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THERMAL CONDUCTIVITY OF SAND UNDER TRANSIENT CONDITIONS

TPSYS02 Thermal Measurement System

Sustainability of shallow geothermal systems.
Applied studies to Southern Europe climates.
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Title

THERMAL CONDUCTIVITY OF SAND UNDER TRANSIENT CONDITIONS
TPSYS02 Thermal Measurement System

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TPSYS02 Thermal Measurement System

Abstract

The growing concerns about the climate change and the negative environmental impacts of heating and cooling systems operated by fossil fuels, have led the technical and the scientific communities to find out more environmentally and sustainable energy alternatives, such is the case of shallow geothermal sources. Shallow geothermal systems use the ground as a heat reservoir, transferring thermal energy from the ground to a building, or vice-versa, with the help of a heat pump, to provide warmth in winter and cooling in summer. A proper evaluation of the thermal properties of the soil is essential in their design process and for a sustainable system. Thermal conductivity is the most important parameter for this evaluation.

Thermal conductivity can be estimated or measured by several methods, namely by means of empirical correlations, and experimentally, either in situ, by means of the well-known Thermal Response Tests (TRT), or in the laboratory, under steady state or transient conditions.

This work presents a series of thermal conductivity measurements on dry sand samples obtained by means of a high accuracy system with reference TPSYS02 Hukseflux equipped with Non-Steady-State Probes (NSSP) (TP02 or TP08). The system was acquired in the aim of an FCT research project.

The work includes the completion of a guide to procedures to the use of the thermal measurement system. Details of its calibration as well as results from tests in soil samples are also presented.

Keywords: Shallow geothermal energy systems / Soil thermal conductivity / Laboratory tests / Thermal needles / Calibration / Guide to procedures

CONDUTIVIDADE TÉRMICA DE AREIA EM CONDIÇÕES TRANSITÓRIAS

Sistema de Medição Térmica TPSYS02

Resumo

As preocupações crescentes com as alterações climáticas e os significativos impactos ambientais negativos dos sistemas de aquecimento e arrefecimento utilizando combustíveis fósseis, têm impulsionado, por parte das comunidades técnica e científica, a procura de fontes de energia menos poluentes, como as que recorrem à geotermia superficial.

Os sistemas geotérmicos superficiais utilizam o solo como reservatório térmico transportando, com auxilio de uma bomba de calor, a energia do solo para um determinado edifício, ou vice-versa, para o seu aquecimento no inverno ou arrefecimento no verão. Uma correta avaliação das propriedades térmicas do solo é essencial para um adequado dimensionamento energético e estudo da sustentabilidade destes sistemas, sendo a condutividade térmica o parâmetro mais importante desta avaliação.

A condutividade térmica do solo pode ser estimada e medida usando vários métodos, designadamente por recurso a relações empíricas ou experimentalmente, por via de ensaios de campo, pelos designados Testes de Resposta Térmica (TRT), ou por ensaios de laboratório, em regimes estacionário ou transitório.

No presente trabalho são apresentados resultados de determinações da condutividade térmica em amostras de areia seca por via de um sistema de medição (condutivímetro) de elevada precisão com referência TPSYS02 Hukseflux, equipado com sondas térmicas para medições em regime transitório (TP02 e TP08). O sistema foi adquirido no âmbito de um Projeto de Investigação da FCT.

O estudo inclui a apresentação de um breve manual de procedimentos para utilização deste dispositivo, sendo iniciado com ensaios de calibração do aparelho e incluindo resultados de ensaios de laboratório.

Palavras-chave: Aproveitamentos de energia geotérmica superficial / Condutividade térmica de solos / Ensaios laboratoriais / Condutivímetro / Calibração / Manual de procedimentos

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1 | Introduction

Geothermal energy can be defined as the energy incorporated as heat in the ground (Barbier, 2002). It can be extracted or discharged from the ground, since the ground has a nearly constant temperature, which tends to increase when moving toward the Earth's centre (Zheng ,2015).

Geothermal energy resources can be classified up to their operation temperature in three groups; (i) low temperature resources (T<90ºC), (ii) medium temperature resources (90°C<T<150°C), and (iii) high temperature resources (T>150°C) (Lopes, 2014).

With the continuous increase in the primary energy consumption, geothermal energy resources can play an important role in reducing energy consumption throughout using the heat capacity contained in the Earth. For instance, geothermal energy foundations can reduce the fossil fuel demand and consequently reduce carbon dioxide emissions (Brandl, 2006). Murphy and Niitsuma (1999) have observed that carbon dioxide emissions of geothermal energy resources can be in the range of 0.01–0.4 kg/kWh, compared to 0.5–1.1 kg/kWh of carbon dioxide from fossil fuels.

According to European Geothermal Energy Council (EGEC), geothermal energy systems can provide more sustainable way of heating and cooling at lower costs, especially if used in residential, commercial and industrial buildings. In such cases, a very shallow depths can provide an efficient resource of heat, which can be called shallow geothermal energy systems.

Shallow geothermal energy systems (SGEs) can extract heat from the ground using geothermal heat pumps, substituting the demand of fossil fuels by greener and more renewable source of energy (Lund et al., 2004). Their performance is influenced by several conditions with a relevant potential for ground characteristics, particularly when considering its primary circuit, which is in direct contact with the ground. Hence, it is essential to study several features that can affect both the design and the performance of the shallow geothermal energy systems (Vieira et al., 2017).

This report is focused on measuring soil thermal conductivity using a high accuracy measuring system by the transient hot-wire method, based in the line-source solution. For that purpose, initially a brief explanation regarding the soil thermal behaviour is presented. Afterwards, in the third chapter, the theory, in which the measuring method is based, will be described. In the fourth chapter, the thermal sensor device TPSYS02 manufactured by Hukseflux (2003) Supplier with its thermal probes TP02, TP08 and other components will be described. The fifth chapter explains the procedures of the thermal device operation to be followed in the sixth chapter by the presentation of the calibration process of the device using a standard material. Furthermore, the seventh chapter will describe preparation procedures of some sand samples and the results of the measurements will be presented in the eighth chapter. Finally, this report will be concluded by a brief guideline of procedures needed to study the thermal behaviour of some materials using that equipment.

2 | Soil thermal characterisation

Shallow geothermal energy systems as an inventive technology can extract heat in winter and inject it in summer. SGES' components are the primary circuit, secondary circuit and heat pump connecting between the two circuits. The primary circuit is formed to connect the system with the ground taking advantage of its nearly stable temperature to heat or cool the buildings. Afterwards, the heat pump transfers the absorber fluid to the secondary circuit to distribute heat in the building (Brandl, 2006).

SGES performance can depend on several variables related to the location, the building needs of energy, the climate, among other factors that can be related to the ground and the primary circuit. In fact, the ground thermal properties can play an important role in the design of shallow geothermal systems, in particular thermal conductivity of the ground, which can be a dominant factor in the heat transfer process between the ground and the primary circuit (Vieira et al, 2017).

Since the soil is a multi-phase system (e.g. solid, gaseous and liquid), ground heat transfer mechanism can be involved in different main processes, which are: (i) Conduction, (ii) Convection, (iii) Radiation (Carslaw and Jaeger, 1959). Conduction mechanism can be considered the most dominant factor in heat transfer process in the soil in case of thermo-active foundations. In conduction, energy moves from one place to another by means of molecular transfer which is more stable in solid soil phase compared to other physical soil phases. However, following Fourier law, heat flux can be expressed in the following constitutive equation:

$$q = -k\frac{dT}{dx} \qquad (1)$$

Where k is soil thermal conductivity, which is the proportionality constant between a gradient (*T*) and the heat flow (*q*) ($W.m^{-2}$). Thermal conductivity in its turn can be affected by many factors related to the ground itself, such as soil structure and composition, soil compactness, ground temperature field, and soil saturation degree. Several studies evaluated soil thermal conductivity considering the ground characteristics. For example, Hiraiwa and Kazbuchi (2000) showed that thermal conductivity increases by increasing the ground temperature. Cosenza et al. (2003) have observed that soil's thermal conductivity tends to increase by increasing soil's moisture content. Misra et al. (1995) concluded that thermal conductivity has a direct correlation with the soil density. Indeed, increasing soil density leads to increase the contact between soil particles and consequently decrease the pores volume in the soil.

Soil thermal conductivity can be evaluated by means of either laboratory or in-situ tests. In-situ measurements can be performed by Thermal Response Tests (TRT). Laboratory tests can follow either steady-state or transient methods. In steady state methods, the soil sample should maintain a steady temperature during the measurement, which can require a considerable time after applying the initial temperature difference. On the other hand, in transient methods, temperature changes with time, which does not require waiting for a steady state temperature to be achieved (Farouki, 1981). In this work, sand thermal conductivity will be measured following the transient method using thermal probes provided with the system (TPSYS02). The following chapter will present a detailed description of the test theory here applied in this report.

3 | Theoretical basis of the test method

The first application of the thermal probe as a transient method to measure thermal conductivity of the soils was carried out by Hooper and Lepper in 1950 (Farouki, 1981). The theory of this method is based on a line heat source introduced in a semi-infinite, homogeneous and isotropic medium. This method permits idealising a radial heat flux released along the line heat source. The medium temperature variations with time can be expressed in Fourier law using the medium thermal diffusivity (α):

$$\frac{\partial T}{\partial t} = \alpha \frac{\partial^2 T}{\partial^2 x} \qquad (2)$$

Where x is the direction of heat flow, and T is the temperature in time t. In cylindrical coordinates, the temperature evaluation with time can be presented as follows:

$$\frac{\partial T}{\partial t} = \alpha \left(\frac{\partial^2 T}{\partial^2 r} + \frac{1}{r}\frac{\partial T}{\partial r}\right) \qquad (3)$$

Assuming the radial heat flow is a constant density of power per unit length q (W.m^{-1}), the rise of medium temperature from the initial time t = 0 till the end of the measurement t = heating time, can be presented as:

$$\Delta T = \frac{q}{4\pi\lambda}\left[-E_i\left(-\frac{r^2}{4\alpha.t}\right)\right] \qquad (4)$$

In which $E_i(x)$ is an exponential integral and λ is the medium thermal conductivity (W.m^{-1}.K^{-1}), r is the radial distance from the heat line source (m), and α is the thermal diffusivity of the sample (m^2.s^{-1}) (Carslaw and Jaeger, 1959).

For large values of time, the temperature variation between two moments t_1 and t_2, can be given as a proportional of the logarithm of time t (sec):

$$\Delta T = T_2 - T_1 = \frac{q}{4\pi\lambda}\ln\left(\frac{t_2}{t_1}\right) \qquad (5)$$

Therefore, the equation of thermal conductivity of the soil can be given in the following equation as function of the rise in temperature from T_1 to T_2 measured in the moment t_1 and t_2, at by means of a temperature-sensitive thermistor located at an equidistant point from the ends of the probe, respectively.

$$\lambda = \frac{Q}{4\pi(T_2 - T_1)}\ln\left(\frac{t_2}{t_1}\right) \qquad (6)$$

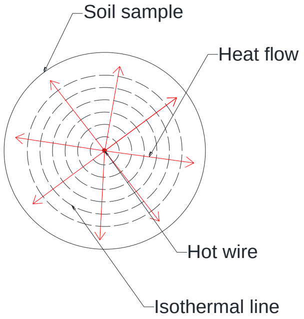


Figure 3.1 – Hot-wire method in a cylindrical soil sample (adapted from Franco, 2007)

In order to idealise an infinite heat line source, the needle probe was designed to have a large length to diameter ratio. For instance, with 150 mm of thermal probe length and 1.5 mm diameter, the ratio (length to diameter) would be equal to 100, a sufficient ratio to idealise an infinite heat line source.

However, there are some factors that can make this method different from the ideal continuous heat line source (hot wire), such as the finite length, radius and heat capacity of the probe, as well as the contact resistance between the probe and the medium (Farouki, 1981).

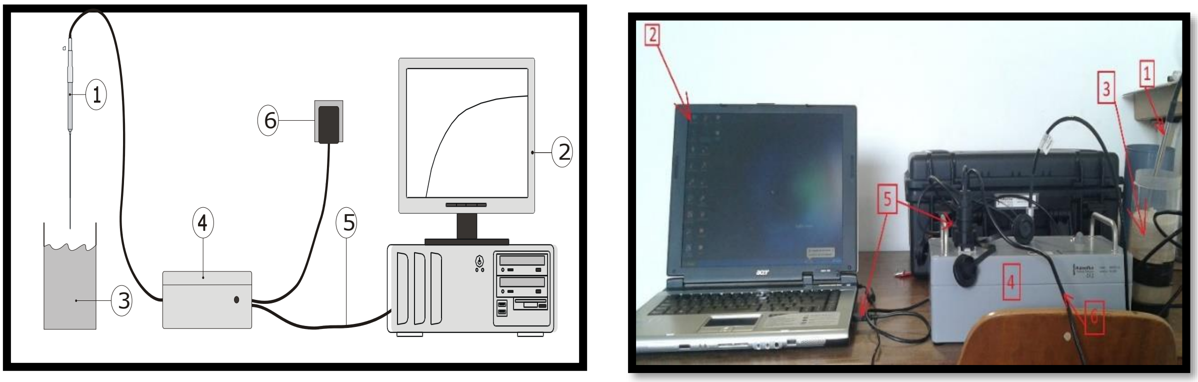
4 | Description of the thermal conductivity measuring system

4.1 System description

The TPSYS02 measurement system is designed to perform high accuracy measurements of thermal conductivity following the transient method of heat conduction using non-steady state probes. Its range of measurements is between 0.1 and 6 ($W.m^{-1}.K^{-1}$). These measurements can be carried out on various materials, such as soils, sediments, powders, thermal backfill and glues. TPSYS02 gives an estimation of average thermal conductivity and a standard deviation (Hukseflux, 2003).

