

# Analysis of thermal probe measurements using an iterative method to give sample conductivity and diffusivity data.

Steven Goodhew and Richard Griffiths\* .

Joint Schools of the Built Environment, University of Plymouth,  
Hoe Centre, Notte Street, Plymouth, Devon, PL1 2AR, UK.

Tel: +44(0)1752 233605, Fax: +44(0)1752 233634 e-mail: [rgriffiths@plymouth.ac.uk](mailto:rgriffiths@plymouth.ac.uk).

\*Author to whom correspondence should be addressed

## Abstract

The application of Microsoft Excel Solver routines to analyze data from a thermal probe placed in a number of samples is described. The successful application of the Solver routine to the thermal probe temperature time data requires the selection of an appropriate time interval. The various critical times are discussed and a method is proposed for selecting the appropriate Solver routine and the time interval over which the experimental data must be analyzed. Values of the sample thermal conductivity, the thermal diffusivity and the probe to sample conductance are reported for a stainless steel probe in unused mineral oil, and paraffin wax and for a copper probe in Magna904 and PTFE. The results for a commercial probe in agar agree with accepted data for water and the results for a clay-straw mixture demonstrates the method applied to a good insulator.

140 words

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## 1. Introduction.

The Centre for Earthen Architecture, in the University of Plymouth School of Architecture, is interested in the conservation of earth walls. The Centre has studied the structural properties, Greer [1], the pathology, Keefe et al [2], the inventoring of earth buildings using GIS, Ford [3] and the thermal properties, Goodhew et al [4].

When modelling the thermal performance of a building material, both the rate of heat transfer, which is a function of the thermal conductivity, and the quantity of heat required to raise the temperature of the material, the thermal capacity, are required. The thermal diffusivity is the thermal conductivity divided by the volumetric heat capacity. This paper will demonstrate that a time-dependent thermal probe technique, together with an iterative method of data analysis, can be used to measure simultaneously the two thermal properties, the conductivity and diffusivity. Goodhew et al [4] described the application of the probe technique with Microsoft Excel Solver routines to analyse the temperature time profiles. In the past, two problems, namely (1) the selection of an appropriate time interval during which the probe data should be analysed, and (2) the unknown probe to sample conductance (H), have hindered the probe technique. A method of data analysis is presented that both objectively identifies the data analysis time interval, and allows the probe to sample conductance (H) to be measured. The materials chosen for this study range from a liquid, a paste, solids and, finally, an insulating material. An underlying aim of the work is to produce a cheap and reliable method for determining the thermal properties of earth walls in Devon.

Simple home-made copper and stainless steel probes and a commercially available thermal probe from Hukseflux have been used.

2. The thermal model and the probe method.

The time-dependent thermal probe technique employs a line heat source supplied with constant power  $Q$  (Watts). The probe has a mass  $m$  (kg), thermal capacity  $c_p$  ( $J\ kg^{-1}K^{-1}$ ), a length  $L$  (m) with an external radius  $r$  (m). The probe is placed in good thermal contact with a homogeneous specimen with thermal conductivity  $\lambda$  ( $W\ m^{-1}K^{-1}$ ) and thermal diffusivity  $\alpha$  ( $m^2s^{-1}$ ). This specimen is assumed to be of infinite extent and at a uniform initial temperature  $\theta_i$  (C). Between the probe heater, which is placed along the axis of the metal probe tube, and the specimen there is a thermal contact conductance  $H$  ( $W\ m^{-2}K^{-1}$ ), termed the probe conductance. A thermojunction is attached to the mid-point along the heater. Once the system is in thermal equilibrium, the heater power  $Q$  is switched on and the probe temperature  $\theta$  (C) increases with the elapsed time  $t$  seconds. The rise in probe temperature  $\theta_e$  with time is recorded, and then analysed using the thermal model proposed by Blackwell [5]. Here, it is assumed that the rise in probe temperature as a function of time is given approximately by the following expression, where  $\gamma$  is Euler's constant, with a value 0.57722.

$$\theta = (Q/4\pi\lambda L) \{ \ln(4\alpha t / r^2) - \gamma + 2\lambda/rH + (r^2/2\alpha t) [ \ln(4\alpha t / r^2) - \gamma + 1 - (\alpha mc_p / \pi r^2 L \lambda) (\ln(4\alpha t / r^2) - \gamma + 2\lambda/rH) ] + \text{other terms } (r^2/\alpha t)^2 \} \quad 1$$

