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Title	Thermal Properties Measurement Device Proposal for Soils and Insulation
Student	Beaufait, Robert Peter
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Lecturer	Worlitschek, Jörg
External examiner	Lieball, Kai

Abstract German

Das Konzept der Nutzung des Bodens als Energiespeichermedium ist für Industrie und Energienetzplaner von großem Interesse. Es wurde erfolgreich als Quelle für Wärmepumpen als für die Gebäudeheizung eingesetzt. Der nächste Schritt besteht darin, ein skalierbares Design zu evaluieren, das den Energiespeicherbedarf einer Gemeinde oder potenziell einer Region decken kann. Die Leistung eines solchen Systems erfordert genaue thermophysikalische Messungen von Böden und thermischer Isolierungen. Dieser Bericht untersucht die verschiedenen Methoden und Techniken, um eine Empfehlung für ein Messgerät für Wärmeleitfähigkeit und/oder Temperaturleitfähigkeit für poröse, feuchte Materialien, insbesondere Böden und Wärmedämmung. Keine zwei Methoden oder Techniken sind gleich. Die Bestimmung der besten Methode und Technik für poröse, feuchte Materialien steht im Mittelpunkt dieser Arbeit. Jede Technik wird auf ihre Eignung zur Messung poröser, feuchter Materialien über die Bereiche Temperatur, Feuchtigkeit und Wärmeleitfähigkeit überprüft, die für feuchte Böden und Wärmedämmungen anwendbar sind. Insbesondere werden zwei transiente Techniken zur Messung der Wärmeleitfähigkeit von Bodenproben aus dem Thurgau und Quarzsand aus der Region Perth in Australien getestet und ausgewertet. Die Ergebnisse werden mit Hilfe von Referenzmaterialien und empirischen Studien und Modellen auf Qualität und Genauigkeit analysiert und verglichen. Die Ergebnisse erweisen sich als nicht schlüssig für genaue Messungen, stehen aber im Einklang mit dem, was in anderen Studien gefunden wurde. Ein überarbeiteter Blick auf die Probenvorbereitung und modernste Sensorik bietet die Möglichkeit, genauere Ergebnisse mit besserer Sicherheit zu erzielen.

Abstract English

The concept of using the ground as an energy storage medium has become of great interest to industry and energy network designers. It has been successfully applied as a source for heat pumps as a sustainable method of heating buildings. The next step is to evaluate a scaled-up designs that can handle the energy storage demand of a community or potentially a region. The performance of such a system requires accurate thermophysical measurements of soils and thermal insulations. This report investigates the various methods and techniques to recommend a

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measurement device for thermal conductivity and/or thermal diffusivity for porous moist materials, specifically soils and thermal insulation. No two methods or techniques are the same and favor some materials and measurement conditions over others. Determining the best method and technique applicable to porous moist materials is the focus of this paper. Each technique is reviewed based on its suitability to measure porous moist materials over the ranges of temperature, moisture, and thermal conductivity applicable to moist soils and thermal insulations. More specifically two transient techniques are tested and evaluated for the purpose of measuring thermal conductivity of soil samples taken from the Thurgau region of Switzerland and quartz sand from the Perth region of Australia. The results are analyzed for quality and accuracy using reference materials and empirical studies and models for comparison. The findings prove inconclusive results for accurate measurements but are consistent with what other studies have found. A revised look at sample preparation and state-of-the-art sensor technology provides an opportunity to obtain more accurate results with better confidence.A

Place, date

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Bachelor's Thesis

Thermal Properties Measurement Device Proposal for Soils & Insulation

Robert Beaufait

Grimselweg 10, 6005 Luzern, Switzerland

robertpeter.beaufait@stud.hslu.ch; +41 78 791 19 70

Supervising Lecturers:

Dr. Willy Villasmil, Senior Research Associate

willy.villasmil@hslu.ch; +41 41 349 39 67

Technikumstrasse 21, 6048 Horw, Switzerland

Benjamin Sponagle, Research Associate

benjamin.sponagle@hslu.ch; +41 41 349 33 84

Technikumstrasse 21, 6048 Horw, Switzerland

Expert:

Prof. Dr. Jörg Wortlischek, Chair Competency Center Thermal Energy Storage (CCTES)

joerg.wortlischek@hslu.ch; +41 41 349 39 57

Technikumstrasse 21, 6048 Horw, Switzerland

Lucerne University of Applied Sciences and Arts - School of Engineering and Architecture

Bachelor of Science, Energy Systems Engineering

Horw, Lucerne University of Applied Sciences and Arts – E&A

7 June 2019

Declaration of authorship

I declare that I have written this thesis without any help from others and without the use of documents and aids other than those indicated. All text excerpts used, quotations and contents of other authors are explicitly denoted as such.

Horw, 7 June 2019

Robert Beaufait

Abstract

The concept of using the ground as an energy storage medium has become of great interest to industry and energy network designers. It has been successfully applied as a source for heat pumps as a sustainable method of heating buildings. The next step is to evaluate a scaled-up designs that can handle the energy storage demand of a community or potentially a region. The performance of such a system requires accurate thermophysical measurements of soils and thermal insulations. This report investigates the various methods and techniques to recommend a measurement device for thermal conductivity and/or thermal diffusivity for porous moist materials, specifically soils and thermal insulation. No two methods or techniques are the same and favor some materials and measurement conditions over others. Determining the best method and technique applicable to porous moist materials is the focus of this paper. Each technique is reviewed based on its suitability to measure porous moist materials over the ranges of temperature, moisture, and thermal conductivity applicable to moist soils and thermal insulations. More specifically two transient techniques are tested and evaluated for the purpose of measuring thermal conductivity of soil samples taken from the Thurgau region of Switzerland and quartz sand from the Perth region of Australia. The results are analyzed for quality and accuracy using reference materials and empirical studies and models for comparison. The findings prove inconclusive results for accurate measurements but are consistent with what other studies have found. A revised look at sample preparation and state-of-the-art sensor technology provides an opportunity to obtain more accurate results with better confidence.

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Nomenclature and Acronyms

Q	heat quantity	J
t	time	s
q_i	specific heat flux	W/m ²
λ	thermal conductivity	W/(m·K)
T	temperature	K
x	dimensional length	m
α	overall heat transfer coefficient	W/(m ² ·K)
ε	radiative emissivity	-
σ	Stephan-Boltzmann constant	W/(m ² ·K ⁴)
a	thermal diffusivity	m ² /s
ρ	density	kg/m ³
c_p	specific heat capacity	J/(kg·K)
A_i	area	m ²
l_i	distance	m
\dot{Q}	heating power	W
d	thickness	m
CCTES	Competency Center Thermal Energy Storage	
GHP	Guarded Hot Plate	
HFM	Heat Flow Meter	
GHB	Guarded Hot Box	
THW	Transient Hot Wire	
THS	Transient Hot Strip	
SPHP	Single Probe Heat Pulse	
DPHP	Dual Probe Heat Pulse	
TPS	Transient Plane Source	

1 Introduction

The forward progress of energy management and utilization has supported development of various techniques for energy optimization and utilization. According to an European Union report, significant amounts of thermal energy are produced alongside the typical energy utilization and often goes unused [1]. Thermal energy storage is a progressive field of study investigating solutions designed for optimizing energy supplies from daily to seasonal time scales. This is of great interest to not only the residential and business sectors, but to the industrial sector in the form of peak shaving. A proper assessment is necessary in all cases to design a system that performs competitively with the price of available energy resources (e.g. grid electricity and fossil fuels). Design requirements rely on accurate measurements of the thermophysical properties of storage media and insulation to determine charging and discharging characteristics in addition to the efficiency of a system. Accurate evaluation of insulation and thermal storage media require measurement methods with precise measurements and known uncertainties. Due to the nature of most material properties being dependent on a fundamental characteristic (e.g. temperature, porosity, etc.), there is not a single measurement technique suitable to measure all materials over all possible states and measurement ranges. Therefore, it is imperative to first properly evaluate and classify materials before selecting the method and equipment to determine thermophysical properties (thermal conductivity, thermal diffusivity, and specific/volumetric heat capacity).

1.1 Background

The Competency Center Thermal Energy Storage (CCTES) is conducting a project with an industry partner, Schenk AG, to develop novel water-proof thermal insulation materials suitable for high temperature applications. These materials are by nature porous and may be exposed or eventually retain moisture especially for in ground applications. In addition, another CCTES project is tasked with conducting a simulation-based techno-economic feasibility study for a novel seasonal energy storage concept using soil as shallow geothermal storage media. Soil exhibits varying thermal properties dependent on a number of physical characteristics which influence how these properties can be measured accurately. Both cases require a thermal property measurement method which is able to handle varying degrees of porosity, homogeneity, and moisture content with a high degree of accuracy and acceptable uncertainty.

1.2 Project Aim and Objectives

Measurement of thermophysical properties for moist soil and thermal insulation presents a challenge to the practical application of the developed methods and measurement devices. Established thermal material property values for industry standards are often determined in a test environment that is not representative of many applied conditions. This project aims to study and identify suitable solutions for thermal property measurement of insulation and soil that are porous, in some cases inhomogeneous, and may contain moisture. The project aim is to be achieved by identifying the measurement device requirements and constraints through consultation with CCTES and an extensive literature review. The literature review includes a survey of the current methods to determine the required thermal material properties and the current state-of-the-art equipment available commercially. Furthermore, a practical review of the in-house CCTES measurement device along with a separate device employing a different measurement method are analyzed for comparison.

1.3 Project Goals

The goal of this project is to present a supported recommendation for either the acquisition of a commercial or in-house developed device suited for measuring at least two of the three required thermal properties. In either case, a detailed cost analysis of the best suited device and supported justification. In addition, a method to handle and properly measure the thermal properties of moist porous materials will be explicitly described.

2 Literature Review

An extensive literature review was conducted to research the types of methods to measure thermophysical properties. A basic review of thermophysical properties was conducted to serve as a base for detailing the various measurement methods. Measurement methods are defined as the principals and theories which are the foundations for the development of measurement techniques. Measurement techniques are detailed in their ability to determine thermophysical properties along with other parameters dictating their capabilities and limitations. A selection of state-of-the-art solutions are listed for each applicable technique along with its feasibility for this project.

2.1 Introduction to Thermophysical Properties

Thermophysical properties of a material characterize the way heat interacts with a given substance. The three main properties of a material are the ability to retain heat before changing temperature (heat capacity), the ability to move heat from hot to cold (thermal conductivity), and the ratio of heat transport to heat retention (thermal diffusivity). These properties are affected by the material structure, composition, density, porosity, moisture content, pressure and temperature. Any material has the ability to transfer heat through three distinct mechanisms; conduction, convection, and radiation. These mechanisms and properties are outlined and expanded below.

2.1.1 Basic Theory of Heat Transfer

Heat transfer is characterized by two categories in the time domain three categories with respect to the physical domain. Heat transfer can exist as a steady-state (stationary) process or a transient (non-stationary) process. The steady-state process is defined as unchanged heat transfer with respect to time. The transient process is defined as a variable or changing heat transfer with respect to

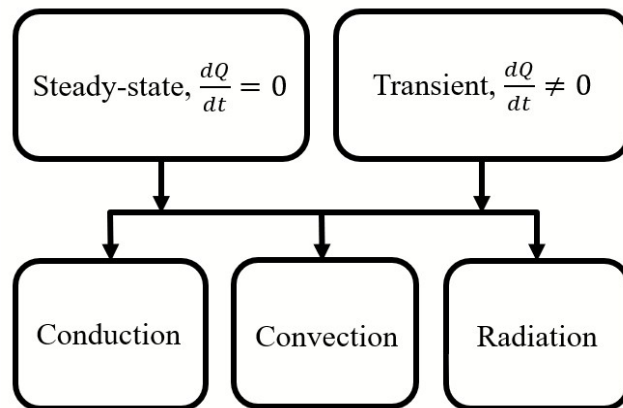


Figure 1: Temporal and physical domains of heat transfer

time. All three physical domain characteristics of heat transfer can exhibit either a steady-state or transient process. The three categories of heat transfer in the physical domain are conduction,

convection, and radiation. Heat conduction is characterized by thermal energy exchange induced by direct contact with adjacent material. Heat convection is characterized by the movement of a fluid increasing the rate of thermal energy transfer between fluid particles. This increases the rate of thermal energy transfer between the fluid and another material when compared with a fluid at rest. Heat radiation is characterized by the energy emitted through electromagnetic waves which are emitted by a material. This category of heat transfer is unlike the previous two as it does not require material contact of any kind to transfer thermal energy [2]. The following sections further detail the three categories within the physical domain.

2.1.2 Conduction

Conduction of heat is the kinetic transfer of thermal energy from a more energetic particle to a less energetic adjacent particle. Heat transfer is the result of a temperature gradient across a given distance and collisions of particles within the material over that distance. Conduction is present in solid, liquid, and gas phase of materials and continues to persist until the temperature gradient no longer exists. The general model of steady-state, one-dimensional heat conduction, is described in the rate equation (1),

$$q_x = -\lambda \cdot \frac{dT}{dx} \left[\frac{W}{m^2} \right] \quad (1)$$

and is known as Fourier's law [3]. The conduction of heat through a material depends on the arrangement of microscopic particles in relation to one another, the amplitude of vibrations of material particles, and how well those vibrations are transferred between neighboring particles. In the case of solids, the types bonds between molecules and availability of free electrons determine the rate of thermal conduction. For example, solids tend to be better conductors of heat as they have constant molecular interaction with a neighboring particle. Moreover, this results in better heat transfer through the material when excited by an energy source. However, not all solid materials are good conductors of heat. The effective conduction of heat is dependent on the path heat flows. This is a function of the material structure and components which define the overall material. There exist materials which do not readily transfer the energy of their neighbors. For example, certain insulations have trapped air bubbles in them that have a much poorer ability to conduct thermal energy thus lowering the overall ability of the material to conduct heat. In general, liquids are generally less conductive than solids and gases less than liquids as the increasing spatial distance between particles reduces the ability to conduct thermal energy efficiently [2].

2.1.3 Convection

Convection heat transfer of energy resulting from the movement of a fluid. There are two categories of convective energy transfer, natural and forced. Natural convection is the movement of a fluid characterized by changes in buoyancy. Buoyancy forces are created when heating of fluid particles in contact with a heat source cause a local decrease in density of the fluid. These particles then move away from the heat source and exchange heat with the surrounding unheated portion of the fluid either by buoyancy or external forcing. It is this second exchange of heat which is considered convective heat transfer [3]. Forced convection is the same concept as natural convection with the difference being that the motion of the fluid is not driven by buoyancy forces, but rather external (e.g. wind, ventilator, pump). Both types of convection are described by equation (2),

$$q = \alpha \cdot (T_{source} - T_{\infty}) \left[\frac{W}{m^2} \right] \quad (2)$$

known as Newton's law of cooling where α is the overall heat transfer coefficient and T_{∞} is the free stream temperature of the fluid outside the direct influence of the heat source [4]. The heat transfer coefficient is dependent on numerous factors with the type of fluid flow being most important (e.g. laminar or turbulent).

2.1.4 Radiation

Thermal radiation is created by the energetic state of a material defined by temperature. It is dependent on the type of material, the type of surface a material has, and by the emissivity or the ability of a material to emit radiation relative to a blackbody (i.e. an ideal emitter). The amount of thermal radiative energy emitted by the surface of a material is offset by the amount being received or absorbed. Thermal radiation is described by equation (3),

$$q_{rad} = \varepsilon \sigma (T_{source}^4 - T_{amb}^4) \left[\frac{W}{m^2} \right] \quad (3)$$

which is an extension of the Stephan-Boltzmann law. Thermal radiation takes the form of electro-magnetic radiation in the infrared range with wave lengths between 700 nm to 1 mm [2].

2.2 Thermophysical Properties

Understanding thermophysical properties is necessary for assessing the suitability of a material as a thermal storage medium. The ability of a material to conduct, store, and diffuse thermal energy fall into two categories, transport and thermodynamic properties. Transport properties describe the movement of thermal energy within a material relative to unit length and temperature (thermal conductivity) and by the ability to diffuse thermal energy relative to time (thermal diffusivity). Thermodynamic properties are a result from the state of the material being considered. Density and specific heat capacity are two thermodynamic properties important for the evaluation of thermophysical properties.

2.2.1 Thermal Conductivity

Previously mentioned in section 3.1.1 Fourier's law of heat conduction is the basis for evaluating how thermal energy flows in a material. Rearranging the Fourier equation for heat conduction, we get equation (4),

$$\lambda = -\frac{q}{(dT/dx)} \left[\frac{\text{W}}{\text{m}\cdot\text{K}} \right] \quad (4)$$

Thermal conductivity is given the units $\text{W}/(\text{m}\cdot\text{K})$ and is a measure of the ratio of specific heat flux in to the temperature gradient applied over a specific distance in a material [3]. A positive change in the ratio of specific heat to the normal temperature gradient between two surfaces results in an increase in thermal conductivity and vice-versa. Figure 2 below is a summary of the preceding statement.

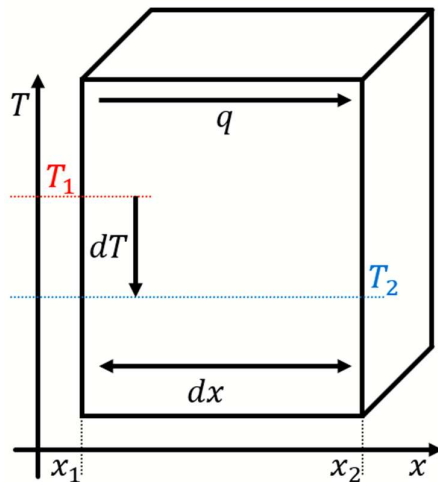


Figure 2: Thermal conductivity

2.2.2 Thermal Diffusivity

Thermal diffusivity is the ratio of the ability to conduct thermal energy to the ability to store thermal energy. It is dependent on both transport and thermodynamic properties and has the units m^2/s . Thermal diffusivity relates to thermal conductivity, density, and specific heat capacity in equation (5).

$$a = \frac{\lambda}{\rho \cdot c_p} \left[\frac{\text{m}^2}{\text{s}} \right] \quad (5)$$

Materials which have a large thermal diffusivity will be more sensitive to environmental changes and are able to quickly reach a new state of equilibrium. Precise and accurate thermophysical properties are necessary to calculate thermal diffusivity especially for low values of thermal conductivity.

2.2.3 Specific Heat Capacity

Specific heat capacity is the measure of thermal energy a material can store per unit mass before experiencing a temperature change of 1 K. It is a measure of the amount of energy a material can accept before incurring an increase in temperature. How a material stores this energy is dependent on the interactions between individual molecules within a material. Specifically, the ability of thermal energy to free electrons for heat transfer in addition to the heat transfer from harmonic oscillations of molecules ultimately defines the specific heat capacity of a material [3].

2.3 Definition of Material Characteristics

An accurate determination of the thermal properties of a material require proper material characterization. The physical characteristics affect the thermal behavior of a sample material. A sample material entirely made up of one or more substances in the same phase uniformly distributed is considered homogenous [5]. Furthermore, a homogenous material that exhibits equal thermophysical properties in all directions relative to an axis is considered isotropic. Often, we encounter materials that vary in some material characteristic over the volume being studied or applied. In nature many materials are considered inhomogeneous and anisotropic. By definition these thermophysical characteristics (i.e. thermal properties) are not uniform over a material volume. It is therefore, critical one clearly define the sample material being analyzed with regards to characteristics affecting thermal properties.

2.3.1 Density

Density is defined as the amount of mass per unit volume and is important in the calculation of heat transfer. It scales the ratio between how well a material conducts thermal energy, thermal conductivity, and how well a material stores thermal energy. Generally, a material of a higher density is indicative of a solid or liquid. Bulk density is the measure of mass per unit volume including any spaces not occupied by matter within the evaluated volume.

2.3.2 Porosity

Porosity is the measure of distributed space that is open inside a solid or loosely packed material. These regions not occupied by the evaluated material can be closed or open to the environment and are occupied by a fluid (e.g. air or water). The physical orientation of the pores and the material which occupies them are crucial to understanding how heat flows through the bulk of a material. Figure 3 below shows two cases of heat flow through a two-component material in which the thermal conductivity of each component is not equal [4].

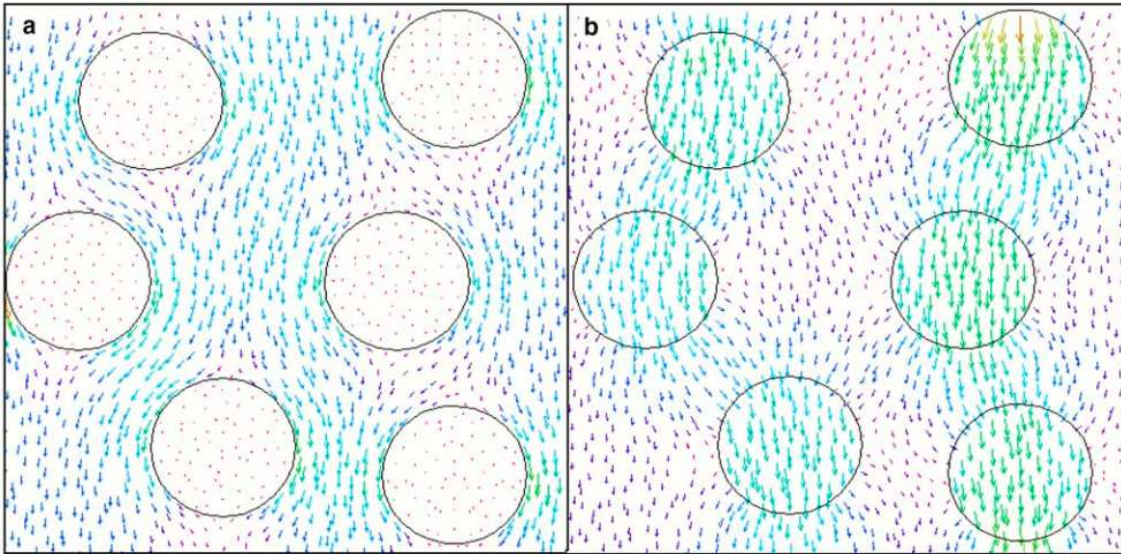


Figure 3: Heat flow through a two-phase material [4]

In case a) the thermal conductivity of the continuous phase is higher than the discontinuous phase and in case b) the opposite is true. Both cases are representative of a matrix of material that maintains the same thermophysical properties in all directions (isotropic). Furthermore, each case is considered to be a disconnected-internal pore structure where there are no connective links between pores and a fluid is unable to flow between pores or to the environment. However, there are many types of pore structures which lead to different thermal transport characteristics.

For the case of connected external pores, a fluid phase can move within a material as well as exchanged with the surrounding environment. Connected internal pores are connected inside a material volume but do not have pathways for exchange with the environment [4]. Pore structures are not limited to the above four cases. Many materials possess a mixture of pore structures in their arrangement.

Porosity has a direct effect on the thermal transport properties such as thermal conductivity and thermal diffusivity. Porosity affects the path thermal energy travels through a material and can vary within the same material at different locations [6]. This is especially true for materials with a large difference in thermal conductivity between phases or materials.

2.3.3 Moisture Content

The moisture content of a porous material influences the thermal transport properties of a material by altering the media heat travels through. Moisture is able to occupy voids in the bulk of the material and may cause the overall specific heat capacity as well as the overall thermal conductivity to change (effective thermal conductivity). The magnitude of changes in thermo-physical properties is dependent on porosity and how much of the porous space is occupied by water. In general, specific heat capacity increases along with thermal conductivity increases as water content increases as it displaces air. Figure 4 below shows the relationship between moisture content and specific heat capacity and Figure 5 thermal conductivity for a clay soil from a state of nearly dry to a moisture content of 0.25.

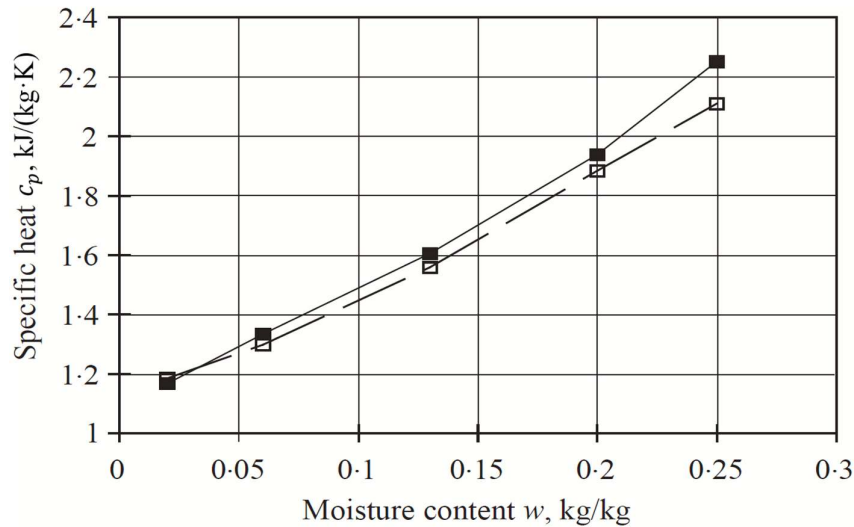


Figure 4: Specific heat capacity versus of moisture content [7]

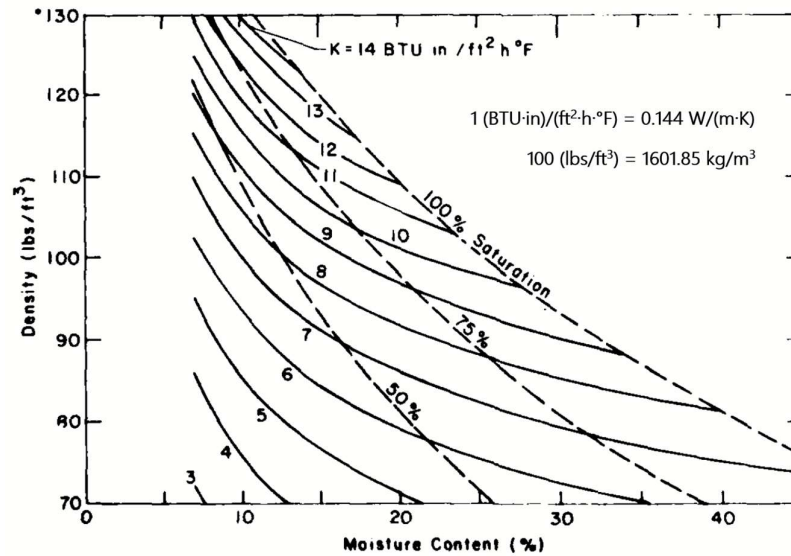


Figure 5: Thermal conductivity versus moisture content and density for clay at 4.44°C [8]

The thermal diffusivity tends to increase the most after gaining moisture from the dry state depending on the material composition, porosity, and size of the pores. A more open pore structure allows moisture to cover the surfaces of material particles faster. This results in a quick rise in thermal diffusivity and thermal conductivity relative to moisture gain than a material more restrictive to water penetrating porous regions [7]. **Error! Reference source not found.** below shows the described behavior of a material open to quick moisture gain along with a material with less ability to accept water.

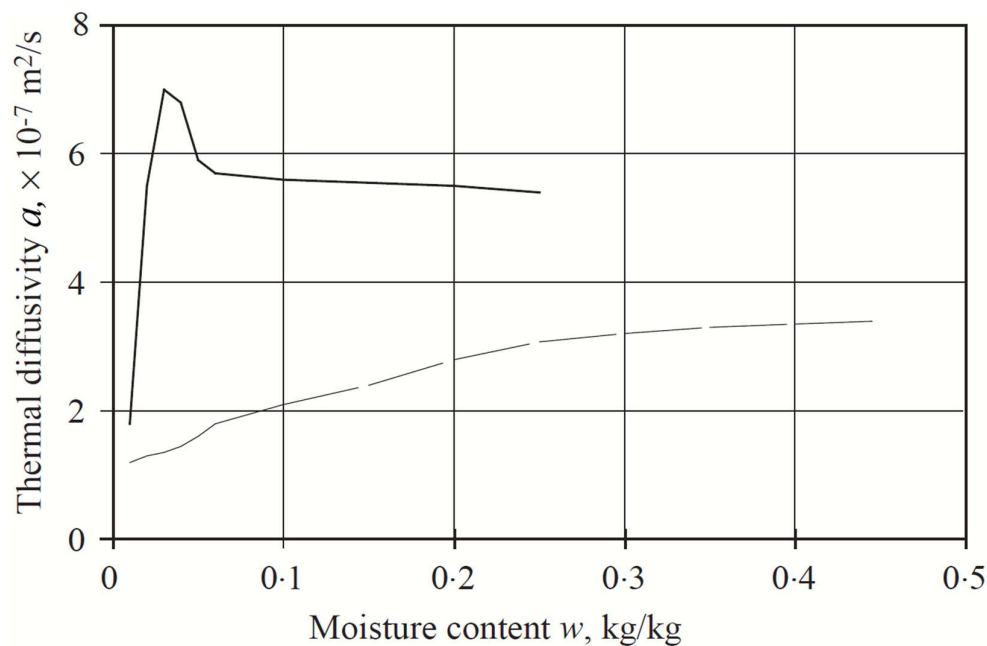


Figure 6: Thermal diffusivity versus moisture content for clay (- -) and sandy (—) soils [9]

2.3.4 Material Composition and Orientation

There have been many studies conducted of the effects of material characteristics and thermal conductivity. These studies consist of empirical measurements paired with an analytical analysis for predicting thermal conductivity for a range of porosities. Assumptions are therefore needed to make an analytical solution possible. It is widely assumed that the thermal conductivity is isotropic in nature and if not, a method of averaging has been applied. In the case of a quartz sand, it is by nature anisotropic regarding thermal conductivity ($11.3 \text{ W}/(\text{m}\cdot\text{K})$ parallel to the optic axis and $6.5 \text{ W}/(\text{m}\cdot\text{K})$ perpendicular) [8]. Another study considers geometric shape as the parameter for predicting thermal conductivity [10]. In addition, material grain size also has the effect of decreasing effective thermal conductivity by reducing grain size. This is the effect of an increased grain surface area which increases the number of thermal barriers heat must cross during its transfer [11].

2.4 Measurement Methods, State-of-the-Art, and Project Suitability

This section describes the various methods and techniques by which thermal conductivity can be measured. The goal of every method is to accurately quantify how heat flows through a material to be measured. Each method has its advantages and disadvantages relative to one another, the material to be measured, and the environment to be tested. Measurement techniques of thermal conductivity fall into one of two types; steady-state and transient. A steady-state method measures the temperature difference across a sample material by a known, constant heat flow. A transient method measures the temperature difference across time at a fixed point in the sample material. Expanded descriptions of the theory, methods, and individual techniques can be found through journal publications and dedicated print materials [12–14]. The following subsections provide introductory and summarized detail of each technique surveyed, availability of commercial equipment, and its suitability for the project aim with an overview table of all methods at the end.

2.4.1 Steady-State Measurement Methods

The steady-state method can be further broken into two measurement types. They are the absolute method and the comparative method. The absolute steady-state measurement method is designed to measure a temperature difference with a constant (or steady) heat flow through a known distance in the material of interest. Once the heat source is turned on all components of the measurement device must come to a state of rest where heat flux across the sample and guard apparatus are constant. This is done by applying a heat source and sink to opposing sides

of a geometrically symmetric sample. This creates the temperature gradient over a specified distance needed to measure the thermal conductivity of the sample. Figure 7 shows a simple example of the absolute steady-state measurement method.

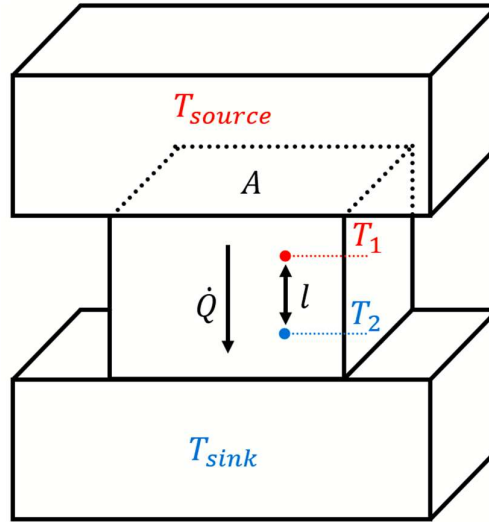


Figure 7: Absolute steady-state measurement method

When applying Fourier's law for one-dimensional heat conduction thermal conductivity is related as follows in equation (6),

$$\lambda = \frac{\dot{Q} \cdot l}{A \cdot \Delta T_{12}} \quad (6)$$

where \dot{Q} is the amount of heat flowing through the sample across the cross-sectional area A over the distance l and temperature differential ΔT_{12} [15].

The comparative steady-state measurement method employs all the concepts above with the addition of a second material of a well-known, similar thermal conductivity to the test sample. The second (reference) material is then placed in series with a test sample ensuring adequate thermal contact between them. A temperature difference is recorded using a minimum of two thermocouples for each material across an equal distance in each material and compared. This type of steady-state method requires a precisely controlled test environment the heat flow across the test sample must nearly match the reference material. Figure 8 below shows the set-up of the comparative steady-state measurement method.

