

## Assessment of long time approximation equation to determine thermal conductivity of high porous materials with NSS probe

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### Abstract

Recent economic changes have increased the focus on energy conservation in buildings. Improve insulation represents one of the biggest challenge to save energy. The TP02 Hukseflux® Non Steady State Probe (NSSP) has been used to determine the thermal conductivity of the building insulation materials. Usually, the long term approximation equation (linear form) is applied to determine the thermal conductivity when using hot wire techniques. Long term approximation has been successfully used to characterize glycerol: a fluid which doesn't present contact resistance and porosity. But, an S-shaped appears for more porous materials. So to generalize the possibility of using this method, glass beads of 2, 8 and 10 mm balls diameters have been tested before characterising insulation materials. Firstly, we define the best portions in time ( $t_1$  and  $t_2$ ) of the S-shaped curve to determine thermal conductivity. Secondly, Comsol Multiphysics® test the influence of parameters of the TP02 Hukseflux® like components of the probe, the contact resistance and electrical power on the S-shaped form.

### Nomenclature

$T$	temperature, $K$	$H$	conductance of the air gap, $W.m^{-2}.K^{-1}$
$t$	time, $s$	$M$	thermal mass of the probe
$r$	the distance from the heat source, $m$	<b>Symboles grecs</b>	
$Q$	lineic electrical power, $W.m^{-1}$	$\gamma$	Euler's constant (0.5772157...)
$R_s$	radius of the probe, $m$	$\lambda$	thermal conductivity, $W.m^{-1}.K^{-1}$
$R_c$	contact resistance ( $= 1/H$ ), $m^2.K.W^{-1}$	$\alpha$	thermal diffusivity, $m^2.s^{-1}$
$C_p$	specific heat, $J.kg^{-1}.K^{-1}$	$\rho$	density, $kg.m^{-3}$

### 1- Introduction

The world currently faces its greatest challenge ever in diminishing natural resources and increasing pollution. Now the largest environmental problem is the atmosphere, where the accumulation of greenhouse gases involves noticeable climate change. The latest report of the International Panel on Climate Change (IPCC) concludes that the increasing carbon dioxide in the atmosphere is the main cause of climate change. The main sector to emit CO<sub>2</sub> in Europe is buildings, besides transport and industry. We are in an age of great innovation,

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where we must find solutions to reduce CO<sub>2</sub> emission and define energy efficiency strategies. Thermal insulation gave us one of the greatest potential for CO<sub>2</sub> reduction compared to other building efficiency measures.

40 to 50% of European energy is dedicated to buildings, and 40 to 60% of this is heating energy. We can reduce this consumption for new building by good design and optimum levels of insulation, but for existing building it requires renovation insulation and glazing. The amount of energy required to cool and heat a building depends on how their structure are thermally designed [1]. The thermal performance of building is determined by the thermal characteristics of the materials.

To access thermal characteristics of insulation materials, in situ measurements, and not just in laboratory conditions, can be considered an important issue. To determine these characteristics in use, only the transient techniques are possible because of their quick measurement, their portability and their relative low cost. They are also interesting because of the low power they need making possible the study of moisture content influence. Hooper and Lepper [2] have already pointed the interest in term of moisture distributions by using Non Steady State Probe (NSSP) compared to Steady State technique like the guarded hot plate technique. Studying insulation materials, Joy [3] found that the linearity of the temperature increase ( $\Delta T$ ) against the logarithm of time was not always achievable if the moisture was not uniformly distributed in the sample and sometimes an S-shaped curve is observed. Batty [4] notes that this S-shaped curve appears not only for moist materials but more generally for mineral wool insulation (19.7 kg/m<sup>3</sup>) even if these materials are dried. Even if hot wire and thermal probe techniques are considered by Hust and Smith in 1989 [5] suitable to determine thermal conductivity of insulation materials (fibre glass insulation, extruded and expanded polystyrene, paraffin wax), Hukseflux actually considers that TP02 technique is not appropriated for characterization of insulation materials (or high porous materials). Hukseflux restrains the use of their probe to a range of thermal conductivity between 0.1 and 6.0 W.m<sup>-1</sup>.K<sup>-1</sup> and defines an expected accuracy at 20 degrees of  $\pm (3\% + 0.02)$  W.m<sup>-1</sup>.K<sup>-1</sup>. The fixed accuracy of 0.02 doesn't allow insulation material characterization with usual thermal conductivity of 0.04 W.m<sup>-1</sup>.K<sup>-1</sup>. Pilkington et al. [6] confirm this point and conclude that the probe technique is unsuitable for materials with thermal conductivity less than 0.07 W.m<sup>-1</sup>.K<sup>-1</sup> because of the nonlinear temperature increase against the logarithm of time.

The objective of this work is, at first, to identify technological barriers **to explain why the experimental kinetic reflecting the temperature increase against the logarithm of time is linear for some materials and non-linear for** insulation materials. Second objective is to suggest improvements in order to determine a well-adapted probe for these high porous materials. Comsol Multiphysics® program has been developed to study the influence of parameters such as the components of the probe (**composition, size ...**), the contact resistance and the thermal properties of materials to reduce S-shaped form.

## 2- Theory of the thermal probe

The non-steady state probe (NSSP) method for thermal conductivity measurement assumed an infinitely long line heat source inserted in an infinite and homogeneous medium and is expressed by the general Fourier equation (1):

$$\frac{\partial T}{\partial t} = \alpha \nabla^2 T \quad (1)$$

Subject to the initial condition: at  $t \leq 0$ ,  $\Delta T(r, t) = 0$

For boundary condition:

at  $r = 0$  and  $t \geq 0$ ,

$$\lim_{r \rightarrow 0} \left[ \frac{rdT}{dr} \right] = - \frac{Q}{2\pi\lambda} \quad (2)$$

at  $r = \infty$  and  $t \geq 0$ ,

$$\lim_{r \rightarrow \infty} [\Delta T(r, t)] = 0 \quad (3)$$

The temperature response of the heat source over time with heat is maintained at a constant rate can be described by Carslaw and Jaeger solution [7]:

$$\Delta T = -\frac{Q}{4\pi\lambda} Ei [u] \quad (4)$$

where

$$u = \frac{r^2}{4\alpha t} \quad (5)$$

The exponential integral function ( $E_i$ ) can be expressed by a series expansion [8]:

$$-E_i(-u) = \int_u^\infty \left[ \frac{e^{-u}}{u} \right] du \quad (6)$$

where

$$-E_i(-u) = -\gamma - \ln(u) - \frac{(u^2)}{2.2!} + \frac{(u^3)}{2.3!} - \frac{(u^4)}{2.4!} + \dots \quad (7)$$