Again, following Blackwell [5], it is assumed that the "other terms  $(r^2/\alpha t)^2$ " can be ignored, since as the time  $t$  increases this term will soon become negligible, and the rise in probe temperature as a function of time simplifies to;

$$\theta = A [ \ln t + B + (1/t) ( C \ln t + D ) ] \quad 2$$

where  $A = Q/4\pi\lambda L$

$$B = \ln(4\alpha/r^2) - \gamma + 2\lambda/rH$$

$$C = (r^2/2\alpha) [ 1 - \alpha mc_p / \pi r^2 L \lambda ] \quad \text{and}$$

$$D = (r^2/2\alpha) [ \ln(4\alpha/r^2) - \gamma + 1 - B\alpha mc_p/\pi r^2 L \lambda ]$$

Previous authors, for example Blackwell [5] and Batty et al [6] and [7], make a further assumption that after a long time the C and D terms with (1/t) can also be ignored, since again at long times these terms become small compared with the A and B terms. Therefore, at long times a graph of the rise in probe temperature against the natural logarithm of the elapsed heating time becomes a straight line. The slope A of the line is a function of the specimen thermal conductivity, while the intercept AB on the rise in probe temperature axis is a function of the thermal diffusivity and the probe conductance (H) between heater and specimen. A regression analysis of the data will give values of A for the slope and AB for the intercept. The thermal conductivity  $\lambda$  is obtained from the value of A. Some authors have assumed that the probe conductance H is large, the probe surface is in excellent thermal contact with the sample over its entire surface area, and therefore the term  $2\lambda / (r H)$  is small and can be ignored. The value of the intercept AB can then be used to determine the thermal diffusivity  $\alpha$  of the specimen.

Clearly, before this technique can be used confidently, two questions need to be explored. Firstly, what time must elapse before the (1/t) terms become sufficiently small, so that they can be ignored? Secondly, what is the value of the probe constant H, and can the term  $2\lambda / (r H)$  be ignored? To explore these questions the thermal model for a stainless steel probe placed in paraffin wax was used to generate typical, but virtual experimental, probe rise temperatures as a function of time. There were two aims for the virtual experiments. Firstly, there was a need to confirm the data analysis method, and secondly, to explore the influence that temperature fluctuations and uncertainties might have on the analysis method. This study of the virtual experiments on paraffin wax are

fully reported in an Internal Report, Goodhew et al [8], and demonstrated the feasibility of the proposed method.

### 3. The Microsoft Excel 97 Solver routine for data analysis.

The present thermal model represents a non-linear mathematical problem since there are products of the variables  $\lambda$ ,  $\alpha$  and H in the expression for the probe temperature versus time. The solutions sought in the iterative method are not constrained in anyway, the results are unknown at the start, and, practically, there are no reasons why any solution should lie in any particular region, except that the solutions should be positive and of appropriate physical magnitude. Microsoft Excel 97 Solver is ideal for this application, and the limitations will be due to the assumptions in the thermal model, that is in the temperature versus time equation 2 above, and in the experimental errors, not due to the Solver routine.

Following Banaszkiwicz et al [9] an iterative method was established to simultaneously determine the three unknowns,  $\alpha$ ,  $\lambda$  and H. Two different Solver routines have been constructed and studied. Solver 2.3 was a Solver routine using only the first two constants, A and B in equation 2, while seeking the values of the three unknowns, hence Solver 2.3. Solver 4.3 employs all four of the constants A, B, C and D in equation 2, and again seeks the three unknowns. For each Solver routine a set of trial values for each of the unknowns  $\alpha$ ,  $\lambda$  and H is used to calculate the trial rise in probe temperature at a given time, say  $\theta_t$  K. At each time the difference  $(\theta_e - \theta_t)$  between the experimental rise in probe temperature  $\theta_e$  and the calculated, or trial, rise in probe temperature  $\theta_t$  is found. This temperature difference is squared, to remove the negative contributions, and then the squared differences are summed. Solver uses an

iterative process to find the minimum of the square root of this sum of the temperature differences squared, or;

$$\text{Sum} = [ \Sigma(\theta_e - \theta_t)^2 ]^{1/2} = 0, \quad \text{or a minimum.}$$

Choosing the appropriate data analysis time interval is not always a simple and routine matter. The various times that control the selection of the appropriate Solver routine, Solver2.3 or Solver4.3, and the time interval will be described and some results discussed.

#### 4. The analysis time interval $t_s < t < t_b$ .

The analysis of the thermal probe data is complicated, since the time interval during which the plot of the rise in probe temperature against the natural logarithm of the elapsed heating time is linear is not always obvious. Blackwell [5] and Vos [10] have addressed this issue and consider short times  $t_s$  or the time that must elapse before the analysis begins. Again, at the extreme end of the time interval, that is at long times, following Blackwell [11] allows the probe axial heat flow error to be calculated. Here the maximum time  $t_b$  is calculated, the time  $t_b$  that may elapse before the magnitude of the axial error becomes a significant fraction of the radial heat flow.

