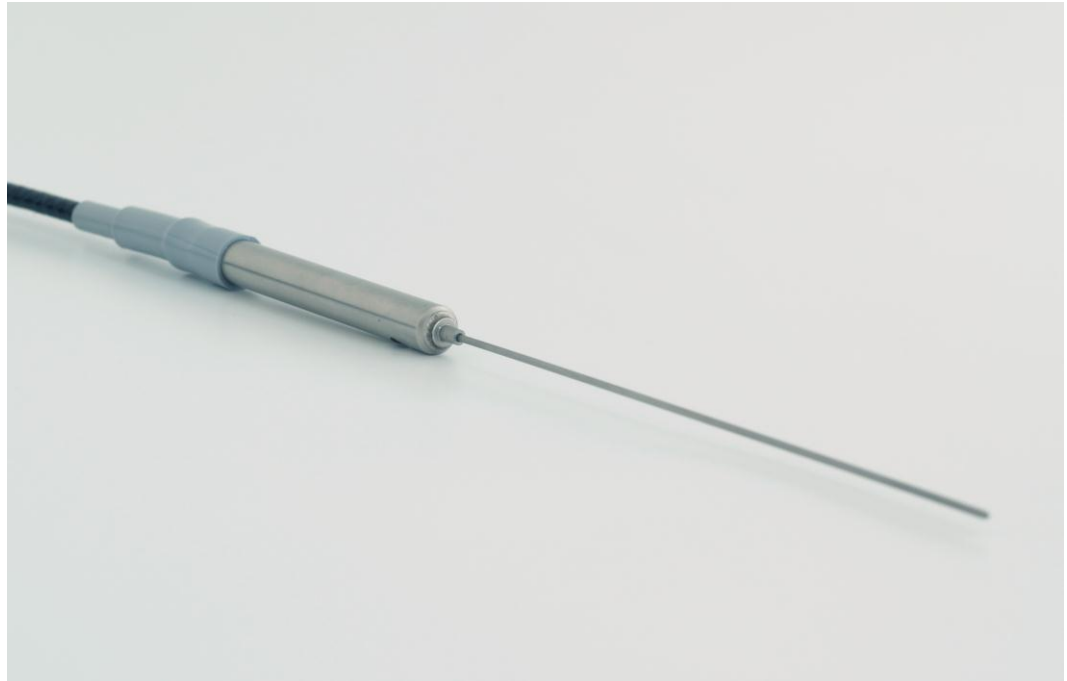


TP02

Non-Steady-State Probe for Thermal Conductivity Measurement



TP02 manual v1209

Edited & Copyright by:

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Warnings:

Putting more than 3 volts across any part of TP02 may result in permanent damage to the sensor.

The user is responsible to take care that TP02 is not overheated during operation.

The TP02 needle is vulnerable. In case of doubt if it can penetrate the sample, the sample should be pre-drilled or use of guiding tubes (GT series) should be considered.

The TP02 needle is vulnerable. When not being used, it is recommended to have the protective cover over the needle.



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List of symbols

Thermal diffusivity	a	m^2/s
Distance from the heater	r	m
Heating cycle time	h	s
Thermal conductivity	λ	W/mK
Voltage output	U	V
Sensitivity	E	V/K
Time	t	s
Response time	τ	s
Temperature	T	K
Differential temperature (main sensor signal)	ΔT	K
Electrical resistance	R_e	Ω
Electrical resistance per meter	R_{em}	Ω/m
Thermal resistance	R_t	m^2K/W
Thickness	H	m
Diameter	D	m
Length	L	m
Area	A	m^2
Volumetric heat capacity	C_v	J/Km^3
Heat capacity	C	J/kg
Density	ρ	kg/m^3
Heat flux	Φ	W/m^2
Current	I	A
Mass	M	kg
Volume	V	m^3
Power	P	W
Power per meter	Q	W/m
Intermediate variables	x, w, z	-
Error in a variable	δ	-

Subscripts

Property of thermopile sensor	sen
Property of the current sensing resistor	current
Property of the heater	heat
Property of the medium	med
Property of the needle	needle
Property of the cold junction	cold
Property of the Pt1000	pt1000
Property, at $t = 0$, at $t = 180$, $t = h$ seconds	0, 180, h