Carlsaw and Jaeger [7] found solutions for equation (1) after considering the assumptions of constant heat flow ( $Q$ ), constant thermal properties, negligible the heat conduction in the radial direction and negligible thermal mass of the heater.

$$\Delta T = -\frac{Q}{4\pi\lambda} \left[ -\gamma - \ln(u) - \frac{(u^2)}{2.2!} + \frac{(u^3)}{2.3!} - \frac{(u^4)}{2.4!} + \dots \right] \quad (8)$$

For small  $u$  value (equivalent to the large values of time), the terms after logarithmic term can be neglected and the temperature change can be approximated by:

$$\Delta T = \frac{Q}{4\pi\lambda} \left[ \left[ \ln \left( \frac{t_2}{t_1} \right) \right] + B \right] \quad (9)$$

where

$$B = \ln \left\{ \frac{4\alpha}{r^2} \right\} - \gamma + \frac{2\lambda}{rH} \quad (10)$$

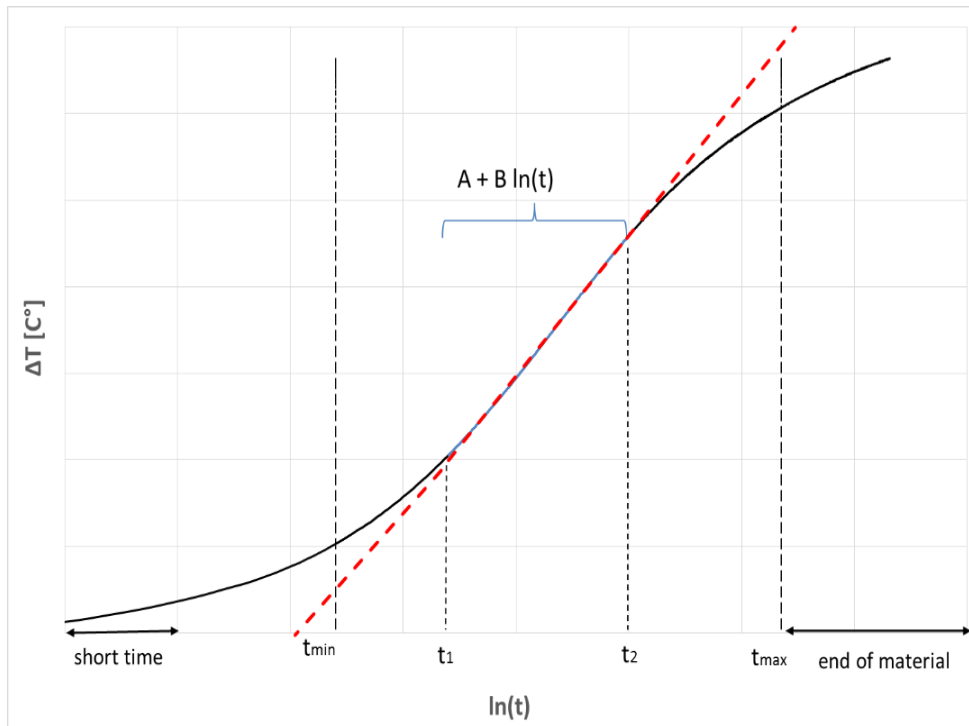
with  $H$  the air gap thermal conductance .

If the early time data are ignored, a graph of  $\Delta T$  against  $\ln(t)$  becomes straight line with a slope equal to  $\frac{Q}{4\pi\lambda}$ , then the two points define a straight line and the thermal conductivity could be found from:

$$\lambda = \frac{Q}{4\pi} \left[ \frac{\ln(t_2/t_1)}{T(t_2) - T(t_1)} \right] \quad (11)$$

The thermal conductivity can then be determined from the slope ( $Q/4\pi\lambda$ ). The intercept  $B$  (equation 9) should determine the thermal diffusivity whether the approximation at long times is satisfied but also whether the conductance  $H$  of the air gap (the inverse of the contact resistance) is known. In practice, the value of  $H$  varies greatly depending on the quality of the contact with the surrounding material requiring a preliminary calibration [9]. Moreover there is a gap between the experimental results and the values from equation 11 allow the determination of thermal conductivity only and not determinate thermal diffusivity.

For a fluid material such as glycerol, the temperature variation with the logarithm of the time is linear so equation 11 can be easily used to determined thermal conductivity but for insulation materials (example in Figure 1 for glass wool) this variation is non-linear so we must select the values of  $t_1$  and  $t_2$  to determine thermal conductivity.



**Figure 1.** Variation of temperature against natural logarithm of the time for glass wool.

For short time ( $< t_{\min}$ ): an initial phase after heating is happening due to the heating through the probe materials. This period is caused by the thermal capacity of the probe, contact resistance and a thermal imbalance between the temperature of the probe and the material. It depends on the thermal properties of the probe and its surrounding medium. Vos [10] defined the duration of this nonlinear transition period by:

$$t_{\min} = \frac{50 \cdot (R_s)^2}{4 \cdot \alpha} \quad (12)$$

where  $R_s$  is the radius of the probe.

For long time ( $> t_{\max}$ ): this nonlinearity can be attributed to the axial losses at the end of the probe and heat exchange with the surrounding atmosphere (when the heating reaches the outer limits of the material). Vos [12] established time  $t_{\max}$  by:

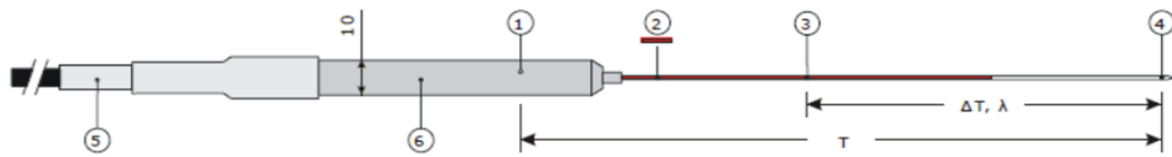
$$t_{\max} = \frac{0.6 (r - R_s)^2}{4 \cdot \alpha} \quad (13)$$

where  $r$  is the sample radius.