The rise in probe temperature versus time  $t$  data should be analysed in an appropriate time interval, so that  $t_s < t < t_b$ .

There are four times that should be discussed in the study of the thermal properties of a sample using the thermal probe. These times are; (a) the Vos [10] start time  $t_s$ , (b) the Vos [10] reflected heat time  $t_r$ , (c) the Blackwell [11] axial flow error time  $t_b$ , and (d) the long time error  $E\% t_l$ , Goodhew et al [8], which enables the selection of the appropriate Solver routine.

- (a) Vos start time  $t_s$ . Vos showed that the linear section of the rise in probe temperature against natural logarithm of the elapsed heating time appears after the time  $t_s$ . Here  $t_s > 50 r^2 / 4 \alpha$ , for a probe with external radius  $r$  metres placed in a material with a thermal diffusivity  $\alpha \text{ m}^2\text{s}^{-1}$ . Practical observations suggest that linear sections are available after 50 to 70 seconds, with some exceptions.
- (b) Vos reflected heat time  $t_r$ . Vos points out that the theory supporting the probe studies assumes that the sample is of infinite extent. In samples of finite size, the probe behaviour will be disturbed by heat returning to the probe from the boundary of the sample. Vos gives an expression for this reflection effect and defines a reflection time  $t_r$  given by  $t_r > 0.6 x^2 / 4 \alpha$ , where  $x$  is the minimum distance from probe to sample boundary. The majority of the samples studied in this work had minimum distances in the range 40 to 70 mm with corresponding Vos reflection times greater than 1500 seconds.
- (c) Blackwell axial flow error time  $t_b$ . A spreadsheet was constructed based upon Blackwell [11] equation 13 on page 416. The aim of this spreadsheet was to calculate the ratio, expressed as a percentage, of axial heat flow to the radial heat flow as a function of both the time and the probe to sample conductance  $H$ . The sheet contained the physical and thermal data for the various probes and samples. For a given probe–sample combination the time was adjusted for a series of  $H$  values, ranging from 60 to 500  $\text{Wm}^{-2}\text{K}^{-1}$ , to identify the time when the magnitude of the axial heat flow was just less than 1% of the radial heat flow. This gave the Blackwell time  $t_b$ . This time represents the maximum time beyond which any analysis of the data will be in error by at least 1% due to the axial heat loss from the probe.
- (d) Long time error  $E\%$   $t_l$ . Ideally, the analysis should be carried out with an elapsed heating time  $t$  so that  $t_s < t < t_b$ . However, a further complication arises in a particular

probe-sample combination namely, when do the  $(1/t)$  terms with constants C and D in the Blackwell expression, equation 2 above, become negligible. This question was explored in the following way. Two theoretical probe temperatures can be found using equation 2 above. The first is calculated using only the two constants A and B, say  $\theta_2$ , while a better theoretical probe temperature is available if all four constants A, B, C and D are used, say  $\theta_4$ .

The relative percentage error E% at a given time t in assuming the four constant model over the two constant model may be written as,

$$E\% = 100 (\theta_4 - \theta_2) / \theta_4 = 100 (C \text{Int} + D) / [(t + C) \text{Int} + Bt + D]$$

The criterion adopted here was that, if an acceptable error value of E% is set at 1%, then the time  $t_i$  can be found using a graph of E% versus t. Both Solver spread-sheets have this graph and allow an estimate of  $t_i$ .

Now the time analysis interval,  $t_s < t < t_b$  can be determined in an objective way, and the appropriate Solver routine can be selected. The Solver 4.3 routine should be used first with t in the range  $t_s < t < t_b$ . The routine can be used to explore the error E% and determine the time  $t_i$ . The Solver 2.3 routine should then be used with  $t_i < t < t_b$ . The Solver 2.3 is the better routine as it is independent of the probe thermal capacity.

## 5. Experimental arrangement.

The home-made simple probes are of two types, and followed the designs used by Batty et al [6] and [7]. First there is a stainless-steel probe 3mm in external diameter, 70mm long. A robust probe is essential for the proposed field studies on cob walls. The second home-made probe used copper tube, again with diameter of about 3mm and 70mm long. These probes usually had one thermojunction placed at the centre of the hairpin heater running along the axis of the probe.

