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# A Laboratory Procedure to Determine the Thermal Properties of Silt Loam Soils Based on ASTM D 5334

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**Abstract** Nowadays, there are three variables measured for the characterization of the thermal properties in porous media and soft rocks. These are the volumetric specific heat capacity, the thermal conductivity and the thermal diffusivity. Recently, Decagon Devices has developed the KD2-Pro meter. This instrument is a useful device, which permits storing more than 4000 thermal data. This company has also improved the design of sensors for this specific use. KD2-Pro uses the infinite line heat pulse method based on current standard ASTM D 5334. Nevertheless, it is not clear how to obtain the required reliability and accuracy in the measurements since neither the standard nor manufacturer's user manual do not include any method or recommendation for that. Therefore, a strong methodology is required to achieve the maximum efficiency when KD2-Pro is used. This work presents the first "stadium" towards the development of a laboratory and field methodology to obtain reliability, accuracy and rapidity in the analytical dataset of thermal properties in porous media.

***Keywords:*** *thermal conductivity, thermal diffusivity, volumetric heat capacity, water content, volume of air*

## 1. Introduction

Since 1822 Fourier published the Theorie Analytique de la Chalour, many authors have presented several scientific works related to the thermal properties of soil, as Carslaw and Jaeger [1] and DeVries [2] for instance.

Fourier's law presents the relationship between heat flux and temperature gradient. This sort of dependence between these variables can only be understood as a heat transfer by conduction mechanism. Typically, three parameters are measured to characterize the thermal properties of any porous media: volumetric specific heat capacity, thermal conductivity and thermal diffusivity. Thermal properties are strongly influenced by physical properties such as bulk density, water content, particle-size distribution, and structural arrangement. Therefore, these factors have to be taken into account when performing measurements at laboratory and field scales.

Recently, Decagon Devices has developed the KD2-Pro meter logger, and two specific sensors; the SH-1 and TR-1. The SH-1 thermal sensor (1.3mm diameter, 30mm long and 6mm spacing) measures the three thermal properties by employing the dual needle heat pulse method (DNHP), whereas the TR-1 thermal sensor ( 2.4mm diameter and 100mm long.) is a single needle employing an infinite line heat pulse method (ILHP). The methodology of these devices is based on ASTM D5334. American's standard D5334 is applicable for both undisturbed and remoulded soil specimens as well as soft rocks specimens, yet this test method is suitable only for isotropic materials [3]. In order to obtain a reliable thermal dataset, a simple field and laboratory methodology needs to be adapted and depicted, according to existing standards and owing to neither the manufacturer nor the standard present whatever methodology. Also, since soil scientists, engineers and other current users are demanding these kinds of data for their different applications. The present work describes the first step towards the development of a laboratory procedure to obtain reliable, accurate and rapid thermal properties dataset in soils, taking into account the current accepted standard [3] as a work method that KD2-Pro is based on.

## 2. Methodology

Samples were obtained from the top soil horizon (0-30 cm depth) of a plot placed at Can Solé Road (Figure 1), located in the Llobregat delta plain (Northeast of Spain).

### 2.1. Soil Properties

To characterize the soil of Can Solé Road, the physical variables, particle size distribution, bulk density, total organic carbon content, and calcium carbonate content were measured. In addition, the residual water content (hygroscopic water) was determined. Particle-size distribution was determined using the wetting sieve method for 2000 to 500μm, and a device by dispersion laser beams (Malvern Mastersizer/E) for particles smaller than 500μm. Bulk density and total porosity were determined from undisturbed sample volumes. Total carbon content was analyzed by loss on ignition at 900°C, and inorganic carbon content by loss on ignition at 200°C, both using a Shimadzu SSM-5000A and solid sample module. These results allowed calculations of both total organic carbon content and calcium carbonate content.

The residual water content was determined by loss in weight after drying the samples at 105°C for 24h.