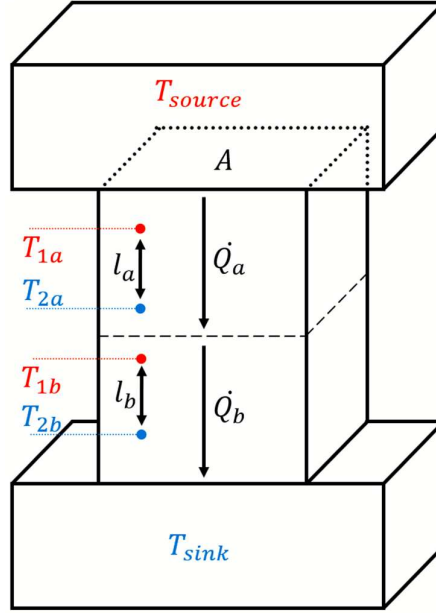


Figure 8: Comparative steady-state measurement method

The comparative steady-state method avoids the requirement to measure the power applied to the sample. The thermal conductivity can be calculated using equation (7),

$$\lambda_a = \lambda_b \cdot \frac{A_b \cdot \Delta T_{12b} \cdot l_a}{A_a \cdot \Delta T_{12a} \cdot l_b} \quad (7)$$

where λ_b is the known thermal conductivity of the reference material, A_i are the cross-sectional areas of the materials, l_i the distances between thermocouples, and ΔT_{12i} the temperature differences between thermocouples. Advantages of both steady-state methods are: (i) the ability to measure low thermal conductivities, (ii) measure samples that are made of more than one material, (iii) heat flow over a larger area allowing for more accurate measurement of anisotropic materials, and (iv) reducing the heat flow to a one-dimensional calculation directly using Fourier's law. Disadvantages include long wait times for the measurement system to reach steady-state, complex sample preparation, and imprecise thermal contact resistance [16]. This can often be several hours up to a day depending on geometry and the conditions being tested. Another major disadvantage is accurately calculating the heat flow through the sample while accounting for parasitic losses. The sample loses heat in the form of radiation and convection to the surroundings and conduction from the couples being used to measure the sample [15]. It is therefore necessary to carefully apply measures to keep heat directed into the sample and insulate the sample from the environment such that heat flow is directed along linear paths from

source to sink [17]. Several techniques are described below applying the steady-state method of measuring thermal conductivity.

2.4.1.1 Guarded Hot Plate (GHP)

The guarded hot plate technique can be used to determine the thermal conductivity of glasses, ceramics, polymers, insulation, liquids, and gases [17]. This absolute technique has a robust measurement temperature range between -190°C and 525°C and relative expanded uncertainties can be reduced approximately to 2 to 3% [18]. However, the guarded hot plate is very sensitive to the homogeneity of a sample. The sample thickness must be a minimum of 10 times greater than the largest material constituent (e.g. pore, crystal, grain, etc.) and must bear a flat plate or disc geometry [17,19]. Additionally, this measurement technique has difficulty measuring moist samples due to convection of the fluid contained in the pores of the material and the long measurement time drying the test material. This measurement method has three major sources of uncertainty, heat flux from losses in the heating plate and sample to the surrounding medium, and contact resistance between the sample material and thermocouples [13,17]. Figure 9 shows the concept of the guarded hot plate measurement technique.

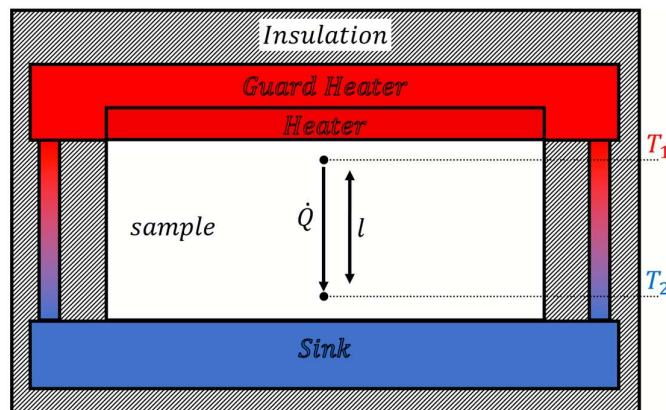


Figure 9: Guarded hot plate measurement technique

The guarded heat plate technique is able to measure a range of thermal conductivity between $0.2 \text{ W}/(\text{m}\cdot\text{K})$ and $200 \text{ W}/(\text{m}\cdot\text{K})$ [20]. Design specifications and build requirements are detailed by ASTM C177, DIN 12667, EN 12939, and ISO 8301/8302 [19]. GHP commercial equipment is available through Netzsch [21] and Taurus [22]. The equipment is large ($\sim 1.5 \text{ m} \cdot 1.5 \text{ m} \cdot 2 \text{ m}$) and heavy ($\sim 270 \text{ kg}$) [23]. The GHP is a very accurate and certain technique to determine thermal conductivity. It is recognized by international standards and can be designed to operate either as an absolute or comparative method. However, this type of technique is very time consuming to build and/or expensive to purchase. The accuracy is strongly dependent on the

ability to guard the heater and the boundary conditions of the sample. Measurement times may take well more than 12 h (up to 36 h) and may require climate control to maintain sample material moisture. This type of measurement technique is not suitable for this project.

2.4.1.2 Heat Flow Meter (HFM)

A heat flow meter (HFM) is able to measure thermal insulation materials, plastics, glasses, ceramics, metals, rocks, and polymers [17,19,24]. The HFM is conceptually the same as the absolute guarded heat plate measurement system except in place of thermocouples, specific heat flux is measured using transducers [17]. An HFM is placed between the heat source and the sample material to measure the amount of power flowing per unit area. Often a second heat flow meter is placed between the sample material and sink. This allows for the radial heat losses along the sample to be calculated [18]. This reduces the amount of time needed for a measurement as the thermal guarding of the sample does not need to be brought to equilibrium as well. Once the target temperatures of the sample are reached, temperatures can be recorded at the heat flux sensors and a determination of thermal conductivity can be made [25]. The HFM technique is considered an absolute steady-state method when measuring a single material and a comparative steady-state method when using a reference material. The advantage of the HFM technique are simple design and operation [17]. The disadvantages are difficulty in reproducibility of results especially at higher temperatures ($> 200^{\circ}\text{C}$) as the HFMs are very sensitive to changes in the surrounding temperature [26]. Design specifications and build requirements are detailed by ASTM C518, ASTM E1530, DIN 12667, and EN 12939 [15,19]. HFMs are available commercially through firms Netzsch [27], and Thermtest [28]. The HFM has shown difficulty in measuring moist samples similar to the GHP technique. A minimum sample size of approximately 200 mm by 200 mm by 51 mm is required for measurements [27]. Sample preparation would not be feasible for with a core samples of soil containing inhomogeneous materials such as rocks. An HFM would not be suitable for this project.

2.4.1.3 Guarded Hot Box (GHB)

The GHB is a measurement device that is typically used to measure thermal resistance of external elements and facades used in buildings. They are capable of measuring the thermal conductivity of insulation materials, plastics, glasses, ceramics, and inhomogeneous structures [19]. As with all steady-state measurement technique, the GHB must be designed to minimize the effects of convection. In addition, the measured sample size must be representative of the sample considered if an inhomogeneous material is to be tested. Figure 10 shows an example of a GHB used to measure a building element.

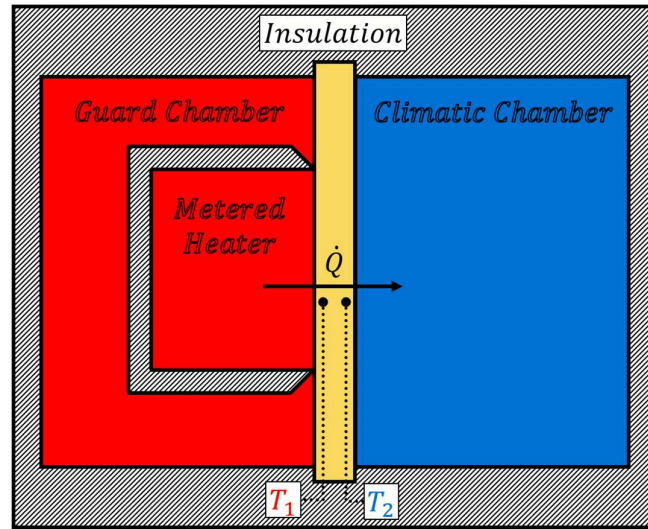


Figure 10: Guarded hot box measurement technique

The measurable thermal conductivity using this technique is approximately from 0.1 to 2.5 W/(m·K) for a 0.5 m thick sample [19,29]. The applied temperature range for this technique is from -48°C to 85°C with the ability to control the climate on both sides of the measured sample. One major disadvantage of this measurement technique is the construction size tends to be on the order of several meters cubed. The device dimensions are required to have enough air to circulate in order to maintain steady-state conditions on both sides of the sample material. The GHB measurement apparatus take a significant time to build based on the size and commercial products are very expensive [30]. The main purpose of this device is to measure building materials that are flat in geometry. Preparation of inhomogeneous soil samples for this type of technique are not feasible therefore, a GHB is not suitable for this project.

2.4.1.4 Other Steady-State Techniques

This section outlines the basic concept of researched techniques that were not feasible to consider for the measurement of porous, moist materials. The cylinder technique requires a long thin sample designed for axial heat flow in materials with high thermal conductivities and metals [17]. The four probe or direct heating technique uses a long thin cylindrical sample that must be homogenous over its entire length and electrically conductive as the material itself is used as the temperature measurement sensor [17,19]. Pipe and hot wire techniques use radial heat flow in a cylindrical sample by embedding a heating element in the sample axis. This steady-state technique induces convective heat transfer of any materials having interconnected pores and drying of the sample [17].

2.4.2 Transient Measurement Methods

The transient measurement methods use an intermittent pulse of energy either in time or amplitude to determine the thermophysical properties of a sample [16]. The development of this method was to avoid the problematic nature of steady-state methods regarding heat losses, contact resistance of temperature sensors, convective effects from prolonged sample heating, and long measurement times. Much less time is needed for transient measurements and the requirement of needing temperature measurements at different positions is replaced with one measurement location [17]. Test times are on the order of a few seconds to a few minutes excluding the relaxation period to allow the sensor and sample material to return to the initial state between measurements. The transient measurement method is suitable for sample materials containing a high moisture content because of the shorter heating times and direct response measurement.

2.4.2.1 Transient Hotwire/Hot Strip (THW/THS)

The THW technique determines the thermal conductivity of a sample material by measuring the temperature rise at a precisely known distance from a linear heat source. This is achieved by embedding a wire in the sample material for heating along with a thermocouple at distance from the wire to measure temperature profile over time. The thermocouple is placed at a radial distance from the center of the heated wire. This allows for the assumption of a one-dimensional radial heat flow for an isotropic, homogeneous sample material treating the heating wire as an infinite line source and an infinitesimally small radius [15]. This assumption leads to equation (8),

$$\lambda = \frac{q}{4\pi(T_2 - T_1)} \ln \left(\frac{t_2}{t_1} \right) \quad (8)$$

where λ is the thermal conductivity, q is the heating power per length of heating wire (W/m), and T_i the temperature of the wire at different points in time. The ability of modern electronics allows for 100 measurement data points in 1 ms to 1 s in liquids and gases, and 10 s for solids. Shorter measurement times in fluids is necessary to avoid convective heat transfer of the sample material. Uncertainties of $\pm 1\%$ for gases, liquids, and solids are achievable [31]. Figure 11 below shows the concept of measuring thermal conductivity using the THW technique.

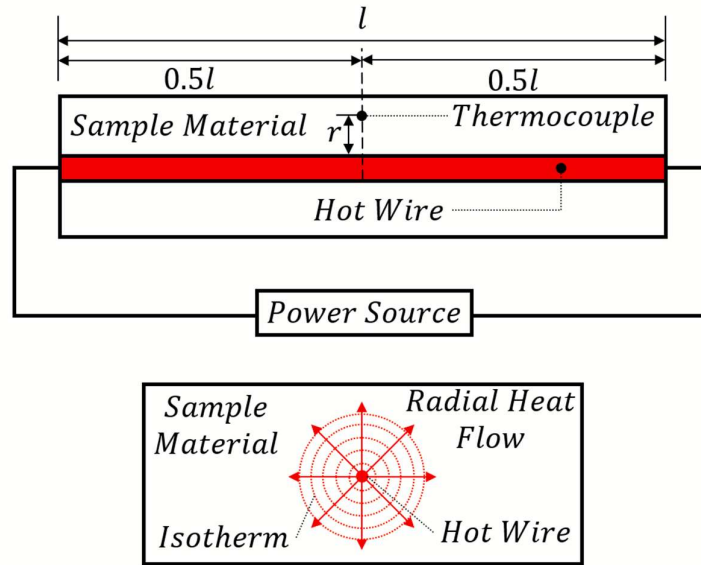


Figure 11: Transient hot wire measurement technique

The thermal hot strip (THS) technique is a modification that works in principle the same as the THW with the exception the use of a strip of thin metal and a mathematical evaluation procedure to account for the radially asymmetric temperature profile [17]. The THW and THS techniques are suited best for measuring solids including earth minerals, building materials, plastics, powders, fluids and gases as long as sufficient contact between sample material and sensor is maintained [19]. The THW and THS techniques require a low thermal contact resistance between the heating element and measured sample [17]. The approximations of THS and THW techniques limit them to the measurement of thermal conductivity only and thermal diffusivity must be calculated using equation (5) previously mentioned in section 2.2.2. The THW was further developed to eliminate the use of a thermocouple by using the heating wire instead of a second wire to measure temperature response via electrical resistance. The advantage of this modification called the parallel wire technique is the ability to measure anisotropic sample materials and an expanded measurement range of thermal conductivity [17]. Overall, the thermal conductivity values which can be measured by this technique range from $0.001 \text{ W}/(\text{m}\cdot\text{K})$ to $20 \text{ W}/(\text{m}\cdot\text{K})$ over a temperature range of -150°C to 200°C . The latest iteration in development of the THW technique is the transient hot bridge (THB). The THB uses a balanced Wheatstone bridge to produce measured variances in voltages across each circuit. This allows for the measurement of thermal conductivity as well as the thermal diffusivity and the ability to derive the volumetric heat capacity as the heating wires are also used as temperature sensors [32,33]. The measurement range of this type of technique is $0.02 \text{ W}/(\text{m}\cdot\text{K})$ to $30 \text{ W}/(\text{m}\cdot\text{K})$ over a temperature range of -100°C to 200°C with an accuracy of less than 3% and uncertainty of less than $\pm 3\%$.

[32]. All of the THW and THS measurement techniques require a much smaller sample size (> 1 mm thick by 30 mm wide by 80 mm long) relative to steady-state methods (> 0.5 mm thick by 300 mm wide by 300 mm long) [16]. The THW design specifications are outlined in ASTM D7984. Commercially available measurement devices exist through Linseis [34], TeKa [35], and Thermtest [36]. There exists the potential to use the thermal THB measurement technique with correctly prepared samples. If planar surfaces could be realized, a THB sensor may be a feasible method of measuring thermal conductivity and thermal diffusivity.

2.4.2.2 Single and Dual Probe Heat Pulse (SPHP & DPHP)

The SPHP and DPHP are fundamentally extensions of the THW techniques. Both SPHP and DPHP can be used on insulation, plastics, soils, ice cores, refractories, fibers, liquids, and powdered sample materials [37]. The SPHP is constructed in the shape of a needle which has a heating wire and a thermocouple in the center of the heated strip. The advantage of the SPHP design allows it to be inserted into loose sample material or into a drilled hole. As long as the minimum dimensions of the sample are present and contact resistance with the probe not too large, the SPHP is able to produce measurements of thermal conductivity with an accuracy of $\pm 2\%$. The SPHP technique is able to measure thermal conductivity over a range of $0.1 \text{ W}/(\text{m}\cdot\text{K})$ to $10 \text{ W}/(\text{m}\cdot\text{K})$ for temperatures of -25°C to 125°C [38]. Furthermore, it is the only transient method which has obtained an ASTM standard for thermal measurements of soil (ASTM D5334) [39]. Other research groups have used this method to study sediments on the ocean floor [40], soils and soft rocks in geothermal applications [41], and thermophysical characteristics of porous materials [10]. The SPHP does have difficulties regarding accuracy in measuring moist samples containing an uneven distribution of water within the sample material and if the contact resistance between probe and sample material is too high [37]. The DPHP is related to the SPHP but with the addition of a second probe. The second probe acts as a secondary sensing element thus the measurement of thermal diffusivity in addition to thermal conductivity is also possible along with the calculation of volumetric heat capacity from a single heat-pulse measurement [42]. An added advantage of the DPHP is the ability to measure the water content of a soil sample. The DPHP sends a heat pulse which measured by the second probe. It has been discovered that volumetric water content of a soil can be accurately estimated from the volumetric heat capacity [43]. It was found that for two different soil samples that the gravimetric method of determining water content agreed within 0.03 to $0.04 \text{ m}^3/\text{m}^3$ over a water content range of 0.10 to $0.45 \text{ m}^3/\text{m}^3$ [44]. The DPHP technique is able to measure thermal conductivities from $0.02 \text{ W}/(\text{m}\cdot\text{K})$ to $2 \text{ W}/(\text{m}\cdot\text{K})$ and thermal diffusivity from $0.1 \text{ mm}^2/\text{s}$ to $1.0 \text{ mm}^2/\text{s}$ in a

temperature range from -50°C to 150°C . Accuracy for both measurements is approximately 10%. Measurement times for the DPHP are on the order of 1 minute and conform to ASTM 5334 and IEEE 442 standards [45]. One major disadvantage of the DPHP is the distance between probes must remain undisturbed. It is imperative that the two probe remain parallel for accurate measurements using this technique [46]. Commercial measurement equipment is available through Thermtest [47], Huskeflux [48], TeKa [35], Metergroup [45], and C-Therm [49]. SPHP and DPHP are the most widely used techniques for measuring thermophysical properties of soil and rock formations. These techniques are well suited for the measurement constraints of this project and the SPHP is further investigated.

2.4.2.3 Hot Disk Transient Plane Source (TPS)

The TPS or hot disk technique is based on the hot strip technique to measure both thermal conductivity and thermal diffusivity from a single measurement. The sensor is encapsulated in a protective coating and placed between two planar surfaces of the sample material. This technique is able to measure thermal properties of liquids, solids, and powders [50]. Measurements are made by heating the sensor with an electrical current while monitoring the resistance for the behavior of temperature over time [51]. Figure 12 shows examples of TPS sensors in three sizes and insulation types.

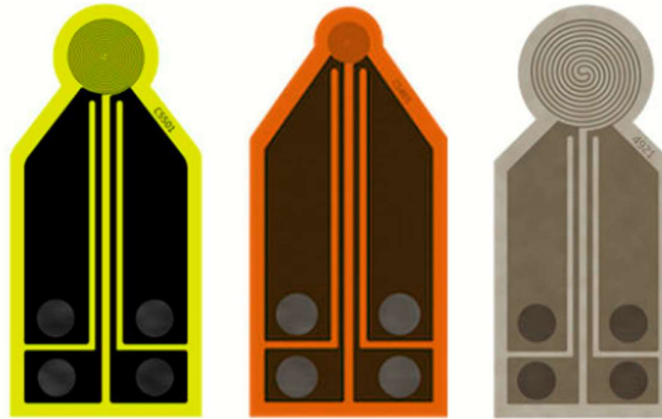


Figure 12: Transient plane technique measurement sensors [52]

The TPS is able to measure a range of materials that are electrically and thermally conductive to insulating materials. The thermal conductivity can be measured over a range from $0.005 \text{ W}/(\text{m}\cdot\text{K})$ to $500 \text{ W}/(\text{m}\cdot\text{K})$ in a temperature range from -243°C to 930°C . ASTM D7984 and ISO 22007-2 give greater detail to the test equipment and TPS measurement technique [53]. Repeated TPS analyses have produced approximate accuracies of 2% for thermal conductivity, 4% for thermal diffusivity, and 6% for volumetric heat capacity [51]. One major advantage of

this system is the elimination of contact resistance. This is advantage is inherently present from the required sample material preparation of having smooth surfaces in contact with the sensor under pressure. Both faces of the sample material must be planar making it quite difficult for loosely packed, unconsolidated or granulated materials to be measured [15]. Commercially available equipment is available through Hot Disk [54], C-Therm [49], and Thermtest [55]. The TPS technique would have difficulty in measuring most soil samples as they exist in nature. A well-planned sample preparation method would be needed to refine samples to an acceptable geometry and surface quality while maintaining a representative sample for measurement using this technique.

2.4.2.4 Laser Flash

The laser flash measurement method works differently from all previous techniques to determine thermophysical properties. It does not need physical contact with the sample material to take measurements of thermal diffusivity with high accuracy. Furthermore, it is non-destructive to the sample material as well [15]. This technique has the ability to measure polymers, glasses, ceramics, metals, liquids and powders without major restrictions on the achievable measurement uncertainty. It is the most often used technique to determine thermophysical properties of solids [17]. The laser flash technique is based on the concept of heating one side of a sample material with a pulse of light in the form of a laser and measuring the temperature signature on the opposite side with an infrared sensor [16]. Figure 13 shows the lay-out of a laser flash measurement device.

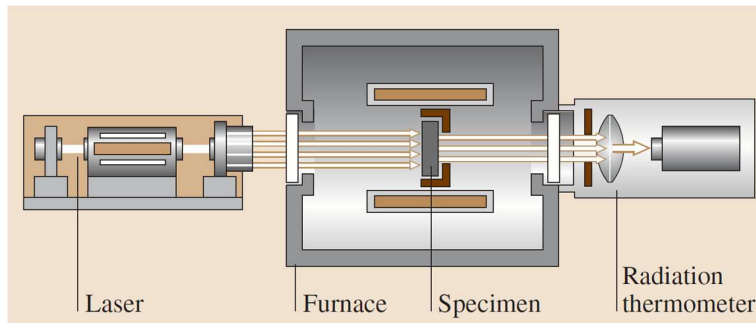


Figure 13: Laser flash measurement device [17]

It is assumed the laser pulse to be instantaneous and the sample held under adiabatic conditions. Using these assumptions allows for equation (9),

$$a = 0.1388 \frac{d^2}{t_{1/2}} \quad (9)$$

where a is the thermal diffusivity, d is the thickness of the specimen, and $t_{1/2}$ is half the time it takes for the full temperature rise of the sample material after being heated by the laser. Using the thermal diffusivity from this relationship along with the specific heat capacity and density, it is possible to calculate the thermal conductivity with equation (10) [16,17].

$$\lambda = a \cdot \rho \cdot c_p \quad (10)$$

One major disadvantage of the laser flash technique is sample preparation. Both sides of the sample material must be flat and parallel and approximately 1-4 mm thick. This rules out discontinuous materials with pores or voids such as thermal insulation or soils. Another disadvantage is difficulty measuring solid samples which are inhomogeneous. Grains that exhibit different thermophysical properties than the surrounding bulk sample material must be less than 5% of the thickness of the overall sample. For feasible sample materials the laser flash has distinct advantages. It is the only technique that does not require a direct temperature measurement. In addition, it is fast, and accurate for a temperature range of -100°C to 3000°C with an uncertainty of 3-5% [16,26]. Laser flash equipment is commercially available through Netzsch [56], TA Instruments [57], and Linseis [58]. The laser flash technique is not suitable for porous, moist materials. The required sample sizes are not feasible with soils containing inhomogeneities that can exceed the entire sample size itself.

2.4.2.5 Photothermal/Photoacoustic

Photothermal and photoacoustic are designed to measure thin films, liquids, and gases. These techniques are still in the development phase and use non-standardized methods of measurement. They require expert knowledge to operate and must have a laser rated building with a precisely controlled environment [26]. Commercial equipment is not available for these techniques as they are still being developed. Not enough information is available to determine whether this method is feasible to measure porous and moist materials.

2.4.3 Measurement Methods and Techniques Summary

Method/ Technique	Temperature range	Uncertainty/ λ , W/(m·K)	Materials	Pros	Cons
Steady-state Methods					
Guarded hot plate [26]	-190 – 525°C	2% $\lambda < 2$	Insulation, plastics, glasses	High accuracy	Long measurement times, large specimen, low conductivity materials
Heat flow meter [26]	-100 – 200°C	3-10% $0.007 < \lambda < 500$ [25]	Insulation materials, plastics, glasses, ceramics	Simple construction & operation	Measurement uncertainty, relative measurement
Guarded hot box [29]	-48 – 85°C	~6% $0.1 < \lambda < 2.5$	Building materials, inhomogeneous materials, composites	climate control	Small temperature range, long measurement times
Comparative [17,25]	20 – 1300°C	10-20% $0.2 < \lambda < 200$	Metals, ceramics, plastics	Simple construction & operation	Measurement uncertainty, relative measurement
Cylinder [26]	-270 – 620°C	2% n/a	Metals	Temperature range	Long measurement time
Four probe [19]	20 – 1600°C	n/a	Thermal conductors	Very high temperatures above most melting points	Homogenous materials, very thin samples
Pipe [17,25]	20 – 2500°C	3-20% $0.02 < \lambda < 2$	Solids, calcium silicates, mineral wool, ceramics	Temperature range	Sample prep, long measurement times
Transient Methods					
Hot wire, hot strip [25,26]	-50 – 500°C	1-10% $0.001 < \lambda < 20$	Solids, liquids, glasses, ceramics, powders, granules	Temperature range, measurement time, accuracy	Limited to low conductivity materials
Line Source [19,38]	-25 – 125°C	3% [37] $0.05 < \lambda < 20$	Soils, polymers, moist materials, powders	Portable or laboratory set up,	Unknown uncertainty
Plane Source [51]	-243 – 930°C	n/a $0.005 < \lambda < 500$	Solids, liquids, pastes, powders	Short measurement times, accuracy, measures thermal diffusivity	Unknown uncertainty
Laser flash [17,25]	-100 – 3000°C	3-5% $0.1 < \lambda < 1500$	Solids, liquids, powders, polymers, ceramics	Temperature range, small sample size, fast	expensive, cannot be used with insulation materials
Photothermal photoacoustic [17,25]	-50 – 500°C	Unknown	Solids, liquids, gases, thin films	thin films, liquids, and gases	Non-standard knowledge regarding accuracy

Table 1: A General overview and comparison of all methods researched for this project.

3 Testing and Validation Procedures

Testing and evaluation were necessary to assess measurement equipment on-loan and in-house. The tests serve as a starting point for the evaluation of specific methods and commercial devices suitable for measuring porous and moist materials. The literature review aided in finding a starting point along with the help of the University of Neuchâtel in providing CCTES an introduction to a device based on the TLS measurement method. The devices considered represent the state-of-the-art solutions for measuring thermal conductivity in their respective techniques with the TLS measurement method focusing on earth minerals and soils. A specific description of each device is presented in the following sections along with testing procedures and reference tests made during this project.

3.1 Instruments Tested

The instruments evaluated for the measurement of porous and moist materials were both transient methods. One of the devices tested was the Linseis THB-100 currently owned by CCTES which uses the transient hot-bridge (THB) technique. The Linseis THB is a modification of the hot-wire hot-strip techniques using 2 pairs of balanced circuits of different lengths at different distances to measure the effects of heat directed to the material over distance and time (see section 4.2.1). It has the capability to simultaneously measure the thermal conductivity and thermal diffusivity of a sample material in a short measurement period (< 2 s for very low conductive materials). This type of sensor uses a variety of sensor geometries made from a 5-10 μm thick foil strip sandwiched between two layers of Kapton for durability. Figure 14 shows the array of sensors offered by Linseis for use with the device.

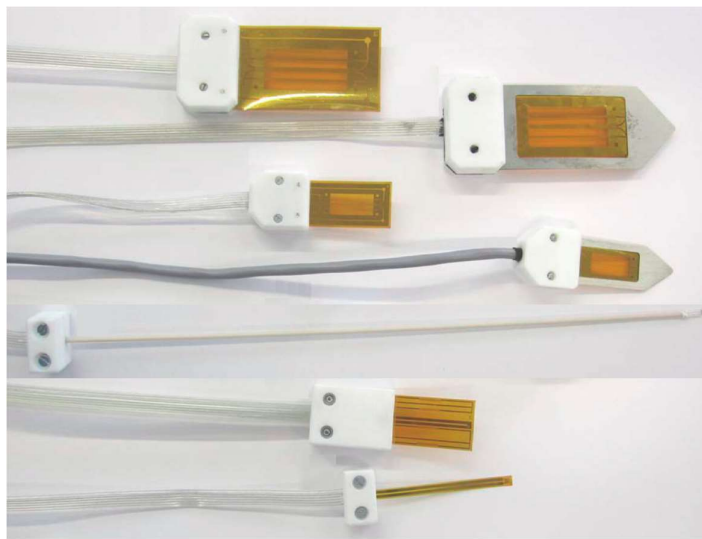


Figure 14: Transient hot bridge sensor types [32]

This type of device was evaluated for its measurable thermal conductivity range as well as the capability to measure thermal diffusivity as well. It is configured with an active heating and cooling system to test materials at temperatures from -100°C to 200°C for the sensors applicable to this project. The thermal conductivity measurement range is between $0.02 \text{ W}/(\text{m}\cdot\text{K})$ and $30 \text{ W}/(\text{m}\cdot\text{K})$ for the selected sensor [32]. This Linseis THB-100 is suitable for measurements of solids, liquids, pastes, powders, and unconsolidated sample materials. Single measurement times for this device can range from several milliseconds to a couple of minutes. After a measurement is taken, it is necessary to allow enough time for the sample material to relax. This allows for the effects of measurement heating to dissipate and for the sample material to return to pre-measurement conditions. Linseis recommends at least 10 fold the amount of time allotted for measurement thus the round-trip measurement time ranges from $\sim 20 \text{ ms}$ to 30 min [32]. The user is able to select the heating current (mA), measurement time (s), number of measurements, and number of temperature drift measurements as a means to obtain a more accurate measurement.

A collaboration with the University of Neuchâtel Laboratory of Geothermics made it possible to evaluate a second measurement device, the TeKa, model TK04. The VLQ measurement sensor is able to measure thermal conductivity from $0.1 \text{ W}/(\text{m}\cdot\text{K})$ to $10 \text{ W}/(\text{m}\cdot\text{K})$ in a temperature range of -25°C to 125°C . The HLQ measurement sensor is able to measure thermal conductivity from $0.3 \text{ W}/(\text{m}\cdot\text{K})$ to $10 \text{ W}/(\text{m}\cdot\text{K})$ in a temperature range of -25°C to 70°C . It is currently on-loan and is available for testing in the HSLU campus lab. This device is another derivation of the hot-wire hot-strip technique called the transient line source. This technique employs the theory of an infinite line source of heat directed into a material using a thin metallic rod or strip. The heating strip and temperature sensor are virtually in the same position which only allows for the measurement of heat over time and not over distance. Therefore, the main drawback of this measurement device is the inability to directly measure a second thermophysical quantity and thus a determination of thermal diffusivity. Extra steps to determine density and specific heat capacity are necessary for complete thermophysical analysis (see section 4.2.2). German manufacturer, TeKa, offers two different sensors for measuring thermal conductivity with their TK04 measurement system. One sensor is a needle probe (full volume sensor, VLQ) that is inserted into the sample material. The second sensor option is a small cylinder with a heating strip (half volume sensor, HLQ) that is placed on the appropriately prepared surface of a sample material. Figure 15 are the two types of measurement sensors of the TeKa measurement system.

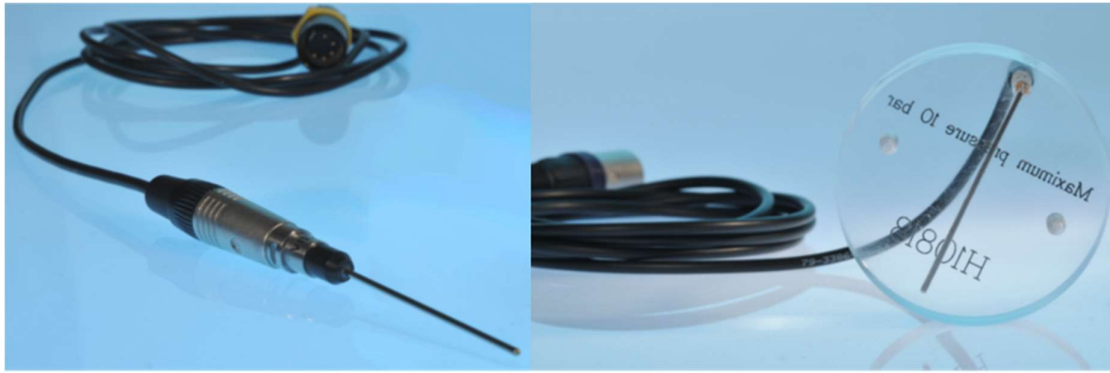


Figure 15: TeKa VLQ sensor left; TeKa HLQ sensor right [38]

This type of device was evaluated for its measurable thermal conductivity range and sample preparation flexibility with the availability of two different sensors. Single measurement times for this device are recommended to last 80 s with a minimum of 30 minutes for the sample to relax between measurements [38]. The user is able to select the heating power (W/m), measurement time (s), number of measurements, and number of temperature drift measurements as a means to obtain a more accurate measurement.