The Hukseflux thermal probe, type TP02, is 150mm long and 1.5mm in diameter. The external sheath of the probe is stainless steel and contains two internal thermojunctions, and the 100mm long hairpin heater. At the base is a platinum resistance thermometer to determine the ambient temperature. The two internal thermojunctions are used to measure the rise in temperature of the probe above the main body of the sample. The application of this needle probe to the clay-straw had two advantages. Firstly, the probe could be pushed gently into the sample, and secondly, here was an example of the technique being applied to a highly insulating material. It is expected that the thermal conductivity of the clay-straw would be close to that of thatch, and therefore about  $0.07 \text{ Wm}^{-1} \text{ K}^{-1}$ , as suggested by the CIBSE Environmental Design Guide A [12]. The specific heat capacity is likely to be that of straw with a small contribution due to the clay.

The probe heater current was supplied from a lead-acid battery via a simple control circuit. The data was logged using a DataTaker 615 datalogger, again powered by a lead-acid battery, and the logger was controlled using DeLogger Plus, revision 3 software operating in Window NT. The data was downloaded to a Microsoft Excel spread-sheet for analysis. Using low voltage battery supplies for the probe and logger had two advantages. Firstly, the experimental arrangement was independent of mains voltage fluctuations, and secondly, the kit was readily portable for the proposed field work.

## 6. Experimental and analytical procedure.

There are many unknowns in the work, such as (i) the values of the three parameters  $\lambda$ ,  $\alpha$  and  $H$ , (ii) which Solver routine should be used, and (iii) during what times are

they applicable? As a result a standard procedure for obtaining the results from the probe data using the Solver routines has been adopted.

- a. Allow sample and probe to reach thermal equilibrium, say over 24 hours.
- b. Follow probe rise temperature for 400 seconds, with heater off to confirm thermal equilibrium between sample and probe.
- c. Switch heater on with a constant power per m to give a 3 to 5C rise in probe temperature over 400 seconds of heating.
- d. Inspect a graph of rise in probe temperature versus natural logarithm of time, and if there is a linear section after 50 to 70 seconds use a regression analysis to determine the thermal conductivity of the sample.
- e. Run the Solver 4.3 routine over the time interval 60 to 200 seconds to find the preliminary values of  $\lambda$ ,  $\alpha$  and H. Determine the 2or4 constant error time  $t_i$  from the graph on the Solver 4.3 spread-sheet.
- f. Run the Blackwell axial error spread-sheet with  $\lambda$ ,  $\alpha$  and H from the initial Solver 4.3 routine. Determine the Blackwell axial error time  $t_b$ .
- g. Use Solver 2.3 in time interval  $(t_i + 10) < t < (t_b - 10)$ , and use Solver 4.3 in time interval  $60 < t < (t_b - 10)$ .
- h. Compare thermal conductivity from the various routines, including the regression if used.
- i. Repeat the experimental work with three different heater power per m. Limit the maximum probe temperature rise over 400 seconds of heating to between 8 to 10C.
- j. When the values of the three constants  $\lambda$ ,  $\alpha$  and H become more certain, re-run the Blackwell axial error spread sheet for  $t_b$ . Re-check the 2or4 constant error time  $t_i$ .

Repeat analysis of all data obtained under  $i$  using the Solver routines with their appropriate time intervals.

k. Compute means and standard deviations for thermal conductivity, thermal diffusivity, volumetric heat capacity and probe to sample conductance from the values obtained in the final analysis step  $j$ .

#### 7. Data analysis time-intervals.

Table 1 shows the various times for three different probes. Under the heading for each probe the Vos  $t_s$  or start time, the Blackwell end time  $t_b$  and the E% error time  $t_i$  are all given in seconds. Many of the E% error times have been generated from the Solver sheets, resulting from actual studies. Where a particular combination of sample and probe has not been studied then the values followed by the letter a are calculated values. The final section of Table 1 shows the probe with the selected Solver routine and the appropriate time interval identified for the analysis of the data. It should be remembered that if the Solver 2.3 routine is applicable then so is the traditional regression analysis. Comparison of the regression analysis with the Solver 2.3 results enables a cross-check.

Before looking in detail at the results in this table, it is important to stress the pessimistic view of the start times  $t_s$  given by the Vos expression. Having observed the behaviour of a range of probe sample combinations, a time  $t_s$  of the order of 50 to 70 seconds are more acceptable, as was stated earlier. The importance of the diameter of the probe can be seen by comparing the start times for the stainless steel and copper probes with those for the fine stainless-steel probe.

To demonstrate the use of these times for selecting the appropriate Solver routine and the associated time intervals for the analysis, the examples in the table for unused mineral oil, Magna 904, paraffin wax and PTFE will be discussed.

