There is no set rule for frequency of control checks. The procedure at the Innisfail plant requires a control check each time an apparatus is adjusted for a new specimen thickness, and each time the apparatus has not been used for some time (i.e., when a secondary reference specimen is tested before a new test series).

Primary and secondary reference specimens are identified using an alpha-numeric code. Marks are placed on the upper front surface to facilitate orientation of the specimen during test. Internal company certificates are issued for each calibrated specimen to signify the importance of the calibration program. These specimens are stored in rigid containers made of corrugated cardboard to protect them from damage.

CONCLUSION

This paper describes the procedure established by an insulation manufacturer to calibrate the heat flow meter apparatus used for its quality control operation. It identifies the characteristics of apparatus and testing procedure most important in calibration and calibration maintenance. The development of these procedures increased the confidence that quality control could meet the thermal resistance requirements specified by building codes and materials standards.

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