The different parts which compose this system are the following (Figure 4.1):

- the CR1000 measurement and control unit (MCU) (4);
- the thermal probes TP02 or TP08 (1);
- a PC connected to control unit (2);
- the sample where the probe will be placed (3);
- a cable which connects the MCU to the PC (5).



a) b)

Figure 4.1 – TPSYS02 components: systematic scheme (a) and laboratory set-up of the system (b)

The equipment is equipped with two thermal probes: TP02 (0382) and TP08 (0338). These sensors are described in more details bellow. Details concerning the equipment installation and operation are also described below.

For measurements with TPSYS02 system, the MCU must be connected to a PC. This connection is enabled by LoggerNet software, which must be installed and tested on the PC previously. Details of the installation procedures can be found in the system manual (Hukseflux, 2003), and in Annex I (Installation). In Figure 4.2 is shown the principal dialog box of LoggerNet version 4.4.1.

LoggerNet 4.4.1

Build 4,4,1,4
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Figure 4.2 – LoggerNet v.4.4.1 software main dialog box

The user interface for communicating with TPSYS02 is CR1000 connected with LoggerNet**.** This interface will enable the control of the system during normal operation (Figure 4.3).

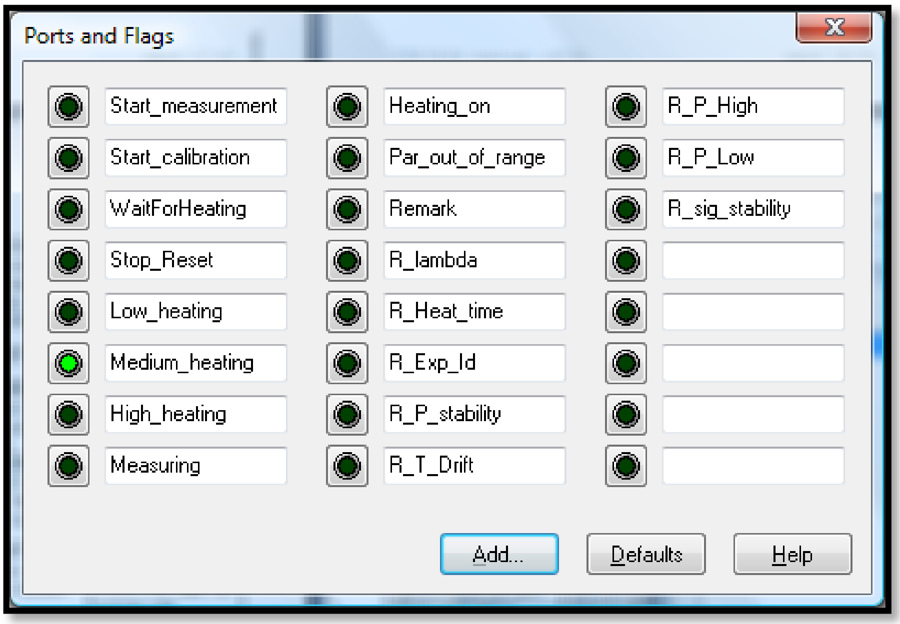

Figure 4.3 – Displayed ports and flags window

The following flags can be controlled by the user:

- Start measurement: It will be turned on with a green light once the measurement starts,
- Start calibration: it will be turned on once the calibration starts,

- Heating level (low, medium or high): it is used to define the power level to be applied during the measurement,
- Measuring: it is turned on automatically during the measurement time only,
- Heating on: it is turned on after activating the button Start measurement and only during the heating time,
- Par_out_of_range: it is turned on automatically once the parameter resulted from the measurement is out of the logical range.

4.2 Measurement and Control Unit (MCU)

This data-logger can store data from the measure sensors, controlling their operation. It is connected to a PC, which can store the data collected by the software LoggerNet. This unit starts working by pressing the grey button (1) and when it is working the red button (2) will brighten up (Figure 4.4).

Figure 4.4 – Initial screen of LoggerNet v.4.4.1

It is recommended to turn off the MCU once the measurement ends and after storing the data and transfer it to the PC, in order to avoid losing information of the measurements data.

4.3 Probes description

The thermal probes enable performing fast measurements of temperature in the medium (sample) in which they are inserted. These probes are called Non-Steady-State Probes (NSSP) as their measurements are performed in transient conditions (based on line source theory). For performing a measurement, the TP02 (or TP08) must be connected to the MCU.

TP02 needle is used to perform a fast measurement of thermal conductivity throughout inserting it inside the sample and connecting it to the CR1000 MCU. Its performance is compatible with the principles and procedures presented in the standards ASTM D5334 – 00 and IEEE442-1981. Studies have affirmed its suitability and applicability to soils and other materials, such as thermal backfill materials, sediments, foodstuff, powders, sludge and various other materials. This large size non-steady state probe has 150mm of length and 1.5mm diameter. Thus, its response time is very small due to the large ratio between the length and the diameter, which equals to 100. A fact that allows performing fast and practical measurements with time spans as reduced as 100s, 200s and 300s. This probe consists of the following components (as shown in Figure 4.5):

- Reference temperature sensor which measures temperature Pt1000) (1);
- Heating wire that occupies two thirds of the probe length (coloured in red) (2);
- The hot joint that measures T_hot (3);
- The cold joint at the tip which stays at a stable temperature T_cold (4);
- Plastic wire connects the needle with the MCU (5);
- The base with 10mm diameter, where the sensor is mounted (6).

Note: This probe has two thermocouple junctions, which are the hot joint in the first third of the needle length (3) and the cold joint at the tip (4).

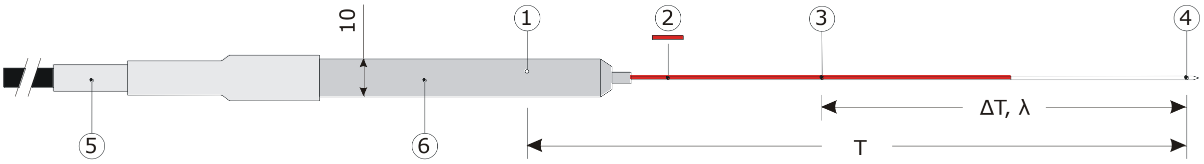

Figure 4.5 – TP02 components

In the design of TP02, the base (6) aims to measure the ambient temperature by means of the reference temperature sensor. The measured values here depend on the evolution in the temperature differences between the cold joint (4) and the hot joint of the needle (3). The heating wire runs only along the upper 100 mm of the thermal probe. A thermocouple junction for measuring temperature of the heated part of the needle is located at half-length of the heating wire. The temperature measured in this point is the so-called T_hot temperature (3).

This design permits this probe to have measurements with the following advantages:

- superior accuracy when measuring at high and low temperatures;
- optimal accuracy independent of the medium temperature;
- minimal sensitivity to thermal gradients;
- high sensor stability and the possibility to use normal cables and connectors.

TP08 probe is a small version of TP02. It is recognised as a small-size probe since its length is 70mm, presenting some differences on its design with regard to the large-size probe TP02. It has approved suitability and applicability in various types of materials. This probe is formed by the following components (Figure 4.6):

- A plastic isolated wire that serves not only to connect the probe with MCU, but also to isolate the probe from temperature perturbations that occurs when the tester touches the probe (1);
- The base with 10mm diameter, where the sensor is mounted (2);
- The needle with 70mm length (3);
- Reference temperature sensor (Pt1000) (4);
- Heating wire that occupies the entire probe length (coloured in red) (5);
- The single thermocouple junction, which is the hot joint at 17mm from the tip of the needle (6).

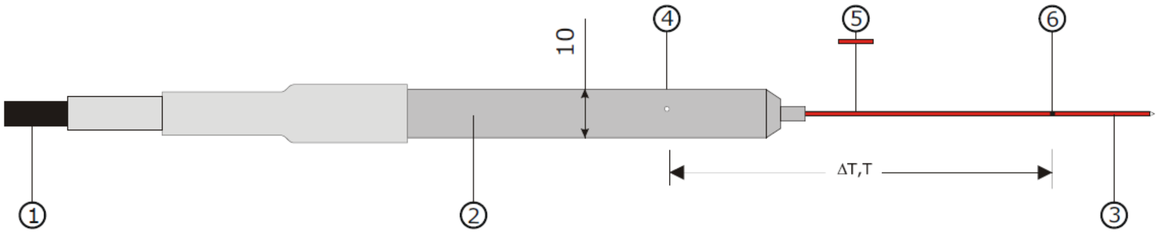

Figure 4.6 – TP08 components

Note: in the small-size probe TP08, the reference thermocouple junction (4) is located in the base (2) Pt1000. Hence, to register the evolution in the temperature’s differences between the base (4) and the hot joint of the needle (6) during the heating time, the junction (2) must be inserted in the soil.

5 | Operation procedures

The test method described in this work enables the determination of thermal conductivity using a transient heat method which can be used in undisturbed or remoulded specimens. The thermal conductivity is determined by a variation of the line source test method using a needle probe with a large height to diameter ratio. As above-mentioned the measurements complies with standards IEEE 442 (IEEE, 1981) and ASTM D 5334-00 (ASTM 5334, 2000).

Several checks must be performed before conducting a standard measurement with TPSYS02 measuring system. Initially, an overall system check must be carried out. Subsequently, after powering the system, the MCU must be connected to the PC and to the measurement control data logger CR1000. Details of the steps to be taken are described in Annex I.

Two modes of operation are possible with TPSYS02: the standard operation mode (St mode) used when analysing batches of similar materials and when the measurement accuracy is not too critical, and the scientific operation mode (Sc-mode), recommended when the measurement needs to be as accurate as possible (Hukseflux, 2003).

The measuring system should be calibrated before its regular use for a specific thermal needle. On calibration tests materials with known conductivities in the system’s measuring range must be used. Details of the standard calibration tests are presented in the following chapter.

Before conducting laboratory tests, the specimen must come to equilibrium with the room temperature. For that a sufficiently long period of time, until all the temperature sensors stabilize must precede the test. Also preceding the application of the thermal load, the probe must be inserted into the specimen. In all situations a perfect contact between the needle probe and the soil sample must be guaranteed.

After reaching thermal equilibrium a known constant current should be applied to the heater. A selection of a proper heating level and of the time cycle must be undertaken.

As the test takes place all the measurements are displayed in the PC screen enabling an automatic analysis of the test taking readings every 0.5 second. The plot of the temperature data as a function of the logarithm time will enable the estimation of thermal conductivity. Furthermore, the raw measurements data can be downloaded from the CR1000 to the PC, see Annex I for more details.

6 | Calibration of TPSYS02 system

Before starting its use, calibration testing of TPSYS02 system must be carried out. Two alternatives are proposed in the manual, either the use of a Calibration Reference Cylinder (CRC), if available, or of a reference material such as glycerine (ASTM 5334, 2008). In this work, the second alternative was followed using the glycerine as a reference material with a known thermal conductivity λ (equal to 0.285 ($W.m^{-1}.K^{-1}$) for 25ºC) (Lide, 2004).

When using the glycerine as the material for tests' calibration it is recommended to mix it with plastic fibres in order to avoid the convection effects which could affect the measurements. Moreover the thermal conductivity of glycerine (0.285 ($W.m^{-1}.K^{-1}$)) is very close to that of the polyester fibres (0.24 ($W.m^{-1}.K^{-1}$)). The quantity of the plastic fibres was indicated in the TPSYS02 manual as 2% of the glycerine mass used in the calibration. Calculations are provided in Annex II.

After preparing the recipient with the glycerine and distribute the fibres homogeneously, the needle TP02 was positioned inside the glycerine for its entire length, applying a low heating level (0.85 ($W.m^{-1}$)) and setting the heating time to 100 seconds. The calibration test was repeated considering different levels of heating: medium (2.5 ($W.m^{-1}$)) and high (4.23 ($W.m^{-1}$)).

After verifying the heating time and setting it to tc = 100s, the calibration test started with the command ''Start Calibration''. The calibration time will double the heating time, 2x tc = 200s. During the test the ''Countdown'' button started counting from 200s until zero, announcing the end of the calibration test.

The resulting average thermal conductivity ''Lambda'' will be given together with the corresponding standard deviation ''Lambda-sd''. After the end of the calibration countdown time, the command ''Cal-OK'' would display ''Yes'' if the measured lambda equals the literature value within a coefficient of variation of ± 5%.

A calibration factor was then calculated using the thermal conductivity of the reference material and the thermal conductivity measured by the equipment according to (ASTM 5334, 2008):

$$C_f = \frac{\lambda material}{\lambda measured} \tag{7}$$

This factor will be used as an equivalent factor which will be applied to the measured values of thermal conductivity in order to get the final corrected values of thermal conductivity of the tested material.