Figure 1 represents variation of temperature against natural logarithm of the time for insulation materials (glass wool). To apply equation 11 in order to determine thermal conductivity, we must select experimental values between  $t_1$  and  $t_2$  where there is a linear part of the curve according to the ASTM recommendation [11].

### 3- Thermal probe of Hukseflux® TP02

Our tests were carried out from the TP02 probe of Hukseflux® (Figure 2) with a length of 150 mm and a diameter of 1.5 mm. It is composed of several layers of material in the radial direction, whose properties are shown in Table 1. Note that the hot wire provides heat only on 2/3 of the probe length.



**Figure 2.** TP02 Hukseflux® probe

Hukseflux® TP02 has a length to radius ( $L/2R_s$ ) ratio of 100. This ratio is enough to overcome the axial heat losses and respects Blackwell [12] recommendation (minimum  $L/2R_s$  value of 25) in order to reduce losses in the axial direction of the probe. Hot wire heater is made from constantan. This material has been chosen because of its high electrical resistivity and its low temperature coefficient. Stainless steel has been used for the outer face due to its low thermal diffusivity.

Table 1: Thermal properties of the layers of the probe.

Details of layer	Thickness [mm]	Thermal conductivity [W.(m.K) <sup>-1</sup> ]	Density [kg.m <sup>-3</sup> ]	Specific heat [J.(kg.K) <sup>-1</sup> ]
Constantan (hot wire)	0.065	19.50	8910	390
Glass pearl (insulant)	0.355	0.16	1600	800
Stainless steel (Outer face)	0.330	16.00	7900	500

Temperature measurements are made via the hot junction positioned 50 mm from the base (thermocouple 3 in figure 2), the cold junction at the end of the probe (thermocouple 4 in figure 2). These thermocouple junctions are connected, producing a voltage output  $U_{sen}$ , proportional to the differential temperature of the two junctions. TP02 has a reference temperature sensor (Pt<sub>1000</sub>) built into the base of the probe. This reference serves as a “cold junction” measurement for establishing the absolute medium temperature  $T$ . The base temperature (the Pt<sub>1000</sub>) is used as a cold junction to compensate the temperature for the cold thermocouple junction at the tip. The main sensor signal is  $\Delta T$ , the differential temperature, measured between the hot and the cold junctions by:

$$\Delta T = \frac{U_{sen}}{E_{sen}} \quad (14)$$

where the temperature dependence ( $\Delta T$ ) of the thermocouple thermoelectric power is determined by  $E_{sen}$  ( $\mu V.K^{-1}$ ) defined by measuring the base temperature  $T$  using the Pt<sub>1000</sub> as:

$$E_{sen} = 39.40 + 0.05 T - 0.0003 T^2 \quad (15)$$

#### 4- Experimental measurements with TP02 for different materials.

The TPSYS02 control interface allows the power setting test conditions and visualization of measurement during time data acquisition. An electric power per unit length  $Q$  is kept constant during the duration of the test.

##### 4-1- Glycerol test

Glycerol has been chosen to calibrate the experimental process. This fluid is considered by Hukseflux as a reference material. Fluids are interesting to calibrate because they present no contact resistance and no porosity. Test has been done in a tube of 2.2 cm diameter. According to glycerol thermal diffusivity of  $9.10^{-8} \text{ m}^2.\text{s}^{-1}$ , a maximum time ( $t_{max}$ , equation 13) of 180s has been determined [13 and 14]. Linear variation of increase temperature against logarithm of time is observed (Figure 3). Applying the  $4.44 \text{ Wm}^{-1}$  high flow, a slope of

1.1863 is obtained for time between  $t_{\min}=10$ s and 150s. The slope value is then introduced in equation 11 to determine thermal conductivity.

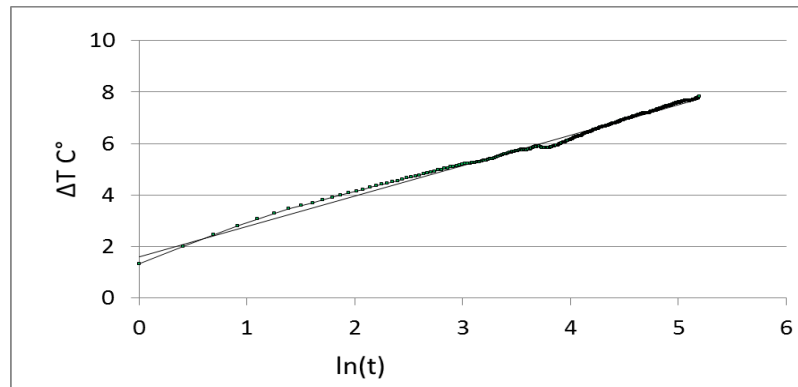


Figure 3: Variation of temperature against natural logarithm time for glycerol heat flow: 4.44 W/m

Maximum time and heat flux values have been modified. Thermal conductivity reference value (handbook) for glycerol at 25 ° C is  $0.29 \text{ W (m.K)}^{-1}$ . Considering the expected accuracy defined by Hukseflux at 20 degrees of  $\pm (3\% + 0.02) \text{ W.m}^{-1}.\text{K}^{-1}$ , experimental values are consistent with the reference value. However, the lower heat flux generates the most important difference with the reference value.

Table 2: Experimental results for thermal conductivity ( $\text{W (mK)}^{-1}$ ) of glycerol

Test time [s]	High heat flux [4.44W.m <sup>-1</sup> ]	medium heat flux [2.64 W.m <sup>-1</sup> ]	Low heat flux [0.87 W.m <sup>-1</sup> ]
60	0.30± 3%	0.31± 3%	0.34± 3%
120	0.30± 3%	0.31± 3%	0.33± 3%
180	0.29± 3%	0.30± 3%	0.34± 3%
240	0.28± 3%	0.30± 3%	0.32± 3%

#### 4-2- Glass beads test

Three diameters (2 mm, 8 mm and 10 mm) glass beads have been studied (Figure 4). For each bead type, the total porosity by water saturation (Table 3) is determined in a well-defined volume. Porosity increases with diameter of glass balls. These experimental values of porosity are in agreement with the literature [15, 16].