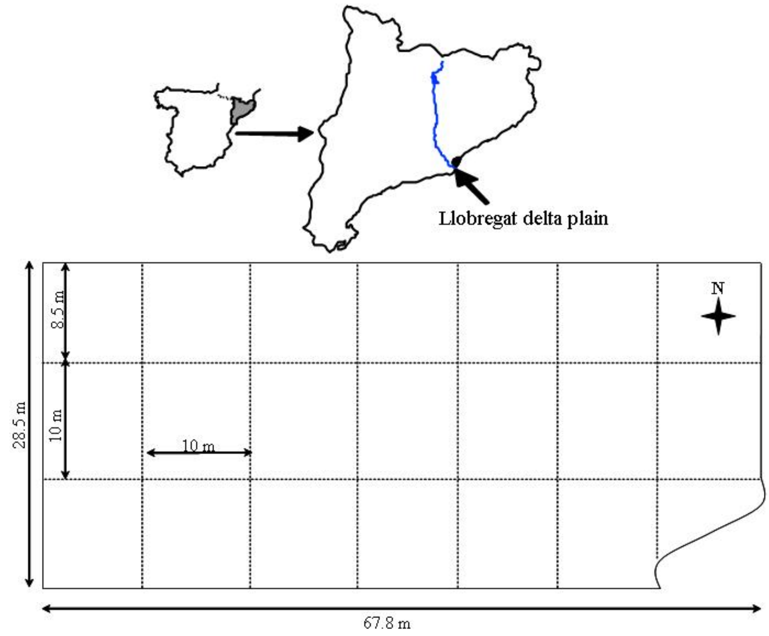


**Figure 1.** Map of the Can Solé Road at Llobregat delta plain, showing location of the sampling plot

Measurements of thermal-hydrodynamic properties were made on soil columns (10cm height and 13cm diameter), constructed specifically for this experiment. Several sensors were placed inside of the device, allowing control of moisture content and thermal properties as well as temperature of the sample.

To determine the thermal properties, two thermal sensors, one small dual-needle sensor (SH-1) and one single needle sensor (TR-1) were employed. These kinds of sensors use the heat pulse methodology and yield reliable soil thermal data. Using a SH-1 sensor, a data set with thermal diffusivity ($\alpha$), thermal resistivity (R) and the inverse thermal conductivity ($\lambda$) and volumetric specific heat capacity (Cv) estimations are obtained. On the other hand, the TR-1 sensor only measures the thermal resistivity and the inverse thermal conductivity.

The thermal data were collected using a KD2-Pro reader-logger. The KD2_Pro resolves 0.001ºC in temperature. It uses special algorithms to analyze measurements made during a heating and a cooling interval. It also uses special algorithms to separate out the effects of the heat pulse from ambient temperature changes. The algorithms are based on the transient line heat source analysis given in [1,10]. Thus, from both thermal properties the volumetric specific heat capacity is calculated. To determine the volumetric water content ($\theta$), the soil column was monitored with $ECH_2O$ EC-5 frequency domain probe. A Decagon Devices Em-5b data-logger was required to collect the water content and temperature isothermal chamber data.

## 2.2. Field Sampling Design

The first step in developing a protocol to measure the thermal properties of soil begins for the field sampling design, i.e. to choose a representative unit for sampling. Field observations and preliminary prospection must be performed. In this work, disturbed samples from a silt loam soil were taken.

Some considerations must be taken during this stage:

- Calibration of the thermal sensor
- Definition of the thermal sensor placement
- Position of the needle with respect to surface
- Extraction of the sample
- Determination of bulk density *in situ*
- Determination of water content and thermal properties *in situ*.

## 2.3. Analytical Laboratory Procedure

This method is applicable for both, unaltered and repacked soil specimens, which are suitable only for isotropic materials. Hetero-metric materials must be taken into account for repacking soil samples.

After the sample is air-dried, it is sieved to 2000µm and repacked inside the column device to a target bulk density. In this case, the bulk density should be similar to the value measured in the field. If the sample presents large quantity of coarse elements, these must be taken into account when the sample is repacked.

Once the soil sample column is ready the next step will be to place the thermal sensors inside the device. Usually, we recommend inserting more than one TR-1 or SH-1 thermal sensor for each column device. The experience indicated that few measurements are required to obtain reliable results, and assessing the uncertainty of the measurements. A comparison between both sensors was not capable owing to the different volume fraction measured for each type of sensor.

On the other hand, KS-1 thermal sensor is strongly not recommended for these types of samples, since KS-1 is only useful and recommended to measuring thermal conductivity in liquids.

To wet up the sample, we use two different techniques; (i) dynamic technique, where thermal properties and water content measurements are taken as water rises by capillarity from the bottom of the column; and (ii) static technique, where measurements are taken after water was added to the soil, mixed thoroughly and repacked. Afterwards, thermal sensors were placed in two different positions, vertically and horizontally and left up to reach the steady state conditions for each scenario or water content. One of the most important steps is to avoid the evaporation fluxes between the surface of column device and environment. The time required to reach equilibrium inside the column device depends on the temperature of both the sample and water [9]. To avoid convective fluxes the device was placed inside an isothermal chamber which maintained the experiment temperature around 17°C.