The main results of the calibration tests carried out in this study are presented in the Table 6.1.

Table 6.1 – Calibration tests results applying low heating power (0.85 ($W.m^{-1}$)) before using vacuum

Test Nº	**1**	**2**	**3**	**4**	**5**	**6**	**7**	C_f**Average**
λ measured	0.280	0.228	0.227	0.229	0.287	0.281	0.289	
C_f	1.0178	1.2500	1.2555	1.2445	0.99303	1.01423	0.98616	1.108746

As explained before, the calibration calculations were repeated applying different values of heating power, such as medium and high power available in the TPSYS02 system.

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1 A more detailed description of these steps for the TPSYS02 system will be provided in section 9. (Guide to procedures – see pages. 33-39 of the manual).

In the following calibration tests measurements vacuum was used to extract air form the mixture of glycerine and fibres. The main results are presented in Tables 6.2 and 6.3.

Table 6.2 – Calibration tests results applying medium heating power (2.50 (W.m^{-1}))

Test Nº	1	2	3	4	5	6	7	C_fAverage
λ measured	0.2849	0.2851	0.2808	0.2807	0.2835	0.2869	0.2886	
C_f	1.0004	0.9996	1.0149	1.0153	1.0053	0.9934	0.9875	1.0023

Table 6.3 – Calibration tests results applying high heating power (4.23 (W.m^{-1}))

Test Nº	1	2	3	4	5	6	7	C_fAverage
λ measured	0.2855	0.2883	0.2873	0.2862	0.2915	0.2885	0.2908	
C_f	0.9982	0.9886	0.9920	0.9958	0.9777	0.9878	0.9801	0.9886

It can be concluded that the elimination of air from the mixture has helped stabilising the thermal conductivity values of the glycerine. The values shown in Tables 6.2 and 6.3 have ranged between 0.2807 (W.m^{-1}.K^{-1}) and 0.2915 (W.m^{-1}.K^{-1}), whereas in Table 6.1 the thermal conductivity varied between 0.227 (W.m^{-1}.K^{-1}) and 0.289 (W.m^{-1}.K^{-1}). In fact, stabilising the thermal conductivity values has helped obtaining better calibration factors of almost 1. On contrary, the calibration factor obtained before using vacuum was averagely 10% higher than 1.

Calibration was also repeated after vacuum using low heating level (0.85 (W.m^{-1})), the obtained results are presented in Table 6.4:

Table 6.4 – Calibration tests results using vacuum and applying low heating power (0.85 (W.m^{-1}))

Test Nº	1	2	3	4	5	6	7	C_fAverage
λ measured	0.2716	0.2912	0.2792	0.2915	0.2837	0.2822	0.2774	
C_f	1.0493	0.9787	1.0208	0.9777	1.0045	1.0099	1.0274	1.0097

It can be observed that also for the lower heating level the homogenous mixture of glycerine with less air voids leads to values of thermal conductivities closer to the literature ones, which resulted in better calibration factors.

7 | Sample preparation

A series of precautions should be taken in the sample preparation before conducting a thermal response test. Regarding its dimensions, the size of the tube/recipient must have a minimum diameter, with the purpose of approaching the dimensionless condition of the heating wire, and a minimum height, in order to attain a sufficiently low diameter to height ratio (for approaching the infinite medium condition).

Undisturbed and remoulded soil specimens can be tested with TPSYS02 system. In all the cases, a good contact between the specimen and the probe should be guaranteed. For the case of undisturbed soil specimens ASTM D 5334 – 08 standard indicates a minimum diameter of the sampling recipient containing the soil specimen of 51 mm. On the other hand, TP02 manual provided by Hukseflux Supplier indicates that the recipient radius should not be less than 15 times the needle probe radius. Therefore, for the case of TP02 and TP08 probes, with diameters of 1.5mm and 1.2mm, respectively, the recipients' diameters shall not be less than 22.5mm and 18mm, correspondingly.

The length of the recipient is critical, since it permits inserting the probe in the soil sample allowing a good contact with the soil along the entire probe. Thus, a cut of 200 ±30 mm long section of recipient is indicated which will allow having both needles inserted in the soil sample especially in TP08. In fact, in the small size probe TP08, it is required to have it entirely immersed, since the cold joint is located in the base, which must be sensible to the soil sample temperature.

IEEE standard and recent studies (Vieira et al., 2017) refer that the moisture migration which might occur previously to the thermal test (during the sample preparation and the elapsed time until temperature is stabilized) is a major factor affecting thermal conductivity measurements, especially in granular soils. Globally, soil in situ conditions must be preserved in order to be obtained reliable values of thermal conductivity.

In the context of this work a series of tests were performed in *Fontainebleau* sand for different densities. Firstly, before performing the thermal tests, maximum and minimum (γ_{dmin}, γ_{dmax}) densities were determined, following standard ASTM D 4253-00. The obtained results are presented in Table 7.1:

Table 7.1 – Maximum and minimum density and particles dry unit weight results

Soil reference Nº	Υ_s (kN.m^{-3})	γ_{dmin} (kN.m^{-3})	e_{max}	γ_{dmax} (kN.m^{-3})	e_{min}
5118	26.18	14.11	0.855	17.20	0.521

Soil samples were afterwards prepared in dry conditions for five different densities, corresponding to five different relative densities ratios D_r (0%, 30%, 45%, 60% and 100%).

The range of dry unit weights was in the range [14.11-17.20] (kN.m^{-3}). Densities' determinations followed the ASTM D 4253-00 standard expressions. Table 7.2 shows some index properties of the tested samples on which thermal conductivity tests were performed.

Table 7.2 – Index properties of the samples used in the thermal conductivity measurments

Relative density	100	60	45	30	0
Void ratio (e)	0.521	0.655	0.705	0.755	0.855
Porosity (n)	0.343	0.396	0.413	0.430	0.461
Dry weight kN.m^{-3}	17.20	15.81	15.35	14.91	14.11
Dry mass kg.m^{-3}	1750	1610	1560	1520	1440

To achieve this range of densities, specific techniques were carried out. To reach the minimum density of *Fontainebleau* sand and since it is a poorly graded sand, a sandy shower was used to get the minimum density in the recipient. Specific care was subsequently required to keep the soil with the desired density since any movement could lead to changes in its density.

For higher densities a selection of an appropriate compaction technique was required. For instance, to achieve densities higher than the minimum dry weight mass (1520, 1560, and 1610 (kg.m^{-3})), the recipient was divided to three equal layers by its height and each layer was prepared for the same desired density. For this purpose, the weight of the soil in each layer was calculated considering the desired density and manual compaction was carried out. This technique allowed having almost a homogenous density along the recipient.

For the case of the maximum dry weight mass (1750 (kg.m^{-3})), standard ASTM D 4253-00 was followed using the vibratory table. The recipient was positioned on the vibratory table and fixed by its apparatuses. Figure 7.1 shows some photos of maximum density determination.

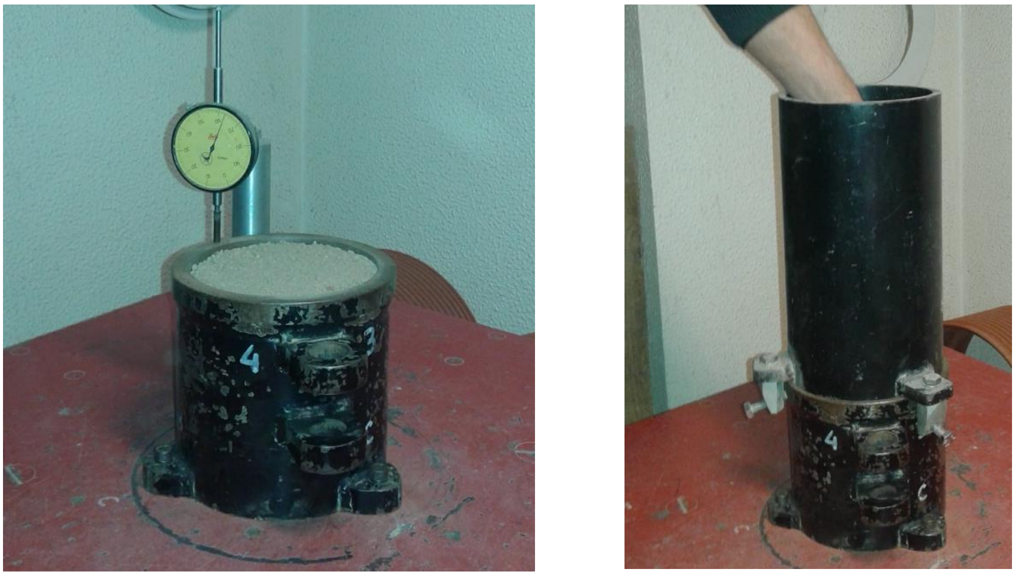

Figure 7.1 – Maximum density preparation following the standard ASTM D 4253-00

In case of very dense/rock specimens, a predrilled hole must be done before inserting the thermal probe. However, care should be taken to ensure that the thermal probe shaft is fully embedded in the specimen and not left partially exposed.

After weighing the specimen and verify the density calculations, the thermal probe was inserted in the specimen pushing the probe into the soil specimen. Thermal tests were performed in all samples (from the loosest to the densest specimen). The obtained results are presented in the following chapter.

8 | Thermal test results

Several thermal tests using TPSYS02 and thermal probe TP02 were performed. The tests, with duration of 200s, consisted on the application of a thermal flux 0.89 $W.m^{-1}$ (low heating power) through the thermal probe where a heating wire runs and the systematic measurement of the induced temperature in time in the thermal sensors. As described in chapter 3, with the evolution of the temperature with time thermal conductivity will be estimated. Each test was repeated several times; and the values presented are average values. Each sand sample was prepared in a metal recipient with 1958 cm^3 volume (Figure 8.1).

Figure 8.1 – Recipient used in thermal conductivity measurements

Different densities starting by the minimum dry weight mass of 1440 $kg.cm^{-3}$ and ending by the maximum dry weight mass of 1750 $kg.cm^{-3}$ were considered and prepared in this recipient following the compaction techniques explained previously. For each sample a thermal test was afterwards carried out. Table 8.1 are shown the thermal conductivity results for all *Fontainebleau* sand samples.

Table 8.1 – Thermal conductivity measurements of Fontainebleau Nº 5118 in the recipient 1

Recipient Number	Dry weight mass of ($kg.cm^{-3}$)	Porosity (n%)	Void index (e)	Thermal conductivity ($W.m^{-1}.K^{-1}$) in centre	Thermal conductivity ($W.m^{-1}.K^{-1}$) after using Cf
Nº 1	1440	46%	0.855	0.218	0.220
	1520	43%	0.755	0.236	0.238
	1560	41%	0.705	0.255	0.257
	1610	39%	0.655	0.272	0.275
	1750	34%	0.521	0.332	0.335

In Figure 8.2, the results of a typical thermal test carried out by means of the system TPSYS02 are shown. It can be observed that thermal conductivity of the dry sand increases with increasing its density which is related to the removal of air from the soil voids and to the increase in the contact area between the soil particle, which in its turn increases the conductivity of the soil sample. In the same figure, the quadratic regression, to which the experimental data were fitted, is shown.

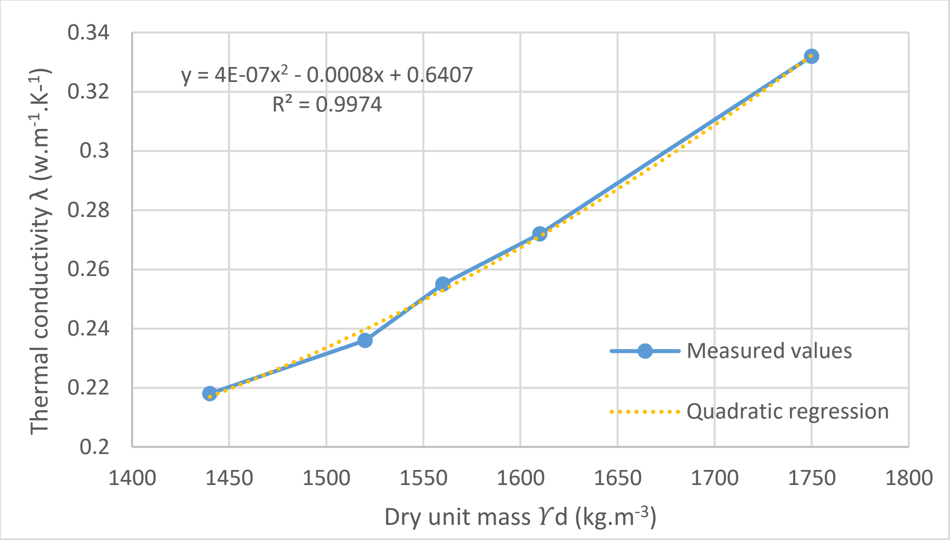

Figure 8.2 – Thermal conductivity measurements vs. sand sample dry unit mass (λ vs. ϒs)

Consequently, thermal conductivity decreases by the increase in soil porosity due to the increase of air in the specimen, which represents an obstacle to heat conduction.