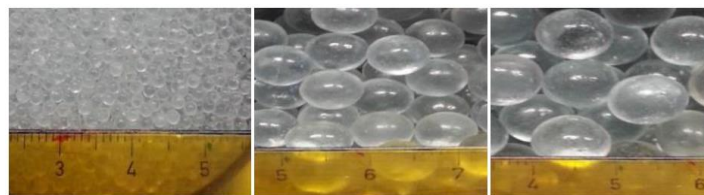


Figure 4: Glass beads 2, 8 and 10 mm diameters

A low heat flux of  $0.87 \text{ W.m}^{-1}$  and maximum time of 600s have been defined as test conditions. For 2mm diameter glass balls, a linear curve is observed (Figure 5). But, non-linear variation in form of S-shaped curves is obtained for glass diameters of 8 and 10 mm. The S-shaped form can be assigned to heat transfer conduction and thermal inertia contrasts in the air and in the glass bead and the edge effect. Taking into account all these phenomena, thermal conductivity can be considered as an equivalent value.

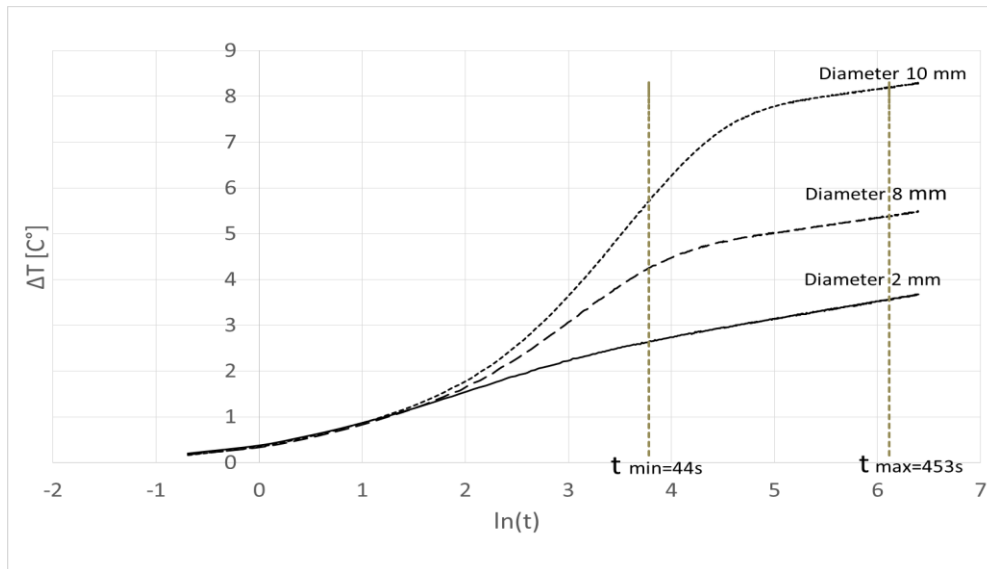


Figure 5: Variation of temperature against natural logarithm time for the different glass beads diameter with heat flow 0.87 W/m.

Thermal conductivities have been found from the S-shaped curve time period between  $t_{\min}$  and  $t_{\max}$  defined respectively by equation 12 and equation 13. Thermal conductivity's values decrease when porosity increases which is scientifically checked. Coherent value has been found in literature [15, 16] with our experimental values for 2mm glass bead diameter. But, for 8 and 10 mm diameters no value has been found in literature. So, measurement using guarded hot plate has been done for 8 mm diameter.

Table 3: Properties of glass beads

Glass beads diameter mm	Total porosity %	Thermal conductivity (measured) $W (mK)^{-1}$	Thermal conductivity (reference) $W(mK)^{-1}$
2	36.55	$0.18 \pm 3\%$	0.17 [16] and 0.20 [15]
8	40.30	$0.18 \pm 3\%$	0.14*
10	41.00	$0.09 \pm 3\%$	

(\*) Determined in the laboratory by using the guarded hot plate method

#### 4-3- Study of different insulating materials

Insulation materials representative of the main categories of sold insulation have been chosen: powdery mineral insulator (vermiculite) or panel (glass wool) and synthetic insulation of chemical (XPS). Vermiculite is a powdered material having a porosity of about 90% and the size of the grains is between 1 and 4 mm. The glass wool is a structured material with fiber assemblies thereby obtaining a higher porosity (about 96%) [17]. Extruded polystyrene has a porosity similar to the glass wool but the pores are closed thereby reducing the transfer of heat into the air (Table 4). The maximum time (1500s) allowed by the TPSYS02 control interface has been selected. This value respects maximum time conditions (Equation 13).

Table 4: properties and time intervals ( $t_{\min}$ ,  $t_{\max}$ ) for thermal characterization insulation materials

Insulation material	Diffusivity $m^2 \cdot s^{-1}$	Porosity %	$t_{\min}$ s	$t_{\max}$ s
Vermiculite	$69 \cdot 10^{-8}$	90	10	527
Glass wool	$63.5 \cdot 10^{-8}$	96	11	573
XPS	$56 \cdot 10^{-8}$	96	13	650

As before for glass beds, low heat flux has been applied on 10 cm diameter cylindrical form samples. Non-linear variation in S-shaped form appears for each curve of insulation material (Figure 6). Due to the more

important value of porosity, gradient of temperature is higher for insulation materials than for glass beds. It is appropriate to be careful because the radiative phenomena can intervene in this case for glass wool.

Note that Hakansson et al in 1988 [18] already observed the non-linearity of the  $\Delta T/\ln t$  curve for thermal conductivity less than 0.07 W/ (m.K). He attributes the origin of the S-shaped curve to the thermal diffusivity of the samples. For Pilkington and Grove [19] this non-linearity depends on the pores of the cellular structure of the materials. They remark the shape of the curve is S-shape for polyisocyanurate foam (PIR) ( $0.018 < \lambda < 0.025$  W/ (m.K)) and linear for the polytetrafluoroethylene (PTFE) (thermal conductivity of 0.25 W/ (m.K)). For Batty et al. [6], the structure of the material is less important than the contact resistance. They reveal the importance of the contact resistance between the probe and the glass wool fibers compared to the contact between the fibers. In addition to these parameters, the non-linear variation in form of S-shaped curves is affected by the contrast capacity ratio of the thermal mass of the materials to the thermal mass of the probe (M) where  $M = (\rho.C_p)_{material} / (\rho.C_p)_{probe}$  [20]. This ratio is very low for insulation materials (0.014 for glass wool, 0.012 for XPS and 0.025 for vermiculite) while this ratio is important 0.78 for glycerol (linear variation).

