

An inter-comparison of a steady-state and transient methods for measuring the thermal conductivity of thin specimens of masonry materials

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Abstract

Recent standardization developments within the European Union relating to masonry and block systems now allow calculation of thermal performance to be undertaken based on the block configuration and reliable values of the material thermal conductivity. In general, to attain the optimum thermal resistance of a block a minimum amount of material is used such that the webs and faces bounding the air spaces are usually of the order of 6–8 mm. However, the current standard measurement methods are not suitable or precise enough for direct measurement on thin specimens cut from blocks. As a result the masonry industry requested that CEN TC89 investigate and recommend candidate alternative methods suitable for standardization.

A working group was established and following consideration of the issues involved, including, in particular, the thermal conductivity range, simplicity, small size, and short operating times, two methods were proposed for further investigation. The first was the steady-state guarded heat flow meter and the second the transient hot strip in both its linear and disc configurations. In order to verify their applicability both methods were used in an initial inter-comparison involving a total of nine organizations. Measurements were made at or close to room temperature on uniform thin test specimens fabricated from a batch of one typical masonry material.

This article contains details of the distinctive features and suitability of each method for the task, the participants, the material and test criteria, and includes a summary and discussion of the results that includes unexpected effects due to anisotropy in the particular material. Overall, the results were very promising such that a further

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series of intercomparison measurements is now underway on some typical materials within the total conductivity range for masonry type products.

Keywords

thermal conductivity, thermal diffusivity, transient methods, masonry, standards, clay, transient hot strip, guarded hot plate, transient hot disc, intercomparison

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Background

Masonry materials and systems are a major source of building envelope construction worldwide and with emphasis now placed on energy savings and related issues thermal performance becomes a critical factor in the design, operation and performance of buildings. A recent European study involving 11 organizations was carried out on the thermal and moisture properties of masonry construction materials using available standard test methods. The study was undertaken to provide the European Union with additional tools to satisfy the Construction Products Directive for these products (Salmon et al., 2002). The essential goals of the study were fulfilled satisfactorily, especially in confirming the use of calculation techniques using reliable property values.

However, a particular measurement problem issue was highlighted by the difficulty faced by the six laboratories involved in obtaining consistent thermal conductivity results on one specific material. This was a brick material where the maximum practical thickness of the available specimen cut from the block faces and having the lateral dimensions for the standard apparatus hot plates being used was of the order of 6–7 mm. This is not thick enough for measurements using the methods recommended in EN1745.

This thickness range is unsuitable for reliable measurements on such types of solid materials using a standard guarded hot plate or heat flow meter apparatus, as the specimen thermal conductance due to their high thermal conductivity range will normally be outside the recommended limits for the methods. Furthermore, for hard solids the surface temperature measurements require direct instrumentation of the surfaces, which result in further uncertainties in the actual measurement thickness. This problem is compounded further by the fact that additional surface preparation is required to attain the optimum degree of flatness and uniformity of thickness to minimize contact resistances.

The remaining applicable standard method that could be considered is based on the use of one of the hot box techniques but this requires large complex test specimen assemblies, long times of test, and is thus very expensive.

Based on the above issues and at the request of TC 125 on Masonry, CEN/TC89 on thermal insulation test methods established a working group, WG11, containing members from both groups having the necessary experience in masonry materials and thermal properties measurements. The scope of their task was to recommend and validate one or more possible candidate measurement techniques suitable to be

used for masonry materials, especially those in the form of specimens no greater than 10 mm in thickness and of sufficient areal size suitable for more than one method.

It was realized that specimen size limitations would restrict the use of any method to materials where the maximum aggregate dimension would be of the order of one-tenth of the thickness and also that due to possible material inhomogeneities, density differences and moisture content of multi-specimens from the same or different blocks would have to be evaluated. These factors would apply to any chosen method and an agreed test protocol would be essential.

Candidate measurement solutions

The composition of the working group (WG) is shown in Table 1. Also included are details of the various measurement apparatuses that each has available. Following their review of the steady-state and transient techniques that could satisfy the basic criteria of simplicity of concept and operation, small size, short

Table 1. Details of the working group participants.