Others aspects taken into account were the quantity of observations, and the number of measurements for each observation. An observation is defined as one thermal sensor placed in the sample several measurements were collected per sensor to ensure sufficient data to determine significance of the values (Table 1). Thermal sensor TR-1 measured every 15 minutes, and it used 2 minutes to perform every determination. Thermal sensor SH-1 measured every 30 minutes, and used 2 minutes to estimate the measure as well. Each TR-1 thermal sensor collected 7 measures per water content, i.e. a total of 21 measures per scenario.

On the other hand, the SH-1 thermal sensor recorded 630 data using a KD2-Pro continuous mode. As far as the water content is concerned, several scenarios were determined using a data-logger and FDR probe. The measures were collected at the same instant as that of the thermal data. Thus, thermal data and water content could be related. The average temperature was 17.3°C.

**Table 1. Paired samples T-test calculated for each moisture content scenario. To compare the thermal resistivity values between vertical and horizontal sensor placement. Each scenario means one group class of soil moisture defined by FDR probe**

| Moisture | Correlation | Std. Desv. |
|---|---|---|
| Scenario 1 | .845 | 1.362 |
| Scenario 2 | .959 | 1.531 |
| Scenario 3 | .826 | 0.096 |
| Scenario 4 | .689 | 0.288 |
| Scenario 5 | .170 | 0.158 |

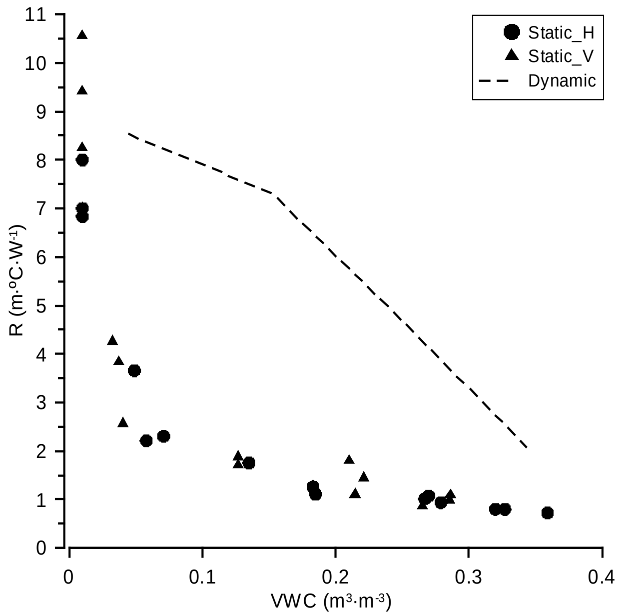


**Figure 2.** Comparing different wetting processes to determine the thermal resistivity (R) as a function of volumetric water content (VWC) for a silt loam soil. H: parallel to surface; V: perpendicular to surface.

The data obtained with the perpendicular sensor showed large thermal resistivity values when the sample was air-dried. However, the thermal resistivity values were lower than the data obtained with the parallel sensor when the water content was close to saturation. Therefore, for a silt loam soil, the thermal resistivity (R) showed a gradual decrease insofar as water content increased [5]. The soil presented a strong reaction when soil moisture was higher than 10% vol·vol$^{-1}$ for the static technique, and close to 20% vol·vol$^{-1}$ for the dynamic technique. This fact did suggest a discrete wetting front occurred with the dynamic method. Therefore, the greatest decrease in R occurred during the wetting process range, assuming a constant slope [6,7]. Similar results were showed by Al Nakshabandi and Kohnke [6] with the same type of soil textural class.

Often, a common approach to present soil thermal properties has been to plot these properties as a function of water content. But less commonly, thermal properties have been plotted as a function of volume fraction of air (Φ, m3·m-3) [8]. An experiment using SH-1 thermal sensor was carried out. This type of thermal sensor estimates the three thermal properties; i.e. thermal conductivity, thermal diffusivity and volumetric specific heat capacity [7]. SH-1 uses a dual needle heat pulse. Dual needle allows estimate thermal conductivity and thermal diffusivity to calculate the volumetric specific heat capacity.

Figure 3 shows the relationship between λ, α and Cv vs Φ. Volume fraction of air was calculated once water content and particle density were known, since the sum of

# 3. Results and Discussion

## 3.1. Soil Properties

The studied soil from Can Solé Road was classified as silt loam textural class [4], with a particle size distribution for silt content, always, higher than 60%, mean sand content about 34%, and mean clay content about 4%. Mean bulk density is 1.47g·cm$^{-3}$ and total porosity 45%. Mean total organic carbon content was about 3.1%, mean calcium carbonate content was 40.3%.