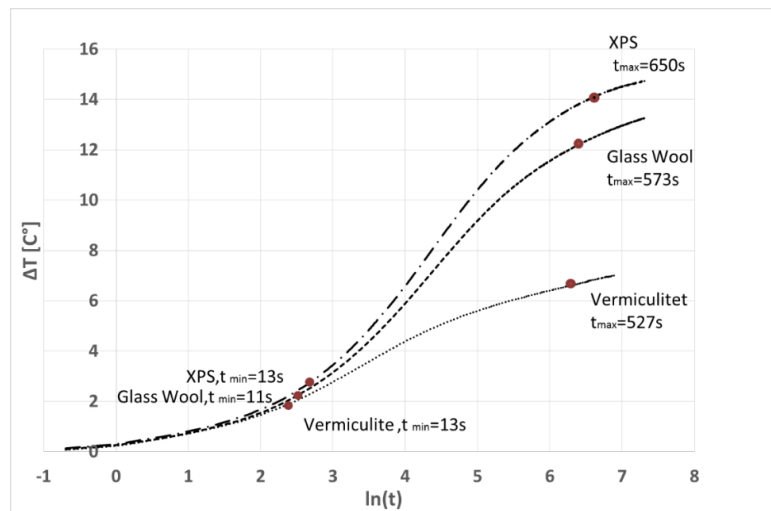


Figure 6: experimental kinetic different insulating materials at time 1500s and low flow  $0.87 \text{ Wm}^{-1}$

To determine thermal conductivity, we have defined three cases. First case (time 1), the entire kinetic has been selected to determine the slope ( $0 < \ln(t) < \text{end}$ ). Second case (time 2), according to Vos [10], we consider the part between  $t_{min}$  and  $t_{max}$ . Third case (time 3), according to ASTM recommendation [11], we chose  $t_1$  and  $t_2$  to obtain the best straight line between  $t_{min}$  and  $t_{max}$ . Despite these considerations and considering the uncertainty of measurement defined by Hukseflux ( $\pm 3\% + 0.02$ ), a good consistency between the measured values and those from the literature is found (Table 5) when we take all the time ( $0 < \ln(t) < \text{end}$ ) on the S-curve to find thermal conductivity for insulation materials.

Table 5: measured and reference thermal conductivity values of insulation materials

Insulation material	Thermal conductivity (measured)[W(mK) <sup>-1</sup> ]	Thermal conductivity (reference)[W(mK) <sup>-1</sup> ]	Time intervals
vermiculite	$0.071 \pm 3\%$	0.076 (*)	<b>Time 1 :</b> $0 < \ln(t) < \text{end}$
Glass wool	$0.033 \pm 3\%$	0.035 (**)	
Extruded p.(XPS)	$0.029 \pm 3\%$	0.028 (**)	
vermiculite	$0.045 \pm 3\%$	0.076 (*)	<b>Time 2 :</b> $t_{min} < \ln(t) < t_{max}$
Glass wool	$0.026 \pm 3\%$	0.035 (**)	
Extruded p.(XPS)	$0.023 \pm 3\%$	0.028 (**)	
vermiculite	$0.052 \pm 3\%$	0.076 (*)	<b>Time 3 :</b> $30s < \ln(t) < 150s$
Glass wool	$0.021 \pm 3\%$	0.035 (**)	
Extruded p.(XPS)	$0.018 \pm 3\%$	0.028 (**)	

(\*) Determined at the laboratory by using the guarded hot plate method

(\*\*) Technical agreement value issued from the manufacturer of the product



For glass wool,  $t_1$  is equal to 30s and  $t_2$  is equal to 150s. The experimental results showed that the first case ( $0 < \ln(t) < \text{end}$ ) was more accurate and closed to the references value ( $\lambda = 0.033 \text{ W (m.K)}^{-1}$ ). While the other cases give us values of  $\lambda = 0.026 \text{ W (m.K)}^{-1}$  and  $\lambda = 0.021 \text{ W (m.K)}^{-1}$  respectively as shown in figure 7.

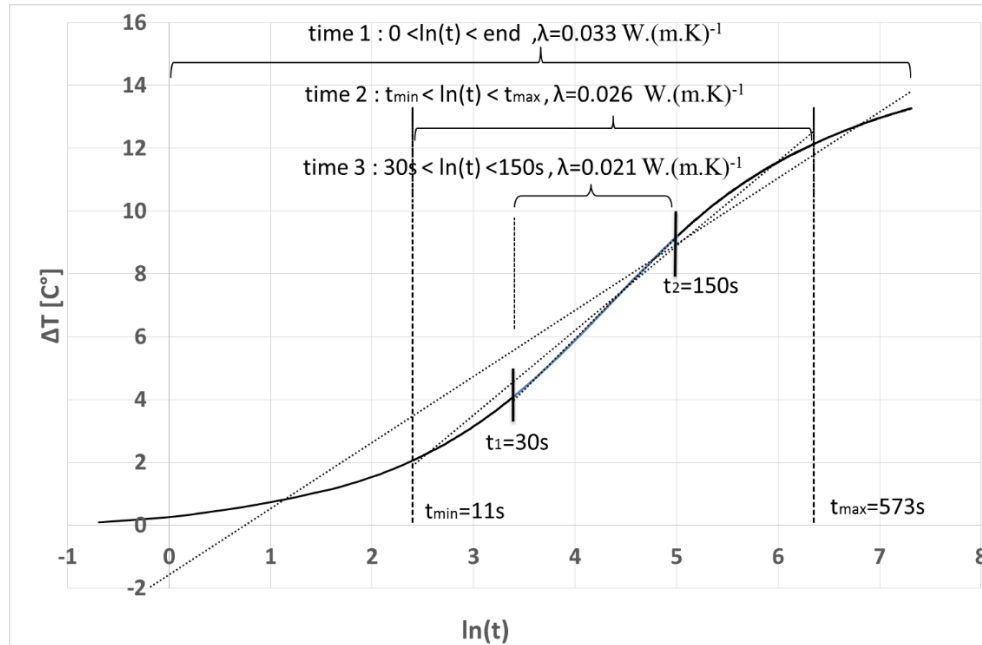


Figure 7: three ways to select  $\ln(t)$  on the S-curve to find thermal conductivity for glass wool.