Name	Organisation	Country	GHP	GHFM	Transient plane source	Hot strip bridge	Other transient
D Salmon* (Convenor)	NPL	UK	×	×	×		
R Tye	NPL	UK					
H Anton*	FIW	Germany	×				
U Hammerschmidt*	PTB	Germany	×			×	
O Dupont*	CTMNC	France		×			
D Palenzuela*	CTMNC	France		×			
G Pettit	Tarmac	UK					
P Primmer	Hanson	UK					
J Regrettier	Bouyer-Leroux	France					
H Mayr*	BTI	Austria		×			
A Earker*	BTI	Austria		×			
B Barthou	CERIB	France					
B Adl-Zarrabi*	SP	Sweden			×		
L Kubicar**	Slovakia Tech Uni	Slovakia					×
M Kogler**	Wienerberger Ziegel	Austria					×

*Original measurement partner.

**Measurement partner joined after original measurements completed.

measurement times, and potentially acceptable precision of $\pm 5\%$ for the expected thermal conductivity range, two basic techniques were identified as candidates satisfying all of the major criteria. These were:

The 50-mm diameter guarded heat flow meter

The method, based on steady-state heat flow conditions, is similar in principle to the heat flow meter method used for thermal insulations except that specimen size is smaller and the thermal resistances are much lower such that corrections need to be made to account for surface contact resistances between the hard surfaces (Tye and Coumou, 1981). This is accomplished by careful calibration using several reference materials having different thicknesses/thermal resistances. It has already been in use for quality control (QC) purposes by some members of TC 125 and typical uncertainties have been found to be of the order of $\pm 5\%$ or better for a broad range of material types.

The transient plane source technique as single strip or bridge and disc forms

These are two forms of the basic line source transient method based on the principle of creating a small disturbance by a heat pulse or a step-wise heat flux in a specimen at a steady temperature and recording the temperature response with time (Gustafsson et al., 1979; Gustafsson, 1991). They are similar in concept except in the form and size of the heat pulse generator, either a long thin strip or a small thin circular heater, embedded in the specimen and in the analytical solution of the temperature response, based on a solution of the basic heat transfer equation together with appropriate assumptions, to provide the measured property.

These techniques had been in general use for a number of years on all types of materials, mostly other than masonry, and also have the advantage of being multi-property in concept. In addition, considerable developments had occurred with time in upgrading the use of and improving the accuracy of both techniques to an acceptable order of at least $\pm 5\%$. This had been accomplished by undertaking improvements in the type, form and size of sensor, the mode of operation, and the model used for evaluation of anisotropic thermal property values from the experimental data for the circular heater form (Gustafsson et al., 1994, Sabuga and Hammerschmidt, 1995) and for the linear strip form which had evolved through a tandem strip of unequal lengths to minimize end losses (Hammerschmidt et al., 2005) finally to a multi-strip bridge form to attain more heating uniformity (Hammerschmidt and Meier, 2006).

Suitability of methods

The essential methodology of each candidate already had the added advantage of being the subject of standardization in some form. The steady-state guarded heat

flow meter had been standardized as ASTM E-1530 (ASTM E 1530-99, 1999) for many years while both transients are the subject of recommended standard prepared by an international task force for the whole group of transient techniques based on the line-source (Tye et al., 2005), the hot disc part of which is now the subject of an international standard under development by the ISO/TC 61/SC10 Committee on Plastics. Finally, user-friendly commercial models based on the techniques and all having acceptable performances were either available or coming into the market place to provide ready means to undertake properties measurements on appropriate smaller specimens.

A specific benefit of the disc form of plane source transient technique is that it can be used on the same specimen configuration as that for the guarded heat flow meter. A second and more important issue, which became apparent during this intercomparison, is that, due to the use of the circular heater form and the consequent difference in the heat flow direction and distribution within a test specimen to that for the strip and all the other forms being used, the disc transient can also be used to determine anisotropy in a test specimen, providing the volumetric specific heat is measured or known (Sabuga and Hammerschmidt, 1995).