Introduction

TP02 is a probe that offers the possibility to perform a practical and fast measurement of the thermal conductivity (or thermal resistivity) of the medium in which it is inserted at the highest accuracy level. It works in compliance with the ASTM D5334-14, D5930-97 and IEEE 442-1981 standards. The standard TP02 probe has proven suitability in soils, thermal backfill materials, sediments, foodstuff, powders, sludges, paints, glues and various other materials. The Non-Steady-State Probe (NSSP) measurement method (also known as transient line source, thermal needle, hot needle, heat pulse- and hot wire technique) has the fundamental advantages that it is fast and absolute while the sample size is not critical.

Hukseflux is specialised in NSSP design. Special models have been developed for in-situ field experiments. For permanent installation in soils, a dedicated model, TP01, is available. TP02 has been designed and tested in collaboration with the Applied Physics Group of Wageningen University.

The TP08 is a small version of type TP02, made for situations where the length of TP02 poses a problem.

Chapter 1 contains information about theory and design of the NSSP, chapter 2 summarises the instrument specifications. The remaining chapters contain information about installation, connection, programming and maintenance.

For performing a measurement, TP02 has to be combined with a Measurement and Control Unit (MCU). The required specifications are treated in the appendix on MCU specifications.

One of the most cost-effective solutions to the problem of measurement and control is to use the data loggers of Campbell Scientific, type CR10X or CR1000. More detailed information on this (programs and wiring diagrams) can be obtained at Hukseflux.

Turn key systems are offered for sale: see TPSYS, separate brochure and manual.





The end result of the measurement is preferably calculated by analysis of the measured data in a spreadsheet (like MS-Excel) or a mathematical program. Alternatively, but not recommended because it is less reliable, automatic calculation of the end result in the MCU is possible.

Example Excel sheets can be obtained at Hukseflux.

For high accuracy calibration CRC Calibration Reference Cylinders are available. See appendix.

For insertion into hard soils GT Series Guiding tubes can be applied. See appendix.

In soil energy balance studies it is also useful that a very accurate differential temperature measurement is done (before the heating cycle starts) between the tip and the hot joint. This difference in temperature across 100 mm can be determined within 0.01 degrees C. This might be useful for determining the surface energy balance. Response time is very fast because the probe is very thin.

TP02 and TP08 can be used, if the right gasket is used, in pressurised or vacuum environments. The bower end of the base must then be clamped.

In the appendices, several more specialised subjects are treated.

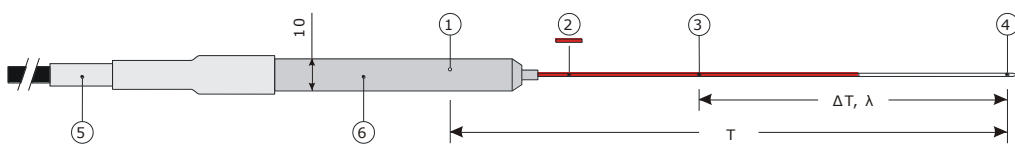


Figure 0.1 *TP02 Non-Steady-State Probe consists of a needle with 2 thermocouple junctions; the hot joint (3), and the cold joint (4) (the cold joint at the tip remains at a stable temperature). A heating wire, (2) runs across 2/3 of the needle length. The needle is inserted into the medium that is investigated. In the base, (6), a reference temperature sensor (pt1000), (1), is mounted. Advantages of this design: an optimal accuracy independent of the medium temperature, a minimal sensitivity to thermal gradients, a high sensor stability and the possibility to use normal cables and connectors. Needle length 150 mm. All dimensions are in mm.*





1 Theory

1.1 General Non-Steady-State Probe (NSSP) Theory

For determining the thermal conductivity of materials various types of measurement equipment can be used. In general one can make a distinction between steady-state techniques in which the investigated sample is supposed to reach a perfect thermal equilibrium, and non-steady-state techniques. In non-steady-state techniques the material properties are determined while the sample temperature still changes.

The main advantage of steady-state techniques is the simplicity of the analysis of stabilised constant sensor signals. The main advantages of non-steady-state techniques are the short measurement time and the fact that the sample dimensions do not necessarily enter the equation.