3.2 Testing Procedure

A testing procedure was developed for the TK04 and Linseis THB-100 to adhere to manufacturer specified measurement conditions. The first step is to select a measurement area which is out of direct sunlight, free of drafts especially from the out-of-doors, and other heat sources (e.g. radiators). The TK04 was positioned in a part of the thermodynamics lab that was near a frequently used door that connected to an area that often has a different temperature and created a strong draft when opened. Therefore, the first step was to assemble an insulated chamber for the TeKa probe and sample to be placed for measurement. 30 mm thick sheets made of swissporXPS 300 GE [59] were used to construct a guard box (0.565 m tall · 0.51 m long · 0.31 m wide) for the purposes of keeping temperature and humidity as stable as possible during measurement runs. Expanding insulation foam sealant was used to bind the sheets together creating a box for moderating the temperature changes of the thermodynamics laboratory. Figure 16 shows the final construction of the guard box.



Figure 16: Guard box with temperature and humidity monitors

The guard box was fitted with an internal temperature/humidity sensor along with an external temperature sensor for comparison using the Elpro ECOLOG TH2. Double lined insulation tape was used to seal the gap between the top and the main housing. The other device tested stationed in a different location.

The Linseis THB-100 is placed in the chemistry laboratory and is the site of testing. A simple polystyrene foam box with lid was used as a buffer against fluctuations in ambient conditions. This environment is much better controlled than the test site of the TK04 and does not need as much protection from changes in ambient conditions. A key to using this test environment is having the windows and door closed. An important step is to bring the sample to be tested to the lab for at least 24 hours prior to the measurements. The best practice for measurement preparation is to have the sensor in the material at least 1 hour before the start of the first run. This ensures that any small temperature drift of the sample material is in-step with the sensor.

Sample material preparation is one of the most important steps to obtaining an accurate measurement. Contact resistance between the probe and the sample material can be a major source of error in making thermal conductivity measurements. The first criterion to consider is the physical state of the sample material. Sample material preparation is dependent on whether a sample material is bound to itself (consolidated) or if the material is loose (unconsolidated) and takes the geometry of the vessel by which it is contained. Equally, different sample material preparations are required for TeKa VLQ and HLQ probes than the THB probe used in this project. Sample material preparation for the VLQ and HLQ probes from TeKa for consolidated material requires preparing a geometrically adaptable surface for thermal contact. VLQ probe requires a cylindrical hole drilled into the sample of a geometry that is large enough to insert

the probe firmly without using more than a thumb and two fingers to grip the handle. Generally, a hole slightly larger than the VLQ dimensions is necessary resulting in a small air gap. The presence of an air gap acts as a thermal resistor and is a major source of measurement error. This requires the use of a thermal interface material (TIM) such as a silicon-based paste. A TIM serves to create better thermal contact between the heat source and sample material by filling the air gap with a thermally conductive layer. The minimum sample material dimensions for the VLQ probe are 40 mm in diameter by 85 mm in height [38]. The other TK04 probe, the HLQ, needs a planar surface for contact with the sample material. The consolidated sample needs a smooth surface to contact the heated portion of the probe. Analogous to the VLQ, a non-planar surface will result in a larger air gap needing to be filled with a TIM. This situation results introduces an additional error by having an uneven thickness of TIM between the probe heat source and sample material. The TK04 measurement system comes with a clamp for applying pressure to the HLQ probe and sample material to reduce thermal resistance. It is recommended that 5 to 10 bars pressure be applied before measurement. The minimum sample material dimensions for the HLQ probe are 88 mm in diameter by 20 mm in height [38]. It is to be noted that there is an advantage to using the HLQ over the VLQ. The advantage of the HLQ probe allows for a visual if not measured inspection of the prepared surface and improvement of the surface if it is not sufficient for measuring. Using the VLQ probe does not allow for this type of inspection. It is possible for materials which are porous in nature to have an unknown sample material cavity that goes undetected and is in line with the inserted VLQ position. Figure 17 shows an example of silicone paste applied to both the VLQ and HLQ probes.



Figure 17: TeKa VLQ sensor with thermal paste (left) and HLQ sensor (right) on a planar surface.

Unconsolidated sample materials must be carefully prepared to control for physical characteristics affecting measurements. Very careful attention must be used in how sample material is

loaded into the container before measurement to ensure uniform distribution of density, porosity, moisture, and material types. TeKa recommends this type of sample material to be measured with the VLQ probe. This type of sample material must be contained in a container which allows for the minimum sample material size. The other device tested needed a different method to prepare consolidated samples while unconsolidated remained the same. The Linseis THB-100 comes with an array of hot bridge sensors able to measure consolidated solids, pastes, and powders. For solids the appropriate probe requires placement between two solid planar measurement surfaces of the same sample material clamped with 0.2 to 0.5 bar of pressure [32]. If a smooth surface cannot be produced, the use of a thin layer of copper paste as a TIM is recommended by the manufacturer to be applied. Pastes and powders require the use of a reinforced probe to prevent damaging the mechanically sensitive sensor allowing for direct placement into the unconsolidated material. Careful attention must be taken in order to not damage the sensor if the sample material contains abrasive particles or elements.

The physical characteristics of a sample material needs to be documented before testing begins. Both Linseis and TeKa systems record a temperature profile during measurement to reference to the results and no further verification is needed. All samples need to be to have moisture content and porosity determined before measurement. Moisture content and porosity affect how the effective thermal conductivity may be determined. Prolonged measurement times may initiate convective heat transfer effects having a significant effect on results (see section 2.3.3). Use of the TeKa measurement system the specific heat capacity and density must also be determined in order to obtain thermal diffusivity.

3.2.1 References Tests

Tests were conducted using the TeKa TK04 measurement system on two types of sample materials with known thermal conductivity. A glass ceramic block with known thermal conductivity provided by TeKa [60] and a well analyzed quartz sand provided by Dr. Arnold were tested [61]. The ceramic block has a perfectly pre-drilled hole and planar side with sufficient dimensions for VLQ and HLQ measurement verification. Measurement verification began with the verification of the VLQ probe and reference block. The reference block was verified first without the silicon thermal paste and second using the thermal paste. It was important to see how large an effect an air gap has with the VLQ results on an idealized sample material. Additional verification using the HLQ probe was made using thermal paste. Figure 18 shows the values and relative dimensions of the ceramic sample material during the HLQ verification.



Figure 18: TeKa ceramic sample material with HLQ probe in pressure clamp

A second material was tested for verification of a material with known physical characteristic and thermophysical properties. The Geotechnical Department at HSLU and Dr. André Arnold provided a nearly pure quartz sand from Western Australia near Perth. The sand has had an extensive material and characteristic analysis presenting a unique opportunity to verify the Linseis THB-100 and TK04 VLQ with an unconsolidated material. The Perth Quartz Sand (PQS) was analyzed during a Ph.D. study which provided initial information on the ranges of porosity, density, grain size distribution, and relative shape of grains [61]. Additional suggestions by Dr. Arnold were given to create samples of sand that had varied with porosity and density. A cylinder with a capped end drilled out with 2 mm holes spaced 2 mm square was used to fill a vessel to a known volume. Figure 19 shows the process of filling a vessel with PQS.

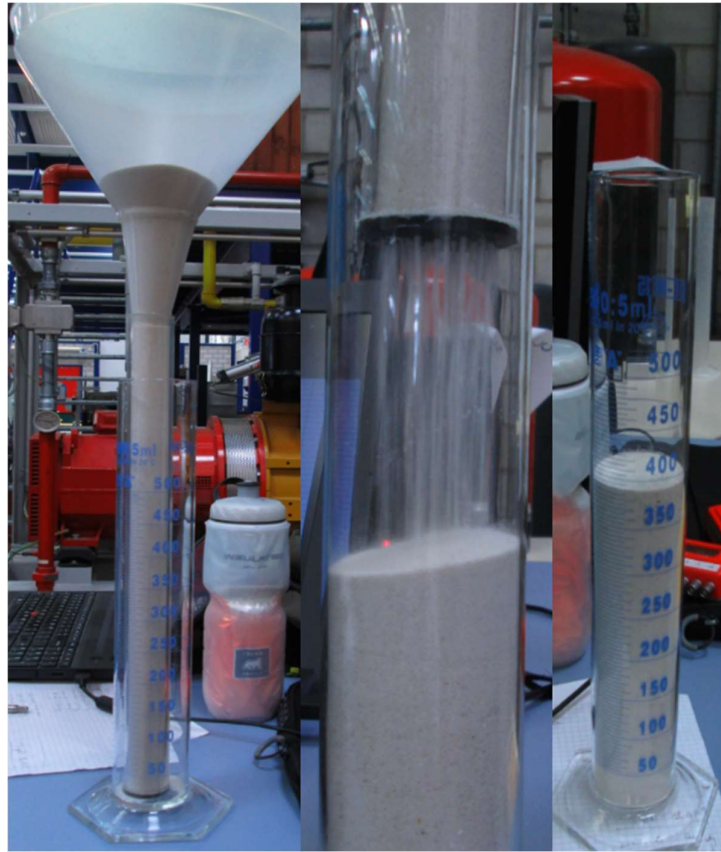


Figure 19: Pre-fill (left), filling (center), filled (right) graduated cylinder with PQS

This method of filling a known volume allowed for the control for the density and porosity of the PQS sample. It also allowed for a level pouring of material creating a uniform density profile of sand. A similar pouring apparatus was also used to fill 5 PQS samples of different porosities for testing with the Linseis THB-100. Finally, the comparison of a PQS sample was made to control for any effects of humidity. A PQS sample was placed in an oven at 100°C overnight and allowed to cool wrapped in cellophane for 24 hours to ambient conditions.

3.2.2 Soil Sample Tests (TeKa)

Two soil samples available for measurement were provided by Schenk AG from the region of Turgau. The two soil samples, B4.3b and B3.1a, were extracted from two separate bore holes representing layers of earth between 9.2 – 9.4 m and 6.7 – 7.0 m respectively [62]. Each bore hole soil sample had a diameter of 150 mm, length of ~110 mm for sample B4.3b, and length of ~160 mm. Both soil samples are geologically described as compacted sub-glacial moraine, very dense ($\sim 2000 \text{ kg/m}^3$), and mechanically unstable when agitated. Furthermore, randomly spaced rocks in various sizes were dispersed throughout the sample material volume. The samples were tested for thermal conductivity using the TeKa TK04 measurement device. Time was

insufficient for a measurement using the Linseis THB-100. The first attempt was made by drilling a hole into sample B4.3b for measurement with the VLQ probe. The necessity to drill a precise hole while keeping the sample intact prompted a design to secure the sample while dampening vibration. Figure 20 shows an in-house made clamp using steel pipe, a wood block, c-clamps, and thermal insulation.



Figure 20: Soil sample clamp and base

The machine shop CNC drilling machine was used to drill a bore hole for insertion of the VLQ probe. The second attempt on soil sample B3.1a was prepared for measurement using the HLQ probe. The mechanical instability of the soil samples required careful planning of the preparation process. Careful steps were taken to reduce one surface of the soil sample sufficient using a variety of methods including wire mesh, a piece of scrap metal, a hand saw, and Dremel tool.

4 Results and Discussion

This section covers the results for the testing and validation procedures outlined in the last section. The following information will give a detailed description of the major challenges of sample preparation and measurement results for the reference materials and soil samples. Charts and tables supporting data results will be located in the appendix and the full files submitted electronically. The following section gives the details of results of the tests performed and a short discussion on test location, sample preparation, and results for each measurement device.

4.1 Guard Box

A location free from drafts, sharp temperature changes, sunlight and other disturbances affecting measurement results is required for use with the TeKa TK04 measurement device. The TeKa TK04 was placed near a doorway and a guard box was used to moderate changes to ambient conditions. The guard box was verified over a period of 12 hours to show dampened internal temperature fluctuations relative to external temperature. The VLQ probe was placed in the guard box along with the ceramic reference block and a 1.5-liter bottle of water as a thermal mass resembling a sample material. Figure 28 in the appendix shows the comparison of internal and external temperature along with the internal relative humidity recorded every 2 seconds over the evening of April 9th and the morning of April 10th, 2019. The results were as expected with internal temperature lagging in time behind external temperature. At approximately 07:00 on April 10th, a sharp external temperature decrease of $\sim 0.8^{\circ}\text{C}$ in 90 s was measured. The internal temperature performed well by responding with a small drop of 0.06°C as shown in Figure 29 in the appendix. Furthermore, the rate of temperature change was charted using a 2-second and 2-minute time step for evaluation. Figure 29 in the appendix shows both 2-second and 2-minute time step plots for same data set. In all cases the 2-minute time change did not exceed the 2-second time change with a max temperature change of $\pm 0.07^{\circ}\text{C}$.

4.2 TeKa TK04

The consolidated reference material (TeKa ceramic reference block) was tested using both the HLQ and VLQ measurement probes. No special preparation was needed before measurement with the exception of placing the reference block inside the guard box for at least 1 hour prior to starting measurements for temperature monitoring. This was to ensure that temperature fluctuations within the sample material and any components in thermal contact associated with measurements were in thermal equilibrium. Figure 30 the appendix shows the reference block temperature monitoring for the period of approximately 1 hour before measurement with the

VLQ probe. The VLQ was further tested with and without thermal paste to determine what type of effect it has on measurement results. The first test used the VLQ measurement probe inserted into the TeKa ceramic block without thermal paste and outside of the guard box for measurement. The default settings of 3 W/m heating power, 80 s measurement time, 30 min relaxation time, and a minimum LET of 4 were used for this measurement. Additionally, a second test was done with thermal paste and inside the guard box using the same default parameters. It was found that the thermal paste and use of the guard box improved accuracy and precision of the measurements with the reference block. Figure 31 in the appendix shows the results of each VLQ test side by side. This was further supported by the results revealing a fluctuating contact resistance for the without thermal paste and a constant contact resistance for the test with thermal paste. The HLQ test of the TeKa reference block used the recommended settings of 1.61 W/m heating power, 80 s measurement time, 60 min relaxation time, and a minimum LET of 4. The HLQ probe requires pressure to keep it in better contact with the measurement surface. A mechanical clamp provided by the manufacturer was used to fix the sensor to the surface as shown in Figure 21 and placed in the guard box for testing.



Figure 21: TeKa clamp with HLQ probe and reference block (left), placed in guard box (right)

Figure 32 in the appendix shows the results of four tests in a box and whisker plot performed with different pressures placed on the TeKa reference block. All tests contained 18 measurements taken where some points overlap and are not apparent in the plot. Measurement points are represented by a whisker or a circle. The middle line in the box represents the median value, the “X” the average value, the bottom box line the division between 1st and 2nd quartile, and the top of the box the division between 3rd and 4th quartile. Top and bottom whiskers represent the highest and lowest measured value of thermal conductivity if the value was contained within \pm

1.5·IQR (inter-quartile range). The IQR is range of values that contains the middle 50% of measurements and is equivalent to subtracting the value of the first quartile from the third quartile. Otherwise, the value is considered an outlier and plotted outside the whisker. The whisker in this case is the next measurement point contained within $\pm 1.5 \cdot \text{IQR}$. All test runs had a consistent contact resistance that was not considered a factor for determining the thermal conductivity. Table 2 shows contact resistance values for each measurement including thermal conductivity.

Table 2: HLQ measurement tests arranged by pressure setting and contact resistance value

meas. #	λ @ 1.5 kN W/(m·K)	CV [-]	λ @ 1.0 kN W/(m·K)	CV [-]	λ @ 0.2 kN W/(m·K)	CV [-]	λ @ 0.1 kN W/(m·K)	CV [-]
1	1.582	10.08	1.588	10.23	1.605	10.16	1.616	10.08
2	1.614	10.08	1.609	10.16	1.623	10.31	1.586	10.08
3	1.587	10	1.625	10	1.595	10.16	1.606	10
4	1.615	10.31	1.603	10	1.608	10	1.625	10
5	1.597	10.16	1.616	10.23	1.638	10.08	1.586	10
6	1.616	10.23	1.621	10.23	1.615	10.16	1.582	10
7	1.589	10.08	1.616	10.23	1.604	10.16	1.594	10
8	1.568	10	1.608	10.23	1.582	10	1.601	10
9	1.572	10.08	1.628	10.08	1.595	10	1.62	10
10	1.644	10	1.605	10	1.601	10	1.582	10
11	1.585	10	1.587	10.08	1.594	10	1.579	10.08
12	1.627	10	1.603	10.08	1.6	10	1.578	10
13	1.617	10	1.628	10.23	1.601	10	1.634	10
14	1.579	10	1.616	10.23	1.595	10	1.604	10
15	1.596	10.16	1.648	10	1.581	10.16	1.627	10.23
16	1.584	10	1.595	10	1.625	10	1.606	10
17	1.592	10	1.591	10	1.605	10.16	1.599	10
18	1.593	10.08	1.633	10.23	1.58	10.16	1.618	10

The second test using 1.0 kN pressure was the most successful having a median value, 1.613 W/(m·K), very close to the average value, 1.612 W/(m·K), indicating a balanced measurement set and having zero outliers. Both are quite close to the supplied reference block value of 1.614 W/(m·K). It is important to note the clamp does not stay fixed for periods of time longer than 12 hours. It was necessary to fix the clamp handle in order to maintain a constant pressure during testing.

The dry unconsolidated reference sample (PQS) was poured into a cylinder with a sieve at the bottom to regulate the density and thus porosity of the sample. From this point forward in the project time did not permit to test the effects of varying moisture or sample material temperature. Ambient conditions were only considered for this report. Only dry material was to produce a uniform distribution of a porous reference material for measurement. The results of oven heating a 3553.8 g sample of PQS overnight resulted in a loss of 2 g. PQS samples measured were therefore considered dry. Results for density and porosity by pouring sand from a very low height above the sand level in a glass graduated cylinder are shown in Table 3.

Table 3: Table results of tube pouring PQS into a glass graduated cylinder

Mass cylinder	Total mass	PQS volume	Density	Porosity
kg	kg	m ³	kg/m ³	-
0.4042	1.1507	4.95E-04	1.508E+03	0.4309
0.4042	1.1303	4.85E-04	1.497E+03	0.4351
0.4042	1.0625	4.43E-04	1.488E+03	0.4386
0.4042	0.9864	3.90E-04	1.493E+03	0.4367
0.4042	0.7671	2.45E-04	1.481E+03	0.4410
0.4042	0.8862	3.25E-04	1.483E+03	0.4403
0.4042	0.9372	3.60E-04	1.481E+03	0.4413
0.4042	0.7782	2.53E-04	1.481E+03	0.4411
0.4042	1.1083	4.75E-04	1.482E+03	0.4406
0.4042	1.1069	4.73E-04	1.487E+03	0.4388
	average (density porosity)		1488.13	0.4384
	standard deviation (density porosity)		9.44	0.0036

The heights for pouring sand were not recorded as the process was done completely by hand using only visual estimation for maintaining consistency. The results were strikingly accurate however, the use of a long thin geometry limited the ability to keep the sand from piling up in the middle. This caused some sand grains to fall farther than others and thus creating an undesired non-uniform density distribution. This feature is not reflected in the calculations above as they are an average for the bulk volume of the material. The decision was made to use a wider vessel in the form of a glass beaker with a larger volume avoiding the feature of “piling” or “heaping” in the middle of the sample material during preparation.

The consolidated material tested was the main focus of this project. Two soil samples from different bore holes were prepared for measurement with the TeKa TK04. The TK04 has drill bits included for creating a pilot hole suitable to insert the VLQ probe into the sample material.

The results of using the CNC machine into the first sample (B4.3b [62]) were not successful. In order to maintain the composition of the sample, no cooling fluid could be used nor water. The drill bit failed on a material level and the CNC was only able to make a ~ 2 mm deep hole at 10 μm increments. Figure 22: Drilling attempt on soil sample B4.3b below is a close up of the drilling attempt using the CNC machine.

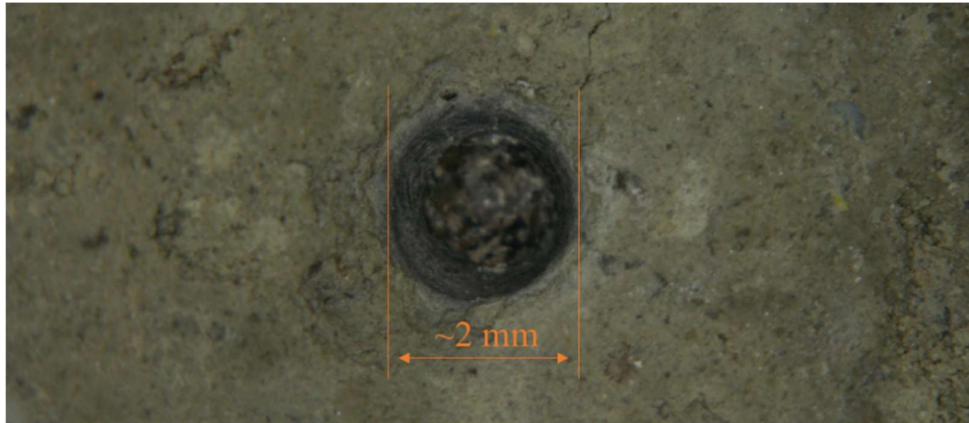


Figure 22: Drilling attempt on soil sample B4.3b

At this point it was decided that drilling a hole into this material is extremely challenging given the constraints for preparation, time allotted, and equipment. The second option to measure soil samples with the HLQ probe was explored. Attempts were made using rasps, files, hacksaws, and wire screens to finish a surface on soil sample B4.3b. The results were promising until a mechanical breakdown of the material occurred. This soil sample was a rock nearly the entire diameter (150 mm) and most of the depth (~ 80 mm) covered in chunks of soil at one end and on lateral surfaces. The result was a sample which crumbled away to reveal a big rock. Figure 23 shows the result after making the second attempt to finish a surface for use with the HLQ probe.



Figure 23: Soil sample B4.3b after second preparation attempt

Dr. Arnold expressed this is not uncommon occurrence sampling soil consisting of sub-glacial moraine. A second sample was obtained from the HSLU Geotechnik Department from a different bore hole location for preparation. Soil sample B3.1a was prepared by the same method which terminated progress on soil sample B4.3b. Figure 24 shows the finished product of sawing, rasping, and finishing soil sample B3.1a with a Dremel rotary grinding tool.

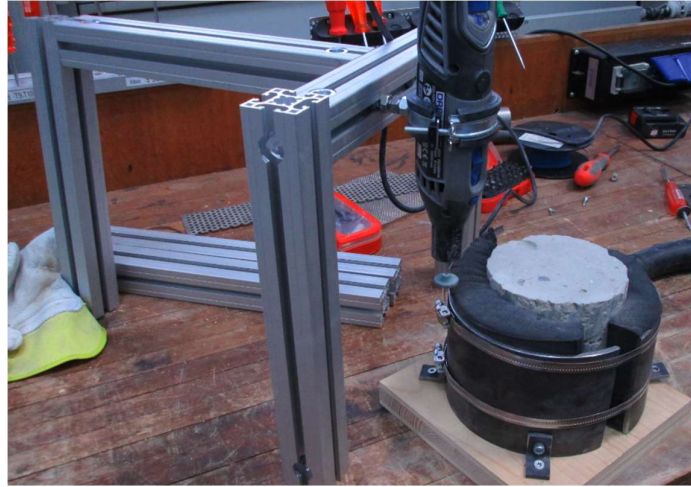


Figure 24: Finished surface of soil sample B3.1a

Measurement results for the TK04 with the VLQ probe are summarized in Figure 33 the appendix showing the results for varying porosities, heating power, and measurement times. All tests were conducted with a temperature increase of $\sim 2^{\circ}\text{C}$ during measurement. Table 4 below shows how individual tests were differentiated and the measured result.

Table 4: Settings for VLQ probe tests on PQS

Glass Beaker Tests with VLQ probe

Test	Heating Power [W/m]	Measurement Time [sec]	Average Contact Value [-]	λ [W/(m·K)]	Porosity [-]
4	0.5	80	16.87	$0.2854 \pm 13.97\%$	0.3931
5	0.7	80	17.14	$0.2830 \pm 2.50\%$	0.3931
6	0.5	80	17.93	$0.3630 \pm 23.51\%$	0.3533
7	0.35	80	16.73	$0.3537 \pm 26.95\%$	0.3533
8	0.5	100	18.12	$0.3639 \pm 3.73\%$	0.3533
9	0.5	120	17.70	$0.3610 \pm 13.81\%$	0.3533
10	0.5	100	18.27	$0.2951 \pm 3.34\%$	0.3969
11	0.5	100	17.99	$0.3656 \pm 7.19\%$	0.3459
13	0.5	100	17.64	$0.2757 \pm 3.60\%$	0.4058

It was found the heating power between 0.35 W/m and 0.7 W/m was sufficient to measure PQS. However, it was not clear from the tests why there was such a large variability in the range of results. The literature review indicated contact resistance is a source of measurement error as well as possible convective heat transfer in the air occupying the porous voids between grains of sand. These results were sent for review to TeKa with the response confirming contact resistance values were too high and inconsistent to support reliable measurement results. It was also expressed that one should not be attempting to measure sand with the TK04 which brought measurements with the VLQ to a stop.

The results for the TK04 using the HLQ probe with soil sample B3.1a were split into two groups. The first group of results are for the soil sample with a manually finished surface for contact with the measurement probe. Various heating powers were tested to generate measurement results along with the recommended settings of 80 s measurement time and minimum LET of 4. The temperature increase during measurement was $\sim 2^{\circ}\text{C}$. The first attempt results from this method as it were nearly unusable. The HLQ probe is very sensitive to the finished quality of the planar surface on the soil sample. This resulted in an air gap between the probe and soil sample discovered after measurement. Contact resistance values were found to be high (~ 17 to 19) and there were numerous instances where measurements were not at all possible. Table 5 shows an abbreviated version of results from this first measurement attempt using different heating powers.

Table 5: Sample of results from hand finished soil sample using HLQ probe

First attempt to measure soil sample B3.1a, hand finished surface, porosity 0.3366

Test/ meas. #	Heating Power [W/m]	λ W/(m·K)	Contact Value [-]	Clamp pressure [kN]	# of solu- tions
1/1	1.61	1.390	17.42	1.5	33
1/2	1.61	-	17.50	1.5	-
1/3	1.61	-	17.58	1.5	-
2/1	1.1	1.235	17.66	1.5	2
2/2	1.1	-	17.66	1.5	-
2/3	1.1	1.236	17.66	1.5	19
3/1	0.8	1.320	17.81	1.5	32
3/2	0.8	1.285	17.81	1.5	51
3/3	0.8	1.279	17.89	1.5	52
4/1	0.6	1.088	18.28	1.5	35
4/2	0.6	1.154	18.44	1.5	88
4/3	0.6	1.160	18.52	1.5	11

Dashes in the thermal conductivity column represent a measurement that was not possible due to poor contact with the sample material producing LET values below the minimum threshold ($LET = 4$).

The second group of results were generated after the refinishing of the soil surface for soil sample B3.1a by changing the heating power. A more planar surface was achieved during the sample preparation using a support holding a leveled grinding tool. Table 6 shows an abbreviated set of results for each heating power tested.

Table 6: Sample of results from machine finished soil sample using HLQ probe

Second attempt to measure soil sample B3.1a, machine finished surface, porosity 0.3366

Test/ meas. #	Heating Power [W/m]	λ W/(m·K)	Contact Value [-]	Clamp pressure [kN]	# of solu- tions
1/1	1.8	1.792	10	1.5	121
1/2	1.8	1.770	10	1.5	27
1/3	1.8	1.780	10	1.5	261
2/1	2.2	1.720	10	1.5	45
2/2	2.2	1.730	10	1.5	19
2/3	2.2	1.688	10	1.5	27
3/1	1.4	1.668	10	1.5	50
3/2	1.4	1.807	10	1.5	188
3/3	1.4	1.709	10	1.5	95
4/1	1.5	1.704	10	1.5	3
4/2	1.5	1.730	10	1.5	159
4/3	1.5	1.738	10	1.5	167
5/1	1.6	1.739	10	1.5	105
5/2	1.6	1.729	10.63	1.5	167
5/3	1.6	1.653	10	1.5	107

The second attempt had near perfect contact values and an increased number of solutions for each measurement. It was inconclusive to deduce which measurement was the most accurate result as these measurements were made for a material with randomly dispersed inhomogeneous material parts in the form of small rocks. An inhomogeneous material field creates a non-uniform temperature profile in the material during measurement which is outside the approximation method used for this measurement technique. Figure 34 in the appendix shows the full results of measurements made with a refined sample surface.

4.3 Linseis THB-100

The Linseis THB-100 is located in the chemistry laboratory. Ambient temperatures were monitored with a handheld temperature sensor to confirm stable conditions before starting a measurement. It was found that data taken in the chemistry lab was suitable for measurement tests.

The Linseis THB-100 comes with pre-set calibrations defined by the manufacturer that automatically recommend the heating current and measurement time needed to measure a sample material. A list of choices for different thermal conductivities can be made from a drop-down menu before measurement begins to select a material of a similar thermal conductivity. For the PQS this was PMMA which has a listed thermal conductivity of $0.193 \text{ W/(m}\cdot\text{K)}$ [32]. From this point forward in the measurement procedure, it is up to the user to define a region of stability in terms of thermal conductivity versus heating current and measurement time for a sample material with unknown thermal conductivity. The goal is to find a region where reasonably small changes in the heating current ($\sim 5 \text{ mA}$) and measurement time ($\sim 10 \text{ s}$) do not change the measured thermal conductivity and thermal diffusivity. It was planned to test 5 different porosities of sand however, due to limited time, only one sample of PQS was tested for thermal conductivity and thermal diffusivity using the THB-100. The PQS sample had a porosity of 0.411 and was measured with the THB6N/MFR sensor with metal support. The metal support is necessary to keep the delicate sensor from bending and potentially breaking. Figure 25 shows the THB sensor used and how it was fixed before placement in the PQS sample.



Figure 25: THB sensor used to measure PQS sample (left), sensor fixed to cork donut for support (right)

Measurement settings were chosen using the Linseis recommended measurement time of 100 to 300 s and measurement current between 50 to 80 mA for the PQS sample. Each data point

shown represents the average of three measurements taken during tests. Below are the results of measuring thermal conductivity and thermal diffusivity for several measurement times (Figure 26) and measurement currents (Figure 27).

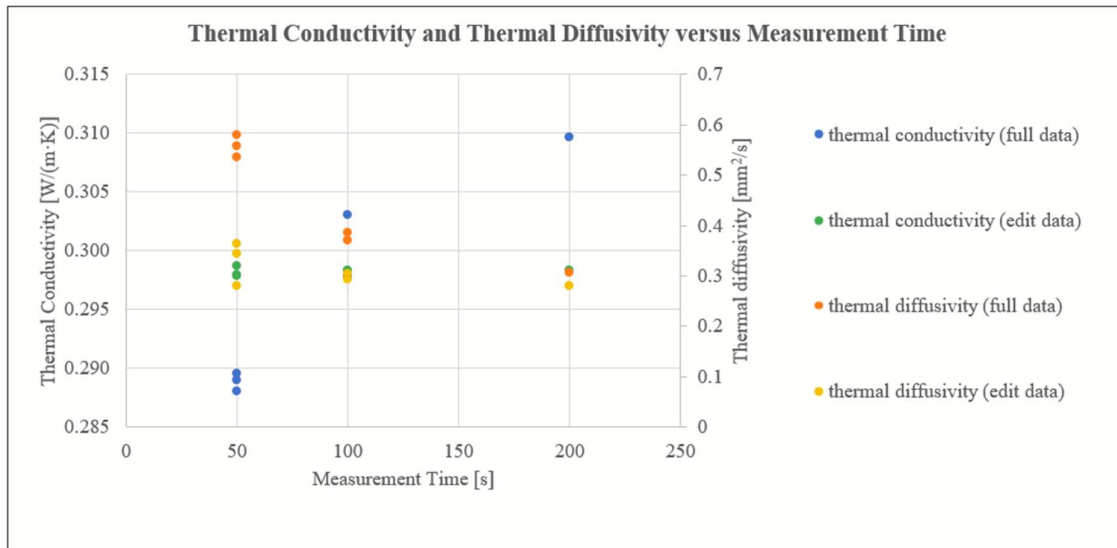


Figure 26: Thermal conductivity and thermal diffusivity for PQS and porosity 0.411

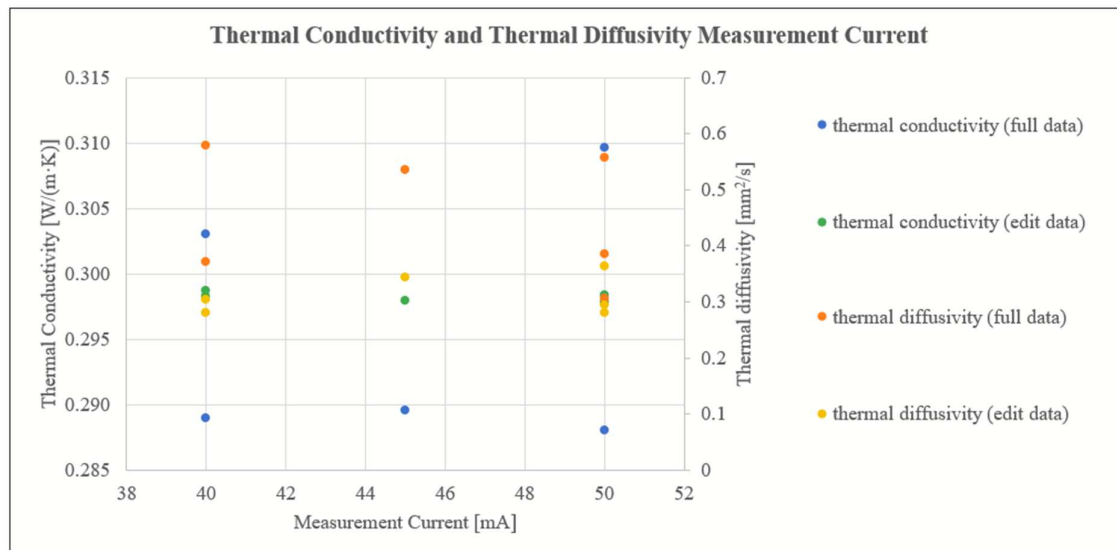


Figure 27: Thermal conductivity and thermal diffusivity for PQS and porosity 0.411

Each individual test did not exceed a 1.5°C temperature increase during measurement with the smaller differences associated with low measurement times and measurement currents. Results from the Linseis THB-100 use a subjective data management process after a measurement has been taken. The initial measurement period (usually < 2.5 s) contains voltage fluctuations from the power supply which create noise not associated with the measurement of the sample material. It is therefore essential to remove these data points to have a clear measurement signal.