Figure 8.3 illustrates the relation between soil thermal conductivity measurements and the sand porosity together with the quadratic regression of the measured data fitted with a high value of determination coefficient R^2=0.9995.

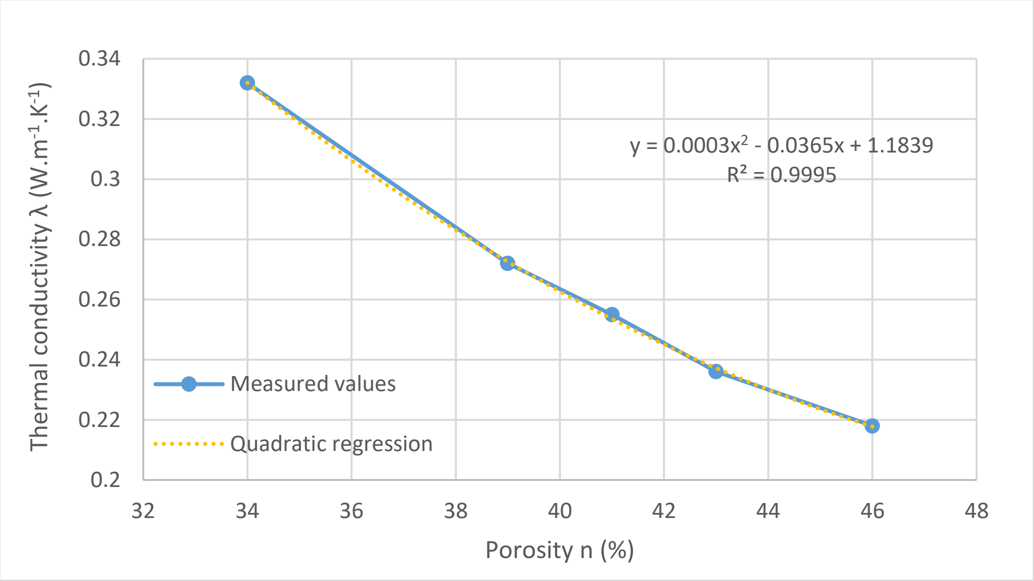

Figure 8.3 – Thermal conductivity measurements vs. soil sample porosity (λ vs. n%)

Studies, such as García et al., (1989), Tong et al., (2016) and Mady and Shein (2018), are in line with these results confirming that the soil thermal conductivity relation with its density are better fitted in a polynomial regression than in a linear regression. These studies have observed also a quadratic regression between thermal conductivity and soil porosity, obtaining a high determination coefficient between the curves and the measured values.

9 | Guide to procedures

To perform thermal measurements using TPSYS02 thermal sensors device, a brief guideline is presented below, which should be followed respecting the order listed:

- Connect the MCU to the PC which has the LoggerNet program installed and as well as to the electricity using its cable.
- Insert the needle probe (e.g. TP02) into the soil sample and connect it to the MCU.
- Wait until the sample temperature stabilises as soon as thermal balance between the sample and the room temperature is achieved.
- Select a level of heating power to be used in the measurements. Selecting this power level is related to the resistivity of the material tested by this system. Some materials need experience to determine the power level. However, normally the materials with low resistivity require high level of heating power.
- Select a specific heating time to be applied in the measurements, which is normally 100s. In this case the countdown will be double of this time to allow the thermal probe to stabilise its temperature before heating. This time can be set to higher values considering the material type and resistivity, among other factors.
- Start the measurement and wait until the countdown parameter stops counting.
- In this system, it is not required to register the data (Temperature vs. Time), since the data are automatically registered and saved in the system. The time interval used to register the data can be changed and set by the user. For instance, the reads registered by the system can be done each 0.5 seconds or by an interval of 1 seconds.
- Collecting the data must be done before disconnecting the system in order to avoid losing them, see Annex II.
- Analysing the data in this system can be performed by the Excel spreadsheet sent from the Hukseflux Supplier, which permits the user obtaining the thermal conductivity of the material more accurately, see Annex II.

Note: For a detailed guideline of operating this system and performing thermal measurements, see the Annex I and Annex II.

10 | Final remarks

This report was focused on the TPSYS-02 thermal measurement system acquired in the context of research project Success (PTDC/ECM-GEO/0728/2014). Following a brief description of the theoretical basis of the line-source solution, from which was derived the method used to evaluate thermal conductivity, the system operation and its calibration were also presented.

A series of measurements of thermal conductivity were performed in dry samples of *Fointainebleau* sand for different relative densities. The evolution of conductivity of the dry sand samples shows a consistent evolution, i.e., an increase with density and a very high coefficient of determination for a quadratic fitting.

The report also contains a guide to procedures for a standard thermal test and a detailed explanation of the main procedures to be followed.

Lisbon, LNEC, December of 2018

APPROVED

The head of Geotechnics Department

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Annexes

ANNEX I
(Detailed procedures)

TPSYS02 must be connected to electricity and to the LoggerNet program installed on a PC in order to read out the data directly and check-up the temperature changes with time during the heating time. A detailed guideline is presented below to help users operate the system and perform measurements correctly.

- Open LoggerNet v.4.4.1 program and the main dialog box will appear which has all the main lists of this program, click on ‘’Setup’’ to perform setup screen as presented in Figure AI.1.

Figure AI. 1 – LoggerNet program icon and Dialog box of LoggerNet with its Main menu

- In the ‘’Setup’’ screen, click on ‘’ComPort’’ and select the correct COMPort or add immediately a COMPort if needed. In this case, click on ‘’Add Device’’, and add a ‘’CR1000’’ to the ComPort, as presented in Figure AI.2.

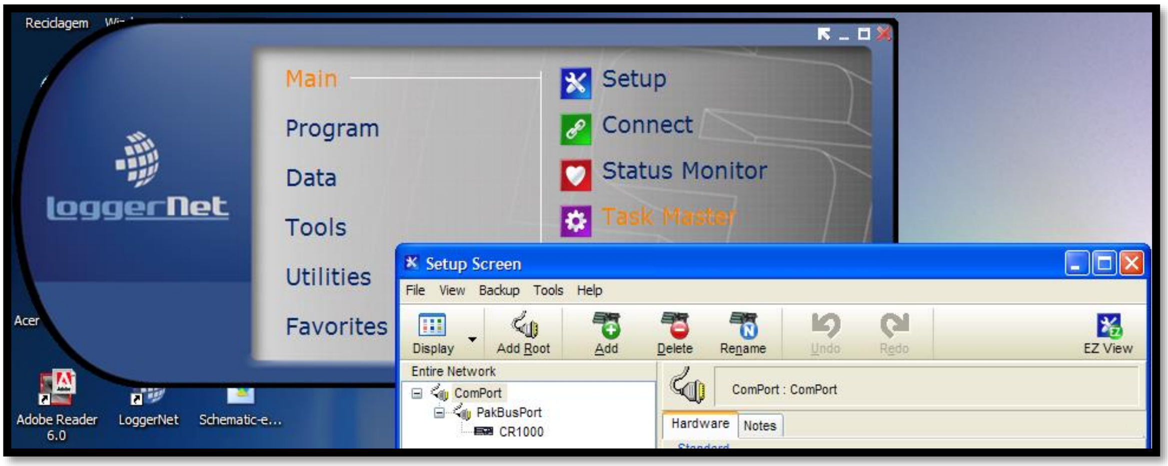
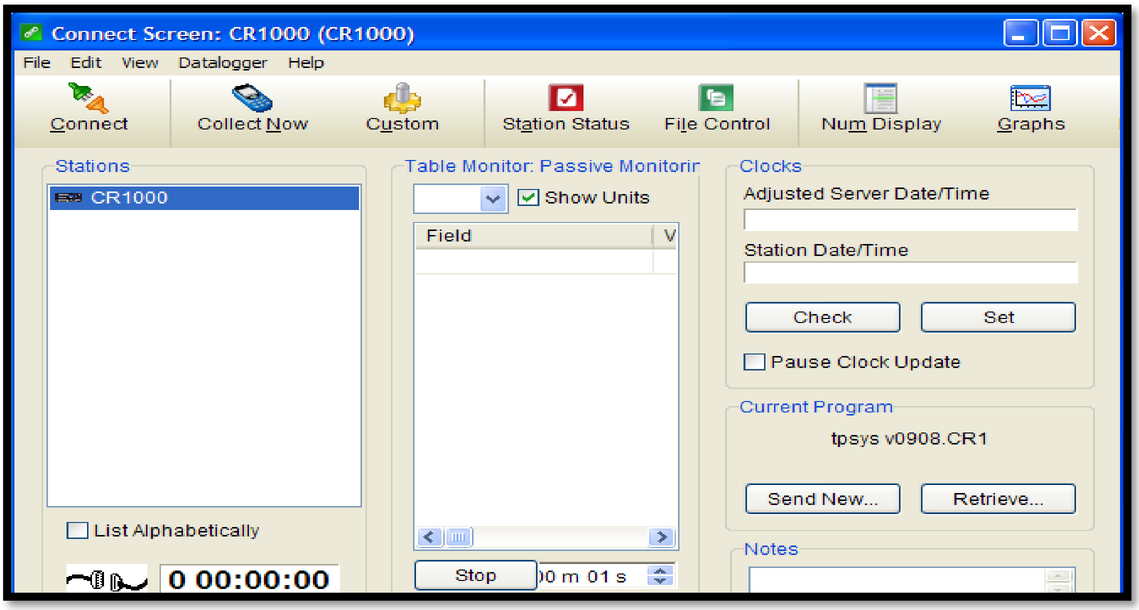


Figure AI. 2 – Setup screen

- Again in the main screen of the LoggerNet (see Figure 12.1), click on ‘’Connect’’ to perform connect screen which is presented in Figure AI.3.
- In the connect screen, select CR1000 and click on connect to connect the CR1000 and start operating the system once the time (00:00:00) in the left bottom starts counting.
- In connect screen, click on ‘’Num Display’’ to display numerical screen and to monitor the parameters that would be analysed during the measurements, see Figure AI.4.



☐ List Alphabetically

0 00:00:00

☑ Show Units

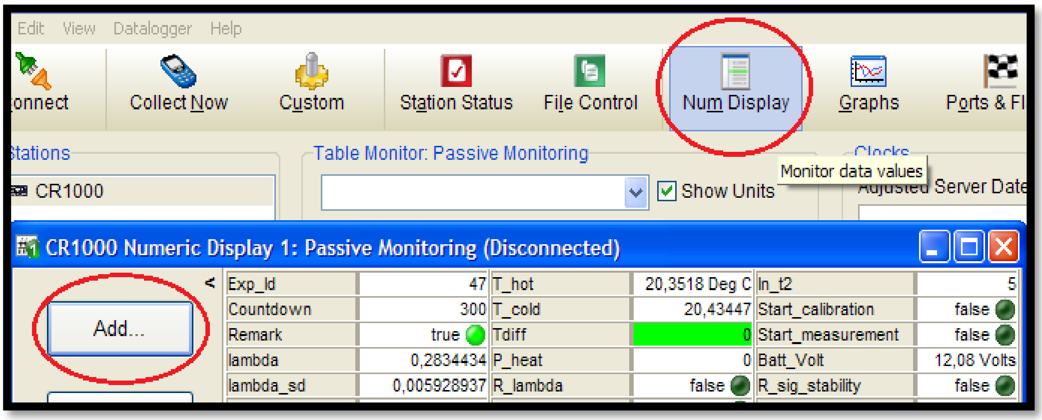
)0 m 01 s

☐ Pause Clock Update

Current Program: tpsys v0908.CR1

Figure AI. 3 – Connect screen with its Num Display, Graphs and Ports and Flags buttons

- To add parameters in the "Numeric Display" screen, put the cursor in the cell where it is preferred to insert the specific parameter and click on "Add" and then, "Add Selection" dialog box will appear, as presented in Figure AI.4 and Figure AI.5.



Exp_Id	47	T_hot	20,3518 Deg C	In_t2	5
Countdown	300	T_cold	20,43447	Start_calibration	false ●
Remark	true ●	Tdiff	0	Start_measurement	false ●
lambda	0,2834434	P_heat	0	Batt_Volt	12,08 Volts
lambda_sd	0,005928937	R_lambda	false ●	R_sig_stability	false ●

Figure AI. 4 – Numeric Display screen to add parameters

- In "Add Selection" dialog box, select "Public" and choose the parameters required to monitor the data during the measurement/calibration and then click on "Paste" to add it to the numerical screen, see Figure AI.5.
- To display another numerical screen to perform the results, it is advised to repeat the previous steps and in "Add selection" choose "Results" and select the results that would be analysed and then click "Paste".

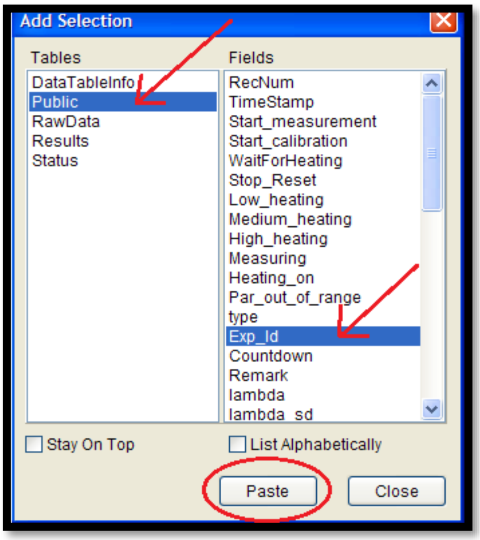


Figure AI. 5 – Add selection dialog box to add specific parameters

- It is possible to perform and display three numerical displays and three charts as well, as presented in Figure AI.6 and Figure AI.7.