Thus, if there appears to be differences between the results obtained by all other methods from those by the hot disc then the effects of anisotropy have to be considered and can be examined using the hot disc. Since masonry blocks consist of a series of webs between the faces and edges, unless the material is known or considered to be homogeneous and isotropic the thermal conductivity for the different heat flow directions in these sections must be known in order to calculate the thermal conductance of the complete block.

Interlaboratory comparison

Participants

Following the selection of candidate methods an initial interlaboratory program of comparative testing was planned to determine the viability and reliability of the chosen techniques. This was to be carried out initially on one typical material provided by a group member. Measurements would be undertaken by members of the group together with additional invited member(s) each using one or more of the individual techniques available to them (Table 1).

Material and specimens

The selected material was fabricated and supplied by CTMNC. A mix of clay typically used for manufacture of clay blocks was cast as two plates large enough to machine specimens 250 mm² by 40 mm thick; both plates were then fired in an oven. It was designed to be a typical clay block as used in the building

industry with a density of around 1950 kg/m^3 . It was supplied initially in the form of two 'large' sample pieces marked A and B each approximately 250 mm square and 40 mm thick. These were required to provide appropriate machined specimens for all subsequent measurements as shown in Table 2.

Three organizations were to undertake measurements by the standard guarded hot plate method to provide accurate baseline values in the approximate temperature range of $10\text{--}30^\circ\text{C}$. For these, two pieces were machined to provide a pair of uniform 200 mm square and 40 mm thick specimens with flat parallel faces in accordance with the standard requirements for measurement by NPL and FIW. One of these specimens was then machined to a 100 mm diameter and 15 mm thick flat specimen for the PTB smaller hot plate.

The remaining pieces were machined to provide the smaller specimens required for the other methods. A total of eight specimens were obtained, four each from specimens A and B, 50 mm in diameter and 10 mm thick (apart from one which was 12 mm thick) flat uniform discs both for BTI, CTMNC, and NPL, using the guarded heat flow meter, and for NPL, SP, SV and WZ using the disc and strip source transient techniques. One pair of specimen pieces 100 mm by 30 mm and 5 mm thick was machined for the PTB hot bridge transient method.

Test conditions

All the tests were to be carried out under the following conditions:

Specimen conditioning. All specimens to be tested immediately following drying to constant mass at 105°C .

Temperature settings. For steady-state methods, measurements to be made at one or both mean temperatures of 10°C and 23°C with a temperature difference of approximately 15°C and for the transient methods, measurements required at one or both mean temperatures of approximately 10°C and 23°C .

Other requirements

- (1) Measurements of the dry density based on the mass and dimensions were to be carried out on the respective test specimens by the recipients prior to and after the thermal properties measurements.
- (2) For the guarded hot plate measurements, it was required that temperature sensors had to be attached to the specimen surfaces or be in them to ensure reliability of surface temperature measurements in this hard material.
- (3) For measurements by other methods, results would be the mean of at least three measurements under the same conditions.

Table 2. Details of cutting plan for test specimens and test programme.

Action	Specimen name	Specimen thickness (m)	Length (m)	Width (m)
1 NPL cut specimens to size shown	A	40	201	201
	B	40	201	201
2 NPL and FIW measure the pair A and B in GHPs. Then send to PTB.				
3 PTB cut specimens A and B to sizes shown	A1	40	201	90
	A2	40	201	108
	B1	40	201	90
	B2	40	201	108
4 PTB measure using transient method pairs A1/A2 and B1/B2. PTB send A1 and B1 to BTI				
5 PTB cut specimens to sizes shown	A2.1	40	108	108
	A2.2	40	90	108
	B2.1	40	108	108
	B2.2	40	90	108
6 PTB cut specimens A2.1 and B2.1 to correct diameter and measure using GHP. PTB measure A2.2 and B2.2 using transient probe				
7 PTB cut transient specimens to reduced thickness as shown and remeasure A2.2 and B2.2 using transient probe	A2.2	5	90	108
	B2.2	5	90	108
8 BTI, using specimens A1 and B1, cut out three 50 mm discs from each piece and reduce thickness to 10 mm thick. Note locations of specimens on original uncut piece for reference later.	A1.1	10	50 mm diameter	
	A1.2	10	50 mm diameter	
	A1.3	10	50 mm diameter	
	B1.1	10	50 mm diameter	
	B1.2	10	50 mm diameter	
	B1.3	10	50 mm diameter	
9 BTI measure all samples using 50 mm GHFM apparatus and send specimens to CTMNC				
10 CTMNC complete measurements and send specimens to NPL				
11 NPL measure specimens and send specimens to SP, Kubicar and Weinberger for final measurements				
12 NPL produce final report				