However, this process is completely user driven and there is not a set procedure for how disturbed a data point must be to determine its removal. This step is completed graphically by the user and not very well defined overall.

4.4 PQS Sample Data and Model Comparisons

The TeKa TK04 and Linseis THB-100 were used to measure a material with well-studied thermophysical properties. The results of the TK04 and THB-100 for PQS were plotted against some empirical studies and models on porous quartz sands for comparison. Figure 35 in the appendix shows a graphical comparison of the results for both devices, studies, and models [8,11,63,64]. All external studies raise the importance of contact resistance and how this can affect the measurement result. TeKa explicitly expressed that measuring sand is not reliable with the TK04 measurement device. The communication did not elicit what the critical threshold for contact resistance is with the TK04. Measurements made with the TK04 were within range of other studies conducted using TLS measurement equipment and mathematical models. The one measurement point for the THB-100 makes it impossible to determine its suitability for an unconsolidated dry material such as sand across a range of porosities. The data points plotted in Figure 35 represent an initial test of using the system and are not useful for comparison. Manufacturers for both measurement devices claim their instruments can measure porous materials but do not specify what type of porosities are measurable nor a threshold for contact resistance. Measurement accuracy is defined by user experience and sample preparation to determine if a material can be measured by these instruments. Therefore, without an extensive study considering sample preparation and more time to work through all possible parameter setting applicable for measuring PQS, it is impossible to determine if the measurement devices tested are functioning as intended by the manufacturer.

4.5 Cost Analysis

Cost analysis is always a significant decision factor when considering scientific measurement equipment acquisition. Repeated attempts were made by e-mail to contact manufacturers supplying commercial equipment for price quotes. All manufacturers did not provide a quote, rather they requested the attendance of webinars or in-person viewing of equipment. At this time, we do not have an economic assessment for a measurement device to measure thermophysical properties.

5 Conclusion & Future steps

This section contains conclusions based on the literature review, lab testing, and communication with manufacturers. It provides a recommendation for measurement method and technique for measuring porous, moist materials with a focus on soils. Not all investigations were taken to completion and they are outlined further in the future steps subsection.

5.1 Conclusion and Recommendation

Precise measurement of thermophysical material properties are required for the design and performance of thermal energy storage media. Thermophysical performance is crucial to evaluating whether a material is suitable for an intended use. Most methods and techniques over the past 70 years have been developed to measure thermal conductivity. A minority of techniques able to measure thermal diffusivity in addition to thermal conductivity have been developed since 1990. Steady-state techniques are time consuming and require a relatively large sample size. Transient techniques are often favored for their shorter measurement times and simplified methods of simplifying contact resistance between sensor and sample material, and a smaller required sample size. Therefore, it is recommended to use a transient measurement technique for measuring soil and insulations. However, transient techniques require a specific, often exact, surface quality and/or sample material geometry to produce quality measurements.

The measurement experience using the TK04 and Linseis THB-100 during the testing phase demonstrated how important sample preparation is for a quality result. The measurement results taken are inconclusive and requires further investigation. Comparison of the measured values with empirical experiments and models indicate the progress made during this project is in the correct direction. However, there remain unknown uncertainty in the measurements. The contact resistance threshold for poor measurements is not well defined in literature nor by the manufacturer. The sand samples which were tested are a perfect example of this challenge. In addition, inhomogeneous materials present an unknown source of error from a non-uniform temperature field during measurement. The soil samples tested during this project had various sizes and quantity of rocks mixed into the bulk material. The number of options for measurement probes increases the chances of a match between sample material preparation and measurement probe. Therefore, the C-Therm Trident is recommended as it employs three separate measurement probes (transient line source, transient plane source, modified transient plane source) in one device. This device alongside the Linseis THB-100 would give CCTES four different options to measure a sample material.

5.2 Future Steps

The next steps to ensure certain and accurate thermal conductivity and/or thermal diffusivity measurements requires further investigation. A closer inspection of the data needed for modeling must be well defined as it has a great impact on how a sample can be prepared. The investigating team must ask the following questions:

- (i) What are the preparation limitations of the material to be measured?
- (ii) What are the measurement constraints of the project, model, simulation or study?
- (iii) Is it possible to manipulate the structure of the material while maintaining a representative sample for measurement?

The answers to these questions serve as a guide for the number options to prepare a sample with the geometry and surface quality required for an accurate and precise measurement. An example of this would be to grind all the elements of a soil sample to a fine powder. Then a preparation step of shaping, compressing, and humidifying the soil and rock powder into the proper density, geometry, and moisture content before measurement. A high quality, homogeneous sample material would give the best chances for accurate measurements.

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7 Appendix

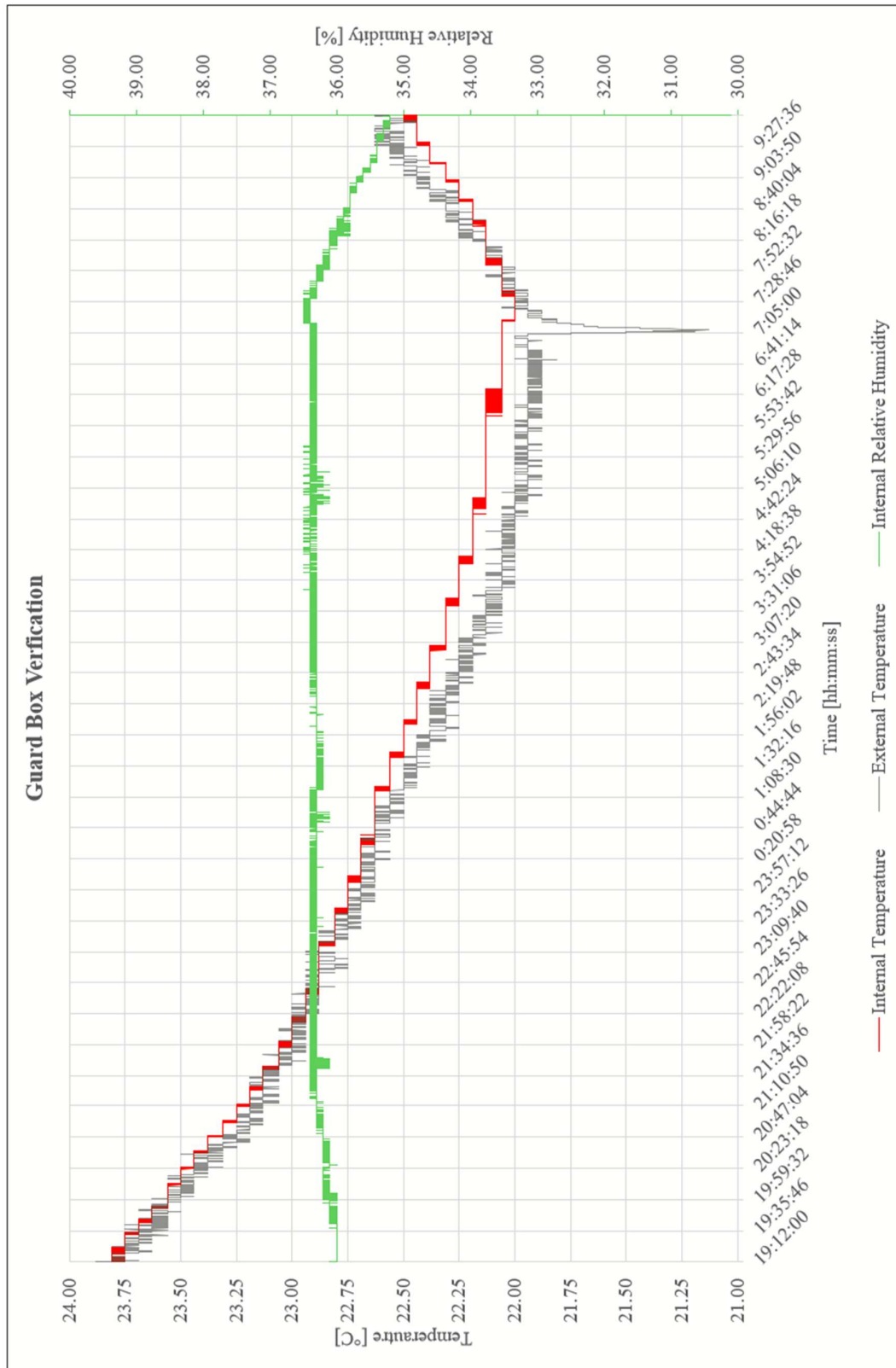


Figure 28: Plot of guard box internal temperature, external temperature, and internal relative humidity

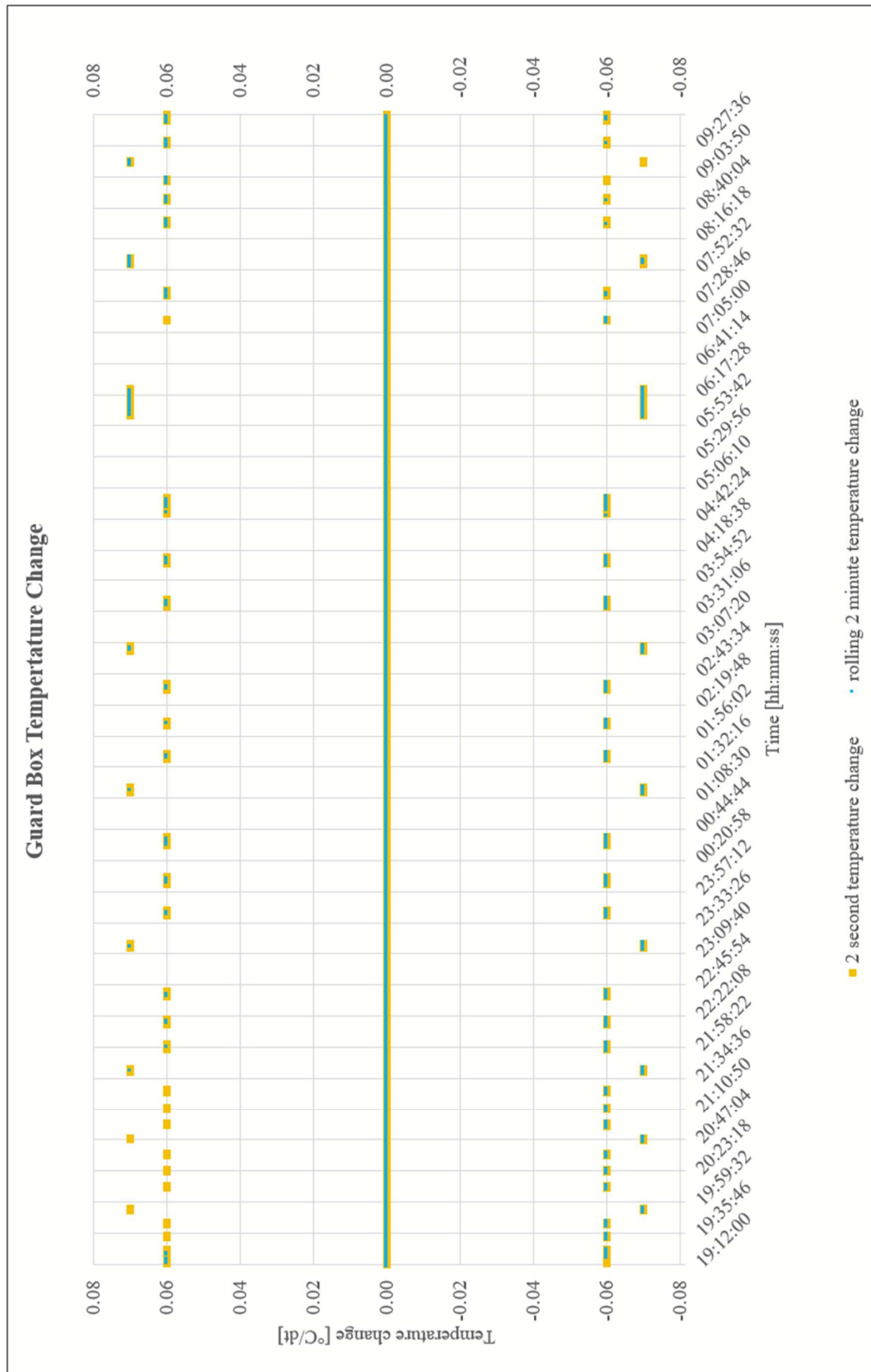


Figure 29: Plot of temperature change over 2 seconds and 2 minutes

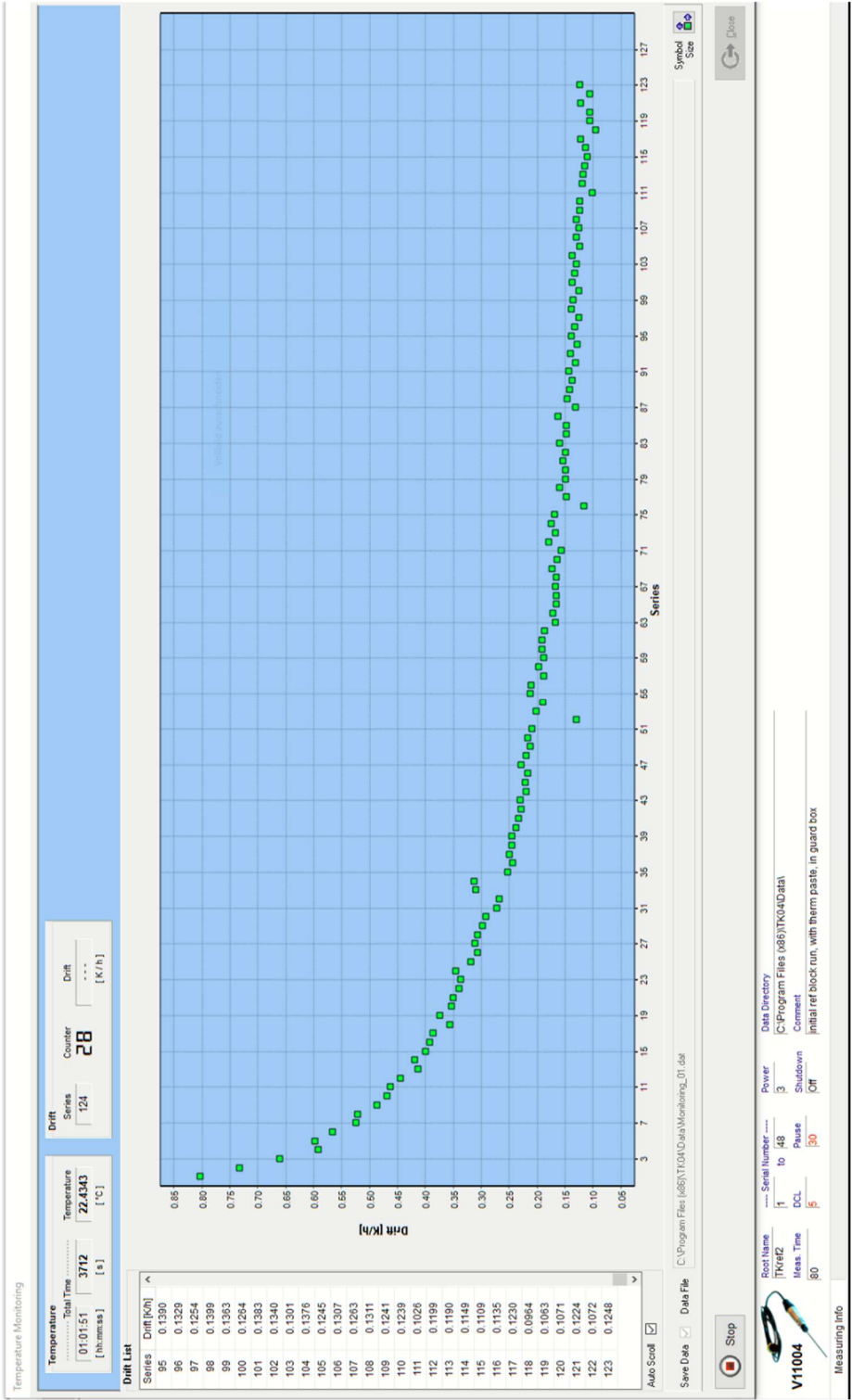


Figure 30: Temperature monitoring of TeKa reference block before measurement

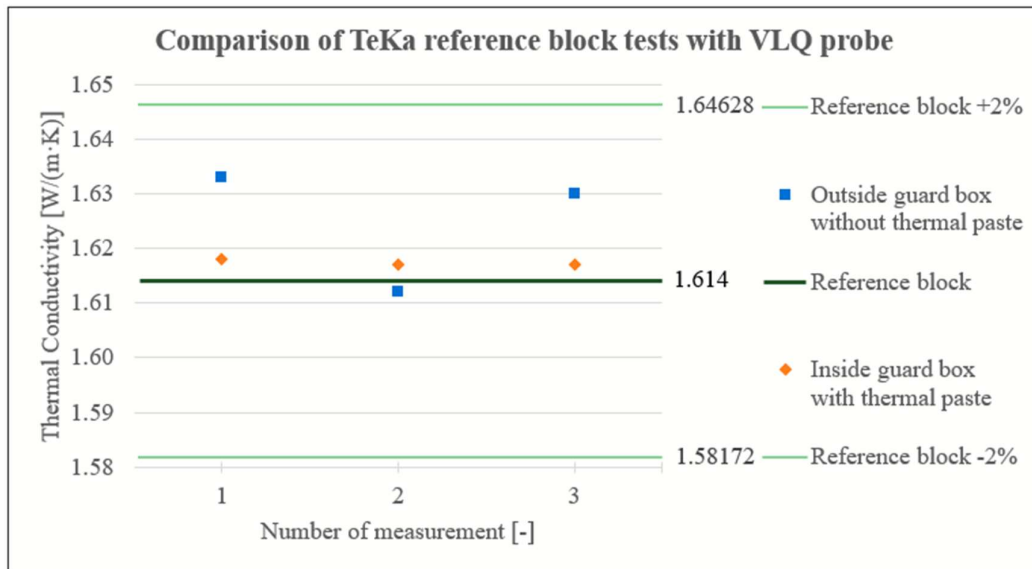


Figure 31: Comparison of VLQ reference block tests

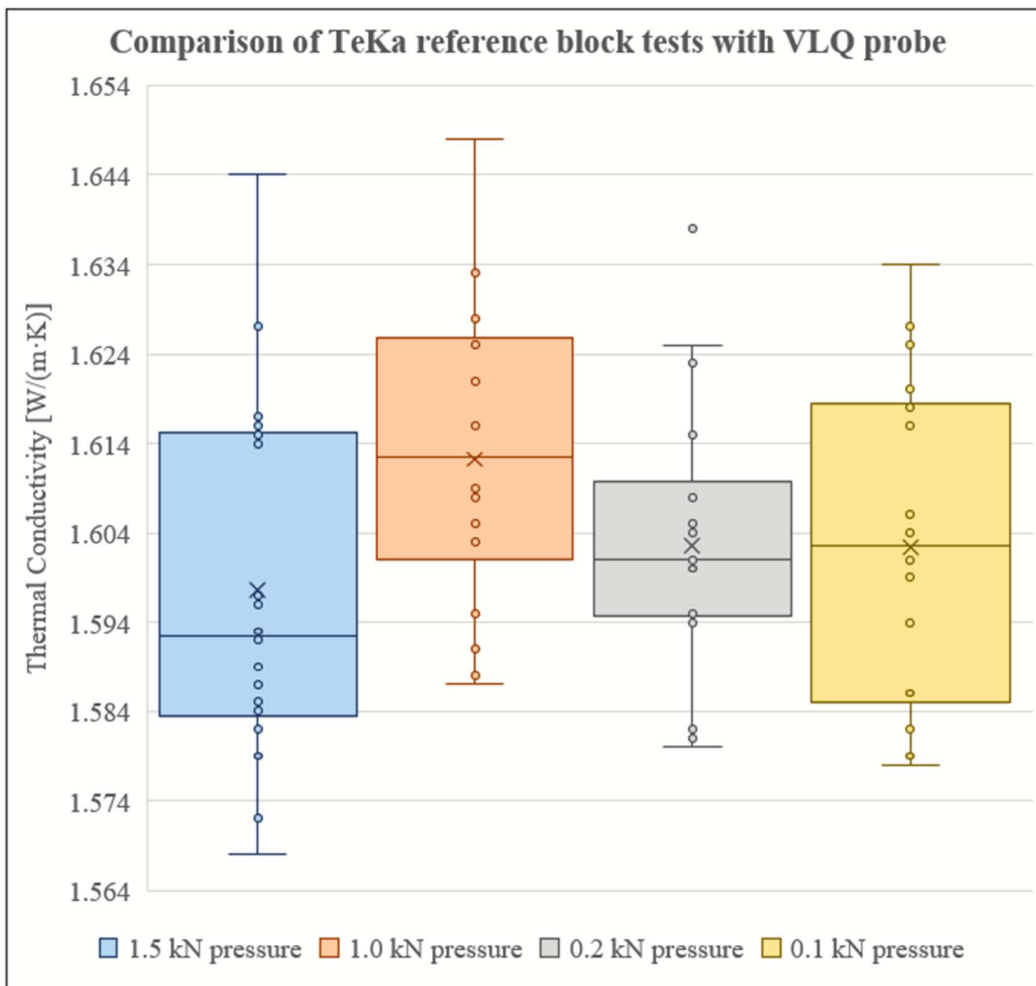


Figure 32: Comparison of HLQ reference block tests

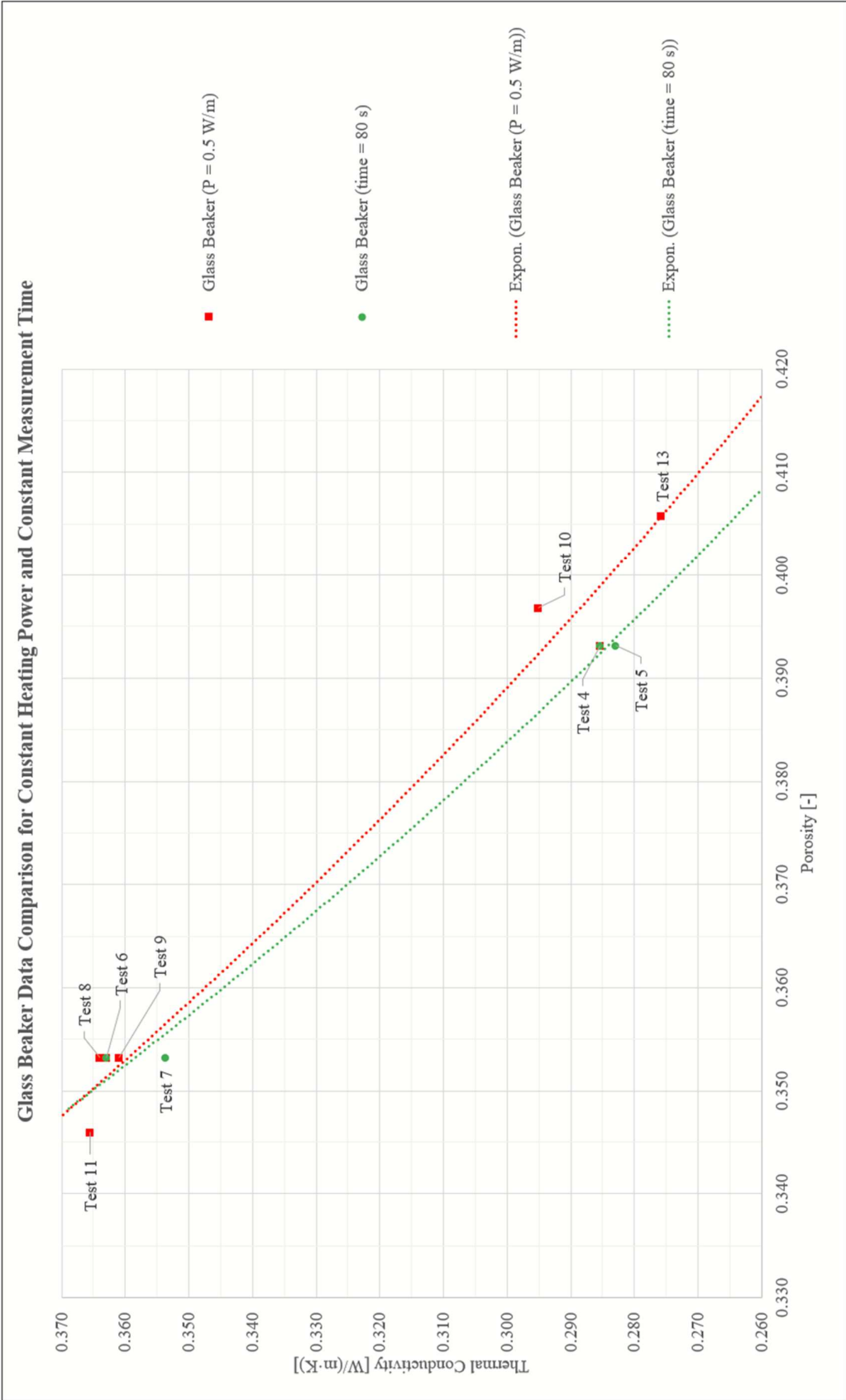


Figure 33: VLQ measurements data

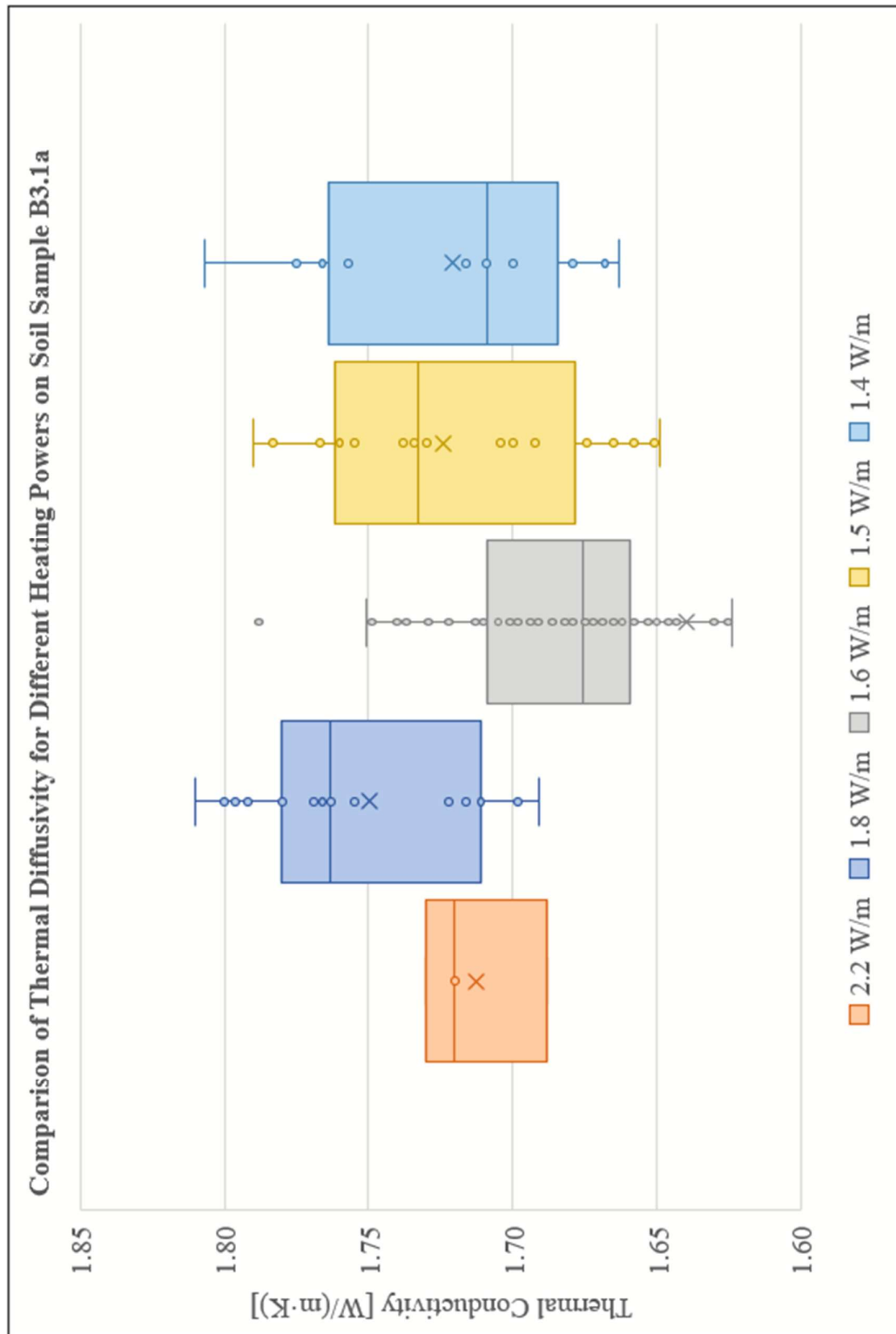


Figure 34: Comparison of thermal conductivity using HLQ probe for machine finished surface on soil sample B3.1a

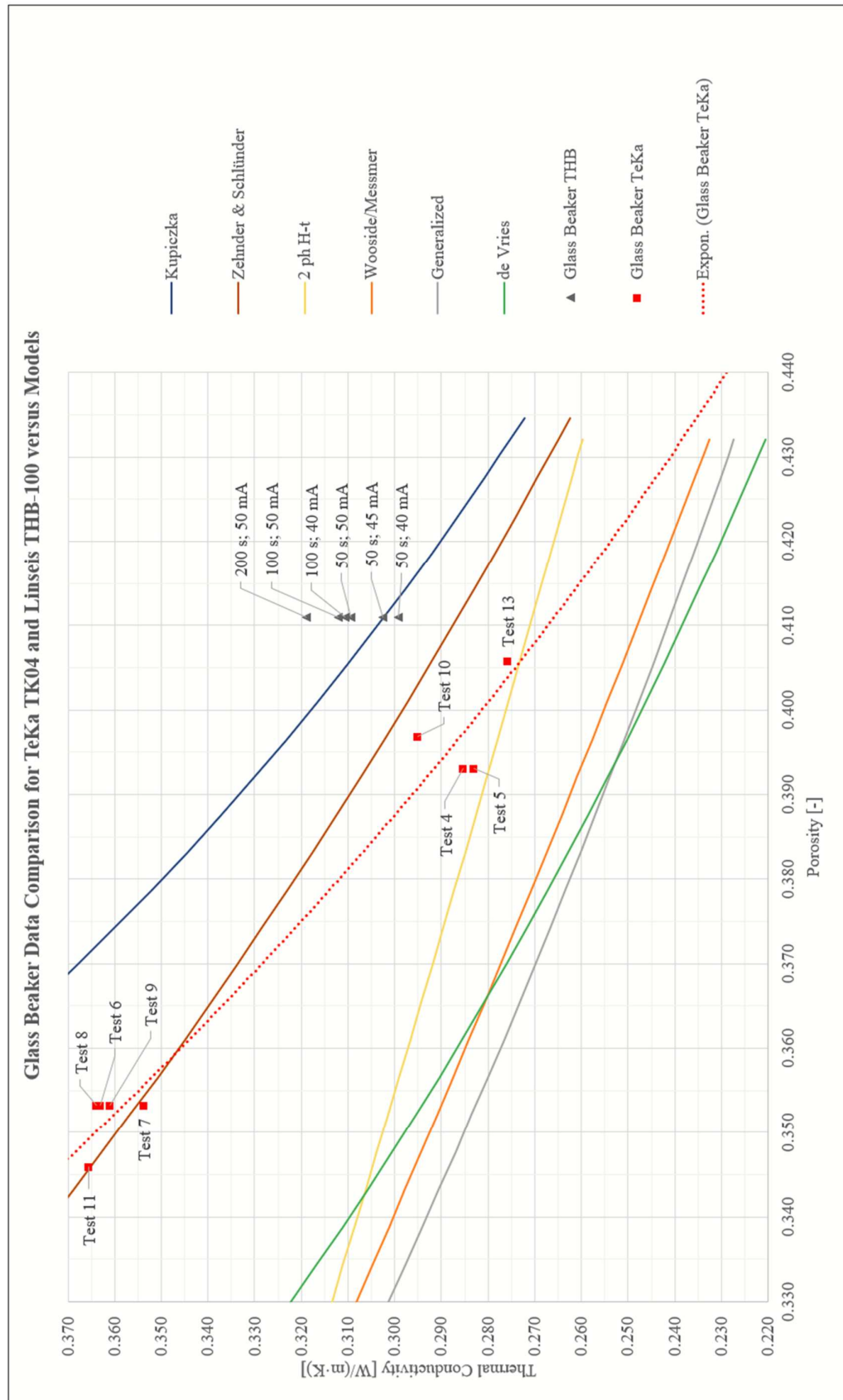


Figure 35: Comparison of VLQ, THB-100, and external studies and models

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Bachelor's Thesis

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Bachelor of Science, Energy Systems Engineering**

**Horw, Lucerne University of Applied Sciences and Arts – E&A
7 June 2019**

Bachelor's Thesis

Thermal Properties Measurement Device Proposal for Soils & Insulation

Robert Beaufait

Grimselweg 10, 6005 Luzern, Switzerland

robertpeter.beaufait@stud.hslu.ch; +41 78 791 19 70

Supervising Lecturers:

Dr. Willy Villasmil, Senior Research Associate

willy.villasmil@hslu.ch; +41 41 349 39 67

Technikumstrasse 21, 6048 Horw, Switzerland

Benjamin Sponagle, Research Associate

benjamin.sponagle@hslu.ch; +41 41 349 33 84

Technikumstrasse 21, 6048 Horw, Switzerland

Expert:

Prof. Dr. Jörg Wortlischek, Chair Competency Center Thermal Energy Storage (CCTES)

joerg.wortlischek@hslu.ch; +41 41 349 39 57

Technikumstrasse 21, 6048 Horw, Switzerland

Lucerne University of Applied Sciences and Arts - School of Engineering and Architecture

Bachelor of Science, Energy Systems Engineering

Horw, Lucerne University of Applied Sciences and Arts – E&A

7 June 2019

Declaration of authorship

I declare that I have written this thesis without any help from others and without the use of documents and aids other than those indicated. All text excerpts used, quotations and contents of other authors are explicitly denoted as such.