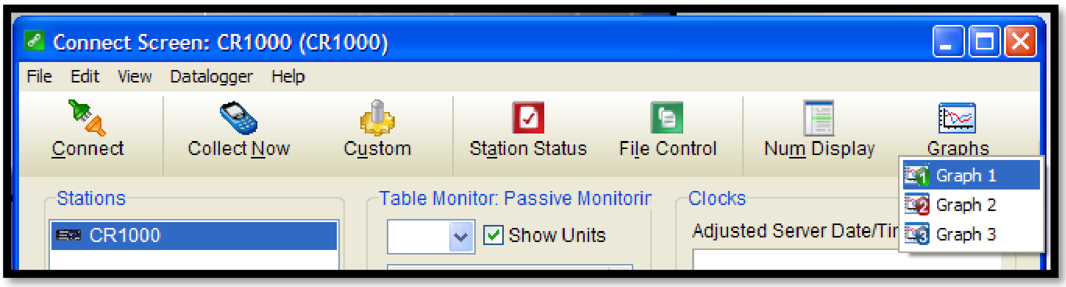


Figure AI. 6 – Connect screen with its Graphs list to create three graphs

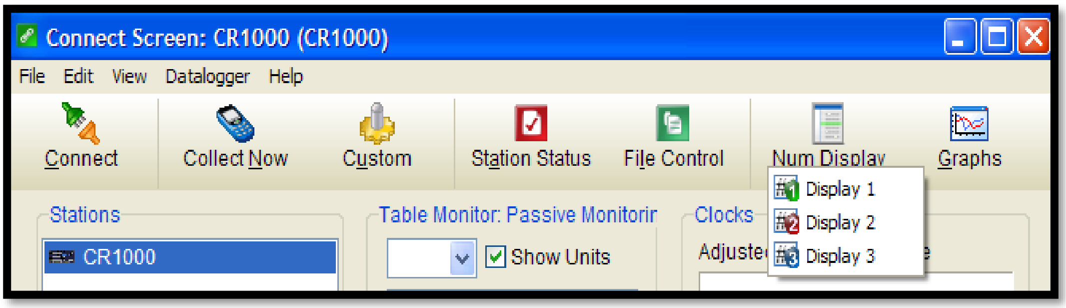
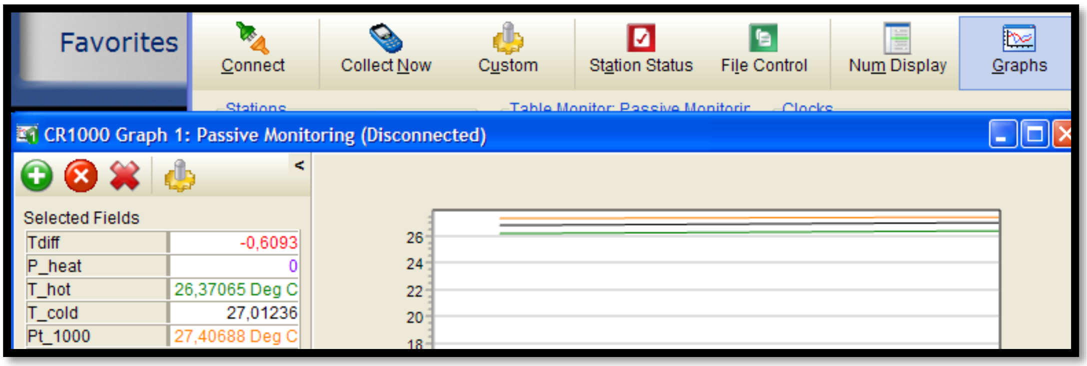


Figure AI. 7 – Connect screen with its Num Display list to create three numerical displays

- To monitor the measurement graphically, click on ‘‘Graphs’’ in the ‘‘Connect’’ screen and select Graph 1 for instance. Then Graph 1: ‘‘Passive Monitoring’’ will be displayed.
- To add parameters to be monitored in this dialog, click on a cell where the parameter is supposed to be inserted (e.g. T_cold for instance) and click on the green plus ⊕ button in the top left corner and then another dialog of ‘‘Add Selection’’ similar to the previous one will appear.

- Select the specific parameter to be monitored and click “Paste” and close, as presented in Figure AI.5 to appear the required parameters in Graph 1 as presented in Figure AI.8.



Tdiff	-0,6093
P_heat	0
T_hot	26,37065 Deg C
T_cold	27,01236
Pt_1000	27,40688 Deg C

Figure AI. 8 – Graph1 window and how to create a graph and add its parameters

- In this dialog “Ports and Flags”, it is possible to define the potential of energy to be applied and here there is three options; (i) Low_heating which applies 0.85 W/m, (ii) Medium_heating that applies 2.45 W/m and (iii) High_heating with a power potency of 4.5W/m, see Figure AI.9.
- After determining the heating power to be used in the measurement, the heating time “Heat_time” must be set in the “CR1000 Numeric Display 1: Passive Monitoring”. Once the heating time is set to 100s for instance, the countdown will be automatically updated to the value 2x100s=200s.
- To start the measurement, it is required to call a dialog box called “Ports&Flags” in the connect screen and then click on “Start_measurement” to start measurement or “Start_calibration” to start a calibration.

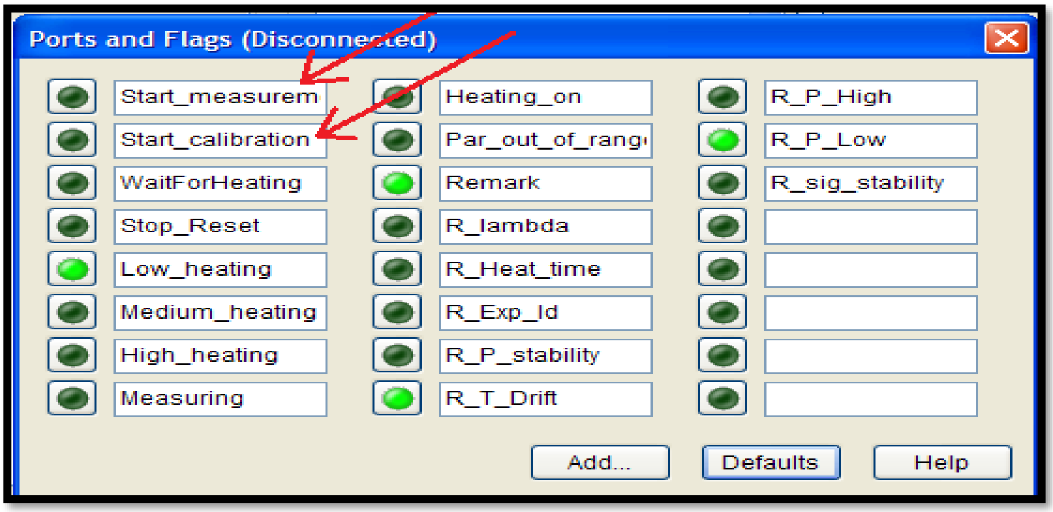


Figure AI. 9 – Ports and Flags window to control the measurements

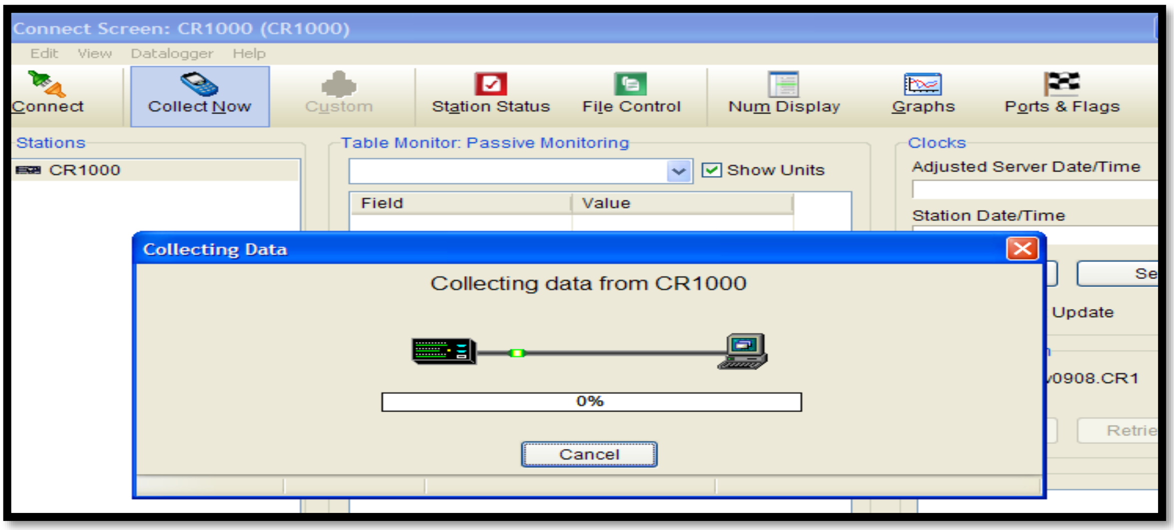
Once the “Countdown” has ended, the heating will stop and the thermal conductivity of material tested would be displayed in the lambda parameter in the Numeric Display by which the test is monitored.

After performing the thermal tests, the data will be collected and stored on the PC to be analysed later using the Excel spreadsheet provided by Hukseflux Supplier. Annex II provides detailed procedures of collecting data and analysing data.

ANNEX II
(Collecting and displaying procedures)

To analyse the results of any measurement, the spreadsheet sent form the must be used after retrieving the results from the CR1000 to the PC. The steps bellow must be followed to retrieve the data from the MCU to the PC:

- Open Setup screen and select the CR1000 and click on data files;
- Choose the Raw data and Results and choose the extension and the type of files where to save data. It is recommended to use TOA5 extension file;
- Open connect screen and click in Collect Now to collect the data from the CR1000 measurement and control unit (MCU) and the data will be collected as in Figure AII.1.



Connect Screen: CR1000 (CR1000)

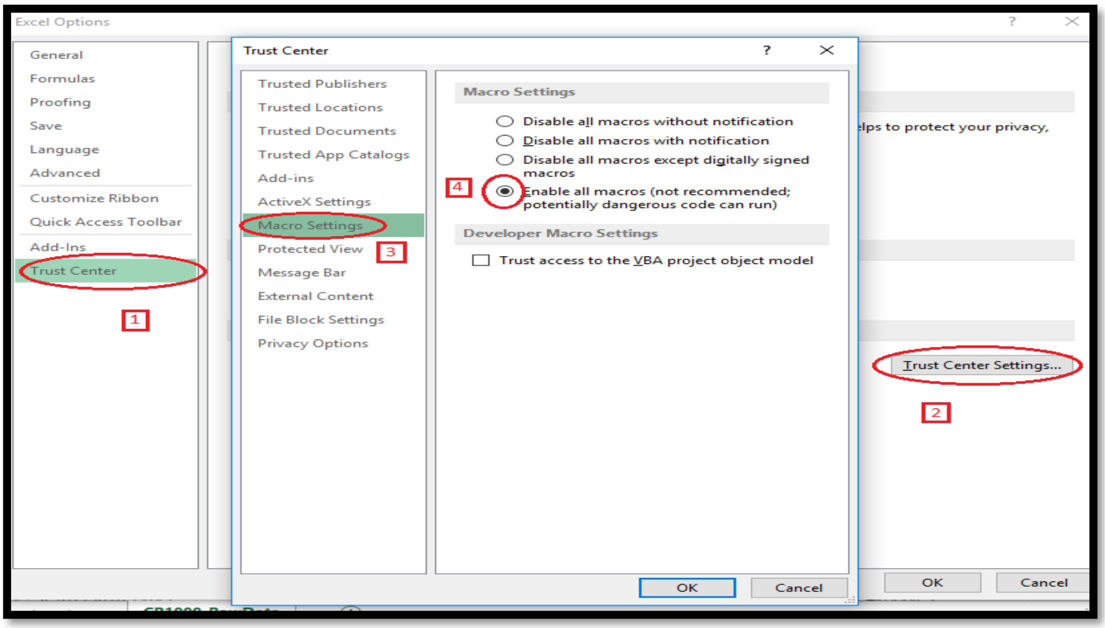
Collecting Data

Collecting data from CR1000

0%

Figure AII. 1 – Collecting data from CR1000 to PC

- To set macro settings: →Open a blank excel file → open File menu → click on → Trust Centre → Trust Centre settings → Macro settings → Enable all macro's → OK, as presented in Figure AII.2.