(i). For the stainless steel probe in unused mineral oil the long time error  $t_l$  was 50s while the Blackwell end time  $t_b$  was 480s. This means that Solver 2.3 was applicable after 50 seconds and the axial errors would be greater than 1% after 480 seconds. However, after about 150 seconds of heating convection currents in the oil influenced the temperature time data from the probe. The rise in probe temperature per unit time became much smaller as convection removed the heat more rapidly. The graph of the rise in probe temperature versus natural logarithm of the elapsed heating time was linear up to about 130s. Therefore, the Solver 2.3 routine was applied between 80 and 120 seconds.

(ii). Before giving the relevant times for Magna, or Heatban, it is important to stress the unusual behaviour of this material and why it was chosen for study in this work. Magna 904 is manufactured by Magna International [13]. Magna 904 is a commercial material used in the automobile industry for controlling the heat flow away from remedial welding work in vehicle body panels. The material is designed to have relatively high thermal conductivity and high thermal capacity. As a paste the insertion of the probe should lead to good thermal contact and reproducible values of probe conductance  $H$ . The original aim was to use this paste as an intermediate step between liquid and solid samples. The supplier of Magna stated that the thermal conductivity was between  $0.61$  and  $0.63 \text{ Wm}^{-1}\text{K}^{-1}$ , and the manufacturer would not give any further details. This study began assuming that Magna would behave like water with conductivity of about  $0.60$  and high specific heat capacity. Some preliminary studies of the specific heat capacity of Magna, using a micro-calorimeter,

indicated that the specific heat capacity was  $2000 \text{ Jkg}^{-1}\text{K}^{-1}$  at  $30^\circ\text{C}$  rising rapidly to  $6000 \text{ Jkg}^{-1}\text{K}^{-1}$  at  $70^\circ\text{C}$ . This is a material that will have a variable specific heat when the probe temperature rises by a few degrees, and should be an interesting vehicle for these probe studies. The copper probe was used with Magna. This had the interesting effect of limiting the analysis time to a small interval, since here a very good conductive material as probe is in a specimen of a relatively good conductor of heat. It is therefore not surprising that the Blackwell axial error time was relatively short at 160 seconds. Initial applications of the Solver 4.3 routine showed that the error  $E\%$  was only less than 1% after a time  $t_i$  of 105 seconds. This means that the Solver 2.3 routine was applicable after 110 seconds and that the analysis should be completed by 150s. A further complication arose since the specific heat capacity will be increasing with the probe temperature during the probe measurements, and the time interval, and therefore the temperature rise should be reduced to a minimum. The Solver 2.3 routine was used over 40s from 110 to 150s, and the Solver 4.3 routine was used between 80 and 120s. These times are shown in Table 1.

(iii). For the stainless steel probe in paraffin wax the long time error  $t_i$  was 270s while the Blackwell end time  $t_b$  was 480s. This means that Solver 2.3 was applicable after 270 seconds and the axial errors would be greater than 1% after 480 seconds. The graph of rise in probe temperature against the natural logarithm of the elapsed heating time showed a linear section after about 250 seconds and both the regression and Solver 2.3 were applied in the interval 300 to 450 seconds. Here 300s as the start time is chosen to be greater than the 270s for the  $t_i$ , while the end time of 450 seconds is less than the axial error time  $t_b$  of 480 seconds. The Solver 4.3 routine was applied to the data between 60 and 400 seconds.

(iv). With the copper probe in the PTFE cylinder the long time error  $t_l$  was 140s while the Blackwell end time  $t_b$  was 310s. This means that Solver 2.3 was applicable after 140 seconds and the axial errors would be greater than 1% after 310 seconds. The Solver 2.3 routine was applied from 180 to 270 seconds, while the Solver 4.3 routine was applied from 100 to 200 seconds.

(v). For the Hukseflux probe placed in agar the long time error  $t_l$  was 60s, while the Blackwell end time  $t_b$  was 960s. The results for agar show that the regression analysis and Solver 2.3 can be applied after 60s have elapsed and that axial heat flow errors will only become significant after 960s. Both the Solver routines were applied to the agar data.

(vi). The results obtained for the Hukseflux probe in the clay-straw pads are more interesting. For the probe placed in clay-straw pads the long time error  $t_l$  was 630s, while the Blackwell end time  $t_b$  was 280s. This suggests that the regression analysis can be used after 630s, but by 280s the axial errors will be greater than 1%. The data for these pads cannot be analysed using the regression analysis, or Solver 2.3. However, Solver 4.3 was applied between 60s and 180s, where the potential axial errors will be minimised. It is reassuring that the study of these times, which suggests that no linear section is possible, is confirmed by the observed probe behaviour.