Horw, 7 June 2019

Robert Beaufait

Abstract

The concept of using the ground as an energy storage medium has become of great interest to industry and energy network designers. It has been successfully applied as a source for heat pumps as a sustainable method of heating buildings. The next step is to evaluate a scaled-up designs that can handle the energy storage demand of a community or potentially a region. The performance of such a system requires accurate thermophysical measurements of soils and thermal insulations. This report investigates the various methods and techniques to recommend a measurement device for thermal conductivity and/or thermal diffusivity for porous moist materials, specifically soils and thermal insulation. No two methods or techniques are the same and favor some materials and measurement conditions over others. Determining the best method and technique applicable to porous moist materials is the focus of this paper. Each technique is reviewed based on its suitability to measure porous moist materials over the ranges of temperature, moisture, and thermal conductivity applicable to moist soils and thermal insulations. More specifically two transient techniques are tested and evaluated for the purpose of measuring thermal conductivity of soil samples taken from the Thurgau region of Switzerland and quartz sand from the Perth region of Australia. The results are analyzed for quality and accuracy using reference materials and empirical studies and models for comparison. The findings prove inconclusive results for accurate measurements but are consistent with what other studies have found. A revised look at sample preparation and state-of-the-art sensor technology provides an opportunity to obtain more accurate results with better confidence.

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Nomenclature and Acronyms

Q	heat quantity	J
t	time	s
q_i	specific heat flux	W/m ²
λ	thermal conductivity	W/(m·K)
T	temperature	K
x	dimensional length	m
α	overall heat transfer coefficient	W/(m ² ·K)
ε	radiative emissivity	-
σ	Stephan-Boltzmann constant	W/(m ² ·K ⁴)
a	thermal diffusivity	m ² /s
ρ	density	kg/m ³
c_p	specific heat capacity	J/(kg·K)
A_i	area	m ²
l_i	distance	m
\dot{Q}	heating power	W
d	thickness	m
CCTES	Competency Center Thermal Energy Storage	
GHP	Guarded Hot Plate	
HFM	Heat Flow Meter	
GHB	Guarded Hot Box	
THW	Transient Hot Wire	
THS	Transient Hot Strip	
SPHP	Single Probe Heat Pulse	
DPHP	Dual Probe Heat Pulse	
TPS	Transient Plane Source	

1 Introduction

The forward progress of energy management and utilization has supported development of various techniques for energy optimization and utilization. According to an European Union report, significant amounts of thermal energy are produced alongside the typical energy utilization and often goes unused [1]. Thermal energy storage is a progressive field of study investigating solutions designed for optimizing energy supplies from daily to seasonal time scales. This is of great interest to not only the residential and business sectors, but to the industrial sector in the form of peak shaving. A proper assessment is necessary in all cases to design a system that performs competitively with the price of available energy resources (e.g. grid electricity and fossil fuels). Design requirements rely on accurate measurements of the thermophysical properties of storage media and insulation to determine charging and discharging characteristics in addition to the efficiency of a system. Accurate evaluation of insulation and thermal storage media require measurement methods with precise measurements and known uncertainties. Due to the nature of most material properties being dependent on a fundamental characteristic (e.g. temperature, porosity, etc.), there is not a single measurement technique suitable to measure all materials over all possible states and measurement ranges. Therefore, it is imperative to first properly evaluate and classify materials before selecting the method and equipment to determine thermophysical properties (thermal conductivity, thermal diffusivity, and specific/volumetric heat capacity).

1.1 Background

The Competency Center Thermal Energy Storage (CCTES) is conducting a project with an industry partner, Schenk AG, to develop novel water-proof thermal insulation materials suitable for high temperature applications. These materials are by nature porous and may be exposed or eventually retain moisture especially for in ground applications. In addition, another CCTES project is tasked with conducting a simulation-based techno-economic feasibility study for a novel seasonal energy storage concept using soil as shallow geothermal storage media. Soil exhibits varying thermal properties dependent on a number of physical characteristics which influence how these properties can be measured accurately. Both cases require a thermal property measurement method which is able to handle varying degrees of porosity, homogeneity, and moisture content with a high degree of accuracy and acceptable uncertainty.

1.2 Project Aim and Objectives

Measurement of thermophysical properties for moist soil and thermal insulation presents a challenge to the practical application of the developed methods and measurement devices. Established thermal material property values for industry standards are often determined in a test environment that is not representative of many applied conditions. This project aims to study and identify suitable solutions for thermal property measurement of insulation and soil that are porous, in some cases inhomogeneous, and may contain moisture. The project aim is to be achieved by identifying the measurement device requirements and constraints through consultation with CCTES and an extensive literature review. The literature review includes a survey of the current methods to determine the required thermal material properties and the current state-of-the-art equipment available commercially. Furthermore, a practical review of the in-house CCTES measurement device along with a separate device employing a different measurement method are analyzed for comparison.

1.3 Project Goals

The goal of this project is to present a supported recommendation for either the acquisition of a commercial or in-house developed device suited for measuring at least two of the three required thermal properties. In either case, a detailed cost analysis of the best suited device and supported justification. In addition, a method to handle and properly measure the thermal properties of moist porous materials will be explicitly described.

2 Literature Review

An extensive literature review was conducted to research the types of methods to measure thermophysical properties. A basic review of thermophysical properties was conducted to serve as a base for detailing the various measurement methods. Measurement methods are defined as the principals and theories which are the foundations for the development of measurement techniques. Measurement techniques are detailed in their ability to determine thermophysical properties along with other parameters dictating their capabilities and limitations. A selection of state-of-the-art solutions are listed for each applicable technique along with its feasibility for this project.

2.1 Introduction to Thermophysical Properties

Thermophysical properties of a material characterize the way heat interacts with a given substance. The three main properties of a material are the ability to retain heat before changing temperature (heat capacity), the ability to move heat from hot to cold (thermal conductivity), and the ratio of heat transport to heat retention (thermal diffusivity). These properties are affected by the material structure, composition, density, porosity, moisture content, pressure and temperature. Any material has the ability to transfer heat through three distinct mechanisms; conduction, convection, and radiation. These mechanisms and properties are outlined and expanded below.

2.1.1 Basic Theory of Heat Transfer

Heat transfer is characterized by two categories in the time domain three categories with respect to the physical domain. Heat transfer can exist as a steady-state (stationary) process or a transient (non-stationary) process. The steady-state process is defined as unchanged heat transfer with respect to time. The transient process is defined as a variable or changing heat transfer with respect to

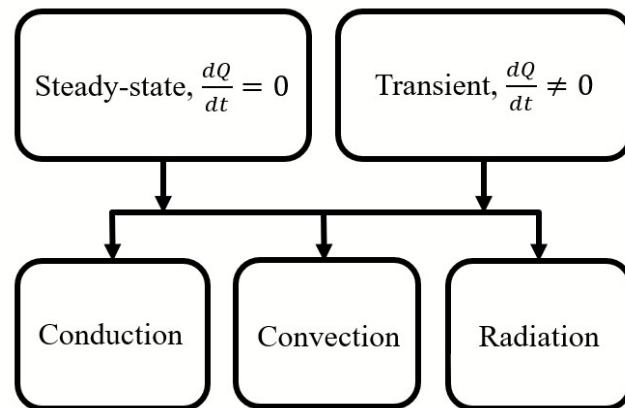


Figure 1: Temporal and physical domains of heat transfer

time. All three physical domain characteristics of heat transfer can exhibit either a steady-state or transient process. The three categories of heat transfer in the physical domain are conduction,

convection, and radiation. Heat conduction is characterized by thermal energy exchange induced by direct contact with adjacent material. Heat convection is characterized by the movement of a fluid increasing the rate of thermal energy transfer between fluid particles. This increases the rate of thermal energy transfer between the fluid and another material when compared with a fluid at rest. Heat radiation is characterized by the energy emitted through electromagnetic waves which are emitted by a material. This category of heat transfer is unlike the previous two as it does not require material contact of any kind to transfer thermal energy [2]. The following sections further detail the three categories within the physical domain.

2.1.2 Conduction

Conduction of heat is the kinetic transfer of thermal energy from a more energetic particle to a less energetic adjacent particle. Heat transfer is the result of a temperature gradient across a given distance and collisions of particles within the material over that distance. Conduction is present in solid, liquid, and gas phase of materials and continues to persist until the temperature gradient no longer exists. The general model of steady-state, one-dimensional heat conduction, is described in the rate equation (1),

$$q_x = -\lambda \cdot \frac{dT}{dx} \left[\frac{W}{m^2} \right] \quad (1)$$

and is known as Fourier's law [3]. The conduction of heat through a material depends on the arrangement of microscopic particles in relation to one another, the amplitude of vibrations of material particles, and how well those vibrations are transferred between neighboring particles. In the case of solids, the types bonds between molecules and availability of free electrons determine the rate of thermal conduction. For example, solids tend to be better conductors of heat as they have constant molecular interaction with a neighboring particle. Moreover, this results in better heat transfer through the material when excited by an energy source. However, not all solid materials are good conductors of heat. The effective conduction of heat is dependent on the path heat flows. This is a function of the material structure and components which define the overall material. There exist materials which do not readily transfer the energy of their neighbors. For example, certain insulations have trapped air bubbles in them that have a much poorer ability to conduct thermal energy thus lowering the overall ability of the material to conduct heat. In general, liquids are generally less conductive than solids and gases less than liquids as the increasing spatial distance between particles reduces the ability to conduct thermal energy efficiently [2].

2.1.3 Convection

Convection heat transfer of energy resulting from the movement of a fluid. There are two categories of convective energy transfer, natural and forced. Natural convection is the movement of a fluid characterized by changes in buoyancy. Buoyancy forces are created when heating of fluid particles in contact with a heat source cause a local decrease in density of the fluid. These particles then move away from the heat source and exchange heat with the surrounding unheated portion of the fluid either by buoyancy or external forcing. It is this second exchange of heat which is considered convective heat transfer [3]. Forced convection is the same concept as natural convection with the difference being that the motion of the fluid is not driven by buoyancy forces, but rather external (e.g. wind, ventilator, pump). Both types of convection are described by equation (2),

$$q = \alpha \cdot (T_{source} - T_{\infty}) \left[\frac{W}{m^2} \right] \quad (2)$$

known as Newton's law of cooling where α is the overall heat transfer coefficient and T_{∞} is the free stream temperature of the fluid outside the direct influence of the heat source [4]. The heat transfer coefficient is dependent on numerous factors with the type of fluid flow being most important (e.g. laminar or turbulent).

2.1.4 Radiation

Thermal radiation is created by the energetic state of a material defined by temperature. It is dependent on the type of material, the type of surface a material has, and by the emissivity or the ability of a material to emit radiation relative to a blackbody (i.e. an ideal emitter). The amount of thermal radiative energy emitted by the surface of a material is offset by the amount being received or absorbed. Thermal radiation is described by equation (3),

$$q_{rad} = \varepsilon \sigma (T_{source}^4 - T_{amb}^4) \left[\frac{W}{m^2} \right] \quad (3)$$

which is an extension of the Stephan-Boltzmann law. Thermal radiation takes the form of electro-magnetic radiation in the infrared range with wave lengths between 700 nm to 1 mm [2].

2.2 Thermophysical Properties

Understanding thermophysical properties is necessary for assessing the suitability of a material as a thermal storage medium. The ability of a material to conduct, store, and diffuse thermal energy fall into two categories, transport and thermodynamic properties. Transport properties describe the movement of thermal energy within a material relative to unit length and temperature (thermal conductivity) and by the ability to diffuse thermal energy relative to time (thermal diffusivity). Thermodynamic properties are a result from the state of the material being considered. Density and specific heat capacity are two thermodynamic properties important for the evaluation of thermophysical properties.

2.2.1 Thermal Conductivity

Previously mentioned in section 3.1.1 Fourier's law of heat conduction is the basis for evaluating how thermal energy flows in a material. Rearranging the Fourier equation for heat conduction, we get equation (4),

$$\lambda = -\frac{q}{(dT/dx)} \left[\frac{\text{W}}{\text{m}\cdot\text{K}} \right] \quad (4)$$

Thermal conductivity is given the units $\text{W}/(\text{m}\cdot\text{K})$ and is a measure of the ratio of specific heat flux in to the temperature gradient applied over a specific distance in a material [3]. A positive change in the ratio of specific heat to the normal temperature gradient between two surfaces results in an increase in thermal conductivity and vice-versa. Figure 2 below is a summary of the preceding statement.

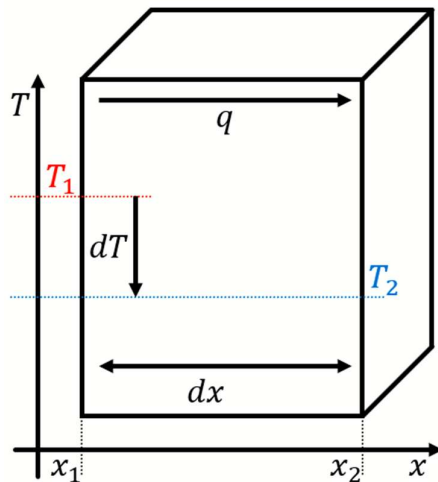


Figure 2: Thermal conductivity

2.2.2 Thermal Diffusivity

Thermal diffusivity is the ratio of the ability to conduct thermal energy to the ability to store thermal energy. It is dependent on both transport and thermodynamic properties and has the units m^2/s . Thermal diffusivity relates to thermal conductivity, density, and specific heat capacity in equation (5).

$$a = \frac{\lambda}{\rho \cdot c_p} \left[\frac{\text{m}^2}{\text{s}} \right] \quad (5)$$

Materials which have a large thermal diffusivity will be more sensitive to environmental changes and are able to quickly reach a new state of equilibrium. Precise and accurate thermophysical properties are necessary to calculate thermal diffusivity especially for low values of thermal conductivity.

2.2.3 Specific Heat Capacity

Specific heat capacity is the measure of thermal energy a material can store per unit mass before experiencing a temperature change of 1 K. It is a measure of the amount of energy a material can accept before incurring an increase in temperature. How a material stores this energy is dependent on the interactions between individual molecules within a material. Specifically, the ability of thermal energy to free electrons for heat transfer in addition to the heat transfer from harmonic oscillations of molecules ultimately defines the specific heat capacity of a material [3].

2.3 Definition of Material Characteristics

An accurate determination of the thermal properties of a material require proper material characterization. The physical characteristics affect the thermal behavior of a sample material. A sample material entirely made up of one or more substances in the same phase uniformly distributed is considered homogenous [5]. Furthermore, a homogenous material that exhibits equal thermophysical properties in all directions relative to an axis is considered isotropic. Often, we encounter materials that vary in some material characteristic over the volume being studied or applied. In nature many materials are considered inhomogeneous and anisotropic. By definition these thermophysical characteristics (i.e. thermal properties) are not uniform over a material volume. It is therefore, critical one clearly define the sample material being analyzed with regards to characteristics affecting thermal properties.

2.3.1 Density

Density is defined as the amount of mass per unit volume and is important in the calculation of heat transfer. It scales the ratio between how well a material conducts thermal energy, thermal conductivity, and how well a material stores thermal energy. Generally, a material of a higher density is indicative of a solid or liquid. Bulk density is the measure of mass per unit volume including any spaces not occupied by matter within the evaluated volume.

2.3.2 Porosity

Porosity is the measure of distributed space that is open inside a solid or loosely packed material. These regions not occupied by the evaluated material can be closed or open to the environment and are occupied by a fluid (e.g. air or water). The physical orientation of the pores and the material which occupies them are crucial to understanding how heat flows through the bulk of a material. Figure 3 below shows two cases of heat flow through a two-component material in which the thermal conductivity of each component is not equal [4].

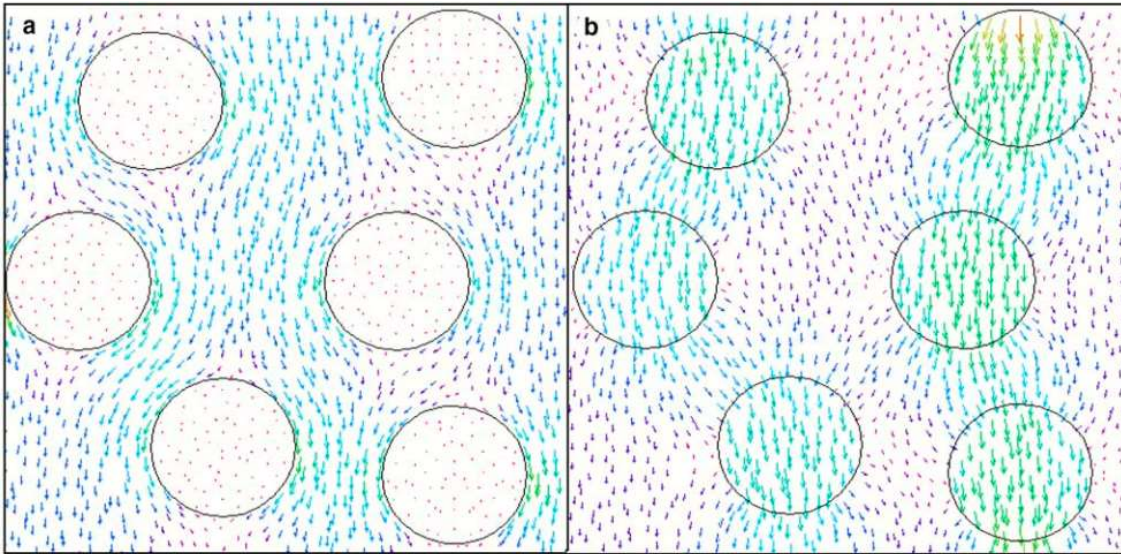


Figure 3: Heat flow through a two-phase material [4]

In case a) the thermal conductivity of the continuous phase is higher than the discontinuous phase and in case b) the opposite is true. Both cases are representative of a matrix of material that maintains the same thermophysical properties in all directions (isotropic). Furthermore, each case is considered to be a disconnected-internal pore structure where there are no connective links between pores and a fluid is unable to flow between pores or to the environment. However, there are many types of pore structures which lead to different thermal transport characteristics.

For the case of connected external pores, a fluid phase can move within a material as well as exchanged with the surrounding environment. Connected internal pores are connected inside a material volume but do not have pathways for exchange with the environment [4]. Pore structures are not limited to the above four cases. Many materials possess a mixture of pore structures in their arrangement.

Porosity has a direct effect on the thermal transport properties such as thermal conductivity and thermal diffusivity. Porosity affects the path thermal energy travels through a material and can vary within the same material at different locations [6]. This is especially true for materials with a large difference in thermal conductivity between phases or materials.

2.3.3 Moisture Content

The moisture content of a porous material influences the thermal transport properties of a material by altering the media heat travels through. Moisture is able to occupy voids in the bulk of the material and may cause the overall specific heat capacity as well as the overall thermal conductivity to change (effective thermal conductivity). The magnitude of changes in thermo-physical properties is dependent on porosity and how much of the porous space is occupied by water. In general, specific heat capacity increases along with thermal conductivity increases as water content increases as it displaces air. Figure 4 below shows the relationship between moisture content and specific heat capacity and Figure 5 thermal conductivity for a clay soil from a state of nearly dry to a moisture content of 0.25.

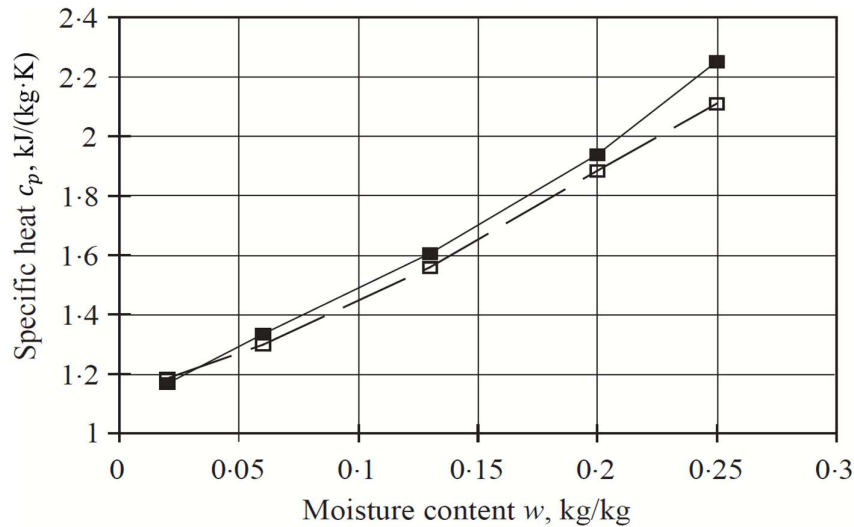


Figure 4: Specific heat capacity versus of moisture content [7]

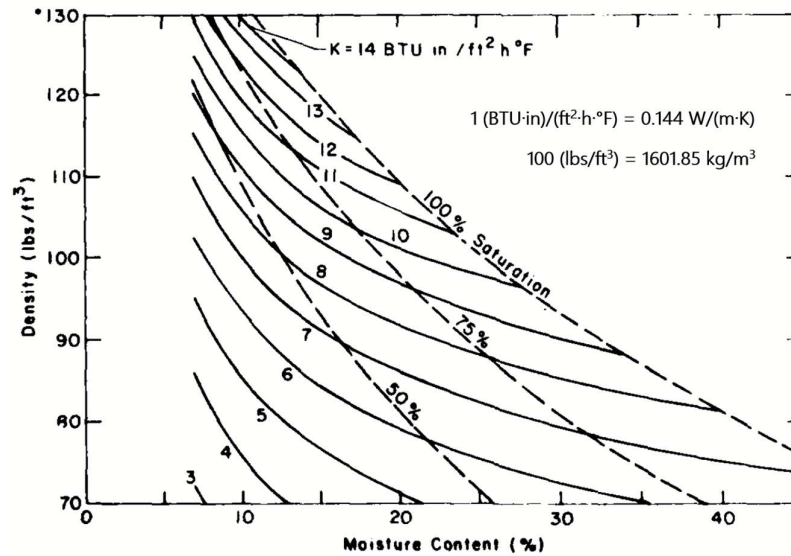


Figure 5: Thermal conductivity versus moisture content and density for clay at 4.44°C [8]

The thermal diffusivity tends to increase the most after gaining moisture from the dry state depending on the material composition, porosity, and size of the pores. A more open pore structure allows moisture to cover the surfaces of material particles faster. This results in a quick rise in thermal diffusivity and thermal conductivity relative to moisture gain than a material more restrictive to water penetrating porous regions [7]. **Error! Reference source not found.** below shows the described behavior of a material open to quick moisture gain along with a material with less ability to accept water.

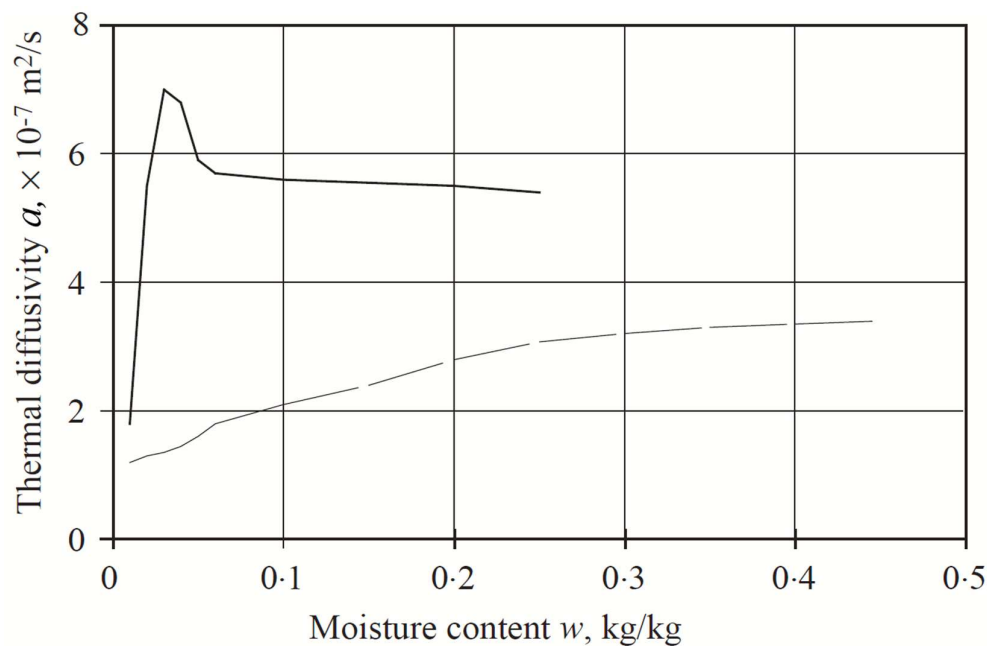


Figure 6: Thermal diffusivity versus moisture content for clay (- -) and sandy (—) soils [9]

2.3.4 Material Composition and Orientation

There have been many studies conducted of the effects of material characteristics and thermal conductivity. These studies consist of empirical measurements paired with an analytical analysis for predicting thermal conductivity for a range of porosities. Assumptions are therefore needed to make an analytical solution possible. It is widely assumed that the thermal conductivity is isotropic in nature and if not, a method of averaging has been applied. In the case of a quartz sand, it is by nature anisotropic regarding thermal conductivity ($11.3 \text{ W}/(\text{m}\cdot\text{K})$ parallel to the optic axis and $6.5 \text{ W}/(\text{m}\cdot\text{K})$ perpendicular) [8]. Another study considers geometric shape as the parameter for predicting thermal conductivity [10]. In addition, material grain size also has the effect of decreasing effective thermal conductivity by reducing grain size. This is the effect of an increased grain surface area which increases the number of thermal barriers heat must cross during its transfer [11].

2.4 Measurement Methods, State-of-the-Art, and Project Suitability

This section describes the various methods and techniques by which thermal conductivity can be measured. The goal of every method is to accurately quantify how heat flows through a material to be measured. Each method has its advantages and disadvantages relative to one another, the material to be measured, and the environment to be tested. Measurement techniques of thermal conductivity fall into one of two types; steady-state and transient. A steady-state method measures the temperature difference across a sample material by a known, constant heat flow. A transient method measures the temperature difference across time at a fixed point in the sample material. Expanded descriptions of the theory, methods, and individual techniques can be found through journal publications and dedicated print materials [12–14]. The following subsections provide introductory and summarized detail of each technique surveyed, availability of commercial equipment, and its suitability for the project aim with an overview table of all methods at the end.

2.4.1 Steady-State Measurement Methods

The steady-state method can be further broken into two measurement types. They are the absolute method and the comparative method. The absolute steady-state measurement method is designed to measure a temperature difference with a constant (or steady) heat flow through a known distance in the material of interest. Once the heat source is turned on all components of the measurement device must come to a state of rest where heat flux across the sample and guard apparatus are constant. This is done by applying a heat source and sink to opposing sides

of a geometrically symmetric sample. This creates the temperature gradient over a specified distance needed to measure the thermal conductivity of the sample. Figure 7 shows a simple example of the absolute steady-state measurement method.

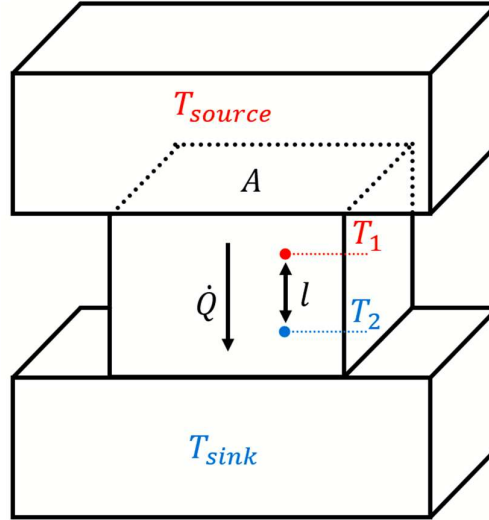


Figure 7: Absolute steady-state measurement method

When applying Fourier's law for one-dimensional heat conduction thermal conductivity is related as follows in equation (6),

$$\lambda = \frac{\dot{Q} \cdot l}{A \cdot \Delta T_{12}} \quad (6)$$

where \dot{Q} is the amount of heat flowing through the sample across the cross-sectional area A over the distance l and temperature differential ΔT_{12} [15].

The comparative steady-state measurement method employs all the concepts above with the addition of a second material of a well-known, similar thermal conductivity to the test sample. The second (reference) material is then placed in series with a test sample ensuring adequate thermal contact between them. A temperature difference is recorded using a minimum of two thermocouples for each material across an equal distance in each material and compared. This type of steady-state method requires a precisely controlled test environment the heat flow across the test sample must nearly match the reference material. Figure 8 below shows the set-up of the comparative steady-state measurement method.

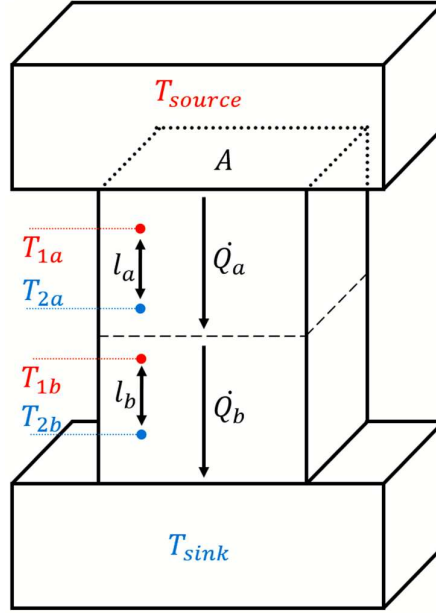


Figure 8: Comparative steady-state measurement method

The comparative steady-state method avoids the requirement to measure the power applied to the sample. The thermal conductivity can be calculated using equation (7),

$$\lambda_a = \lambda_b \cdot \frac{A_b \cdot \Delta T_{12b} \cdot l_a}{A_a \cdot \Delta T_{12a} \cdot l_b} \quad (7)$$

where λ_b is the known thermal conductivity of the reference material, A_i are the cross-sectional areas of the materials, l_i the distances between thermocouples, and ΔT_{12i} the temperature differences between thermocouples. Advantages of both steady-state methods are: (i) the ability to measure low thermal conductivities, (ii) measure samples that are made of more than one material, (iii) heat flow over a larger area allowing for more accurate measurement of anisotropic materials, and (iv) reducing the heat flow to a one-dimensional calculation directly using Fourier's law. Disadvantages include long wait times for the measurement system to reach steady-state, complex sample preparation, and imprecise thermal contact resistance [16]. This can often be several hours up to a day depending on geometry and the conditions being tested. Another major disadvantage is accurately calculating the heat flow through the sample while accounting for parasitic losses. The sample loses heat in the form of radiation and convection to the surroundings and conduction from the couples being used to measure the sample [15]. It is therefore necessary to carefully apply measures to keep heat directed into the sample and insulate the sample from the environment such that heat flow is directed along linear paths from

source to sink [17]. Several techniques are described below applying the steady-state method of measuring thermal conductivity.