Excel Options

Trust Center

Macro Settings

○ Disable all macros without notification
○ Disable all macros with notification
○ Disable all macros except digitally signed macros
● Enable all macros (not recommended; potentially dangerous code can run)

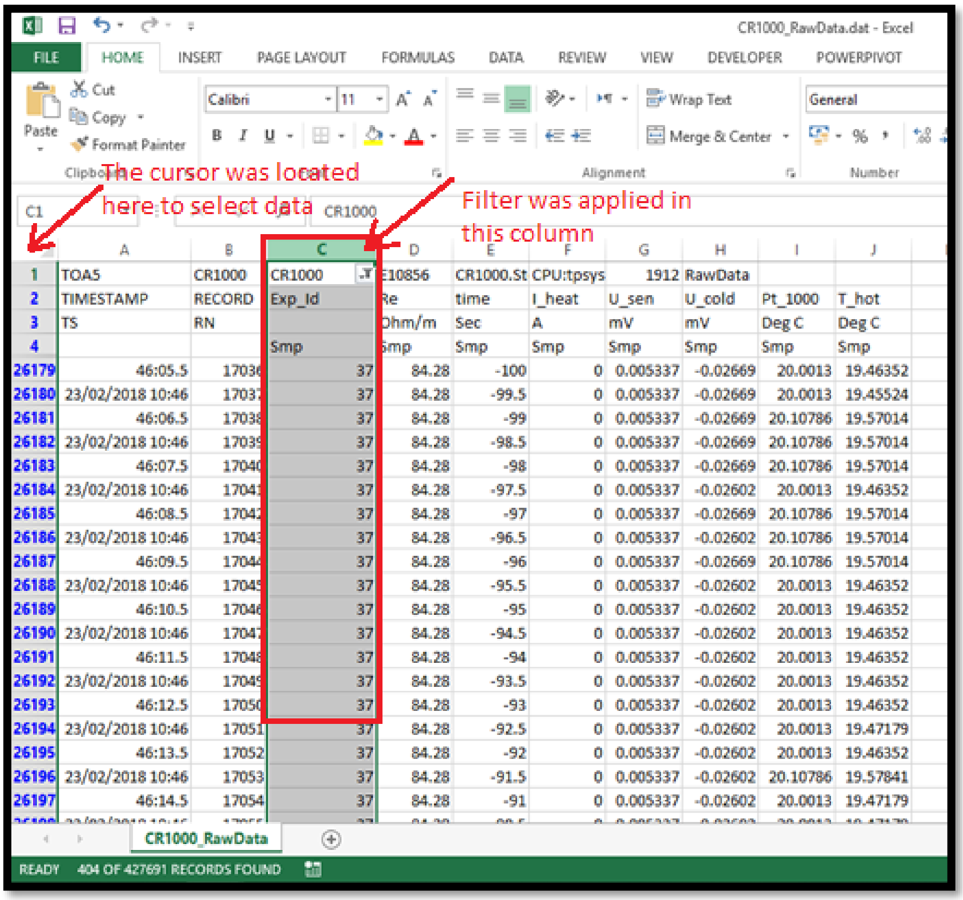
Developer Macro Settings

☐ Trust access to the VBA project object model

Figure AII. 2 – Procedures to edit Macro settings

After collecting the data and enabling Macro to open all the files, the data can be analysed in the excel spreadsheet designed by the Hukseflux Supplier, which will look like the one presented in Figure AII.3.

- To open the results in the spreadsheet, it is required to copy the raw data values from the file called ''CR1000_Raw data'' which was transferred and collected from the MCU.
- Before copying the raw data and after opening the file as shown before in an excel file, a filter can be applied on the column (CR1000/Exp_Id) to define a specific measurement to be analysed. For instance, the experiment Id = 37 was selected. However, by positioning the cursor on the left top corner of the data table in this file, the data can be selected (see Figure AII.3).



The cursor was located here to select data

Filter was applied in this column

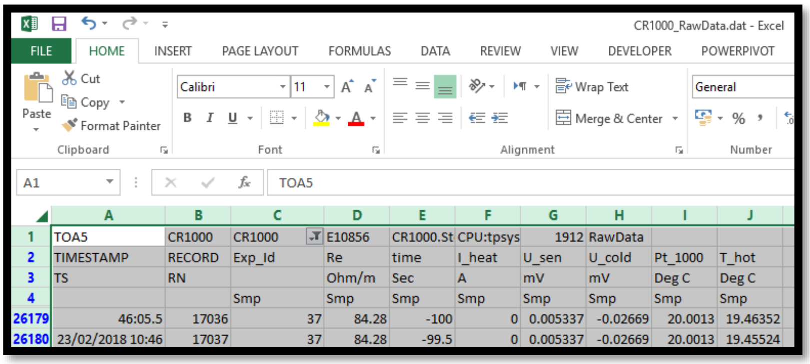
	A	B	C	D	E	F	G	H	I	J
1	TOA5	CR1000	CR1000	E10856	CR1000.St	CPU:tpsys	1912	RawData		
2	TIMESTAMP	RECORD	Exp_Id	Re	time	I_heat	U_sen	U_cold	Pt_1000	T_hot
3	TS	RN		Ohm/m	Sec	A	mV	mV	Deg C	Deg C
4			Smp	Smp	Smp	Smp	Smp	Smp	Smp	Smp
26179	46:05.5	17036	37	84.28	-100	0	0.005337	-0.02669	20.0013	19.46352
26180	23/02/2018 10:46	17037	37	84.28	-99.5	0	0.005337	-0.02669	20.0013	19.45524
26181	46:06.5	17038	37	84.28	-99	0	0.005337	-0.02669	20.10786	19.57014
26182	23/02/2018 10:46	17039	37	84.28	-98.5	0	0.005337	-0.02669	20.10786	19.57014
26183	46:07.5	17040	37	84.28	-98	0	0.005337	-0.02669	20.10786	19.57014
26184	23/02/2018 10:46	17041	37	84.28	-97.5	0	0.005337	-0.02602	20.0013	19.46352
26185	46:08.5	17042	37	84.28	-97	0	0.005337	-0.02669	20.10786	19.57014
26186	23/02/2018 10:46	17043	37	84.28	-96.5	0	0.005337	-0.02602	20.10786	19.57014
26187	46:09.5	17044	37	84.28	-96	0	0.005337	-0.02669	20.10786	19.57014
26188	23/02/2018 10:46	17045	37	84.28	-95.5	0	0.005337	-0.02602	20.0013	19.46352
26189	46:10.5	17046	37	84.28	-95	0	0.005337	-0.02602	20.0013	19.46352
26190	23/02/2018 10:46	17047	37	84.28	-94.5	0	0.005337	-0.02602	20.0013	19.46352
26191	46:11.5	17048	37	84.28	-94	0	0.005337	-0.02602	20.0013	19.46352
26192	23/02/2018 10:46	17049	37	84.28	-93.5	0	0.005337	-0.02602	20.0013	19.46352
26193	46:12.5	17050	37	84.28	-93	0	0.005337	-0.02602	20.0013	19.46352
26194	23/02/2018 10:46	17051	37	84.28	-92.5	0	0.005337	-0.02602	20.0013	19.47179
26195	46:13.5	17052	37	84.28	-92	0	0.005337	-0.02602	20.0013	19.46352
26196	23/02/2018 10:46	17053	37	84.28	-91.5	0	0.005337	-0.02602	20.10786	19.57841
26197	46:14.5	17054	37	84.28	-91	0	0.005337	-0.02602	20.0013	19.47179

READY 404 OF 427691 RECORDS FOUND

Figure AII. 3 – Raw data sheet

- Then, clicking Ctrl + C will copy the entire table of data selected → open the calculation spreadsheet provided by the Hukseflux Supplier → open the sheet called ''Paste raw data here'' and click on the square of the top left corner to select all again and to select where the data will be pasted → click Ctrl + V to paste the raw data values in the Paste raw data sheet;

The raw data sheet when the data will be selected will look like that presented in Figure AII.4.

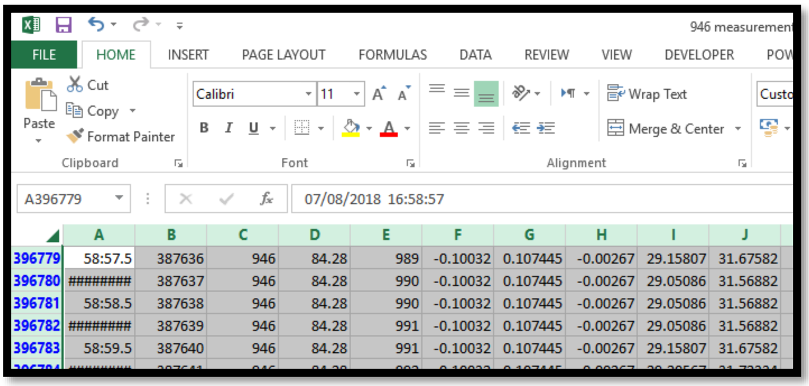


TOA5

	A	B	C	D	E	F	G	H	I	J
1	TOA5	CR1000	CR1000	E10856	CR1000.St	CPU:tpsys	1912	RawData		
2	TIMESTAMP	RECORD	Exp_Id	Re	time	I_heat	U_sen	U_cold	Pt_1000	T_hot
3	TS	RN		Ohm/m	Sec	A	mV	mV	Deg C	Deg C
4			Smp	Smp	Smp	Smp	Smp	Smp	Smp	Smp
26179	46:05.5	17036	37	84.28	-100	0	0.005337	-0.02669	20.0013	19.46352
26180	23/02/2018 10:46	17037	37	84.28	-99.5	0	0.005337	-0.02669	20.0013	19.45524

Figure AII. 4 – Raw data selected in the Raw data sheet

When the raw data are pasted in the calculation spreadsheet, the spreadsheet will look like Figure AII.5.

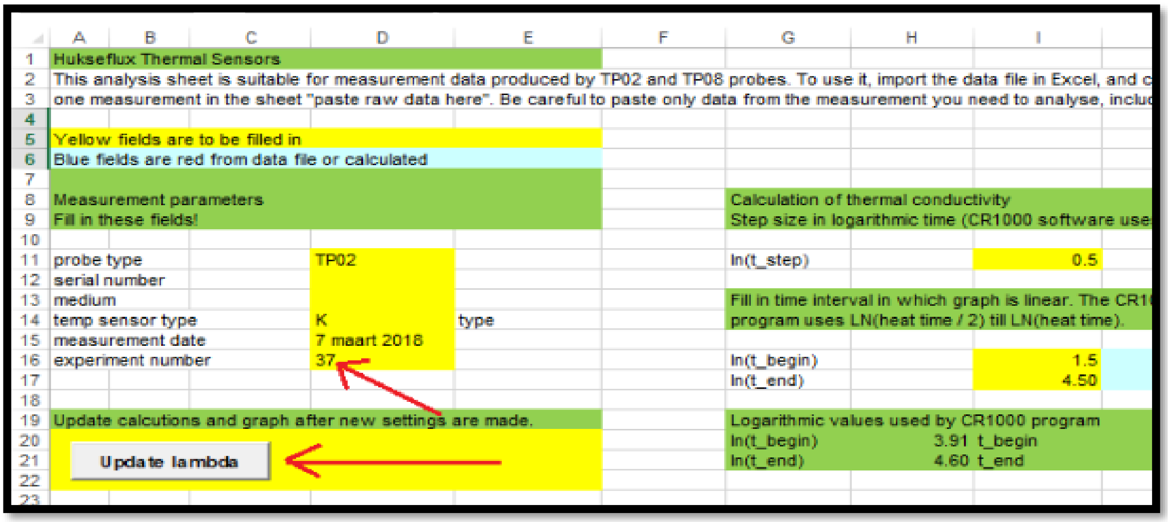


07/08/2018 16:58:57

	A	B	C	D	E	F	G	H	I	J
396779	58:57.5	387636	946	84.28	989	-0.10032	0.107445	-0.00267	29.15807	31.67582
396780	########	387637	946	84.28	990	-0.10032	0.107445	-0.00267	29.05086	31.56882
396781	58:58.5	387638	946	84.28	990	-0.10032	0.107445	-0.00267	29.05086	31.56882
396782	########	387639	946	84.28	991	-0.10032	0.107445	-0.00267	29.05086	31.56882
396783	58:59.5	387640	946	84.28	991	-0.10032	0.107445	-0.00267	29.15807	31.67582

Figure AII. 5 – Raw data pasted in the calculation spreadsheet

The yellow squares in the calculation spreadsheet are to be filled and updated according to the current experiment data here analysed.



1 Hukseflux Thermal Sensors
2 This analysis sheet is suitable for measurement data produced by TP02 and TP08 probes. To use it, import the data file in Excel, and c
3 one measurement in the sheet "paste raw data here". Be careful to paste only data from the measurement you need to analyse, incluc
5 Yellow fields are to be filled in
6 Blue fields are red from data file or calculated
8 Measurement parameters
9 Fill in these fields!
11 probe type: TP02
12 serial number
13 medium
14 temp sensor type: K type
15 measurement date: 7 maart 2018
16 experiment number: 37
19 Update calcutions and graph after new settings are made.