## 3.2. Thermal Properties And Hydrodynamics of The Soil

Figure 2 shows the comparison for different positions of the thermal sensor and different wetting processes, to determine the thermal resistivity. For this example, we have chosen the inverse thermal conductivity variable as it is the most frequently thermal property used in many applications such as civil engineering, basic thermal science, among others. In this experiment the thermal sensor was the TR-1 single needle.

The influence of water content in the thermal resistivity is observed when using either one of the two methods (static and dynamic) and two different sensor placements. The thermal resistivity obtained with the dynamic wetting technique always presented higher thermal resistivity on the whole of the dry out curve values than obtained by static technique. The effect of the position of the sensor inside the soil sample (perpendicular or parallel to sample surface) did not present significant differences between both positions in terms of the thermal resistivity values. In spite of the fact, minimal differences could be observed among the measurements (Table 1).

the volume fraction is 1. The thermal conductivity data in Figure 3 shows the variation in λ can be explained by the variation in Φ values. On the whole, the increase of fraction of air in the soil sample showed an inverse relationship with the thermal properties, except for α values. The thermal diffusivity values did not present a linear dependence on the volume fraction of air. The relationship between λ, Cv and Φ was stronger (r = 0.98, Figure 3) than the relationship between α and Φ (r= 0.95, Figure 3). Therefore, volume fraction of air exerts a limiting effect on thermal conductivity [8] and volumetric heat capacity in these measured conditions for silt loam soil.

On the other hand, the variations in the volume of the air fraction explained much of the variation in the thermal diffusivity data compared to other variables.

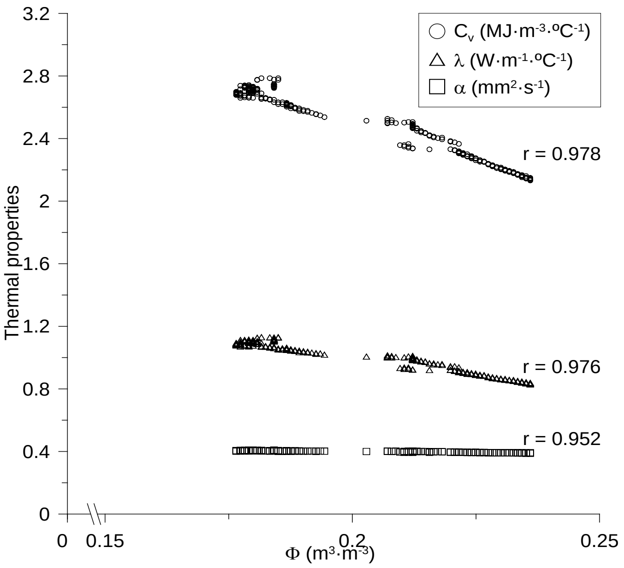


**Figure 3.** Thermal conductivity (λ), volumetric heat capacity (Cv), and thermal diffusivity (α) versus volume fraction of air (Φ) for the studied soil. R indicates the coefficient of correlation between Φ and thermal properties data-set

## 4. Conclusions

Sampling is a crucial stage in the evaluation of soil thermal properties, and the correct decision must be taken following a validated procedure. In fact, it is especially important to obtain reliable results and a lower uncertainty of the registered data.

Preliminary results showed that the new procedure was suitable for all cases, although the soil properties measured in situ were not relevant for the studied case. Only, the different characteristics of each porous media must be taken into account. The method also can be used where the structure of the porous media is relevant to evaluate the hydrodynamic properties.

On the other hand, the special design of the column device was highly effective. The experiment showed several interesting features. Thermal properties showed an acceptable relationship with water content. Also, these measurements could be described as a decreasing linear function of the air-filled porosity.

Indeed, one of the most relevant contributions in this work was about the dynamic method. It is not useful especially when measuring interval time is shorter, thus it is not recommended. This is because it is not possible for the system to reach the equilibrium. The evaporative fluxes as well as the wetting up process presented discrepancies about the thermal resistivity values when these were compared with the static method to wet up the sample.

In spite of the fact it would be beneficial to continue the investigations of the soil thermal behaviour, by studying more variables, which can be especially sensitive. These types of variables include the bulk density, mineralogy and stony porous medium. Even though the experiment was carried out with isotropic samples, a new approach in thermal properties for anisotropic and hetero-metric media should be performed.

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