2.4.1.1 Guarded Hot Plate (GHP)

The guarded hot plate technique can be used to determine the thermal conductivity of glasses, ceramics, polymers, insulation, liquids, and gases [17]. This absolute technique has a robust measurement temperature range between -190°C and 525°C and relative expanded uncertainties can be reduced approximately to 2 to 3% [18]. However, the guarded hot plate is very sensitive to the homogeneity of a sample. The sample thickness must be a minimum of 10 times greater than the largest material constituent (e.g. pore, crystal, grain, etc.) and must bear a flat plate or disc geometry [17,19]. Additionally, this measurement technique has difficulty measuring moist samples due to convection of the fluid contained in the pores of the material and the long measurement time drying the test material. This measurement method has three major sources of uncertainty, heat flux from losses in the heating plate and sample to the surrounding medium, and contact resistance between the sample material and thermocouples [13,17]. Figure 9 shows the concept of the guarded hot plate measurement technique.

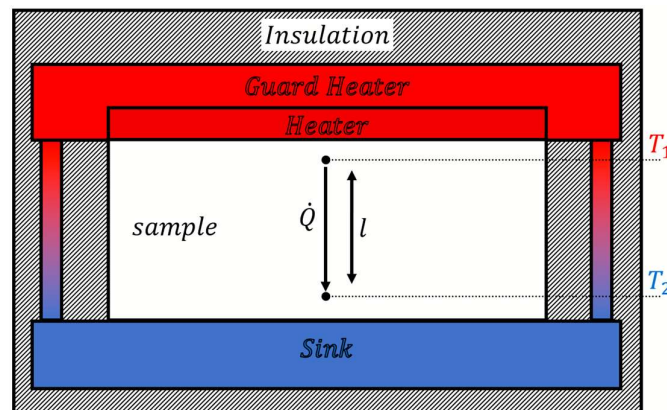


Figure 9: Guarded hot plate measurement technique

The guarded heat plate technique is able to measure a range of thermal conductivity between $0.2 \text{ W}/(\text{m}\cdot\text{K})$ and $200 \text{ W}/(\text{m}\cdot\text{K})$ [20]. Design specifications and build requirements are detailed by ASTM C177, DIN 12667, EN 12939, and ISO 8301/8302 [19]. GHP commercial equipment is available through Netzsch [21] and Taurus [22]. The equipment is large ($\sim 1.5 \text{ m} \cdot 1.5 \text{ m} \cdot 2 \text{ m}$) and heavy ($\sim 270 \text{ kg}$) [23]. The GHP is a very accurate and certain technique to determine thermal conductivity. It is recognized by international standards and can be designed to operate either as an absolute or comparative method. However, this type of technique is very time consuming to build and/or expensive to purchase. The accuracy is strongly dependent on the

ability to guard the heater and the boundary conditions of the sample. Measurement times may take well more than 12 h (up to 36 h) and may require climate control to maintain sample material moisture. This type of measurement technique is not suitable for this project.

2.4.1.2 Heat Flow Meter (HFM)

A heat flow meter (HFM) is able to measure thermal insulation materials, plastics, glasses, ceramics, metals, rocks, and polymers [17,19,24]. The HFM is conceptually the same as the absolute guarded heat plate measurement system except in place of thermocouples, specific heat flux is measured using transducers [17]. An HFM is placed between the heat source and the sample material to measure the amount of power flowing per unit area. Often a second heat flow meter is placed between the sample material and sink. This allows for the radial heat losses along the sample to be calculated [18]. This reduces the amount of time needed for a measurement as the thermal guarding of the sample does not need to be brought to equilibrium as well. Once the target temperatures of the sample are reached, temperatures can be recorded at the heat flux sensors and a determination of thermal conductivity can be made [25]. The HFM technique is considered an absolute steady-state method when measuring a single material and a comparative steady-state method when using a reference material. The advantage of the HFM technique are simple design and operation [17]. The disadvantages are difficulty in reproducibility of results especially at higher temperatures ($> 200^{\circ}\text{C}$) as the HFMs are very sensitive to changes in the surrounding temperature [26]. Design specifications and build requirements are detailed by ASTM C518, ASTM E1530, DIN 12667, and EN 12939 [15,19]. HFMs are available commercially through firms Netzsch [27], and Thermtest [28]. The HFM has shown difficulty in measuring moist samples similar to the GHP technique. A minimum sample size of approximately 200 mm by 200 mm by 51 mm is required for measurements [27]. Sample preparation would not be feasible for with a core samples of soil containing inhomogeneous materials such as rocks. An HFM would not be suitable for this project.

2.4.1.3 Guarded Hot Box (GHB)

The GHB is a measurement device that is typically used to measure thermal resistance of external elements and facades used in buildings. They are capable of measuring the thermal conductivity of insulation materials, plastics, glasses, ceramics, and inhomogeneous structures [19]. As with all steady-state measurement technique, the GHB must be designed to minimize the effects of convection. In addition, the measured sample size must be representative of the sample considered if an inhomogeneous material is to be tested. Figure 10 shows an example of a GHB used to measure a building element.

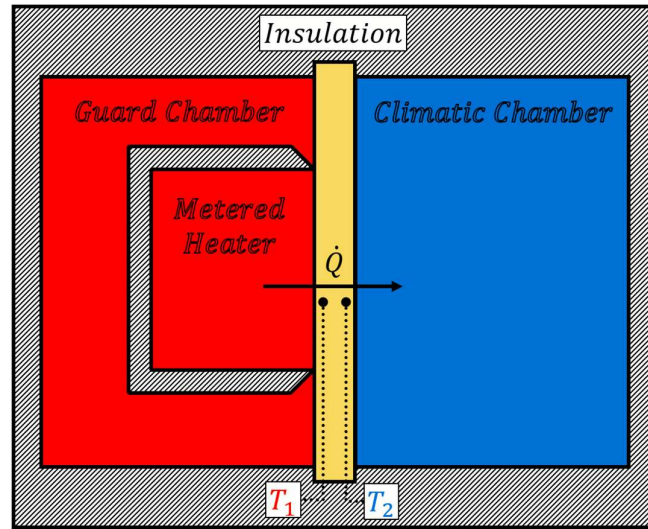


Figure 10: Guarded hot box measurement technique

The measurable thermal conductivity using this technique is approximately from 0.1 to 2.5 W/(m·K) for a 0.5 m thick sample [19,29]. The applied temperature range for this technique is from -48°C to 85°C with the ability to control the climate on both sides of the measured sample. One major disadvantage of this measurement technique is the construction size tends to be on the order of several meters cubed. The device dimensions are required to have enough air to circulate in order to maintain steady-state conditions on both sides of the sample material. The GHB measurement apparatus take a significant time to build based on the size and commercial products are very expensive [30]. The main purpose of this device is to measure building materials that are flat in geometry. Preparation of inhomogeneous soil samples for this type of technique are not feasible therefore, a GHB is not suitable for this project.

2.4.1.4 Other Steady-State Techniques

This section outlines the basic concept of researched techniques that were not feasible to consider for the measurement of porous, moist materials. The cylinder technique requires a long thin sample designed for axial heat flow in materials with high thermal conductivities and metals [17]. The four probe or direct heating technique uses a long thin cylindrical sample that must be homogenous over its entire length and electrically conductive as the material itself is used as the temperature measurement sensor [17,19]. Pipe and hot wire techniques use radial heat flow in a cylindrical sample by embedding a heating element in the sample axis. This steady-state technique induces convective heat transfer of any materials having interconnected pores and drying of the sample [17].

2.4.2 Transient Measurement Methods

The transient measurement methods use an intermittent pulse of energy either in time or amplitude to determine the thermophysical properties of a sample [16]. The development of this method was to avoid the problematic nature of steady-state methods regarding heat losses, contact resistance of temperature sensors, convective effects from prolonged sample heating, and long measurement times. Much less time is needed for transient measurements and the requirement of needing temperature measurements at different positions is replaced with one measurement location [17]. Test times are on the order of a few seconds to a few minutes excluding the relaxation period to allow the sensor and sample material to return to the initial state between measurements. The transient measurement method is suitable for sample materials containing a high moisture content because of the shorter heating times and direct response measurement.

2.4.2.1 Transient Hotwire/Hot Strip (THW/THS)

The THW technique determines the thermal conductivity of a sample material by measuring the temperature rise at a precisely known distance from a linear heat source. This is achieved by embedding a wire in the sample material for heating along with a thermocouple at distance from the wire to measure temperature profile over time. The thermocouple is placed at a radial distance from the center of the heated wire. This allows for the assumption of a one-dimensional radial heat flow for an isotropic, homogeneous sample material treating the heating wire as an infinite line source and an infinitesimally small radius [15]. This assumption leads to equation (8),

$$\lambda = \frac{q}{4\pi(T_2 - T_1)} \ln \left(\frac{t_2}{t_1} \right) \quad (8)$$

where λ is the thermal conductivity, q is the heating power per length of heating wire (W/m), and T_i the temperature of the wire at different points in time. The ability of modern electronics allows for 100 measurement data points in 1 ms to 1 s in liquids and gases, and 10 s for solids. Shorter measurement times in fluids is necessary to avoid convective heat transfer of the sample material. Uncertainties of $\pm 1\%$ for gases, liquids, and solids are achievable [31]. Figure 11 below shows the concept of measuring thermal conductivity using the THW technique.

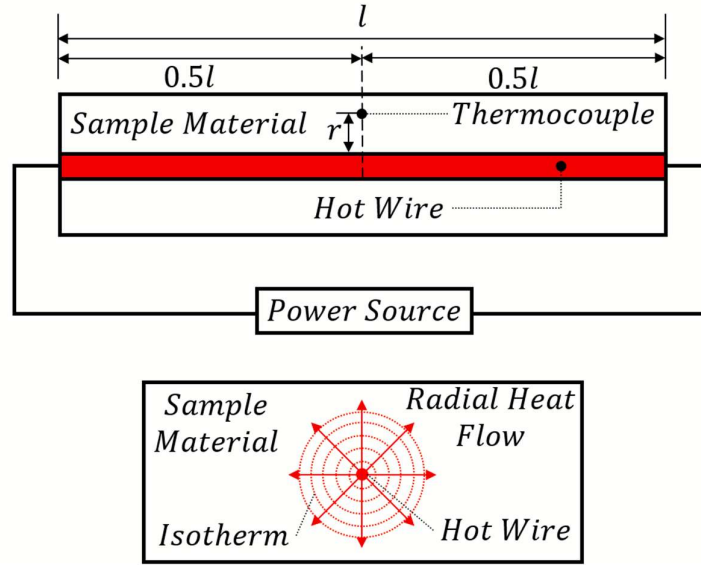


Figure 11: Transient hot wire measurement technique

The thermal hot strip (THS) technique is a modification that works in principle the same as the THW with the exception the use of a strip of thin metal and a mathematical evaluation procedure to account for the radially asymmetric temperature profile [17]. The THW and THS techniques are suited best for measuring solids including earth minerals, building materials, plastics, powders, fluids and gases as long as sufficient contact between sample material and sensor is maintained [19]. The THW and THS techniques require a low thermal contact resistance between the heating element and measured sample [17]. The approximations of THS and THW techniques limit them to the measurement of thermal conductivity only and thermal diffusivity must be calculated using equation (5) previously mentioned in section 2.2.2. The THW was further developed to eliminate the use of a thermocouple by using the heating wire instead of a second wire to measure temperature response via electrical resistance. The advantage of this modification called the parallel wire technique is the ability to measure anisotropic sample materials and an expanded measurement range of thermal conductivity [17]. Overall, the thermal conductivity values which can be measured by this technique range from $0.001 \text{ W}/(\text{m}\cdot\text{K})$ to $20 \text{ W}/(\text{m}\cdot\text{K})$ over a temperature range of -150°C to 200°C . The latest iteration in development of the THW technique is the transient hot bridge (THB). The THB uses a balanced Wheatstone bridge to produce measured variances in voltages across each circuit. This allows for the measurement of thermal conductivity as well as the thermal diffusivity and the ability to derive the volumetric heat capacity as the heating wires are also used as temperature sensors [32,33]. The measurement range of this type of technique is $0.02 \text{ W}/(\text{m}\cdot\text{K})$ to $30 \text{ W}/(\text{m}\cdot\text{K})$ over a temperature range of -100°C to 200°C with an accuracy of less than 3% and uncertainty of less than $\pm 3\%$.

[32]. All of the THW and THS measurement techniques require a much smaller sample size (> 1 mm thick by 30 mm wide by 80 mm long) relative to steady-state methods (> 0.5 mm thick by 300 mm wide by 300 mm long) [16]. The THW design specifications are outlined in ASTM D7984. Commercially available measurement devices exist through Linseis [34], TeKa [35], and Thermtest [36]. There exists the potential to use the thermal THB measurement technique with correctly prepared samples. If planar surfaces could be realized, a THB sensor may be a feasible method of measuring thermal conductivity and thermal diffusivity.

2.4.2.2 Single and Dual Probe Heat Pulse (SPHP & DPHP)

The SPHP and DPHP are fundamentally extensions of the THW techniques. Both SPHP and DPHP can be used on insulation, plastics, soils, ice cores, refractories, fibers, liquids, and powdered sample materials [37]. The SPHP is constructed in the shape of a needle which has a heating wire and a thermocouple in the center of the heated strip. The advantage of the SPHP design allows it to be inserted into loose sample material or into a drilled hole. As long as the minimum dimensions of the sample are present and contact resistance with the probe not too large, the SPHP is able to produce measurements of thermal conductivity with an accuracy of $\pm 2\%$. The SPHP technique is able to measure thermal conductivity over a range of $0.1 \text{ W}/(\text{m}\cdot\text{K})$ to $10 \text{ W}/(\text{m}\cdot\text{K})$ for temperatures of -25°C to 125°C [38]. Furthermore, it is the only transient method which has obtained an ASTM standard for thermal measurements of soil (ASTM D5334) [39]. Other research groups have used this method to study sediments on the ocean floor [40], soils and soft rocks in geothermal applications [41], and thermophysical characteristics of porous materials [10]. The SPHP does have difficulties regarding accuracy in measuring moist samples containing an uneven distribution of water within the sample material and if the contact resistance between probe and sample material is too high [37]. The DPHP is related to the SPHP but with the addition of a second probe. The second probe acts as a secondary sensing element thus the measurement of thermal diffusivity in addition to thermal conductivity is also possible along with the calculation of volumetric heat capacity from a single heat-pulse measurement [42]. An added advantage of the DPHP is the ability to measure the water content of a soil sample. The DPHP sends a heat pulse which measured by the second probe. It has been discovered that volumetric water content of a soil can be accurately estimated from the volumetric heat capacity [43]. It was found that for two different soil samples that the gravimetric method of determining water content agreed within 0.03 to $0.04 \text{ m}^3/\text{m}^3$ over a water content range of 0.10 to $0.45 \text{ m}^3/\text{m}^3$ [44]. The DPHP technique is able to measure thermal conductivities from $0.02 \text{ W}/(\text{m}\cdot\text{K})$ to $2 \text{ W}/(\text{m}\cdot\text{K})$ and thermal diffusivity from $0.1 \text{ mm}^2/\text{s}$ to $1.0 \text{ mm}^2/\text{s}$ in a

temperature range from -50°C to 150°C . Accuracy for both measurements is approximately 10%. Measurement times for the DPHP are on the order of 1 minute and conform to ASTM 5334 and IEEE 442 standards [45]. One major disadvantage of the DPHP is the distance between probes must remain undisturbed. It is imperative that the two probe remain parallel for accurate measurements using this technique [46]. Commercial measurement equipment is available through Thermtest [47], Huskeflux [48], TeKa [35], Metergroup [45], and C-Therm [49]. SPHP and DPHP are the most widely used techniques for measuring thermophysical properties of soil and rock formations. These techniques are well suited for the measurement constraints of this project and the SPHP is further investigated.

2.4.2.3 Hot Disk Transient Plane Source (TPS)

The TPS or hot disk technique is based on the hot strip technique to measure both thermal conductivity and thermal diffusivity from a single measurement. The sensor is encapsulated in a protective coating and placed between two planar surfaces of the sample material. This technique is able to measure thermal properties of liquids, solids, and powders [50]. Measurements are made by heating the sensor with an electrical current while monitoring the resistance for the behavior of temperature over time [51]. Figure 12 shows examples of TPS sensors in three sizes and insulation types.

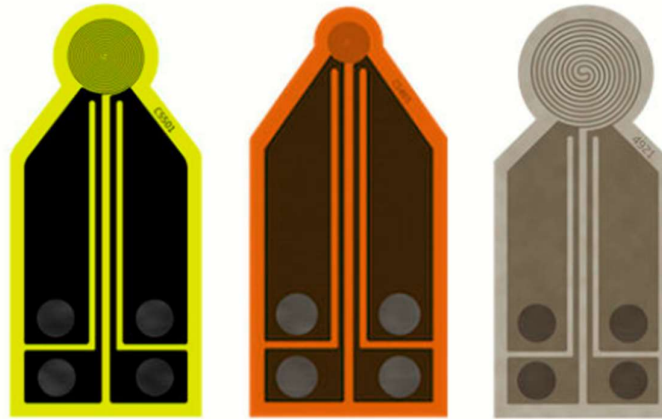


Figure 12: Transient plane technique measurement sensors [52]

The TPS is able to measure a range of materials that are electrically and thermally conductive to insulating materials. The thermal conductivity can be measured over a range from $0.005 \text{ W}/(\text{m}\cdot\text{K})$ to $500 \text{ W}/(\text{m}\cdot\text{K})$ in a temperature range from -243°C to 930°C . ASTM D7984 and ISO 22007-2 give greater detail to the test equipment and TPS measurement technique [53]. Repeated TPS analyses have produced approximate accuracies of 2% for thermal conductivity, 4% for thermal diffusivity, and 6% for volumetric heat capacity [51]. One major advantage of

this system is the elimination of contact resistance. This is advantage is inherently present from the required sample material preparation of having smooth surfaces in contact with the sensor under pressure. Both faces of the sample material must be planar making it quite difficult for loosely packed, unconsolidated or granulated materials to be measured [15]. Commercially available equipment is available through Hot Disk [54], C-Therm [49], and Thermtest [55]. The TPS technique would have difficulty in measuring most soil samples as they exist in nature. A well-planned sample preparation method would be needed to refine samples to an acceptable geometry and surface quality while maintaining a representative sample for measurement using this technique.

2.4.2.4 Laser Flash

The laser flash measurement method works differently from all previous techniques to determine thermophysical properties. It does not need physical contact with the sample material to take measurements of thermal diffusivity with high accuracy. Furthermore, it is non-destructive to the sample material as well [15]. This technique has the ability to measure polymers, glasses, ceramics, metals, liquids and powders without major restrictions on the achievable measurement uncertainty. It is the most often used technique to determine thermophysical properties of solids [17]. The laser flash technique is based on the concept of heating one side of a sample material with a pulse of light in the form of a laser and measuring the temperature signature on the opposite side with an infrared sensor [16]. Figure 13 shows the lay-out of a laser flash measurement device.

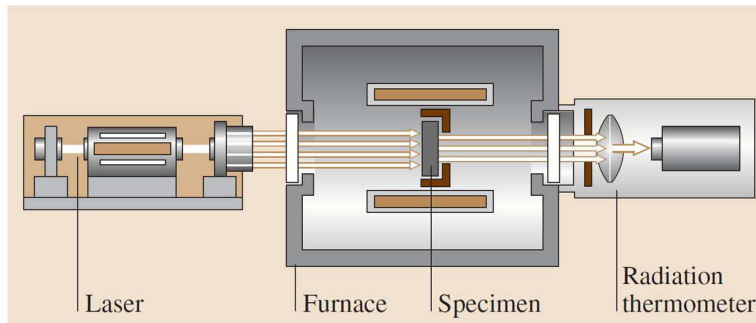


Figure 13: Laser flash measurement device [17]

It is assumed the laser pulse to be instantaneous and the sample held under adiabatic conditions. Using these assumptions allows for equation (9),

$$a = 0.1388 \frac{d^2}{t_{1/2}} \quad (9)$$

where a is the thermal diffusivity, d is the thickness of the specimen, and $t_{1/2}$ is half the time it takes for the full temperature rise of the sample material after being heated by the laser. Using the thermal diffusivity from this relationship along with the specific heat capacity and density, it is possible to calculate the thermal conductivity with equation (10) [16,17].

$$\lambda = a \cdot \rho \cdot c_p \quad (10)$$

One major disadvantage of the laser flash technique is sample preparation. Both sides of the sample material must be flat and parallel and approximately 1-4 mm thick. This rules out discontinuous materials with pores or voids such as thermal insulation or soils. Another disadvantage is difficulty measuring solid samples which are inhomogeneous. Grains that exhibit different thermophysical properties than the surrounding bulk sample material must be less than 5% of the thickness of the overall sample. For feasible sample materials the laser flash has distinct advantages. It is the only technique that does not require a direct temperature measurement. In addition, it is fast, and accurate for a temperature range of -100°C to 3000°C with an uncertainty of 3-5% [16,26]. Laser flash equipment is commercially available through Netzsch [56], TA Instruments [57], and Linseis [58]. The laser flash technique is not suitable for porous, moist materials. The required sample sizes are not feasible with soils containing inhomogeneities that can exceed the entire sample size itself.

2.4.2.5 Photothermal/Photoacoustic

Photothermal and photoacoustic are designed to measure thin films, liquids, and gases. These techniques are still in the development phase and use non-standardized methods of measurement. They require expert knowledge to operate and must have a laser rated building with a precisely controlled environment [26]. Commercial equipment is not available for these techniques as they are still being developed. Not enough information is available to determine whether this method is feasible to measure porous and moist materials.

2.4.3 Measurement Methods and Techniques Summary

Method/ Technique	Temperature range	Uncertainty/ λ , W/(m·K)	Materials	Pros	Cons
Steady-state Methods					
Guarded hot plate [26]	-190 – 525°C	2% $\lambda < 2$	Insulation, plastics, glasses	High accuracy	Long measurement times, large specimen, low conductivity materials
Heat flow meter [26]	-100 – 200°C	3-10% $0.007 < \lambda < 500$ [25]	Insulation materials, plastics, glasses, ceramics	Simple construction & operation	Measurement uncertainty, relative measurement
Guarded hot box [29]	-48 – 85°C	~6% $0.1 < \lambda < 2.5$	Building materials, inhomogeneous materials, composites	climate control	Small temperature range, long measurement times
Comparative [17,25]	20 – 1300°C	10-20% $0.2 < \lambda < 200$	Metals, ceramics, plastics	Simple construction & operation	Measurement uncertainty, relative measurement
Cylinder [26]	-270 – 620°C	2% n/a	Metals	Temperature range	Long measurement time
Four probe [19]	20 – 1600°C	n/a	Thermal conductors	Very high temperatures above most melting points	Homogenous materials, very thin samples
Pipe [17,25]	20 – 2500°C	3-20% $0.02 < \lambda < 2$	Solids, calcium silicates, mineral wool, ceramics	Temperature range	Sample prep, long measurement times
Transient Methods					
Hot wire, hot strip [25,26]	-50 – 500°C	1-10% $0.001 < \lambda < 20$	Solids, liquids, glasses, ceramics, powders, granules	Temperature range, measurement time, accuracy	Limited to low conductivity materials
Line Source [19,38]	-25 – 125°C	3% [37] $0.05 < \lambda < 20$	Soils, polymers, moist materials, powders	Portable or laboratory set up,	Unknown uncertainty
Plane Source [51]	-243 – 930°C	n/a $0.005 < \lambda < 500$	Solids, liquids, pastes, powders	Short measurement times, accuracy, measures thermal diffusivity	Unknown uncertainty
Laser flash [17,25]	-100 – 3000°C	3-5% $0.1 < \lambda < 1500$	Solids, liquids, powders, polymers, ceramics	Temperature range, small sample size, fast	expensive, cannot be used with insulation materials
Photothermal photoacoustic [17,25]	-50 – 500°C	Unknown	Solids, liquids, gases, thin films	thin films, liquids, and gases	Non-standard knowledge regarding accuracy

Table 1: A General overview and comparison of all methods researched for this project.

3 Testing and Validation Procedures

Testing and evaluation were necessary to assess measurement equipment on-loan and in-house. The tests serve as a starting point for the evaluation of specific methods and commercial devices suitable for measuring porous and moist materials. The literature review aided in finding a starting point along with the help of the University of Neuchâtel in providing CCTES an introduction to a device based on the TLS measurement method. The devices considered represent the state-of-the-art solutions for measuring thermal conductivity in their respective techniques with the TLS measurement method focusing on earth minerals and soils. A specific description of each device is presented in the following sections along with testing procedures and reference tests made during this project.

3.1 Instruments Tested

The instruments evaluated for the measurement of porous and moist materials were both transient methods. One of the devices tested was the Linseis THB-100 currently owned by CCTES which uses the transient hot-bridge (THB) technique. The Linseis THB is a modification of the hot-wire hot-strip techniques using 2 pairs of balanced circuits of different lengths at different distances to measure the effects of heat directed to the material over distance and time (see section 4.2.1). It has the capability to simultaneously measure the thermal conductivity and thermal diffusivity of a sample material in a short measurement period (< 2 s for very low conductive materials). This type of sensor uses a variety of sensor geometries made from a 5-10 μm thick foil strip sandwiched between two layers of Kapton for durability. Figure 14 shows the array of sensors offered by Linseis for use with the device.

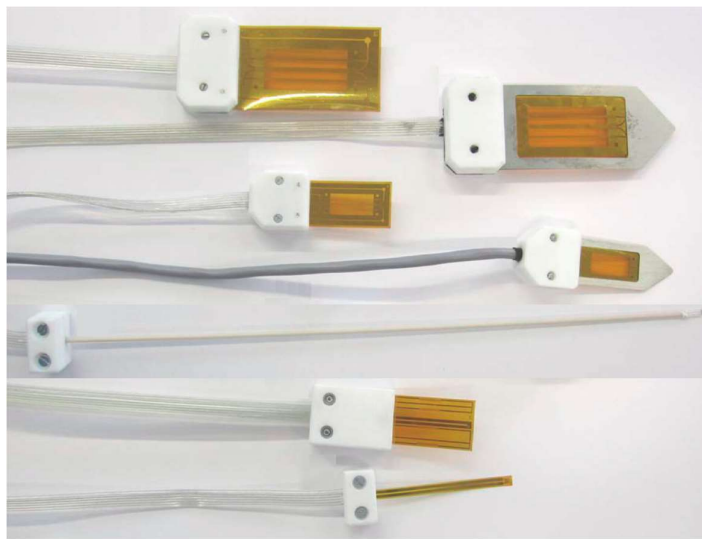


Figure 14: Transient hot bridge sensor types [32]

This type of device was evaluated for its measurable thermal conductivity range as well as the capability to measure thermal diffusivity as well. It is configured with an active heating and cooling system to test materials at temperatures from -100°C to 200°C for the sensors applicable to this project. The thermal conductivity measurement range is between $0.02 \text{ W}/(\text{m}\cdot\text{K})$ and $30 \text{ W}/(\text{m}\cdot\text{K})$ for the selected sensor [32]. This Linseis THB-100 is suitable for measurements of solids, liquids, pastes, powders, and unconsolidated sample materials. Single measurement times for this device can range from several milliseconds to a couple of minutes. After a measurement is taken, it is necessary to allow enough time for the sample material to relax. This allows for the effects of measurement heating to dissipate and for the sample material to return to pre-measurement conditions. Linseis recommends at least 10 fold the amount of time allotted for measurement thus the round-trip measurement time ranges from $\sim 20 \text{ ms}$ to 30 min [32]. The user is able to select the heating current (mA), measurement time (s), number of measurements, and number of temperature drift measurements as a means to obtain a more accurate measurement.

A collaboration with the University of Neuchâtel Laboratory of Geothermics made it possible to evaluate a second measurement device, the TeKa, model TK04. The VLQ measurement sensor is able to measure thermal conductivity from $0.1 \text{ W}/(\text{m}\cdot\text{K})$ to $10 \text{ W}/(\text{m}\cdot\text{K})$ in a temperature range of -25°C to 125°C . The HLQ measurement sensor is able to measure thermal conductivity from $0.3 \text{ W}/(\text{m}\cdot\text{K})$ to $10 \text{ W}/(\text{m}\cdot\text{K})$ in a temperature range of -25°C to 70°C . It is currently on-loan and is available for testing in the HSLU campus lab. This device is another derivation of the hot-wire hot-strip technique called the transient line source. This technique employs the theory of an infinite line source of heat directed into a material using a thin metallic rod or strip. The heating strip and temperature sensor are virtually in the same position which only allows for the measurement of heat over time and not over distance. Therefore, the main drawback of this measurement device is the inability to directly measure a second thermophysical quantity and thus a determination of thermal diffusivity. Extra steps to determine density and specific heat capacity are necessary for complete thermophysical analysis (see section 4.2.2). German manufacturer, TeKa, offers two different sensors for measuring thermal conductivity with their TK04 measurement system. One sensor is a needle probe (full volume sensor, VLQ) that is inserted into the sample material. The second sensor option is a small cylinder with a heating strip (half volume sensor, HLQ) that is placed on the appropriately prepared surface of a sample material. Figure 15 are the two types of measurement sensors of the TeKa measurement system.

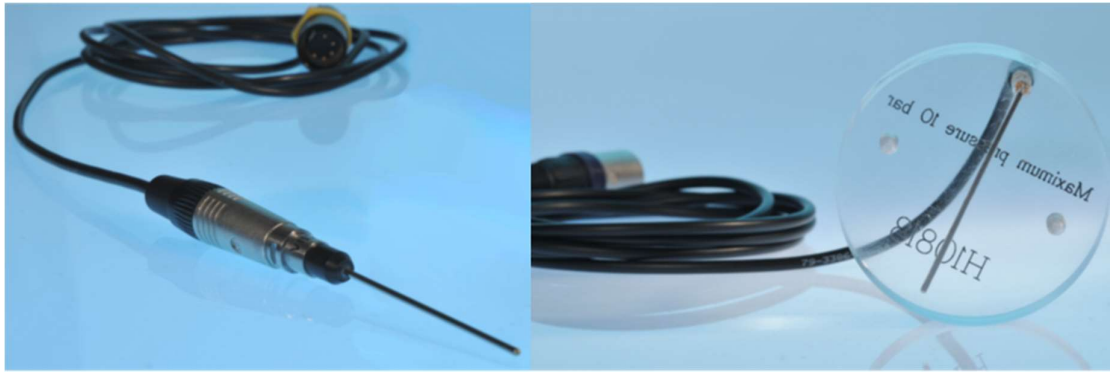


Figure 15: TeKa VLQ sensor left; TeKa HLQ sensor right [38]

This type of device was evaluated for its measurable thermal conductivity range and sample preparation flexibility with the availability of two different sensors. Single measurement times for this device are recommended to last 80 s with a minimum of 30 minutes for the sample to relax between measurements [38]. The user is able to select the heating power (W/m), measurement time (s), number of measurements, and number of temperature drift measurements as a means to obtain a more accurate measurement.

3.2 Testing Procedure

A testing procedure was developed for the TK04 and Linseis THB-100 to adhere to manufacturer specified measurement conditions. The first step is to select a measurement area which is out of direct sunlight, free of drafts especially from the out-of-doors, and other heat sources (e.g. radiators). The TK04 was positioned in a part of the thermodynamics lab that was near a frequently used door that connected to an area that often has a different temperature and created a strong draft when opened. Therefore, the first step was to assemble an insulated chamber for the TeKa probe and sample to be placed for measurement. 30 mm thick sheets made of swissporXPS 300 GE [59] were used to construct a guard box (0.565 m tall · 0.51 m long · 0.31 m wide) for the purposes of keeping temperature and humidity as stable as possible during measurement runs. Expanding insulation foam sealant was used to bind the sheets together creating a box for moderating the temperature changes of the thermodynamics laboratory. Figure 16 shows the final construction of the guard box.



Figure 16: Guard box with temperature and humidity monitors

The guard box was fitted with an internal temperature/humidity sensor along with an external temperature sensor for comparison using the Elpro ECOLOG TH2. Double lined insulation tape was used to seal the gap between the top and the main housing. The other device tested stationed in a different location.