Results

Density

The collected results are shown in Table 3. The overall range for the different specimens is approximately 2.1%. There is a small but definite difference between the two original samples that is manifested in the smaller specimens cut from each. However, there are no large differences within the smaller sections indicating that they are reasonably homogeneous. However, there were small features within the specimens, specifically small particles that were of the order of 1–2 mm across which could affect reproducibility of results of measurements on the smaller specimens especially with the transient techniques due to the probing depth.

Within the industry clay is known to have a layered structure (both microscopically and macroscopically), where the original amount of water used in mixing can affect the final structure. More water used in the mixing results in a smoother, more homogeneous structure while a lesser amount of water in the original mix gives a more disordered structure. Thus it is possible that the material is anisotropic. As a consequence the measurements using the hot disc form were analyzed in the recommended way for such behavior (Sabuga and Hammerschmidt, 1995) using a measured value of specific heat to provide comparable directional values to those obtained with the other methods.

Guarded hot plate

The individual results obtained by the three laboratories to establish the base line for comparison with all the other measurements are shown in Figure 1. It should be noted that each result is an average value of the thermal conductivity for the two

Table 3. Densities in kg/m^3 of specimens as measured by each partner.

Specimen	NPL	FIW	PTB	BTI	CTMNC	STU	SP	WZ
A	1955		1951					
B	1968		1959					
A1			1951					
A2			1959					
A1	1964			1961	1960		1968	
A2				1947	1960			
A3	1943			1942				
A4				1948				
B1	1976			1976	1980			
B2				1962	1960			
B3				1956				
B4	1954			1958				

original blocks A and B. The figure also contains the mean values of all three sets from the slope of which the change in thermal conductivity for a 1° change in temperature can be determined. This value of 0.462 milliW/m·K has been used later to convert all measurements by the other methods to normalized values at 10°C and 23°C.

An analysis was made first using the guarded hot plate results to establish a base line for comparison of all the other thermal conductivity measurements.

Figure 2 shows the percentage deviation of the guarded hot plate values from the mean value of all these results. It can be seen that the agreement between labs is within ±1.5%, which is a good agreement as the measurement uncertainty for this type of specimen is normally between ±3% and ±5%. It is interesting to note that

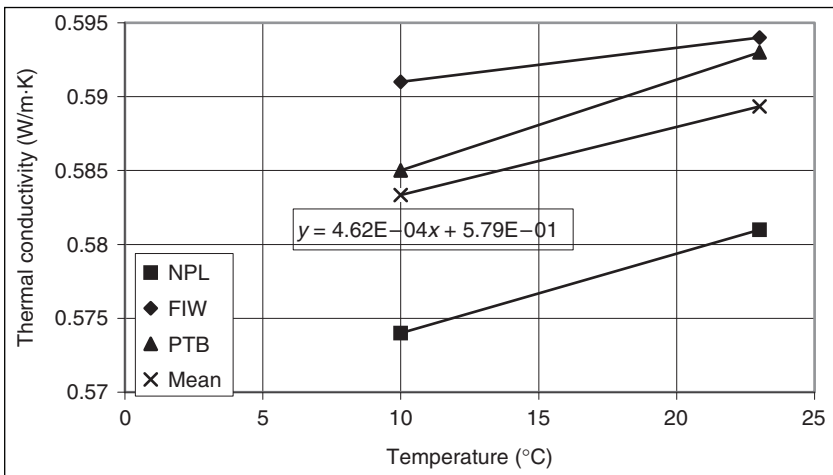


Figure 1. Guarded hot plate thermal conductivity results.