The only Non-Steady-State technique that has officially been standardised is the one using a single needle probe like TP02. The standards of the American Society for Testing and Materials (ASTM) and Institute of Electrical and Electronics Engineers (IEEE) describe proven methods for determining the thermal conductivity of materials.

ASTM D5334-14 and D5930-97 and IEEE Std 442-1981 "Standard Test Methods" specify the use of Non-Steady-State Probes (NSSP) in various applications. In particular, use in plastics, soil and soft rock are mentioned, but the method is not restricted to these materials. More information about these standards can be found in the appendices.



In general a NSSP consists of a heating wire, representing a perfect line source, and a temperature sensor capable of measuring the temperature at this source. The probe is inserted in the medium that is investigated. The NSSP principle relies on a unique property of a line source: after a short transient period the temperature rise, ΔT , only depends on heater power, Q , and medium thermal conductivity, λ :

$$\Delta T = (Q / 4 \pi \lambda) (\ln t + B) \quad 1.1.1$$

With ΔT in K the rise of temperature relative to the moment that the heating starts at $t = 0$, Q in W/m, λ in W/mK, t the time in s and B a constant. By measuring the heater power, and tracing the temperature in time (for TP02 typically heating for 100s), λ can be calculated. The sample size is not critical, as long as a radius around needle is covered that is roughly 15 times the needle radius, in case of TP02: 20 mm.

The measurements of Q , t and ΔT are all direct measurements of power, time, and temperature respectively. These are done without need of reference materials. The measurement with TP02 is absolute.

The thermal conductivity can be calculated from two measurements at t_1 and t_2 . For TP02 both t_1 and t_2 are higher than 60 s, and typically 100 s apart.

$$\lambda = (Q / 4 \pi \Delta T) \ln(t_2 / t_1) \quad 1.1.2$$

With ΔT the rise in temperature between the measurements at times t_1 and t_2 .

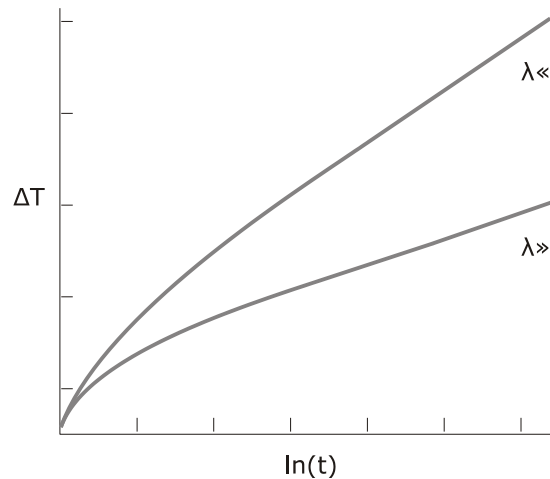


Figure 1.1.1 *The signal of TP02 as a function of the natural logarithm of time. After a transient period the graphs show linear behaviour. In this phase the slope of the graph is inversely proportional to the thermal conductivity λ .*

The formula 1.1.2 is a first order approximation that is only valid under certain conditions.

The most important ones are that the medium has reasonable thermal contact with the probe, and that the sample is thermally stable. More details can be found in the specifications and the directions for performing a correct measurement later in this chapter. More details on the mathematics and literature references can be found in the appendix.

Whether TP02 has passed the transient period is usually checked by visual examination of the graph.

The measurements of Q , t and ΔT are all direct measurements of power, time, and temperature and are done without need of reference materials. The measurement with TP02 is absolute. The main requirement is that the heater resistance per meter is accurately determined before production.





1.2 TP02 theory

What everyone should know about TP02 operation:

TP02 incorporates 2 thermocouple junctions in the needle. They are connected in anti-series, producing a voltage output, U , that is proportional to ΔT , the differential temperature of the two joints. The junction in the tip is not heated.

The main TP02 signal is this differential temperature signal. The hot joint is located at 1/3 of the needle length (seen from the base) and the cold joint at the tip. The heating wire runs across 2/3 of the needle length.

In this configuration the voltage U before starting the measurement is always small, independent of the medium temperature. In designs with only one thermocouple joint in the needle, the signal U will appear on top of a larger signal. The two-junction design has a superior accuracy when measuring at high and low temperatures.