The Linseis THB-100 is placed in the chemistry laboratory and is the site of testing. A simple polystyrene foam box with lid was used as a buffer against fluctuations in ambient conditions. This environment is much better controlled than the test site of the TK04 and does not need as much protection from changes in ambient conditions. A key to using this test environment is having the windows and door closed. An important step is to bring the sample to be tested to the lab for at least 24 hours prior to the measurements. The best practice for measurement preparation is to have the sensor in the material at least 1 hour before the start of the first run. This ensures that any small temperature drift of the sample material is in-step with the sensor.

Sample material preparation is one of the most important steps to obtaining an accurate measurement. Contact resistance between the probe and the sample material can be a major source of error in making thermal conductivity measurements. The first criterion to consider is the physical state of the sample material. Sample material preparation is dependent on whether a sample material is bound to itself (consolidated) or if the material is loose (unconsolidated) and takes the geometry of the vessel by which it is contained. Equally, different sample material preparations are required for TeKa VLQ and HLQ probes than the THB probe used in this project. Sample material preparation for the VLQ and HLQ probes from TeKa for consolidated material requires preparing a geometrically adaptable surface for thermal contact. VLQ probe requires a cylindrical hole drilled into the sample of a geometry that is large enough to insert

the probe firmly without using more than a thumb and two fingers to grip the handle. Generally, a hole slightly larger than the VLQ dimensions is necessary resulting in a small air gap. The presence of an air gap acts as a thermal resistor and is a major source of measurement error. This requires the use of a thermal interface material (TIM) such as a silicon-based paste. A TIM serves to create better thermal contact between the heat source and sample material by filling the air gap with a thermally conductive layer. The minimum sample material dimensions for the VLQ probe are 40 mm in diameter by 85 mm in height [38]. The other TK04 probe, the HLQ, needs a planar surface for contact with the sample material. The consolidated sample needs a smooth surface to contact the heated portion of the probe. Analogous to the VLQ, a non-planar surface will result in a larger air gap needing to be filled with a TIM. This situation results introduces an additional error by having an uneven thickness of TIM between the probe heat source and sample material. The TK04 measurement system comes with a clamp for applying pressure to the HLQ probe and sample material to reduce thermal resistance. It is recommended that 5 to 10 bars pressure be applied before measurement. The minimum sample material dimensions for the HLQ probe are 88 mm in diameter by 20 mm in height [38]. It is to be noted that there is an advantage to using the HLQ over the VLQ. The advantage of the HLQ probe allows for a visual if not measured inspection of the prepared surface and improvement of the surface if it is not sufficient for measuring. Using the VLQ probe does not allow for this type of inspection. It is possible for materials which are porous in nature to have an unknown sample material cavity that goes undetected and is in line with the inserted VLQ position. Figure 17 shows an example of silicone paste applied to both the VLQ and HLQ probes.



Figure 17: TeKa VLQ sensor with thermal paste (left) and HLQ sensor (right) on a planar surface.

Unconsolidated sample materials must be carefully prepared to control for physical characteristics affecting measurements. Very careful attention must be used in how sample material is

loaded into the container before measurement to ensure uniform distribution of density, porosity, moisture, and material types. TeKa recommends this type of sample material to be measured with the VLQ probe. This type of sample material must be contained in a container which allows for the minimum sample material size. The other device tested needed a different method to prepare consolidated samples while unconsolidated remained the same. The Linseis THB-100 comes with an array of hot bridge sensors able to measure consolidated solids, pastes, and powders. For solids the appropriate probe requires placement between two solid planar measurement surfaces of the same sample material clamped with 0.2 to 0.5 bar of pressure [32]. If a smooth surface cannot be produced, the use of a thin layer of copper paste as a TIM is recommended by the manufacturer to be applied. Pastes and powders require the use of a reinforced probe to prevent damaging the mechanically sensitive sensor allowing for direct placement into the unconsolidated material. Careful attention must be taken in order to not damage the sensor if the sample material contains abrasive particles or elements.

The physical characteristics of a sample material needs to be documented before testing begins. Both Linseis and TeKa systems record a temperature profile during measurement to reference to the results and no further verification is needed. All samples need to be to have moisture content and porosity determined before measurement. Moisture content and porosity affect how the effective thermal conductivity may be determined. Prolonged measurement times may initiate convective heat transfer effects having a significant effect on results (see section 2.3.3). Use of the TeKa measurement system the specific heat capacity and density must also be determined in order to obtain thermal diffusivity.

3.2.1 References Tests

Tests were conducted using the TeKa TK04 measurement system on two types of sample materials with known thermal conductivity. A glass ceramic block with known thermal conductivity provided by TeKa [60] and a well analyzed quartz sand provided by Dr. Arnold were tested [61]. The ceramic block has a perfectly pre-drilled hole and planar side with sufficient dimensions for VLQ and HLQ measurement verification. Measurement verification began with the verification of the VLQ probe and reference block. The reference block was verified first without the silicon thermal paste and second using the thermal paste. It was important to see how large an effect an air gap has with the VLQ results on an idealized sample material. Additional verification using the HLQ probe was made using thermal paste. Figure 18 shows the values and relative dimensions of the ceramic sample material during the HLQ verification.



Figure 18: TeKa ceramic sample material with HLQ probe in pressure clamp

A second material was tested for verification of a material with known physical characteristic and thermophysical properties. The Geotechnical Department at HSLU and Dr. André Arnold provided a nearly pure quartz sand from Western Australia near Perth. The sand has had an extensive material and characteristic analysis presenting a unique opportunity to verify the Linseis THB-100 and TK04 VLQ with an unconsolidated material. The Perth Quartz Sand (PQS) was analyzed during a Ph.D. study which provided initial information on the ranges of porosity, density, grain size distribution, and relative shape of grains [61]. Additional suggestions by Dr. Arnold were given to create samples of sand that had varied with porosity and density. A cylinder with a capped end drilled out with 2 mm holes spaced 2 mm square was used to fill a vessel to a known volume. Figure 19 shows the process of filling a vessel with PQS.

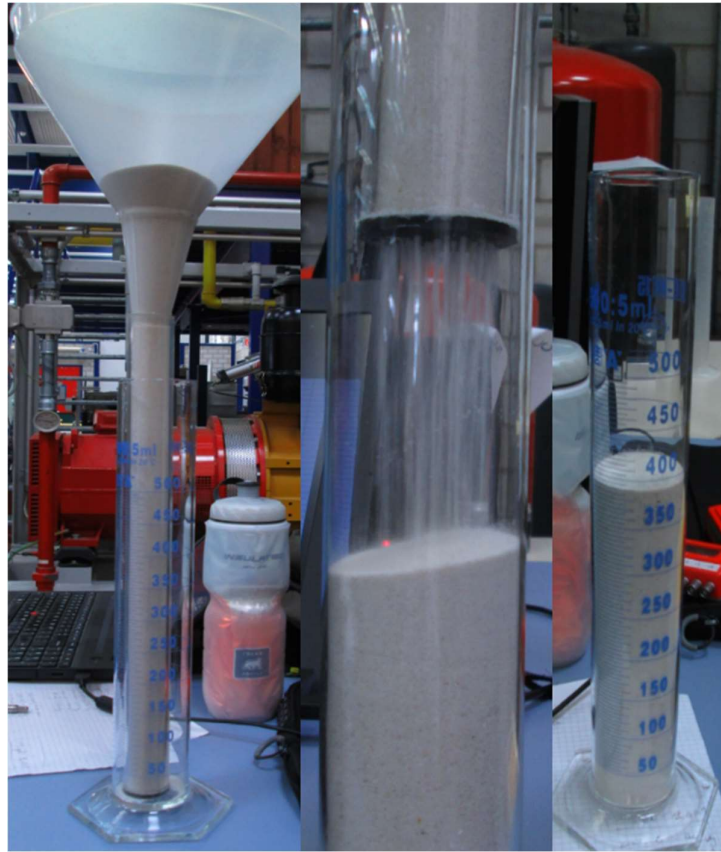


Figure 19: Pre-fill (left), filling (center), filled (right) graduated cylinder with PQS

This method of filling a known volume allowed for the control for the density and porosity of the PQS sample. It also allowed for a level pouring of material creating a uniform density profile of sand. A similar pouring apparatus was also used to fill 5 PQS samples of different porosities for testing with the Linseis THB-100. Finally, the comparison of a PQS sample was made to control for any effects of humidity. A PQS sample was placed in an oven at 100°C overnight and allowed to cool wrapped in cellophane for 24 hours to ambient conditions.

3.2.2 Soil Sample Tests (TeKa)

Two soil samples available for measurement were provided by Schenk AG from the region of Turgau. The two soil samples, B4.3b and B3.1a, were extracted from two separate bore holes representing layers of earth between 9.2 – 9.4 m and 6.7 – 7.0 m respectively [62]. Each bore hole soil sample had a diameter of 150 mm, length of ~110 mm for sample B4.3b, and length of ~160 mm. Both soil samples are geologically described as compacted sub-glacial moraine, very dense ($\sim 2000 \text{ kg/m}^3$), and mechanically unstable when agitated. Furthermore, randomly spaced rocks in various sizes were dispersed throughout the sample material volume. The samples were tested for thermal conductivity using the TeKa TK04 measurement device. Time was

insufficient for a measurement using the Linseis THB-100. The first attempt was made by drilling a hole into sample B4.3b for measurement with the VLQ probe. The necessity to drill a precise hole while keeping the sample intact prompted a design to secure the sample while dampening vibration. Figure 20 shows an in-house made clamp using steel pipe, a wood block, c-clamps, and thermal insulation.



Figure 20: Soil sample clamp and base

The machine shop CNC drilling machine was used to drill a bore hole for insertion of the VLQ probe. The second attempt on soil sample B3.1a was prepared for measurement using the HLQ probe. The mechanical instability of the soil samples required careful planning of the preparation process. Careful steps were taken to reduce one surface of the soil sample sufficient using a variety of methods including wire mesh, a piece of scrap metal, a hand saw, and Dremel tool.

4 Results and Discussion

This section covers the results for the testing and validation procedures outlined in the last section. The following information will give a detailed description of the major challenges of sample preparation and measurement results for the reference materials and soil samples. Charts and tables supporting data results will be located in the appendix and the full files submitted electronically. The following section gives the details of results of the tests performed and a short discussion on test location, sample preparation, and results for each measurement device.

4.1 Guard Box

A location free from drafts, sharp temperature changes, sunlight and other disturbances affecting measurement results is required for use with the TeKa TK04 measurement device. The TeKa TK04 was placed near a doorway and a guard box was used to moderate changes to ambient conditions. The guard box was verified over a period of 12 hours to show dampened internal temperature fluctuations relative to external temperature. The VLQ probe was placed in the guard box along with the ceramic reference block and a 1.5-liter bottle of water as a thermal mass resembling a sample material. Figure 28 in the appendix shows the comparison of internal and external temperature along with the internal relative humidity recorded every 2 seconds over the evening of April 9th and the morning of April 10th, 2019. The results were as expected with internal temperature lagging in time behind external temperature. At approximately 07:00 on April 10th, a sharp external temperature decrease of $\sim 0.8^{\circ}\text{C}$ in 90 s was measured. The internal temperature performed well by responding with a small drop of 0.06°C as shown in Figure 29 in the appendix. Furthermore, the rate of temperature change was charted using a 2-second and 2-minute time step for evaluation. Figure 29 in the appendix shows both 2-second and 2-minute time step plots for same data set. In all cases the 2-minute time change did not exceed the 2-second time change with a max temperature change of $\pm 0.07^{\circ}\text{C}$.

4.2 TeKa TK04

The consolidated reference material (TeKa ceramic reference block) was tested using both the HLQ and VLQ measurement probes. No special preparation was needed before measurement with the exception of placing the reference block inside the guard box for at least 1 hour prior to starting measurements for temperature monitoring. This was to ensure that temperature fluctuations within the sample material and any components in thermal contact associated with measurements were in thermal equilibrium. Figure 30 the appendix shows the reference block temperature monitoring for the period of approximately 1 hour before measurement with the

VLQ probe. The VLQ was further tested with and without thermal paste to determine what type of effect it has on measurement results. The first test used the VLQ measurement probe inserted into the TeKa ceramic block without thermal paste and outside of the guard box for measurement. The default settings of 3 W/m heating power, 80 s measurement time, 30 min relaxation time, and a minimum LET of 4 were used for this measurement. Additionally, a second test was done with thermal paste and inside the guard box using the same default parameters. It was found that the thermal paste and use of the guard box improved accuracy and precision of the measurements with the reference block. Figure 31 in the appendix shows the results of each VLQ test side by side. This was further supported by the results revealing a fluctuating contact resistance for the without thermal paste and a constant contact resistance for the test with thermal paste. The HLQ test of the TeKa reference block used the recommended settings of 1.61 W/m heating power, 80 s measurement time, 60 min relaxation time, and a minimum LET of 4. The HLQ probe requires pressure to keep it in better contact with the measurement surface. A mechanical clamp provided by the manufacturer was used to fix the sensor to the surface as shown in Figure 21 and placed in the guard box for testing.



Figure 21: TeKa clamp with HLQ probe and reference block (left), placed in guard box (right)

Figure 32 in the appendix shows the results of four tests in a box and whisker plot performed with different pressures placed on the TeKa reference block. All tests contained 18 measurements taken where some points overlap and are not apparent in the plot. Measurement points are represented by a whisker or a circle. The middle line in the box represents the median value, the “X” the average value, the bottom box line the division between 1st and 2nd quartile, and the top of the box the division between 3rd and 4th quartile. Top and bottom whiskers represent the highest and lowest measured value of thermal conductivity if the value was contained within \pm

1.5·IQR (inter-quartile range). The IQR is range of values that contains the middle 50% of measurements and is equivalent to subtracting the value of the first quartile from the third quartile. Otherwise, the value is considered an outlier and plotted outside the whisker. The whisker in this case is the next measurement point contained within $\pm 1.5 \cdot \text{IQR}$. All test runs had a consistent contact resistance that was not considered a factor for determining the thermal conductivity. Table 2 shows contact resistance values for each measurement including thermal conductivity.

Table 2: HLQ measurement tests arranged by pressure setting and contact resistance value

meas. #	λ @ 1.5 kN W/(m·K)	CV [-]	λ @ 1.0 kN W/(m·K)	CV [-]	λ @ 0.2 kN W/(m·K)	CV [-]	λ @ 0.1 kN W/(m·K)	CV [-]
1	1.582	10.08	1.588	10.23	1.605	10.16	1.616	10.08
2	1.614	10.08	1.609	10.16	1.623	10.31	1.586	10.08
3	1.587	10	1.625	10	1.595	10.16	1.606	10
4	1.615	10.31	1.603	10	1.608	10	1.625	10
5	1.597	10.16	1.616	10.23	1.638	10.08	1.586	10
6	1.616	10.23	1.621	10.23	1.615	10.16	1.582	10
7	1.589	10.08	1.616	10.23	1.604	10.16	1.594	10
8	1.568	10	1.608	10.23	1.582	10	1.601	10
9	1.572	10.08	1.628	10.08	1.595	10	1.62	10
10	1.644	10	1.605	10	1.601	10	1.582	10
11	1.585	10	1.587	10.08	1.594	10	1.579	10.08
12	1.627	10	1.603	10.08	1.6	10	1.578	10
13	1.617	10	1.628	10.23	1.601	10	1.634	10
14	1.579	10	1.616	10.23	1.595	10	1.604	10
15	1.596	10.16	1.648	10	1.581	10.16	1.627	10.23
16	1.584	10	1.595	10	1.625	10	1.606	10
17	1.592	10	1.591	10	1.605	10.16	1.599	10
18	1.593	10.08	1.633	10.23	1.58	10.16	1.618	10

The second test using 1.0 kN pressure was the most successful having a median value, 1.613 W/(m·K), very close to the average value, 1.612 W/(m·K), indicating a balanced measurement set and having zero outliers. Both are quite close to the supplied reference block value of 1.614 W/(m·K). It is important to note the clamp does not stay fixed for periods of time longer than 12 hours. It was necessary to fix the clamp handle in order to maintain a constant pressure during testing.

The dry unconsolidated reference sample (PQS) was poured into a cylinder with a sieve at the bottom to regulate the density and thus porosity of the sample. From this point forward in the project time did not permit to test the effects of varying moisture or sample material temperature. Ambient conditions were only considered for this report. Only dry material was to produce a uniform distribution of a porous reference material for measurement. The results of oven heating a 3553.8 g sample of PQS overnight resulted in a loss of 2 g. PQS samples measured were therefore considered dry. Results for density and porosity by pouring sand from a very low height above the sand level in a glass graduated cylinder are shown in Table 3.

Table 3: Table results of tube pouring PQS into a glass graduated cylinder

Mass cylinder	Total mass	PQS volume	Density	Porosity
kg	kg	m ³	kg/m ³	-
0.4042	1.1507	4.95E-04	1.508E+03	0.4309
0.4042	1.1303	4.85E-04	1.497E+03	0.4351
0.4042	1.0625	4.43E-04	1.488E+03	0.4386
0.4042	0.9864	3.90E-04	1.493E+03	0.4367
0.4042	0.7671	2.45E-04	1.481E+03	0.4410
0.4042	0.8862	3.25E-04	1.483E+03	0.4403
0.4042	0.9372	3.60E-04	1.481E+03	0.4413
0.4042	0.7782	2.53E-04	1.481E+03	0.4411
0.4042	1.1083	4.75E-04	1.482E+03	0.4406
0.4042	1.1069	4.73E-04	1.487E+03	0.4388
	average (density porosity)		1488.13	0.4384
	standard deviation (density porosity)		9.44	0.0036

The heights for pouring sand were not recorded as the process was done completely by hand using only visual estimation for maintaining consistency. The results were strikingly accurate however, the use of a long thin geometry limited the ability to keep the sand from piling up in the middle. This caused some sand grains to fall farther than others and thus creating an undesired non-uniform density distribution. This feature is not reflected in the calculations above as they are an average for the bulk volume of the material. The decision was made to use a wider vessel in the form of a glass beaker with a larger volume avoiding the feature of “piling” or “heaping” in the middle of the sample material during preparation.

The consolidated material tested was the main focus of this project. Two soil samples from different bore holes were prepared for measurement with the TeKa TK04. The TK04 has drill bits included for creating a pilot hole suitable to insert the VLQ probe into the sample material.

The results of using the CNC machine into the first sample (B4.3b [62]) were not successful. In order to maintain the composition of the sample, no cooling fluid could be used nor water. The drill bit failed on a material level and the CNC was only able to make a ~2 mm deep hole at 10 μm increments. Figure 22: Drilling attempt on soil sample B4.3b below is a close up of the drilling attempt using the CNC machine.

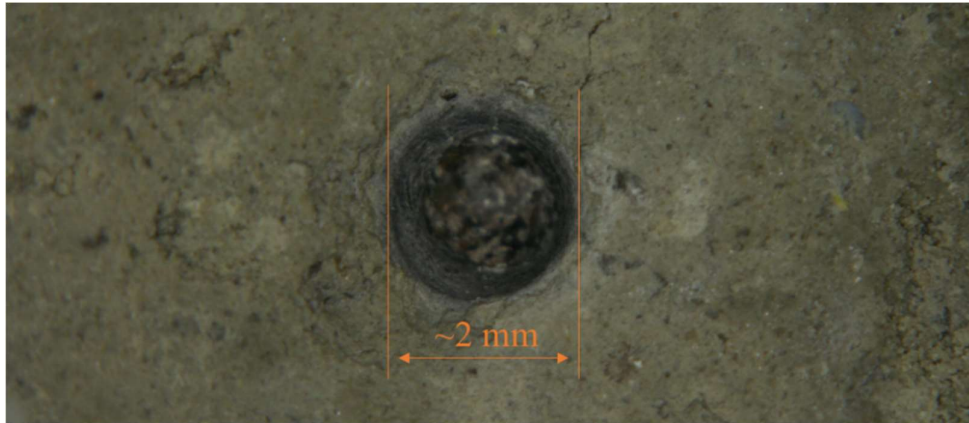


Figure 22: Drilling attempt on soil sample B4.3b

At this point it was decided that drilling a hole into this material is extremely challenging given the constraints for preparation, time allotted, and equipment. The second option to measure soil samples with the HLQ probe was explored. Attempts were made using rasps, files, hacksaws, and wire screens to finish a surface on soil sample B4.3b. The results were promising until a mechanical breakdown of the material occurred. This soil sample was a rock nearly the entire diameter (150 mm) and most of the depth (~80 mm) covered in chunks of soil at one end and on lateral surfaces. The result was a sample which crumbled away to reveal a big rock. Figure 23 shows the result after making the second attempt to finish a surface for use with the HLQ probe.



Figure 23: Soil sample B4.3b after second preparation attempt

Dr. Arnold expressed this is not uncommon occurrence sampling soil consisting of sub-glacial moraine. A second sample was obtained from the HSLU Geotechnik Department from a different bore hole location for preparation. Soil sample B3.1a was prepared by the same method which terminated progress on soil sample B4.3b. Figure 24 shows the finished product of sawing, rasping, and finishing soil sample B3.1a with a Dremel rotary grinding tool.

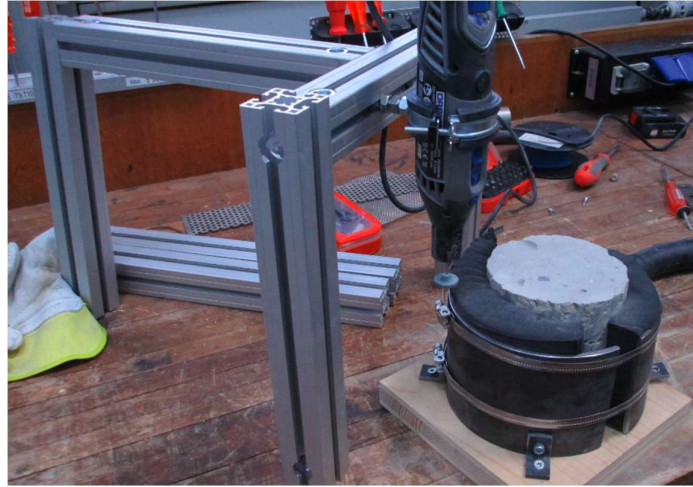


Figure 24: Finished surface of soil sample B3.1a

Measurement results for the TK04 with the VLQ probe are summarized in Figure 33 the appendix showing the results for varying porosities, heating power, and measurement times. All tests were conducted with a temperature increase of $\sim 2^{\circ}\text{C}$ during measurement. Table 4 below shows how individual tests were differentiated and the measured result.

Table 4: Settings for VLQ probe tests on PQS

Glass Beaker Tests with VLQ probe

Test	Heating Power [W/m]	Measurement Time [sec]	Average Contact Value [-]	λ [W/(m·K)]	Porosity [-]
4	0.5	80	16.87	$0.2854 \pm 13.97\%$	0.3931
5	0.7	80	17.14	$0.2830 \pm 2.50\%$	0.3931
6	0.5	80	17.93	$0.3630 \pm 23.51\%$	0.3533
7	0.35	80	16.73	$0.3537 \pm 26.95\%$	0.3533
8	0.5	100	18.12	$0.3639 \pm 3.73\%$	0.3533
9	0.5	120	17.70	$0.3610 \pm 13.81\%$	0.3533
10	0.5	100	18.27	$0.2951 \pm 3.34\%$	0.3969
11	0.5	100	17.99	$0.3656 \pm 7.19\%$	0.3459
13	0.5	100	17.64	$0.2757 \pm 3.60\%$	0.4058

It was found the heating power between 0.35 W/m and 0.7 W/m was sufficient to measure PQS. However, it was not clear from the tests why there was such a large variability in the range of results. The literature review indicated contact resistance is a source of measurement error as well as possible convective heat transfer in the air occupying the porous voids between grains of sand. These results were sent for review to TeKa with the response confirming contact resistance values were too high and inconsistent to support reliable measurement results. It was also expressed that one should not be attempting to measure sand with the TK04 which brought measurements with the VLQ to a stop.

The results for the TK04 using the HLQ probe with soil sample B3.1a were split into two groups. The first group of results are for the soil sample with a manually finished surface for contact with the measurement probe. Various heating powers were tested to generate measurement results along with the recommended settings of 80 s measurement time and minimum LET of 4. The temperature increase during measurement was $\sim 2^{\circ}\text{C}$. The first attempt results from this method as it were nearly unusable. The HLQ probe is very sensitive to the finished quality of the planar surface on the soil sample. This resulted in an air gap between the probe and soil sample discovered after measurement. Contact resistance values were found to be high (~ 17 to 19) and there were numerous instances where measurements were not at all possible. Table 5 shows an abbreviated version of results from this first measurement attempt using different heating powers.

Table 5: Sample of results from hand finished soil sample using HLQ probe

First attempt to measure soil sample B3.1a, hand finished surface, porosity 0.3366

Test/ meas. #	Heating Power [W/m]	λ W/(m·K)	Contact Value [-]	Clamp pressure [kN]	# of solu- tions
1/1	1.61	1.390	17.42	1.5	33
1/2	1.61	-	17.50	1.5	-
1/3	1.61	-	17.58	1.5	-
2/1	1.1	1.235	17.66	1.5	2
2/2	1.1	-	17.66	1.5	-
2/3	1.1	1.236	17.66	1.5	19
3/1	0.8	1.320	17.81	1.5	32
3/2	0.8	1.285	17.81	1.5	51
3/3	0.8	1.279	17.89	1.5	52
4/1	0.6	1.088	18.28	1.5	35
4/2	0.6	1.154	18.44	1.5	88
4/3	0.6	1.160	18.52	1.5	11

Dashes in the thermal conductivity column represent a measurement that was not possible due to poor contact with the sample material producing LET values below the minimum threshold ($LET = 4$).

The second group of results were generated after the refinishing of the soil surface for soil sample B3.1a by changing the heating power. A more planar surface was achieved during the sample preparation using a support holding a leveled grinding tool. Table 6 shows an abbreviated set of results for each heating power tested.

Table 6: Sample of results from machine finished soil sample using HLQ probe

Second attempt to measure soil sample B3.1a, machine finished surface, porosity 0.3366

Test/ meas. #	Heating Power [W/m]	λ W/(m·K)	Contact Value [-]	Clamp pressure [kN]	# of solu- tions
1/1	1.8	1.792	10	1.5	121
1/2	1.8	1.770	10	1.5	27
1/3	1.8	1.780	10	1.5	261
2/1	2.2	1.720	10	1.5	45
2/2	2.2	1.730	10	1.5	19
2/3	2.2	1.688	10	1.5	27
3/1	1.4	1.668	10	1.5	50
3/2	1.4	1.807	10	1.5	188
3/3	1.4	1.709	10	1.5	95
4/1	1.5	1.704	10	1.5	3
4/2	1.5	1.730	10	1.5	159
4/3	1.5	1.738	10	1.5	167
5/1	1.6	1.739	10	1.5	105
5/2	1.6	1.729	10.63	1.5	167
5/3	1.6	1.653	10	1.5	107

The second attempt had near perfect contact values and an increased number of solutions for each measurement. It was inconclusive to deduce which measurement was the most accurate result as these measurements were made for a material with randomly dispersed inhomogeneous material parts in the form of small rocks. An inhomogeneous material field creates a non-uniform temperature profile in the material during measurement which is outside the approximation method used for this measurement technique. Figure 34 in the appendix shows the full results of measurements made with a refined sample surface.

4.3 Linseis THB-100

The Linseis THB-100 is located in the chemistry laboratory. Ambient temperatures were monitored with a handheld temperature sensor to confirm stable conditions before starting a measurement. It was found that data taken in the chemistry lab was suitable for measurement tests.

The Linseis THB-100 comes with pre-set calibrations defined by the manufacturer that automatically recommend the heating current and measurement time needed to measure a sample material. A list of choices for different thermal conductivities can be made from a drop-down menu before measurement begins to select a material of a similar thermal conductivity. For the PQS this was PMMA which has a listed thermal conductivity of $0.193 \text{ W/(m}\cdot\text{K)}$ [32]. From this point forward in the measurement procedure, it is up to the user to define a region of stability in terms of thermal conductivity versus heating current and measurement time for a sample material with unknown thermal conductivity. The goal is to find a region where reasonably small changes in the heating current ($\sim 5 \text{ mA}$) and measurement time ($\sim 10 \text{ s}$) do not change the measured thermal conductivity and thermal diffusivity. It was planned to test 5 different porosities of sand however, due to limited time, only one sample of PQS was tested for thermal conductivity and thermal diffusivity using the THB-100. The PQS sample had a porosity of 0.411 and was measured with the THB6N/MFR sensor with metal support. The metal support is necessary to keep the delicate sensor from bending and potentially breaking. Figure 25 shows the THB sensor used and how it was fixed before placement in the PQS sample.



Figure 25: THB sensor used to measure PQS sample (left), sensor fixed to cork donut for support (right)

Measurement settings were chosen using the Linseis recommended measurement time of 100 to 300 s and measurement current between 50 to 80 mA for the PQS sample. Each data point

shown represents the average of three measurements taken during tests. Below are the results of measuring thermal conductivity and thermal diffusivity for several measurement times (Figure 26) and measurement currents (Figure 27).

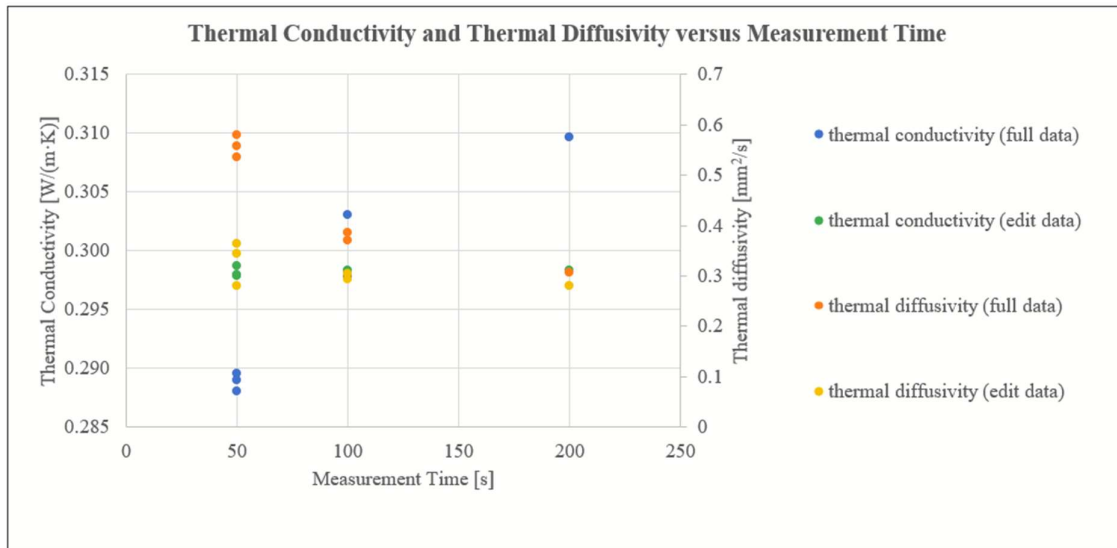


Figure 26: Thermal conductivity and thermal diffusivity for PQS and porosity 0.411

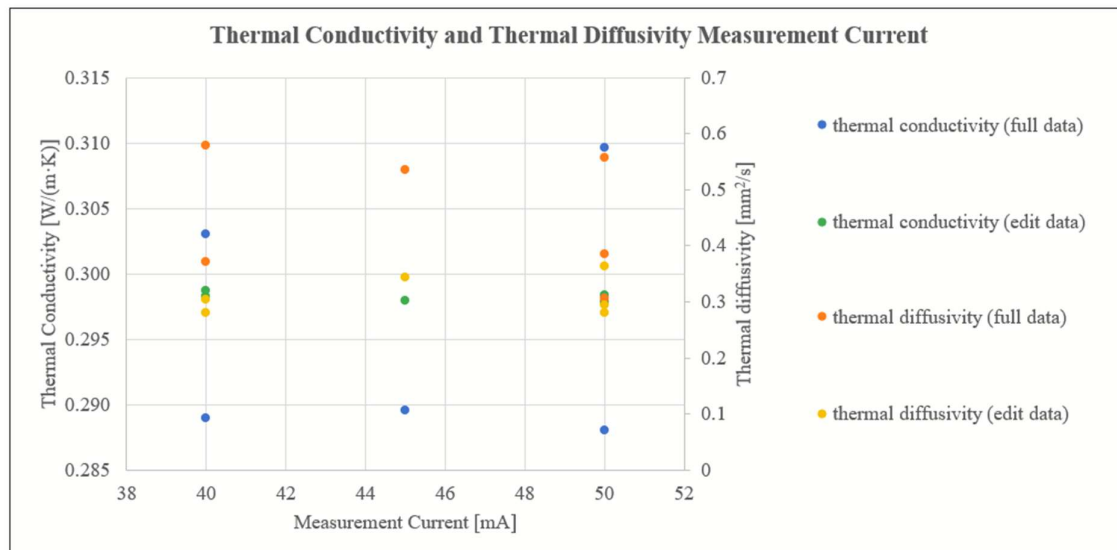


Figure 27: Thermal conductivity and thermal diffusivity for PQS and porosity 0.411

Each individual test did not exceed a 1.5°C temperature increase during measurement with the smaller differences associated with low measurement times and measurement currents. Results from the Linseis THB-100 use a subjective data management process after a measurement has been taken. The initial measurement period (usually < 2.5 s) contains voltage fluctuations from the power supply which create noise not associated with the measurement of the sample material. It is therefore essential to remove these data points to have a clear measurement signal.