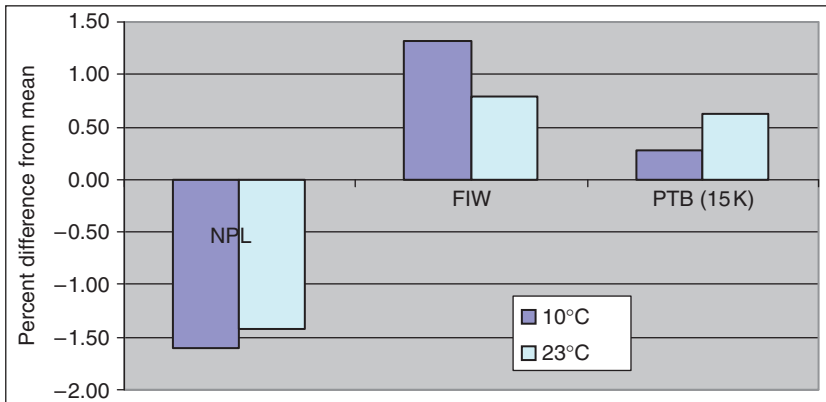


Figure 2. Percentage deviation of guarded hot plate values from mean value.

the slopes of the NPL and PTB graphs are very similar to and quite different from that of FIW.

Guarded heat flow meter

The mean values, obtained from at least three repeat measurements on the individual specimens which varied by less than $\pm 3\%$ overall, are shown in Table 4. The results are remarkably consistent and show very good agreement between participants that measured the same specimens and also it is seen that there is a definite increase in property values with both density and temperature. The results indicate that there are small differences between the two original blocks.

Transient methods

The collected results obtained using the various forms of plane source transient method are shown in Table 5. The agreement between all transients is of the order $\pm 5.5\%$ but for the hot bridge and hot disc techniques it is better than $\pm 3\%$. Initially, the first measurements using the circular heater (disc) form indicated that the thermal conductivity of the material was of the order of 20% greater than that obtained by all other methods. This factor confirmed that the material was anisotropic and, as discussed earlier, the SP and NPL data were subsequently analyzed (Sabuga and Hammerschmidt, 1995) to provide the values in the table for the specific direction perpendicular to the specimen surface.

For this the volumetric heat capacity is required. A value of 1.58 MJ/Km^3 was determined by SP from the measured specific heat of the specimens using a calorimeter combined with the specimen density of 1968 kg/m^3 .

Table 4. Axial thermal conductivity of specimen using guarded heat flow meter method. Values in $\text{W/m}\cdot\text{K}$.

Specimen	NPL			BTI	CTMNC
	10°C	23°C	30°C	10°C	30°C
A1	0.577	0.589	0.595	0.584	0.585
A2				0.534	0.532
A3	0.546	0.553	0.559	0.538	
A4				0.540	0.520
B1	0.569	0.582	0.589		0.589
B2				0.559	0.556
B3				0.560	
B4	0.530	0.539	0.545	0.534	0.534

Discussion

Summary of results from all the measurement methods

Table 6 contains the mean values of thermal conductivity of the material using data from blocks A and B as measured by all laboratories. The highlighted values have been normalized to the relevant temperature using the temperature relationship established with the guarded hot plate measurements.

In general the results, presented in Figure 3 indicate that the guarded heat flow meter values are some 4–5% lower than those of the hot plate values and show the largest percentage deviation.

Table 5. Axial thermal conductivity using transient methods.

Laboratory	Mean thermal conductivity W/m·K		
	10°C	23°C	30°C
PTB (hot bridge)	0.591		0.610
WZ (hot strip)		0.582	
SV (plane source)		0.545	
SP (hot disc)		0.594 (0.584–0.601)	
NPL (hot disc)		0.602 (0.599–0.619)	

Table 6. The mean measured values of all specimens.