8 Calculation of thermal conductivity
9 Step size in logarithmic time (CR1000 software use
11 ln(t_step) 0.5
13 Fill in time interval in which graph is linear. The CR1
14 program uses LN(heat time / 2) till LN(heat time).
16 ln(t_begin) 1.5
17 ln(t_end) 4.50
19 Logarithmic values used by CR1000 program
20 ln(t_begin) 3.91 t_begin
21 ln(t_end) 4.60 t_end

Figure AII. 6 – Procedures to visualise and analyse the calculations in Hukseflux Supplier spreadsheet

To analyse measurement data, the Experimental number must be changed to the ‘‘Exp_Id’’ of the measurement (e.g. 37 for instance) → click ‘‘update lambda’’;

The thermal conductivity shown as ''lambda'' in numeric screen is a calculation made by the TPSYS02, and based on a graph where ΔT from the sensors in the probe is plotted as a function of the natural logarithm of time (ln (t)). After a transient period, this graph will show linear behaviour, where the slope of the graph is supposed to be inversely proportional to the thermal conductivity.

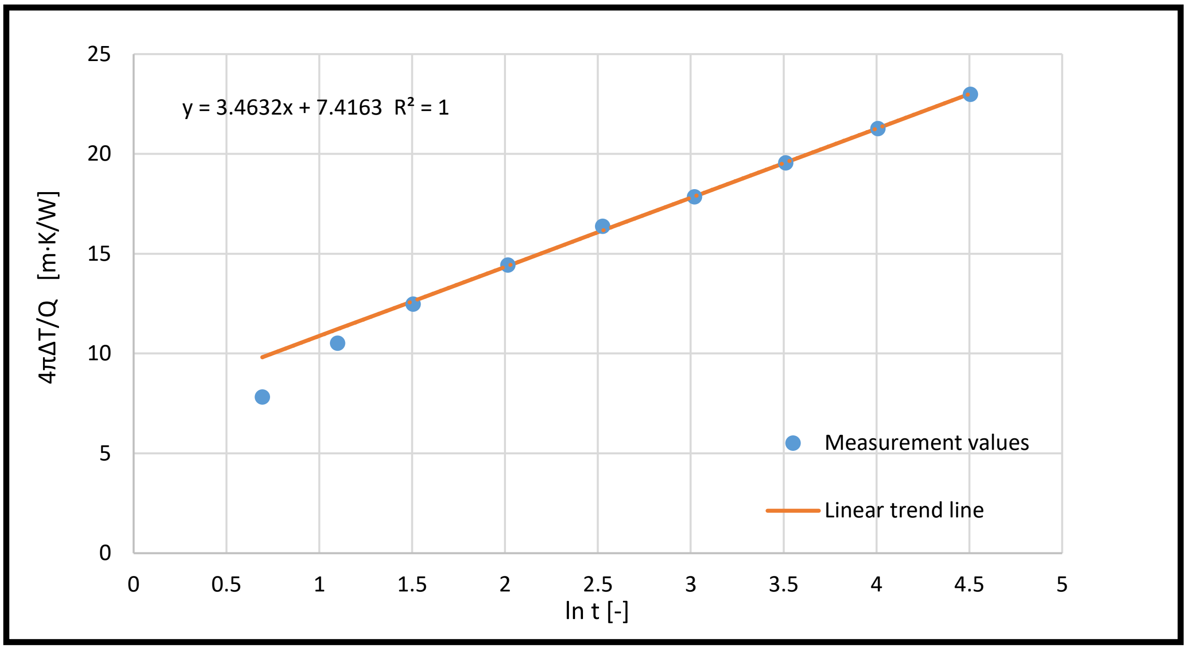
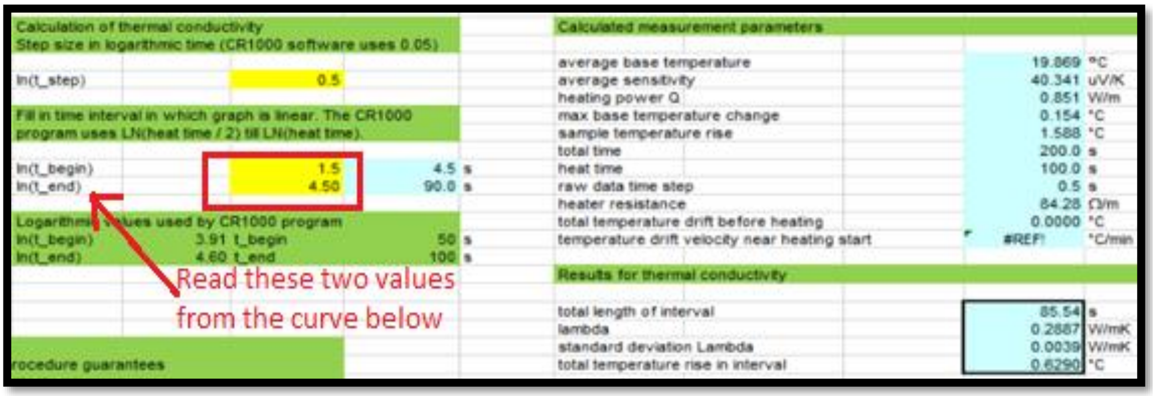


Figure AII. 7 – Visualising the resistance with logarithmic time

Therefore, to obtain the thermal conductivity value, it is required to go to the graph ''Visual analysis in logarithmic time'' and read the two values of logarithmic time between the beginning and the end of the linear part of the curve (see Figure AII.8) → insert these values in ''ln(t_begin)'' and ''ln(t_end)'' in the yellow cells in the TPSYS02 calculation spreadsheet (see Figure AII.6) → click ''update lambda'' again,



Calculation of thermal conductivity
Step size in logarithmic time (CR1000 software uses 0.05)

ln(t_step)	0.5		

Fill in time interval in which graph is linear. The CR1000 program uses LN(heat time / 2) till LN(heat time).

ln(t_begin)	1.5	4.5	s
ln(t_end)	4.50	90.0	s

Logarithmic values used by CR1000 program

ln(t_begin)	3.91	t_begin	50	s
ln(t_end)	4.60	t_end	100	s

Read these two values from the curve below

rocedure guarantees

Calculated measurement parameters

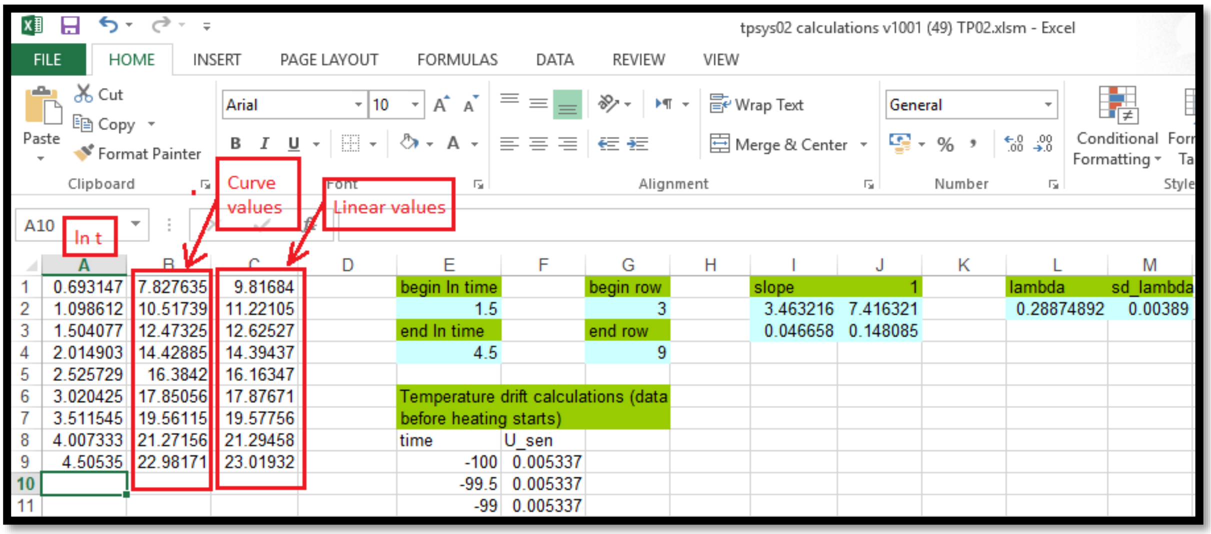
average base temperature	19.869	°C
average sensitivity	40.341	uV/K
heating power Q	0.851	W/m
max base temperature change	0.154	°C
sample temperature rise	1.588	°C
total time	200.0	s
heat time	100.0	s
raw data time step	0.5	s
heater resistance	84.28	Ω/m
total temperature drift before heating	0.0000	°C
temperature drift velocity near heating start	#REF!	°C/min

Results for thermal conductivity

total length of interval	85.54	s
lambda	0.2887	W/mK
standard deviation Lambda	0.0039	W/mK
total temperature rise in interval	0.6290	°C

Figure AII. 8 – Spreadsheet calculating thermal conductivity

In Figure AII.7, the curve is being approximated to a line. Its equation is also presented in in Figure AII.7 ($y = 3.4632x + 7.4163$). Those values are automatically calculated for each measurement and presented in ''Internal auxiliary'' sheet of the excel file as it can be seen in Figure AII.9;



ln t

Curve values

Linear values

	A	B	C	D	E	F	G	H	I	J	K	L	M
1	0.693147	7.827635	9.81684		begin ln time		begin row		slope	1		lambda	sd_lambda
2	1.098612	10.51739	11.22105		1.5		3		3.463216	7.416321		0.28874892	0.00389
3	1.504077	12.47325	12.62527		end ln time		end row		0.046658	0.148085			
4	2.014903	14.42885	14.39437		4.5		9						
5	2.525729	16.3842	16.16347										
6	3.020425	17.85056	17.87671		Temperature drift calculations (data								
7	3.511545	19.56115	19.57756		before heating starts)								
8	4.007333	21.27156	21.29458		time	U_sen							
9	4.50535	22.98171	23.01932		-100	0.005337							
10					-99.5	0.005337							
11					-99	0.005337							

Figure AII. 9 – Initial auxiliary sheet

Read the ‘‘lambda’’ values, which indicates the measured value of the material thermal conductivity and the real one will be calibrated using the calibration factor Cf;

Figure AII.10 represents the power in (mV) per time during the measurement cycle.

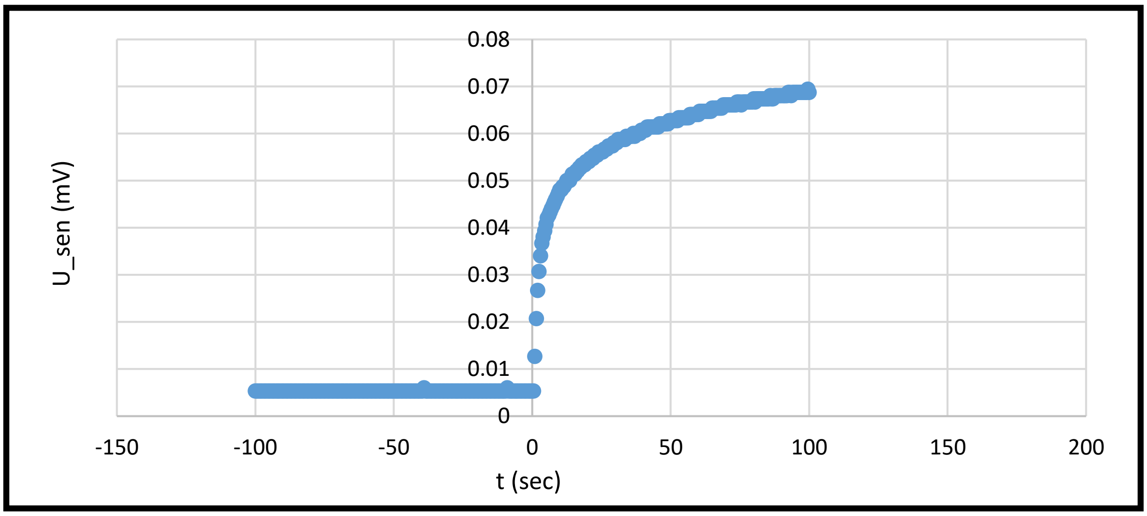
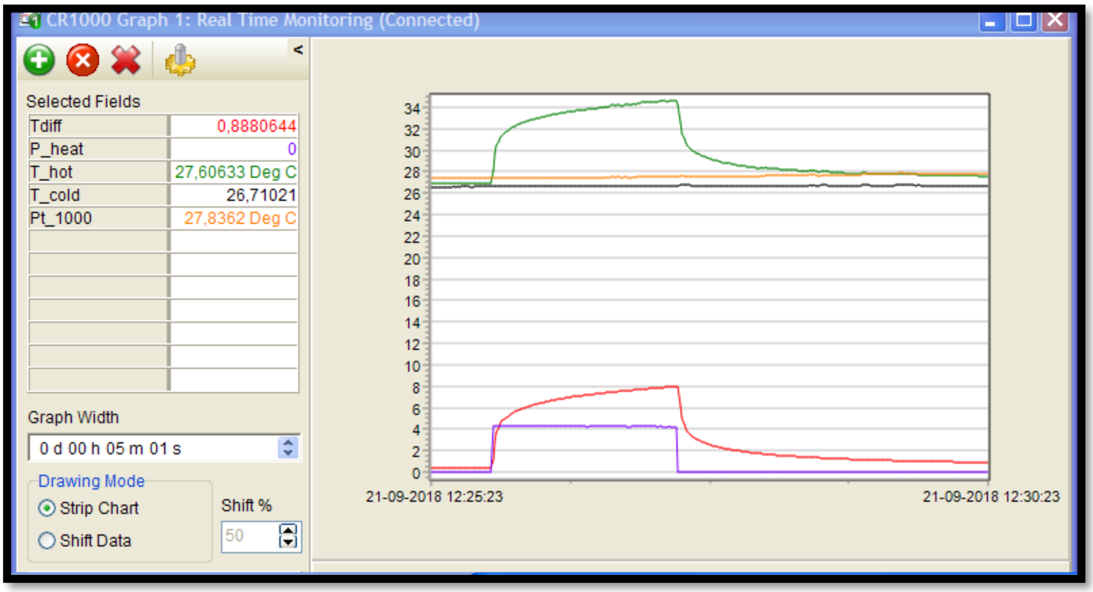


Figure AII. 10 – U_sen (mV) vs. time (sec)

This chart can be also monitored by the Graph 1 displayed as explained before, if the user has selected the correct parameters which are in this case the Heat_time (sec) and the voltage of the sensor (mV).