To complete results from experimental process, Comsol Multiphysics® software has been used. Parameters that cannot be reachable experimentally such as the length to diameter ratio and the effect of changing components of the probe have been studied.

## 5- Numerical modelling of the probe response for glass wool.

Hukseflux® TP02 probe was modelled with Comsol Multiphysics® software to compare experimentally and by simulation the temperature response of the probe. In Comsol, a sensor has been positioned at the probe-material interface representing hot temperature measurement. Axisymmetric module 2-D performing the study in a plane (2D) has been selected. In this module, the radial and the axial directions of the heat transfer are followed and an axis-symmetry in the middle of the probe is considered (Figure 8). Each layer or component of the TP02 probe has been reproduced making possible the study of the effect of changing components of the probe. Physical characteristics of materials have been found in the database of the software. The same power heat flow ( $0.87 \text{ W.m}^{-1}$ ) corresponding to the low experimental heat flux and maximum time (1500s) allowed by the TPsys02 interface have been chosen. To define the boundary conditions, ambient air of  $20 \text{ }^\circ\text{C}$  has been considered surrounding the sample and at the surface of the probe heat power per unit area of the cross section ( $\text{W.m}^{-1}$ ) have been introduced corresponding to the experimental heat flow used. An automatic mesh is realized by Comsol representing the normal size of each component as shown in Figure 9. To represent experimental conditions, a 1 mm air gap has been considered in order to take into account the contact resistance between the probe and sample.

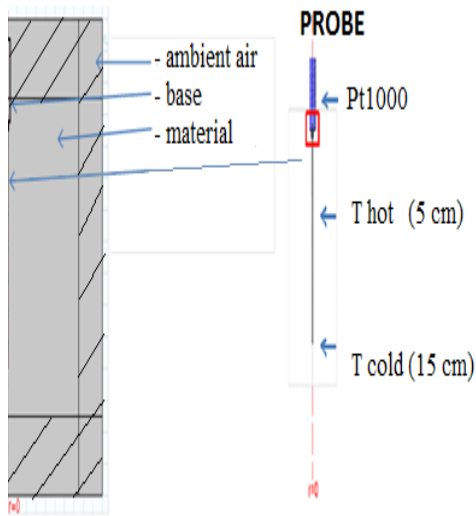


Figure 8: Probe and its environment

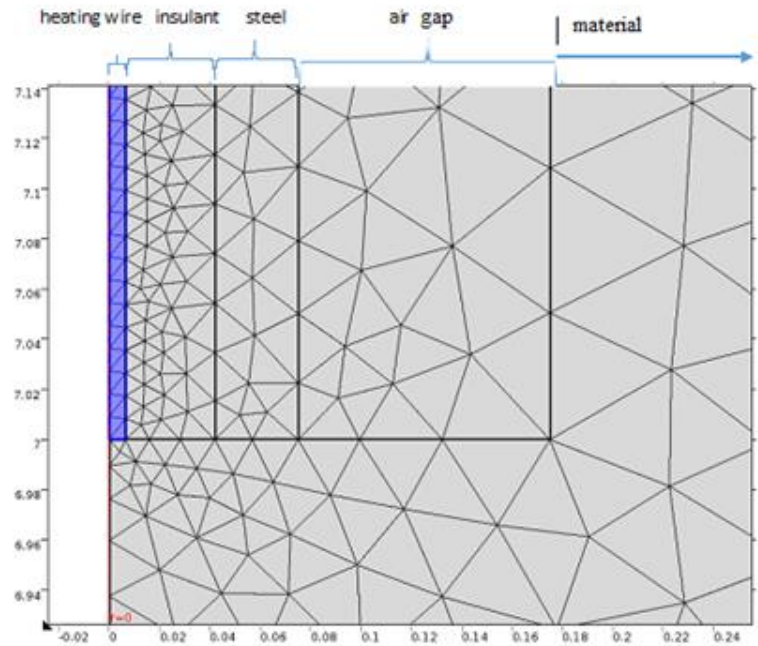


Figure 9: Mesh used in COMSOL model.

### 5-1- Validation of the experimental measurement

Simulation curve has been adjusted to experimental results by changing parameters like contact resistance (we used 1 mm air gap as contact resistance between the probe and the sample). The lack of adjustment can be attributed to difference in the experimental conditions not considered by Comsol® and numerical assumptions used by Comsol. Experimental difference can be assigned to moisture conditions, radiation effects inside the glass wool, accuracy and uncertainty of temperature measurement devices. However, due to the slight difference the simulation approach has been considered validated with experimental values (Figure 10).

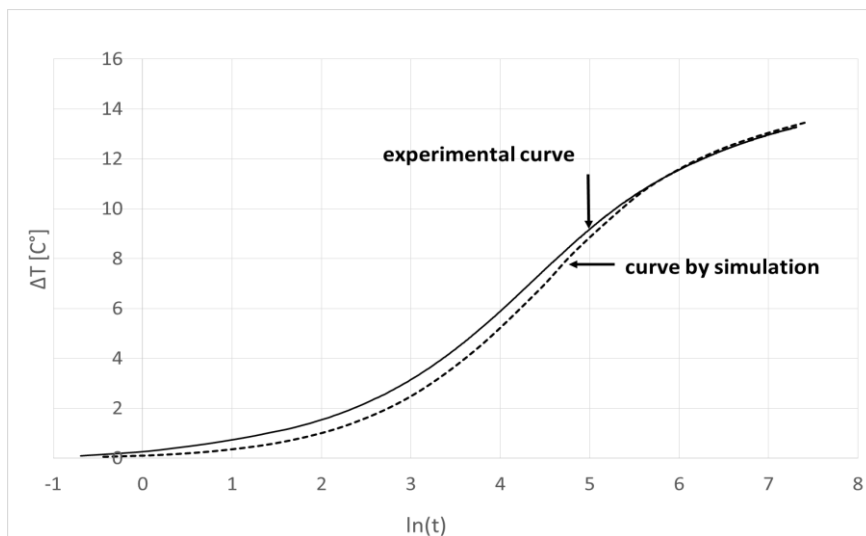


Figure 10: Validation of experimental measurement with simulation of COMSOL for glass wool.