However, this process is completely user driven and there is not a set procedure for how disturbed a data point must be to determine its removal. This step is completed graphically by the user and not very well defined overall.

4.4 PQS Sample Data and Model Comparisons

The TeKa TK04 and Linseis THB-100 were used to measure a material with well-studied thermophysical properties. The results of the TK04 and THB-100 for PQS were plotted against some empirical studies and models on porous quartz sands for comparison. Figure 35 in the appendix shows a graphical comparison of the results for both devices, studies, and models [8,11,63,64]. All external studies raise the importance of contact resistance and how this can affect the measurement result. TeKa explicitly expressed that measuring sand is not reliable with the TK04 measurement device. The communication did not elicit what the critical threshold for contact resistance is with the TK04. Measurements made with the TK04 were within range of other studies conducted using TLS measurement equipment and mathematical models. The one measurement point for the THB-100 makes it impossible to determine its suitability for an unconsolidated dry material such as sand across a range of porosities. The data points plotted in Figure 35 represent an initial test of using the system and are not useful for comparison. Manufacturers for both measurement devices claim their instruments can measure porous materials but do not specify what type of porosities are measurable nor a threshold for contact resistance. Measurement accuracy is defined by user experience and sample preparation to determine if a material can be measured by these instruments. Therefore, without an extensive study considering sample preparation and more time to work through all possible parameter setting applicable for measuring PQS, it is impossible to determine if the measurement devices tested are functioning as intended by the manufacturer.

4.5 Cost Analysis

Cost analysis is always a significant decision factor when considering scientific measurement equipment acquisition. Repeated attempts were made by e-mail to contact manufacturers supplying commercial equipment for price quotes. All manufacturers did not provide a quote, rather they requested the attendance of webinars or in-person viewing of equipment. At this time, we do not have an economic assessment for a measurement device to measure thermophysical properties.

5 Conclusion & Future steps

This section contains conclusions based on the literature review, lab testing, and communication with manufacturers. It provides a recommendation for measurement method and technique for measuring porous, moist materials with a focus on soils. Not all investigations were taken to completion and they are outlined further in the future steps subsection.

5.1 Conclusion and Recommendation

Precise measurement of thermophysical material properties are required for the design and performance of thermal energy storage media. Thermophysical performance is crucial to evaluating whether a material is suitable for an intended use. Most methods and techniques over the past 70 years have been developed to measure thermal conductivity. A minority of techniques able to measure thermal diffusivity in addition to thermal conductivity have been developed since 1990. Steady-state techniques are time consuming and require a relatively large sample size. Transient techniques are often favored for their shorter measurement times and simplified methods of simplifying contact resistance between sensor and sample material, and a smaller required sample size. Therefore, it is recommended to use a transient measurement technique for measuring soil and insulations. However, transient techniques require a specific, often exact, surface quality and/or sample material geometry to produce quality measurements.

The measurement experience using the TK04 and Linseis THB-100 during the testing phase demonstrated how important sample preparation is for a quality result. The measurement results taken are inconclusive and requires further investigation. Comparison of the measured values with empirical experiments and models indicate the progress made during this project is in the correct direction. However, there remain unknown uncertainty in the measurements. The contact resistance threshold for poor measurements is not well defined in literature nor by the manufacturer. The sand samples which were tested are a perfect example of this challenge. In addition, inhomogeneous materials present an unknown source of error from a non-uniform temperature field during measurement. The soil samples tested during this project had various sizes and quantity of rocks mixed into the bulk material. The number of options for measurement probes increases the chances of a match between sample material preparation and measurement probe. Therefore, the C-Therm Trident is recommended as it employs three separate measurement probes (transient line source, transient plane source, modified transient plane source) in one device. This device alongside the Linseis THB-100 would give CCTES four different options to measure a sample material.

5.2 Future Steps

The next steps to ensure certain and accurate thermal conductivity and/or thermal diffusivity measurements requires further investigation. A closer inspection of the data needed for modeling must be well defined as it has a great impact on how a sample can be prepared. The investigating team must ask the following questions:

- (i) What are the preparation limitations of the material to be measured?
- (ii) What are the measurement constraints of the project, model, simulation or study?
- (iii) Is it possible to manipulate the structure of the material while maintaining a representative sample for measurement?

The answers to these questions serve as a guide for the number options to prepare a sample with the geometry and surface quality required for an accurate and precise measurement. An example of this would be to grind all the elements of a soil sample to a fine powder. Then a preparation step of shaping, compressing, and humidifying the soil and rock powder into the proper density, geometry, and moisture content before measurement. A high quality, homogeneous sample material would give the best chances for accurate measurements.

6 References

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7 Appendix

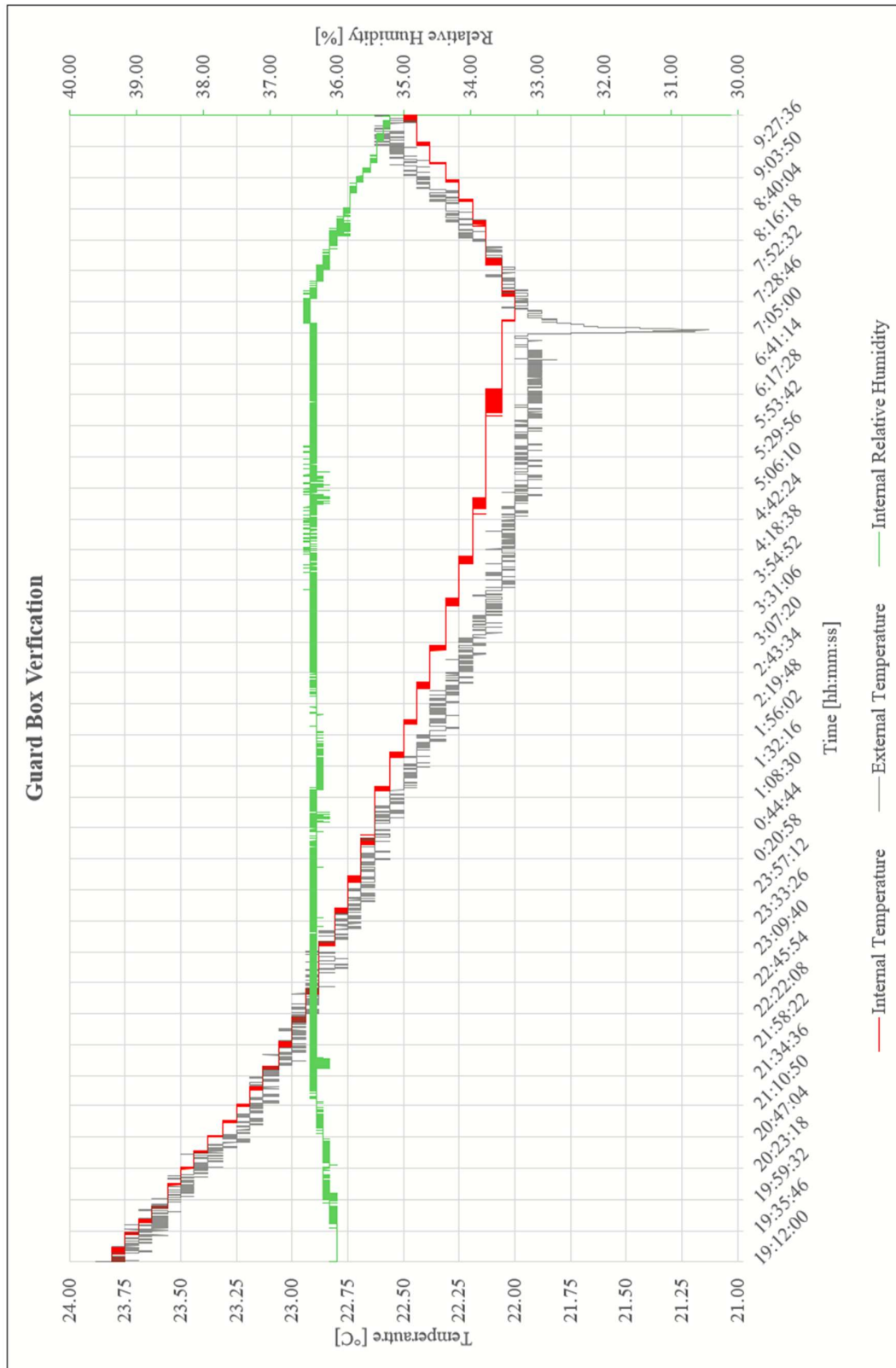


Figure 28: Plot of guard box internal temperature, external temperature, and internal relative humidity

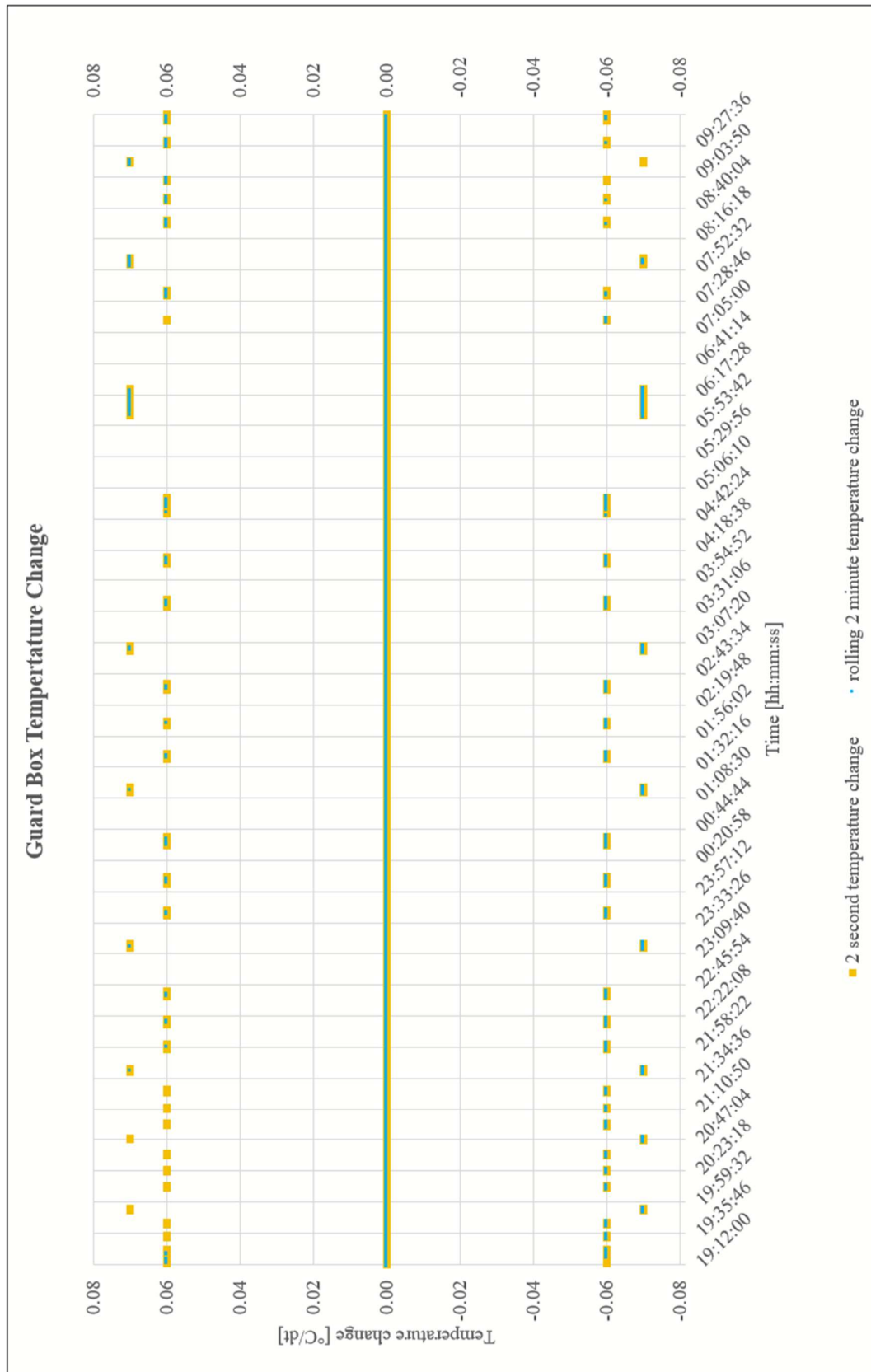


Figure 29: Plot of temperature change over 2 seconds and 2 minutes

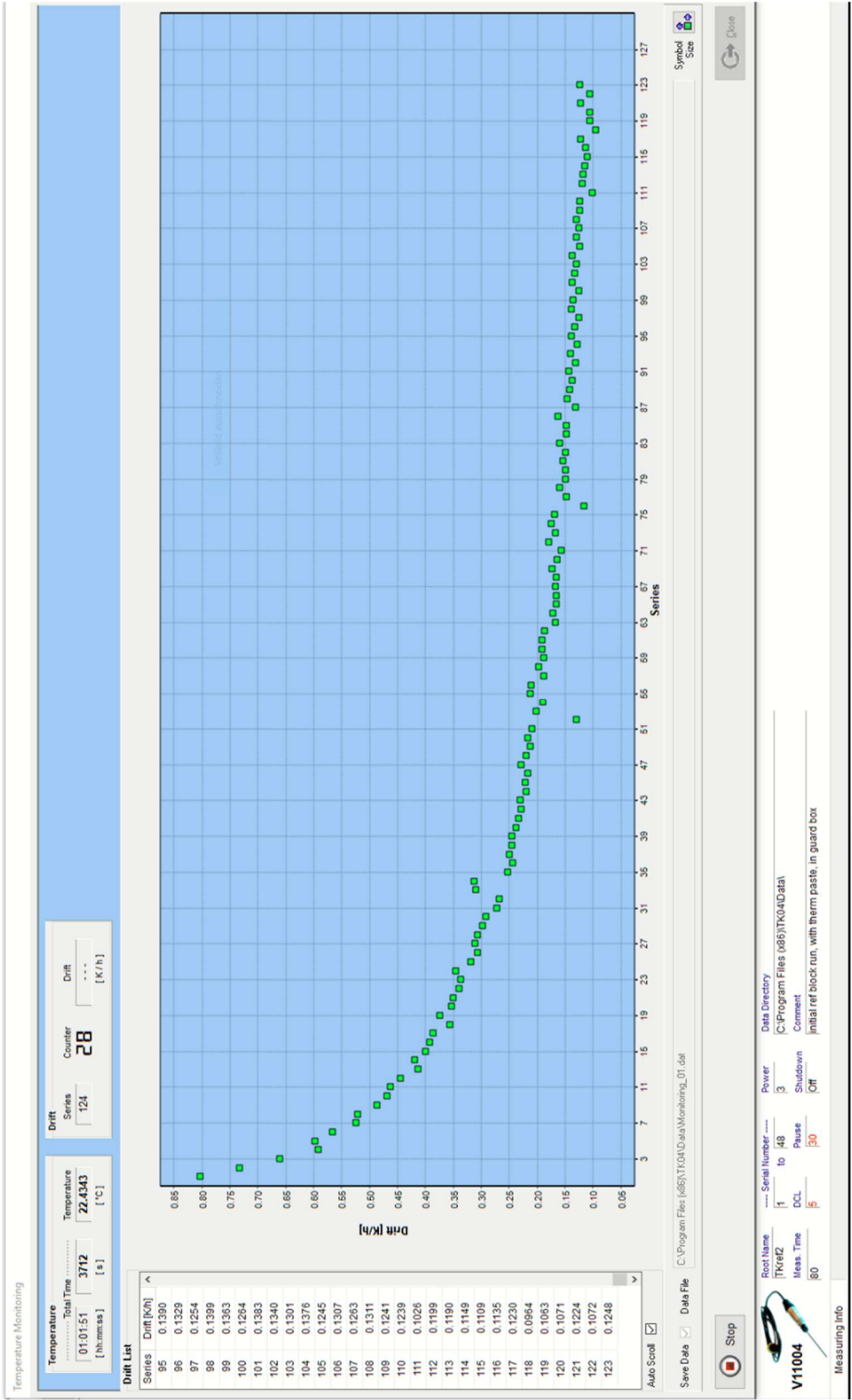


Figure 30: Temperature monitoring of TeKa reference block before measurement

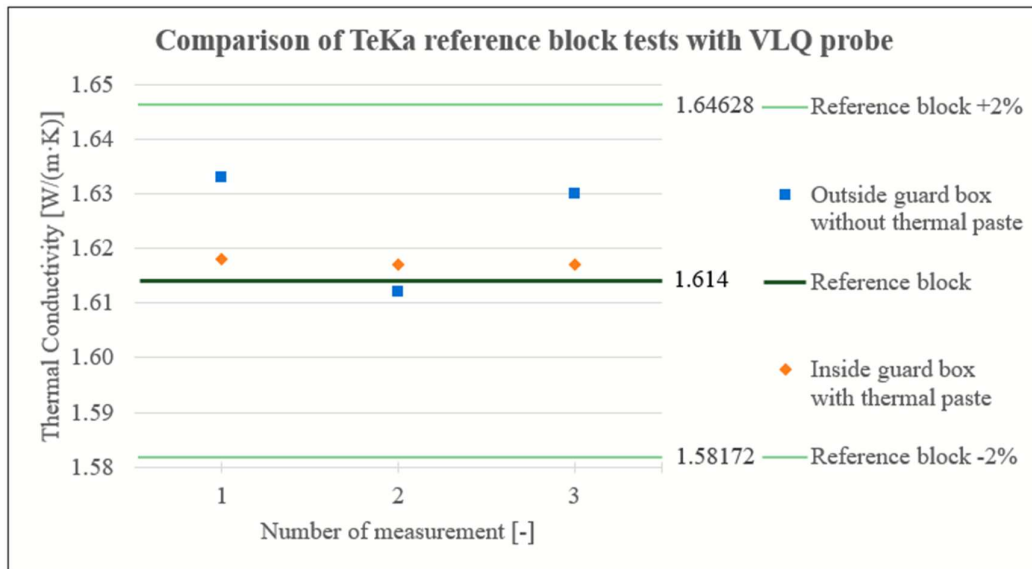


Figure 31: Comparison of VLQ reference block tests

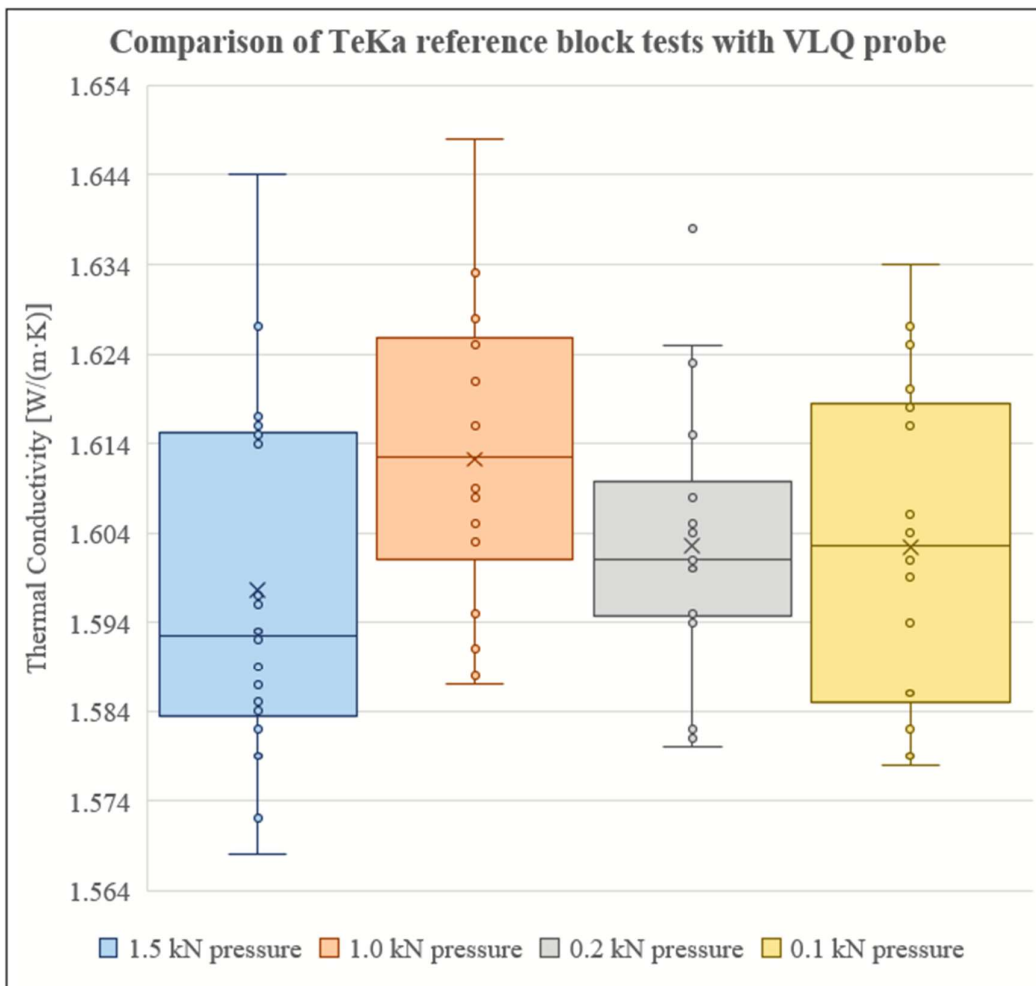


Figure 32: Comparison of HLQ reference block tests

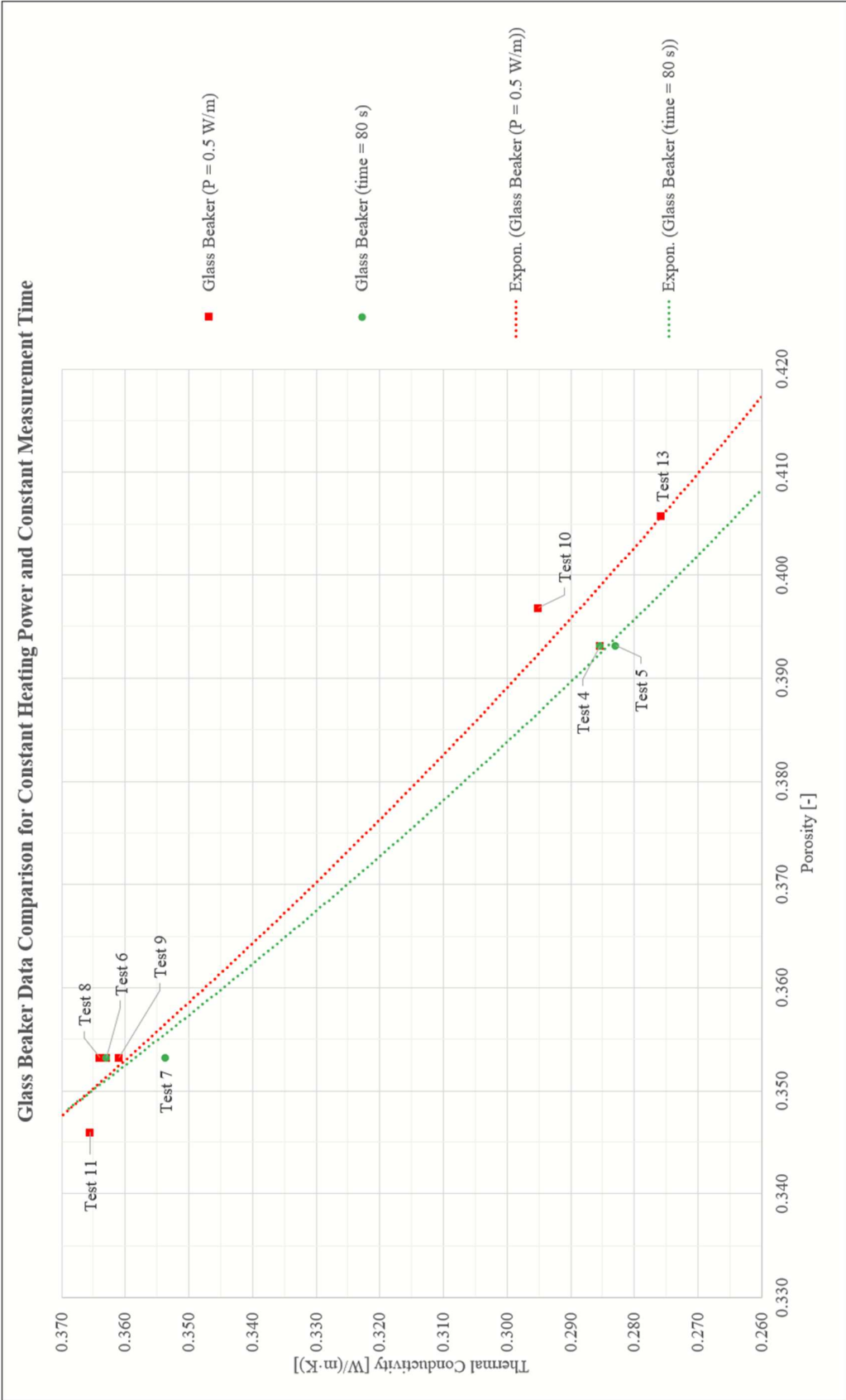


Figure 33: VLQ measurements data

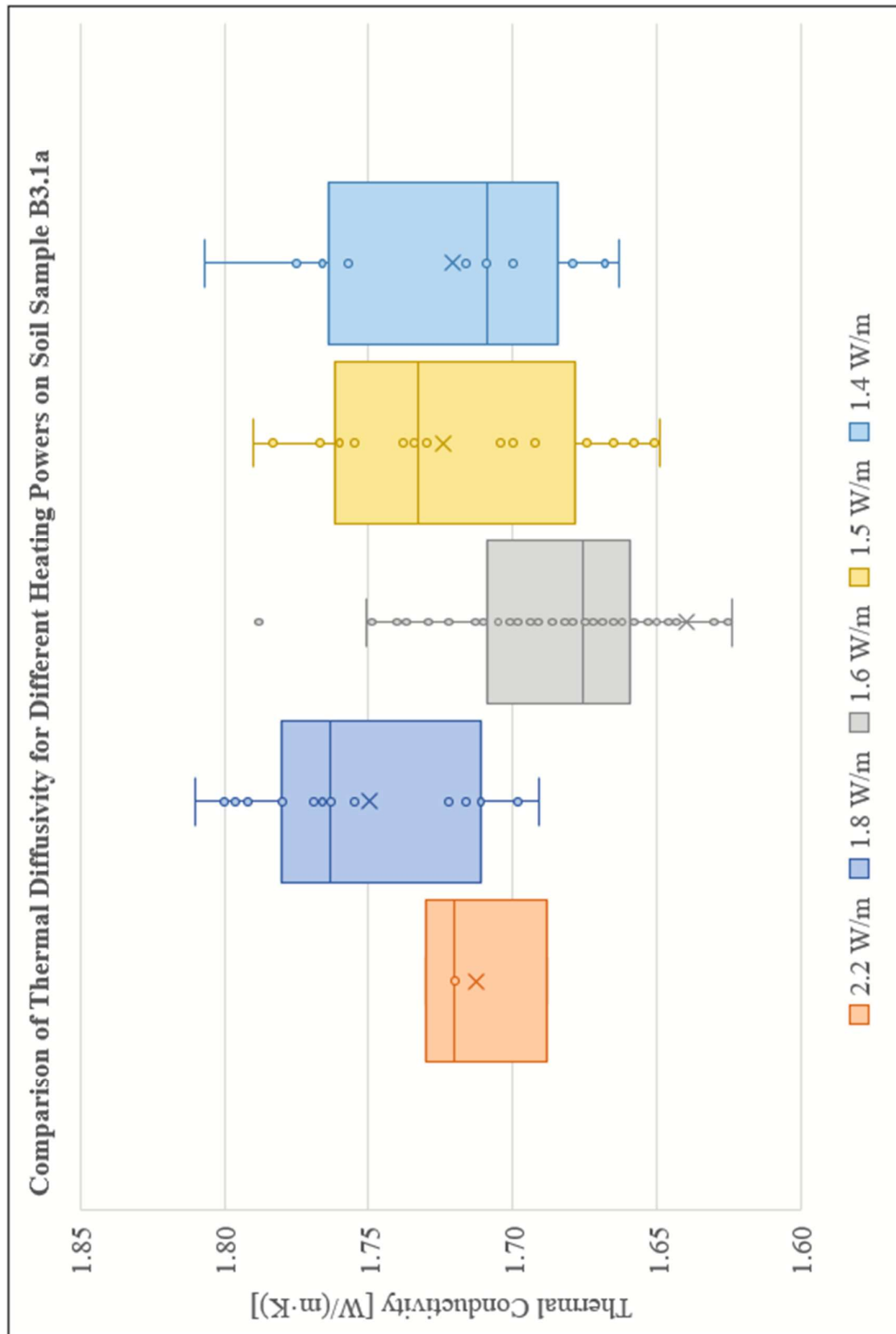


Figure 34: Comparison of thermal conductivity using HLQ probe for machine finished surface on soil sample B3.1a

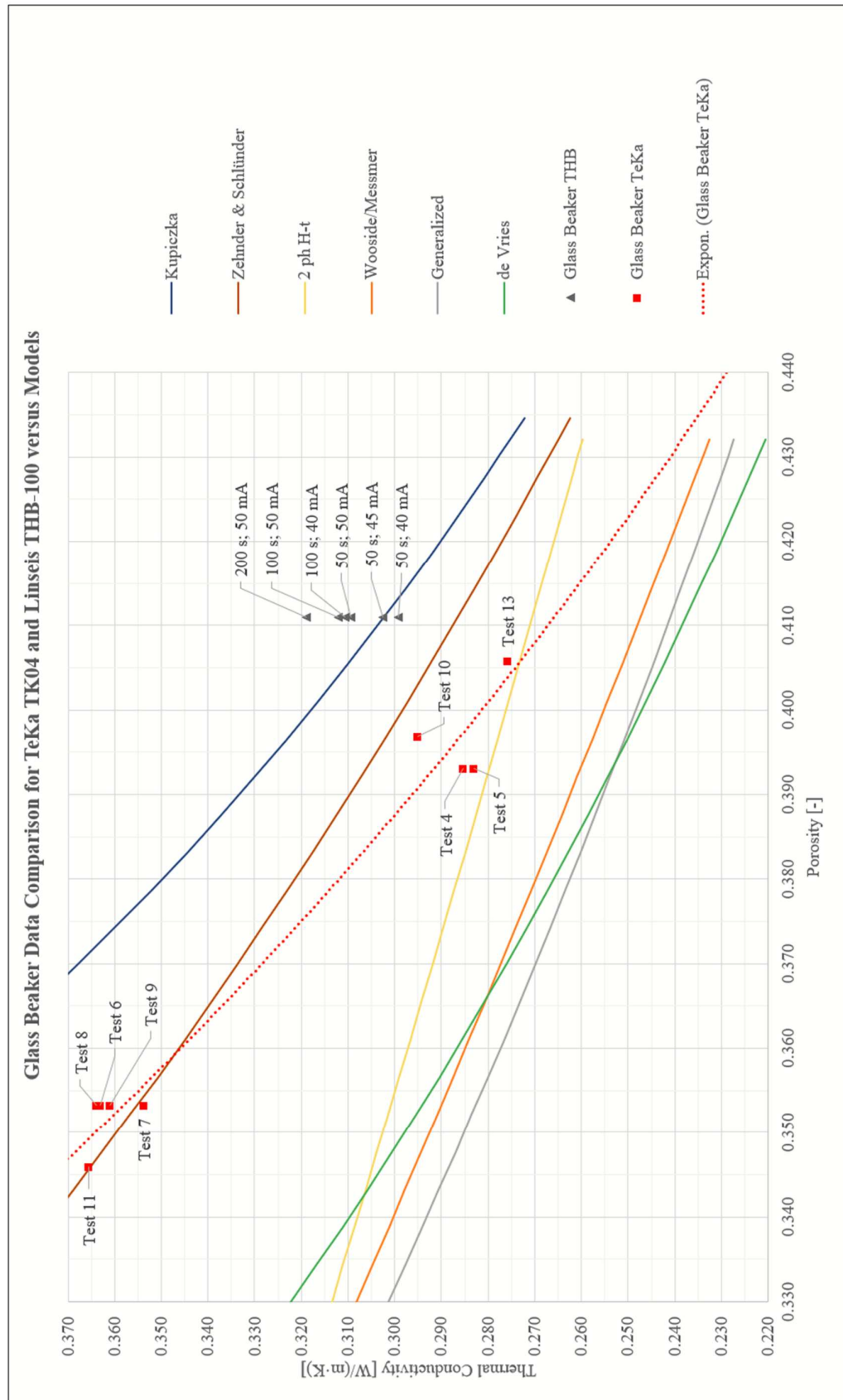


Figure 35: Comparison of VLQ, THB-100, and external studies and models