8A. Results: The thermal properties of unused mineral oil, Magna 904, paraffin wax, and PTFE.

The results of these studies will be given in two sections. The first will report studies of the thermal properties of mineral oil, Magna 904, paraffin wax, PTFE using simple stainless-steel and copper sheathed probes. The second section will report studies of agar and clay-straw pads using a commercial Hukseflux probe.

The sequence of results presented here, and shown in Table 2, illustrates the progression of these probe studies from liquid to paste, and to solids.

Unused mineral oil.

The thermal parameters measured for mineral oil were described in the first report, Goodhew et al [8]. The agreement between the thermal properties of oil as determined by the regression analysis and the Solver 2.3 routine is good, and the results are in agreement with the published data by Incropera and DeWitt [14] page 762.

For the mineral oil study a very early stainless steel probe was used with an additional external thermojunction to allow a rough estimate of the probe to specimen conductance  $H$ , see Goodhew et al [8]. This early work was marred by two factors; (i) an uncertainty in the probe heater power per unit length, and (ii) the very long time weak steady state is only an approximate method because of the axial errors.

Magna904

For the Magna 904 study only the thermal conductivity is available from the manufacturer, who quoted the conductivity as  $0.61$  to  $0.63 \text{ Wm}^{-1}\text{K}^{-1}$ . The error in the heater power would reduce the value presented here for the thermal conductivity of Magna 904 from  $0.66$  to  $0.58 \text{ Wm}^{-1}\text{K}^{-1}$  for the Solver 2.3 result and from  $0.74$  to  $0.65 \text{ Wm}^{-1}\text{K}^{-1}$  for the Solver 4.3 result. The mean value would then be  $0.62 \text{ Wm}^{-1}\text{K}^{-1}$  in agreement with the manufacturer's quoted figure. The observed specific heat capacity values, obtained from the probe studies, are in rough agreement with the values obtained using the micro-calorimeter. However, since Magna 904 has a strongly varying specific heat capacity with temperature commenting further on the results is pointless.

Paraffin wax

The most recent studies of paraffin wax with a stainless steel probe show an acceptable and encouraging agreement with the published values. The agreement between results from the two Solver routines of the thermal conductivity and the diffusivity is acceptable, as can be seen in Table 2. The thermal properties of paraffin wax reported by Incropera and DeWitt [14], page 756, are the thermal conductivity  $0.24 \text{ Wm}^{-1}\text{K}^{-1}$  and diffusivity  $9.2 \cdot 10^{-8} \text{ m}^2\text{s}^{-1}$ . The values obtained in this study are thermal conductivity  $0.24 \text{ Wm}^{-1}\text{K}^{-1}$  with thermal diffusivity of  $9.0 \cdot 10^{-8} \text{ m}^2\text{s}^{-1}$ . The agreement here is acceptable.

#### PTFE

The work on PTFE with and without an oil film to aid the thermal contact showed that there was no observable difference. The thermal conductivity and diffusivity of PTFE are given as  $0.25 \text{ Wm}^{-1}\text{K}^{-1}$  and  $9.0 \cdot 10^{-8} \text{ m}^2\text{s}^{-1}$  by the supplier, Goodfellow Cambridge Ltd. The values obtained here, conductivity  $0.29 \text{ Wm}^{-1}\text{K}^{-1}$  and diffusivity  $10^{-7} \text{ m}^2\text{s}^{-1}$  are high and this is partly due to the copper probe and partly due to the uncertainty in the heater power. Again, the results presented in Table 2 demonstrate that the observed thermal properties of PTFE from both Solver routines are in good agreement.

8B Results: The thermal properties of agar and clay-straw pads.

One aim of this work is to move the technique from the laboratory into the field and make thermal measurements on the cob walls found in Devon. The work with agar and the clay-straw pads allows a more critical demonstration and review of the technique, since agar and the clay straw pads represent the two ends of the thermal property range found in building materials.

Four objectives in this second section are identified. The probe was calibrated in a 1% by weight solution of agar in water. When immersed in the agar jelly, it is

assumed that the probe will be in reproducible thermal contact with the specimen. The observed thermal properties of this agar jelly should be those of water, and the resulting thermal data from the probe temperature profiles will allow three objectives to be met. Firstly, the probe can be calibrated in a material of known thermal parameters, and the probe to specimen conductance can be measured. Secondly, the objective recipe for determining the analysis time interval required for the Solver routines can be subjected to further close scrutiny and appraisal, and thirdly, both the Solver 2 and Solver 4 routines can be demonstrated. The fourth objective is to demonstrate the probe technique with the Solver routines applied to different materials with a range of thermal properties.