### 5-2- Influence of length / diameter (L / D) of the probe

Blackwell [12] has defined a length to diameter ( $L/D$ ) minimum ratio of 25 in order to reduce heat losses in the axial direction of the probe. For  $L/D$  ratio equal to 25, we observe (Figure 11) important axial heat losses in the base-material interface (upper part of the probe) and in the other direction (end of the probe). For  $L/D$  ratio of 100 corresponding to TP02 probe used for experimental runs, we just observed axial heat losses in the upper part of the probe due to the stainless steel (this metal is used to make the base of the probe). It would be preferable to use a more thermally insulating material for this base. As we note that heat is foremost loss from the upper part of the probe, we suggest to follow the temperature from the measurement of the  $Pt_{1000}$  and to keep its increase lower than  $1\text{ }^{\circ}\text{C}$ .

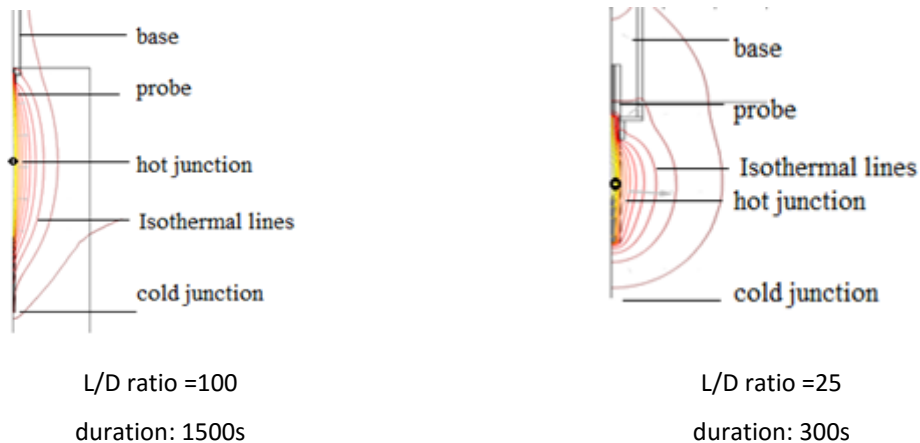


Figure 11: Influence of ratio  $L/D$

### 5-3- Influence of heat power

According to the TP02 probe of Hukseflux® specifications, temperature increase at the outer surface of the probe must be less than  $1\text{ }^{\circ}\text{C}$ . By using the lower heat flux, experimentally we can't respect a temperature increase less than  $1\text{ }^{\circ}\text{C}$ . ASTM Standard D 5334-08 [11] recommended for soil and soft rock that the temperature in the medium must be less than  $10\text{ }^{\circ}\text{C}$  in 1000s. To reduce effect of radiative transfer inside fibrous media especially, temperature variation of  $5\text{ }^{\circ}\text{C}$  maximum must be checked. Van der Held in 1952 [21], [22] observed that the radiation occurs easily in materials which have high porosity. Temperature variation can be reduced by limiting energy and therefore by decreasing the heat flux and the time. Woodside in 1958 [23], Eschner et al in 1974 [24], Pilkington in 2008 [6] and R. Coquard in 2006 [25] also observed this phenomenon.

According to Comsol Multiphysics® simulation (Figure 12), temperature increase less than  $1\text{ }^{\circ}\text{C}$  is only obtained for heat flux lower than  $0.07\text{ W}\cdot\text{m}^{-1}$ . But, this heat power can't be produced by TP02sys interface (the lower heat power level is  $0.87\text{ W/m}$ ). We can note also that this  $0.07\text{ W}\cdot\text{m}^{-1}$  heat flux reduces the S-shaped form of temperature against natural logarithm of the time and the finally obtained thermal conductivity  $0.0347\text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$ , is coherent with the reference value ( $0.035\text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$ ).

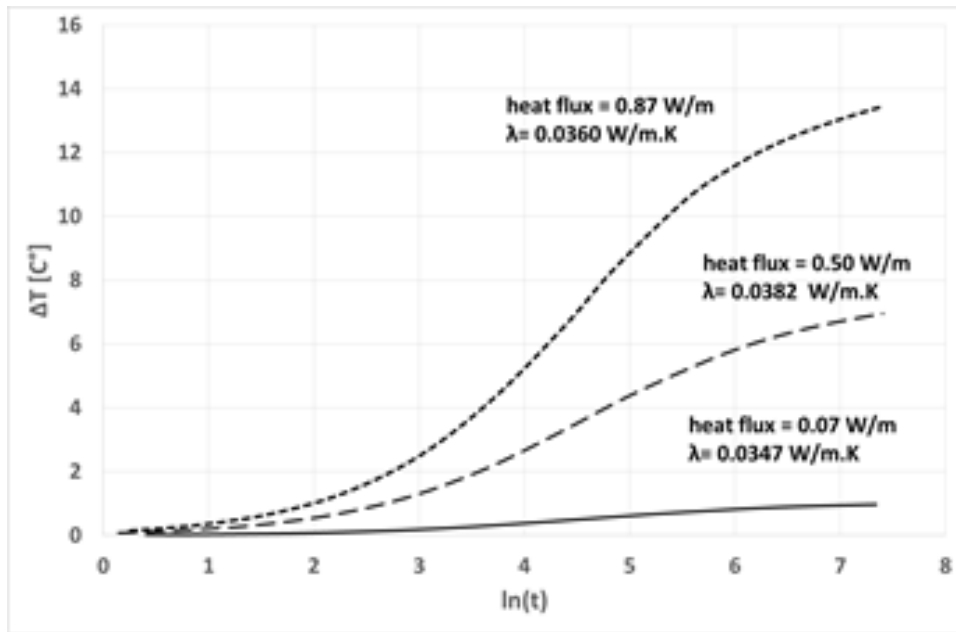


Figure 12: Results for very low heat flow on glass wool

5-4- Phenomena at outer surface of the probe

Hukseflux have used stainless steel as components for the outer surface of the probe at the interface probe-studied material. This metal has the advantage of a lower thermal diffusivity increasing inertia during the heat transfer. But we observed important temperature increase even if a low heat flux is produced (Figure 13). To reduce S-shaped form and to increase temperature at the outer surface of the probe, nickel can be used as Laurent [26] concluded. But, physical characteristics of copper seem to be more interesting.