Lab	Method	Measured thermal conductivity of A and B Calculated using 0.462 mW/m·K ²			Percent deviation from GHP mean	
		Lambda (10°C) /(W/m·K)	Lambda (23°C) /(W/m·K)	Lambda (30°C) /(W/m·K)	10°C %	23°C %
BTI	GHPM	0.554	0.560		–5.0	–5.0
CTMNC	GHPM	0.557	0.563	0.566	–4.6	–4.5
NPL	GHPM	0.555	0.566	0.572	–4.8	–3.9
WZ(T)	Transient	0.576	0.582		–1.3	–1.2
PTB(T)	Transient	0.591	0.610		1.3	3.5
NPL	Trans. disc		0.608			3.23
SP	Trans. disc		0.594			0.85
FIW	GHP	0.591	0.594		1.3	0.8
NPL(G)	GHP	0.574	0.581		–1.6	–1.4
PTB(G)	GHP	0.585	0.593		0.3	0.6
	GHP mean	0.583	0.589			
SV(T)	Transient		0.545			

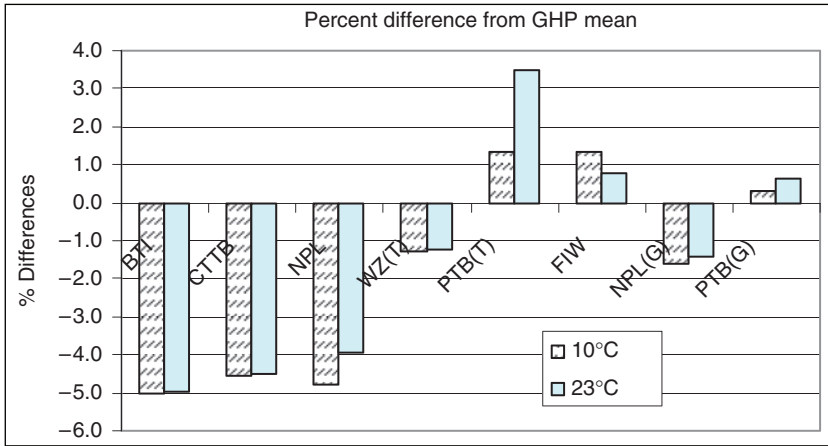


Figure 3. Percentage difference of all results from GHP mean value.

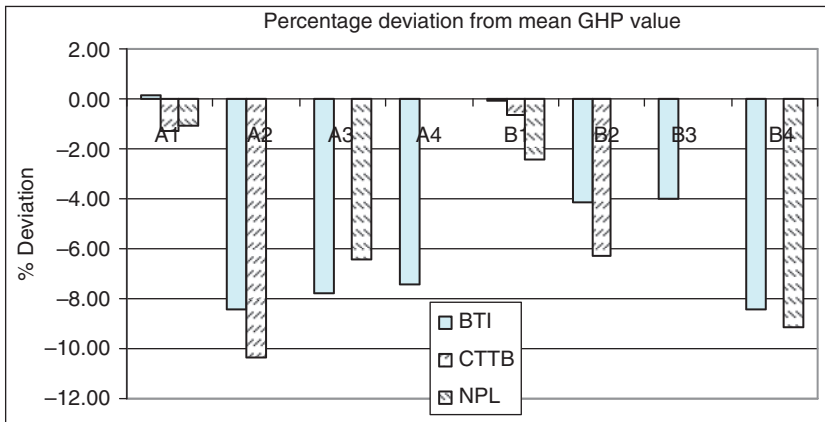


Figure 4. Graph showing the deviation of each specimen from blocks A and B from the mean GHP values.