Figure 1 shows the rise in probe temperature plotted as a function of the logarithm of the elapsed heating time for agar and the clay-straw pads. The observed thermal behaviour of the Hukseflux probe in agar jelly (1% by weight of agar in water) was classical. That is after some 20s a plot of the rise in probe temperature against the natural logarithm of the elapsed heating time was linear, and a simple regression analysis was used to determine the thermal conductivity in the usual way.

The observed thermal behaviour when the probe was inserted into the 150mm cube clay-straw pad showed no linear section between 0s and 950s, as can be seen in figure 1. Attempts to find the thermal properties of the clay straw pads from this data using a regression analysis failed. Applying the Solver 4.3 routine to a portion of this data allowed some preliminary values of the thermal properties of the clay straw material to be obtained. Using this information the time interval for the Solver 4.3 analysis was determined more exactly.

Agar

Table 3 shows the results for the study of agar using the thermal probe TP02. A range of power per unit probe length was used, from  $2.28\text{Wm}^{-1}$  to  $9\text{Wm}^{-1}$ . The manufacturer recommends limiting the power to  $5\text{Wm}^{-1}$ . The rise in probe temperature recorded after 100s was between  $2.7\text{C}$  and  $8.7\text{C}$ , so the thermal properties of agar were measured at a temperature between  $23\text{C}$  and  $30\text{C}$ . The Solver 2.3 and Solver 4.3 routines were used with the analysis time intervals, starting at 60s and ending at 150s, and 200s respectively.

The results for agar, given in table 3, show that the Solver 4.3 values are slightly higher than the Solver 2.3. The thermal conductivity determined using the regression analysis of  $0.60\text{Wm}^{-1}\text{K}^{-1}$ , is in excellent agreement with Solver 2.3 value of  $0.60\text{Wm}^{-1}\text{K}^{-1}$ . Moreover, the Solver 2.3 agar diffusivity together with a density of  $1000\text{kgm}^{-3}$  produces a specific heat capacity of  $4050\text{Jkg}^{-1}\text{K}^{-1}$ , again in reasonable agreement with the standard value for water of  $4200\text{Jkg}^{-1}\text{K}^{-1}$ . The conductivity is determined to within about 2% of the published data for water, while the diffusivity is determined to within 5%. For the Solver 4.3 routine the conductivity is  $0.61\text{Wm}^{-1}\text{K}^{-1}$ , but the specific heat capacity is  $4400\text{Jkg}^{-1}\text{K}^{-1} \pm 700\text{Jkg}^{-1}\text{K}^{-1}$ , a value not so precise. The value of the probe to agar thermal conductance was  $480\text{Wm}^{-2}\text{K}^{-1} \pm 20\text{Wm}^{-2}\text{K}^{-1}$ .

#### Clay-straw pads

The clay-straw pads were produced as part of the Department of Trade and Industry “Partners in Innovation” programme to investigate the potential of “Light Earth Construction” for the UK, see Morgan [15]. The clay-straw pads were approximately 150mm cubes and consisted of cut lengths of straw in layers to the depth of 150mm. The straw was then fixed by allowing a clay water slip to drain through the pads. On drying the clay held the straw together to produce the 150mm cube. The probe was

easily pushed into the pads and the arrangement was allowed to reach thermal equilibrium before the heater power was applied.

With samples of this size it is important to check the Vos [10] reflected heat time  $t_r$ .

The clay straw pads were 150mm cubes and therefore the Vos reflection time  $t_r$  for the fine stainless steel probe placed at the centre of these pads was about 1000s.

Table 4 shows the results for the study of the clay-straw pads. Probe powers per unit length  $Wm^{-1}$  were well within the manufacturers recommended levels, but because the material is a good insulator the temperature rise at 100s was high at 15C for  $2.1Wm^{-1}$ .

There was no linear section in the data, and as a result only Solver 4.3 was used to analyse the data. The time window for the application of the Solver 4.3 routine was from 60s to 180s. The table shows that the thermal conductivity of the pads was  $0.073Wm^{-1}K^{-1}$  with standard deviation of  $0.001Wm^{-1}K^{-1}$ . Consistent values of the volumetric heat capacity of  $46.7 kJm^{-3} K^{-1} +/- 0.8 kJm^{-3} K^{-1}$  were found. Taking the measured density of the pads as  $110kgm^{-3}$  the specific heat capacity of the pads was  $424 Jkg^{-1}K^{-1} +/- 6 Jkg^{-1}K^{-1}$ . However, if the pads were installed in the gap between two leaves of earth walling for example, then the density could be increased by a factor of three due to the self-loading of the clay-straw. The fine stainless steel probe to clay straw pad conductance was  $175Wm^{-2}K^{-1} +/- 18Wm^{-2}K^{-1}$ . This conductance has a magnitude smaller than that found for the same probe placed in agar. This is attributed to the better thermal contact maintained between the probe and agar jelly compared with the thermal contact obtained in the clay-straw pads. It must be remembered that these pads were 30% air by volume.