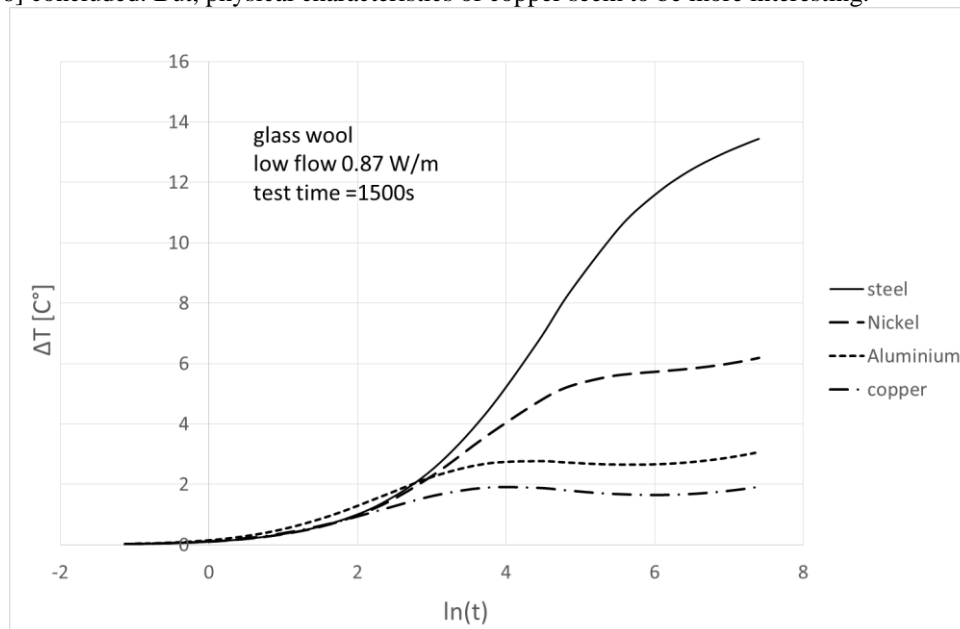


Figure 13: Metal outer surface of the probe

Between the outer surface of the probe and studied material, contact resistance must be taking into account especially for insulation materials. Influence of the contact resistance is considered by introducing air layer

thicknesses of 0 mm (no contact resistance), 1 mm and 2 mm (large contact resistance). We observe (Figure 14) that the contact resistance has no influence in the slope for long time investigation (for  $\ln t > 6$ ). The effect is relatively not sensible on S-shape curve for low heat flux and test time of 1500s.

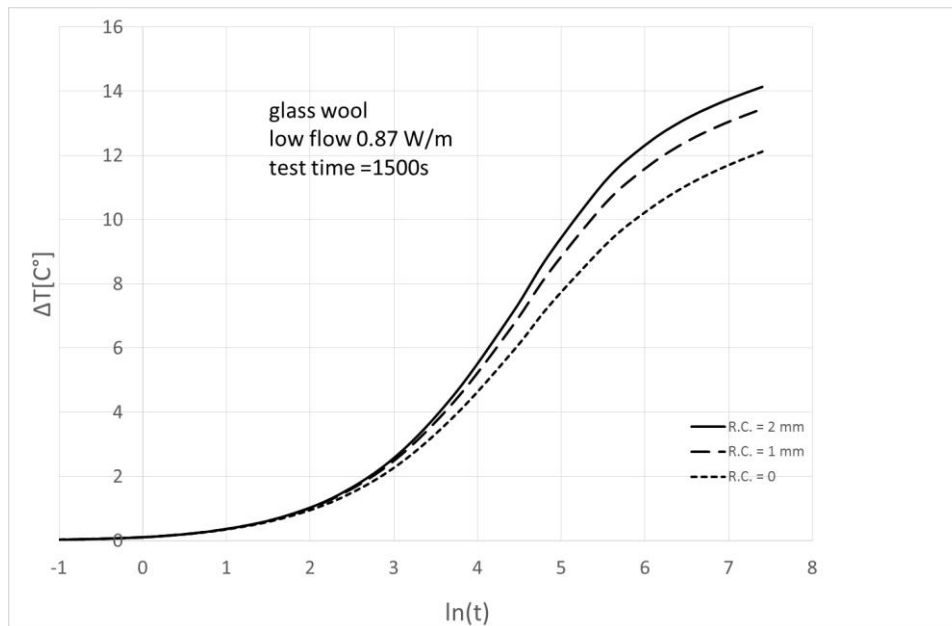


Figure 14: Effect of the thickness of the air gap

## 6- Conclusion

Applying long time approximation relation (equation 11) to determine thermal conductivity requires that the experimental kinetic representing the temperature increase vs. log time is linear. Experimental kinetic is linear for glycerol (a fluid) but as soon as the porosity of studied material is growing, S-shaped curve appears. Method becomes debatable. We observed that selecting the entire kinetics ( $0 < \ln(t) < end$ ) allow to obtain results in agreement with reference values for insulation materials.

For high porous materials, a perfect contact with the probe is not particularly desirable to determine thermal conductivity. Decreasing heat power reduces non-linear variation and provides thermal conductivity values with a good accuracy compared to reference values. We conclude that it is important to carry out tests over long time ( $>1500s$ ) with a very low heat flux ( $<0.1 \text{ W.m}^{-1}$ ). It is also important to check that temperature increase at the hot junction is less than  $5^\circ \text{C}$ . These conditions cannot be achieved with the control interface associated to Hukseflux TP02.

The length to diameter ( $L/2R_s$ ) ratio of the probe is an important value to reduce axial heat flux along the probe and consequently error in thermal conductivity determination. A  $L/2R_s$  ratio of 100 is enough to neglect heat loss in the lower part of the probe. But, we invite user to check if increase temperature at the upper part (with Pt1000 on TP02 Hukseflux for example) of the probe is less than  $1^\circ \text{C}$  during the test. To reduce the S-shape of the kinetics, we recommend the use of an outer surface of copper probe and not steel as the actual design of the TP02 probes.

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