The Graph 1 can also display the relation between sensor’s temperature and the heating power, which is also a curve similar to the curve presented in Figure AII.11.



CR1000 Graph 1: Real Time Monitoring (Connected)

Selected Fields

Tdiff	0,8880644
P_heat	0
T_hot	27,60633 Deg C
T_cold	26,71021
Pt_1000	27,8362 Deg C

Graph Width: 0 d 00 h 05 m 01 s

Drawing Mode
● Strip Chart
○ Shift Data

Shift %: 50

Figure AII. 11 – Graph 1 monitoring T_hot, T_cold, Pt_1000, P_heat and Tdiff

Where: T_hot, T_cold, Pt1000 are parameters explained in the fourth chapter and P_heat is the heating power provided by the systems (W.m^{-1}). Tdiff is explained in the Annex V.

ANNEX III
(Calibration procedures)

Calibration here is applied using a reference material which is glycerin with plastic fibers, following the list of steps below:

- Weight the recipient empty Wr (gr);
- Weight 1 liter of pure glycerin Wg (gr);
- Prepare fibers by weighting 2% of the weight of glycerin Wg, as presented in Figure AIII.1;
- Adding the glycerin slowly to the recipient which has the plastic fibers inside it and confirm that the fibers with the glycerin are making a homogenious mixture.

Table AIII. 1 – Calculations for the preparation of the calibration

Category	Weight (kg)
Recipient Wr	0.0869
1liter of glycerin Wg	1.150
Plastic fibers Wf = %2 x Wg (kg)	0.023

Figure AIII. 1 – Preparation procedures of calibration

- Inserting the needle which is here TP02, and fix it as presented in Figure AIII.1 to avoid touching it during the calibration;
- Display numerical window and insert the parameters as presented in the Figure AIII.2 and insert lambda_ref as 0.285 (W.m^{-1}.K^{-1});
- Set Heat_time to 100s, as recommended in TPSYS02 manual, and then the measurement time will be automatically set to 200s which is countdown time;
- In Ports and Flags window, select Low_heating, and then click start calibration and the calibration will start and the countdown will start counting from 200s to 0s;
- The calibration cycle is, as the measurement cycle, constituted of two phases. The first is 100s, where the system is giving time for the temperature of the needle to stabilise and the sensor is measuring the temperature without applying the power and without any heating. In the following 100s, the Heating_on green light will be turned on automatically, confirming that the heating is going on, and the temperature will increase, and consequently the Tdiff will increase as well;

- At the end of the calibration, the countdown reaches zero and the temperature starts to cool down decreasing the Tdiff and the numerical display will seem as presented in Figure AIII.2 with all the parameters and with the measured Lambda (Lambda) and the lambda of the material that is calculated according to the following equation, considering the temperature coefficient ‘‘ec’’:

$$\lambda_{cal} = \lambda_{ref} + ec * \Delta T$$

Where:

λ_{ref} is a reference value (at zero degrees Celsius);

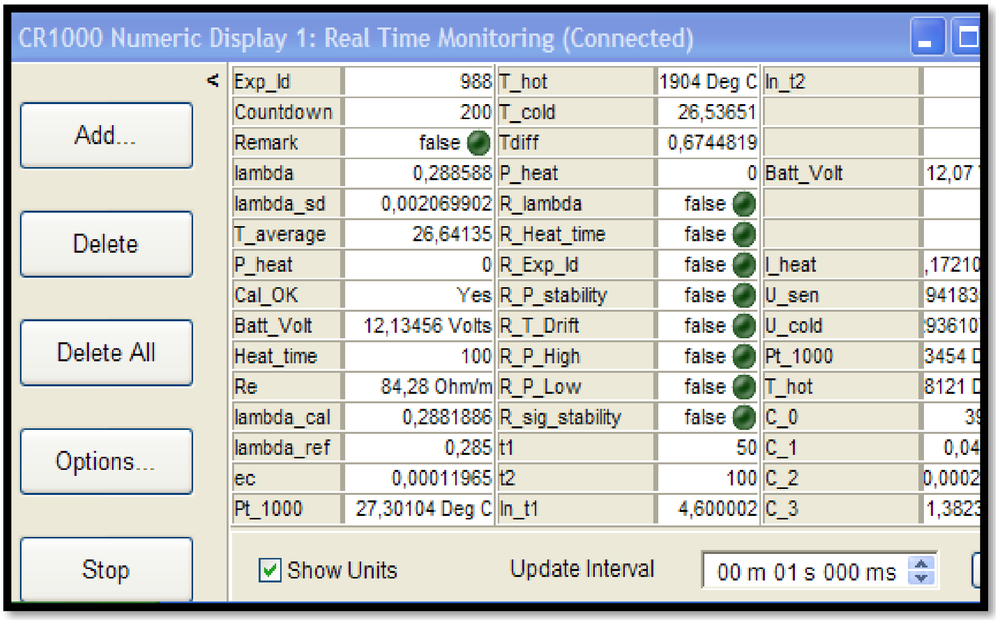
ec is a temperature coefficient. In case the temperature dependence is negligible, “ec” can be set to zero.

ΔT is the temperature of the material in the recipient before the measurement with respect to zero degrees Celsius.

The parameter “Cal_OK” should read “Yes” if the value for Lambda is more or less that set literature value within 5%. Lambda_sd should have a value lower than 0.03.

Note: It is recommended to wait 10 minutes after the measurement to read again the changes of Tdiff per minute which should be less than ±0.1ºC.

Figure AIII.2 represents the calibration results, which were approved by the system, since the previous two conditions are validated and Cal_OK indicates Yes.



CR1000 Numeric Display 1: Real Time Monitoring (Connected)

Exp_Id	988	T_hot	1904 Deg C	In_t2	
Countdown	200	T_cold	26,53651		
Remark	false ●	Tdiff	0,6744819		
lambda	0,288588	P_heat	0	Batt_Volt	12,07
lambda_sd	0,002069902	R_lambda	false ●		
T_average	26,64135	R_Heat_time	false ●		
P_heat	0	R_Exp_Id	false ●	I_heat	,17210
Cal_OK	Yes	R_P_stability	false ●	U_sen	94183
Batt_Volt	12,13456 Volts	R_T_Drift	false ●	U_cold	93610
Heat_time	100	R_P_High	false ●	Pt_1000	3454 D
Re	84,28 Ohm/m	R_P_Low	false ●	T_hot	8121 D
lambda_cal	0,2881886	R_sig_stability	false ●	C_0	39
lambda_ref	0,285	t1	50	C_1	0,04
ec	0,00011965	t2	100	C_2	0,0002
Pt_1000	27,30104 Deg C	In_t1	4,600002	C_3	1,3823

☑ Show Units

Update Interval: 00 m 01 s 000 ms

Figure AIII. 2 – Numerical display once the calibration ends

Therefore, applying the equation (7), calibration factor could be calculated and its values is equal to:

$$C_f = \frac{0.285}{0.2886} = 0.9875$$

This factor will be used to calibrate all the values of thermal conductivity that will be measured of any material by multiplying its value by the average thermal conductivity of the material.

ANNEX IV
(Thermocouple)

The sensors used in TPSYS02 equipment consist of thermocouple type K polynomial. According to American National Standards Institute ANSI MC96.1-1882, the voltage measured in the cold joint of the sensor can be calculated using the following equation:

$$v_{CJ} = v_0 + \frac{(T_{CJ} - T_0)\left(p_1 + (T_{CJ} - T_0)\left(p_2 + (T_{CJ} - T_0)\left(p_3 + p_4(T_{CJ} - T_0)\right)\right)\right)}{1 + (T_{CJ} - T_0)\left(q_1 + q_2(T_{CJ} - T_0)\right)} \tag{8}$$

Where: T_{CJ} is the cold junction temperature, V_{CJ} is the computed cold junction voltage, T_0, V_0, p_i and q_i are coefficients differ from thermocouple to another. Table AIV.1 presents values of these coefficients in case of thermocouple type K (Childs, 2001).

Table AIV. 1 – Coefficients values of a thermocouple type K

Voltage (mV)	Temperature (°C)	To	Vo	p1	p2	p3	p4	q1	q2
-0.778 to 2.851	-20 to 70	2.500 x10^{+1}	1.000 x10^{+0}	4.051 x10^{-2}	-3.879 x10^{-5}	-2.861 x10^{-6}	-9.537 x10^{-10}	-1.395 x10^{-3}	-6.798 x10^{-5}

The voltage calculated in the previous equation must be converted to temperature following the equation presented below:

$$T = a_0 + x * \left[a_1 + x * \left(a_2 + x * \left(a_3 + x * \left(a_4 + x * (a_5)\right)\right)\right)\right] \tag{9}$$

Where: T = Temperature,

x = Thermocouple EMF in Volts,

a = Polynomial coefficients unique to each thermocouple,

n = Maximum order of the polynomial and as (n) increases, the accuracy of the polynomial increase.

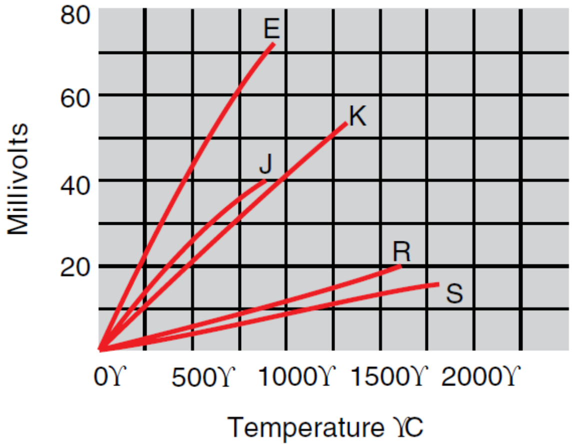


Figure AIV. 1 – Thermocouple temperature vs. voltage graph

Using this thermocouple type K implies using the following equation to calculate sensitivity of hot joint:

$$\text{Sensitivity}_{\text{hot}}\text{joint} = C_6 * T_{hot}^6 + C_5 * T_{hot}^5 + C_4 * T_{hot}^4 + C_3 * T_{hot}^3 + C_2 * T_{hot}^2 + C_1 * T_{hot}^1 + C_0 \tag{10}$$

Where sensitivity coefficients' values are presented in Table AIV.2:

Table AIV. 2 – Sensitivity coefficients' values

C_0	C_1	C_2	C_3	C_4	C_5	C_6
39.455	4.8034x10^{-2}	-2.8925x10^{-4}	1.3823x10^{-6}	-2.8357x10^{-8}	1.7839x10^{-10}	-3.1836x10^{-13}

ANNEX V
(Thermal Conductivity Calculations)

As explained before in the third chapter, calculating thermal conductivity of soils applying the heating power in a hot wire requires calculating Q, the power per meter provided in the hot wire which in this case (the needle probe). Therefore, Ohm law was used to start these calculations applying the following equation:

$$I\ (Amber) = \frac{V\ (volts)}{R\ (Ohm)} \tag{11}$$

Where:

V is the voltage measured across the conductor (Volts),

R is the total resistance in heating wire (Ω).

The power per meter $\left(\frac{W}{m}\right)$ can be given by the following equation:

$$Q = \frac{E * I}{L} \tag{12}$$

Where:

E is the measured voltage,

I is the current flowing through the heating wire,

L is the length of heated needle (m).

And since:

$$E = \frac{V\ (volts)}{l\ (m)} \tag{13}$$

Thus, power per meter can be expressed by in the following equation:

$$Q\left(\frac{W}{m}\right) = \frac{I^2 * R}{L} \tag{14}$$

After determining the power per meter value using the previous equations and difference in temperature between the hot joint and the cold joint must be calculated following the equation presented below:

$$Tdiff = \frac{(U\_sen) * 1000}{Sensitivity\_hot\ joint} \tag{15}$$

Where:

U_sen is the voltage signal from the hot thermocouple junction,

Sensetivity_hot joint is explained in the Annex IV.

Therefore, following the equation (7) provided in the third chapter, thermal conductivity can be calculated as follows:

$$Tdiff = \Delta T = \frac{Q}{4\pi\lambda} \ln\left(\frac{t_2}{t_1}\right) \tag{16}$$