Returning to the four objectives of this section, we conclude as follows. Firstly, the probe was calibrated in agar and gave values of the thermal conductivity and diffusivity for water which were acceptable. The thermal conductivity was within 1%

and the diffusivity within 3%. Moreover the probe to agar conductance was found to be  $480 \text{ Wm}^{-2}\text{K}^{-1} \pm 20 \text{ Wm}^{-2}\text{K}^{-1}$ . This is the first time that the parameter H has been routinely measured, as part of the thermal probe technique. Secondly, the recipe for determining the analysis time intervals required for the Solver routines was demonstrated and shown to be effective. Thirdly, both the Solver 2 and Solver 4 routines were demonstrated as robust and simple tools for the analysis of thermal probe data, when used with appropriate time intervals. The fourth objective was to demonstrate the probe technique with the Solver routines applied to different materials with a range of thermal properties. The thermal conductivity ranged from  $0.60 \text{ Wm}^{-1}\text{K}^{-1}$  for agar to  $0.073 \text{ Wm}^{-1}\text{K}^{-1}$  for the pads. For these two materials it may be said that the agar is a fairly good conductor of heat, in built environment terms at least, while the clay-straw is a good insulator. This study, then, provides an important and significant test of the proposed Solver routines and the probe technique for simultaneously determining the thermal conductivity and thermal diffusivity of both poor and medium thermal conductors. It meets the fourth objective.

## 9. Conclusions.

Finally, some conclusions and the proposed method for the field studies.

Ideally, the temperature rise in the probe should be as small as possible so that the thermal properties are measured at the initial sample temperature. However, practically the rise in the probe temperature should be large enough to ensure an accurate result. Since the Solver routine looks for the difference between the theoretical and experimental rise in probe temperature, finite increases in the experimental rises are essential. The agar studies undertaken with a series of different heater powers suggests that the rise in temperature should be of the order of

3C to 6C. For example, the agar curve in the graph shows that the overall rise in temperature of the probe is about 10C, while the temperature rise is about 1C between 50s and 150s. This temperature profile is proposed as the optimum experimental arrangement.

One disadvantage of using this probe on cob walls in the field will be the need to machine a hole of length 150mm and 1.5mm in diameter in a material where the density changes abruptly and unpredictably. A material like a mixture of clay with woodchips would not be as difficult as cob, a mixture of clay, sand and aggregates. When drilling cob the drill bit is likely to encounter extremely hard flint like materials and be deflected.

Emerging from these studies is a recipe for the application of the thermal probe technique to the cob walls of Devon that is inexpensive, relatively non-destructive, and simple. Fine probes constructed with stainless steel tubes are required, preferably 100mm long or shorter, and with an external diameter of no more than 1.5mm. This will lead to high values of the length to diameter ratio of about 60 that will reduce the axial errors, thereby increasing the analysis time interval. Experience gained so far suggests a cyclic experimental and analytical method should be adopted, so that feed back can be used to gain high quality results. A trial run with the probe at relatively low power is required to assess the approximate values of the thermal parameters. With the approximate values of the thermal conductivity and diffusivity of the sample determined in this trial, the time intervals for the two Solver routines can be determined. Then the power per unit length to the probe heater can be found, that will give about 1 to 2C rise in probe temperature over the time interval 50 to 200 seconds. Following the preliminary stage the thermal behaviour of the probe in the material can be studied carefully, using the Solver 4.3 routine to determine not only the thermal

properties of the sample, but also the error time  $t_i$ . Then the choice of the appropriate Solver routine can be confirmed, use either Solver 2.3 or Solver 4.3 with the associated time interval for the analysis. As the Solver 2.3 routine is independent of the probe thermal capacity it is the favoured routine. Finally, with the appropriate Solver routine and analysis time interval the thermal properties of the sample can be determined with confidence. It is important to remember that the technique does require careful driving and the use of feedback. We have found that the important error times,  $t_i$  and  $t_b$ , can vary appreciably between the preliminary probe trials and the final probe-sample data collection and analysis. Although in effect the experimental data must be analysed twice, the speed with which the Solver routines operate more than out weighs this delay.

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