Guarded heat flow meter results

To compare the GHFM values, the CTMNC results have been normalized to 10°C using the relationship established with the guarded hot plate results. The GHFM measurements were carried out at a mean temperature of 10°C by BTI and 23°C by NPL, and at a mean temperature of 30°C by NPL and CTMNC. Figure 4 shows that the BTI results are consistently between 1 and 2% higher than the CTMNC results, whereas the NPL results show no consistent pattern relative to the other two laboratories. Interestingly specimens A1 and B1 are very close to the ghp mean value.

Table 7. Hot disc measurements by SP (Fire) on specimen pairs at room temperature.

Specimen pairing	Thermal conductivity (W/m · K)	% Difference from GHP mean value
A1-B1	0.595	1.02
A1-B2	0.584	-0.85
A2-B1	0.601	2.04
A2-B2	0.593	0.68
Mean value	0.594	0.85

Transient measurements

For the two transient measurements using the strip configuration it can be seen from Table 1 and Figure 3 that the WZ value is 1.2% lower than the hot plate value, whilst the PTB results are 1.3% higher at 10°C and 3.5% higher at 23°C.

Both SP and NPL partners used the hot disc plane source method on the same specimens as used for the guarded heat flow meter measurements at NPL. These tests showed that the material was anisotropic with thermal conductivities of the order of 0.6 W/m·K through the thickness and 0.9 W/m·K in the plane of the heat source. SP (Fire) carried out measurements on three other specimen pairs in addition to the A2-B2 pair measured by NPL, the results for the through the thickness thermal conductivity of each pair are shown in Table 7. These results show very good agreement with the GHP mean value varying from that value by between -0.85% and +2.04%.

NPL also measured the clay samples A2 and B2 using the transient plane source method, using the same values for the specific heat as SP. The results were 0.61 W/m·K through the thickness and 0.88 W/m·K in the plane. The results showed the same anisotropy as those for SP, compared with the guarded hot plate mean value, the mean through the thickness SP value at 23°C was 0.85% higher and the NPL value was 3.2% higher.

Conclusions

- (1) There was very good agreement between laboratories using the guarded hot plate method with the measured thermal conductivity results all within $\pm 1.5\%$ of the mean values of 0.583 W/m·K and 0.589 W/m·K at 10°C and 23°C, respectively.
- (2) In those cases where more than one laboratory measured the same specimen using the guarded heat flow meter method the agreement was always within 2%. In one case the values for specimen A1 were within 1.5% of the mean guarded hot plate value.

- (3) The results for individual specimens measured in the guarded heat flow meter apparatus showed variations between the samples of the order of 5%. However, some of the differences could be attributed to density variation in the specimens.
- (4) In general the transient values were all higher than the mean guarded hot plate value by as much as 3.2%, the PTB and NPL values agreeing quite closely and the SP result being about 2.5% lower. It is quite feasible that the guarded hot plate values could be slightly low if surface thermal contact resistance has not been fully accounted for in the measurement. There may be additional errors due to the anisotropy. The ratio of thermal conductivity perpendicular to the thickness compared to parallel to thickness is 1.5, which is inside the maximum limit of 2, the ratio allowed by ISO 8302 Guarded Hot Plate Standard.
- (5) There was an overall 2.1% difference in density between the various specimens and a clear difference between values for the two sample pieces.
- (6) Overall, taking into account the inhomogeneity and anisotropy of the material and the measurement uncertainties, the results for the guarded heat flow meter method and the transient methods are generally within $\pm 3\%$ of the mean guarded hot plate values and can be considered to be in good agreement.
- (7) The observed inhomogeneity and anisotropy of this type of masonry sample has implications for the product groups to ensure that adequate sampling procedures are in place to fully represent the mean thermal conductivity of the product, and that directional properties must be measured to provide adequate data for calculation purposes.
- (8) These new methods appear to be appropriate for determining the thermal properties of thin sections of masonry products provided adequate sampling techniques and measurement procedures are established by the product manufacturers.

This limited intercomparison has been successful in that it has shown that there are two potential methods that can be used to measure thin specimens of a masonry material. As a result a more comprehensive study has been planned to establish the range of both material types and thermal conductivity for which they can be applied, and the means to obtain the extent of anisotropy in such materials.

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