In addition, by having both the cold- and the hot junction in the needle, the sensitivity to changing temperature of the medium is minimised.

TP02 has a reference temperature sensor in the base: The sensor in the base (Pt1000) serves as a "cold junction" measurement for establishing the absolute medium temperature T . This is required by ASTM. The medium temperature before starting the measurement is used for temperature correction of the thermocouple sensitivity, again offering superior accuracy across the temperature range.

TP02 has a cable with copper cores (and not thermocouple alloy cores). Putting the Pt1000 temperature sensor in the base allows the use of normal copper core cables and connectors for cable extension of TP02.





To measure the absolute temperature of the medium, the procedure is to measure the base temperature (with the Pt1000), and use this as a cold junction compensation temperature for the cold thermocouple junction at the tip. Most dataloggers can accept such an "external" cold junction measurement. If not, the cold junction temperature can be calculated by

1 measuring the cold junction microvolt signal, and dividing this by 40.35 $\mu\text{V/K}$, to get the temperature of the cold joint relative to the Pt1000. This value can be added to the Pt1000 temperature to get the absolute medium temperature.

or

2 putting the base in the medium, so that the base temperature equals the medium temperature.

The sensitivity of the main sensor signal is 40.35 $\mu\text{V/K}$ at 20 °C. However, the thermocouple sensitivity is temperature dependent and varies with about 0.2%/K. This produces a significant error; working at 10 °C instead of the 20 °C reference temperature produces an error of 2%.

The temperature dependence can be corrected by measuring the base temperature using the Pt1000:

$$E_{\text{sen}} = 10^{-6} (39.40 + 0.050 T - 0.0003 T^2) \quad 1.2.1$$

This approximation will be accurate within 1% between -40 and +100 °C.

The data obtained with TP02 can be processed in any normal spreadsheet program.

An example spreadsheet can be obtained at Hukseflux.





1.3 TP02 design considerations

Further design considerations for TP02 were:

1. to cover a wide range of applications with primary focus on soils and foodstuff
 2. to be as reliable as possible
 3. to have stable long term behaviour
 4. to be compliant with existing standards
 5. to produce relatively simple measurement results, allowing own data processing.
 6. to have a traceable calibration
 7. to be applicable / compatible with commercially available general laboratory equipment
-
1. covering a wide range of applications: The TP02 has an all stainless steel housing. It is able to work in a variety of environments and is more robust than housings that are partially made out of plastic. The measurement with TP02, using two joints in the needle and one absolute temperature measurement in the base, has a superior accuracy over its entire temperature range (see the appendix for details). Also by using two joints, when the medium temperature changes this is automatically compensated for. In the ideal situation because the two junctions are equally affected, the output signal shows no offset.
 2. producing reliable results: A particular advantage over two-needle designs is that bending the needle does not significantly influence the TP02 measurement. By contrast, in two-needle designs bending the needles causes the sensitivity to change.
 3. stable long term behaviour: All the materials in TP02 are highly temperature resistant. It has a full stainless steel needle and base. TP02 is completely sealed. It has a welded tip. This no-compromise sealing guarantees the long term stability of the sensor. Designs with glued sealing or epoxy housings are less reliable in this respect, particularly when working in moist environments.
 4. Complying with standards: For institutes that prefer to work according to standardised procedures: The measurement with TP02 is compliant with the ASTM standards D5334-14 and D5930-97. By contrast, other probe models with dual needles do not comply with these standards.



5. Data processing: a single needle design has a fairly simple signal analysis, only involving the conversion of the signal to a logarithmic scale, and establishing the slope of the curve.
6. Calibration: the TP02 measurement is absolute and traceable to the measurement of the heater resistance and the thermocouple properties. The calibration is traceable to NPL National Physical Laboratory of the UK. Verification of the stability of the total probe can be done by repeated (yearly) testing in Calibration Reference Cylinders (CRC) traceable to NPL. These can be purchased at Hukseflux. As low cost alternatives, both glycerol fluid and agar gel can be used.
7. Application: TP02 can be employed by any laboratory having a high accuracy voltmeter, resistance meter (for Pt1000) and equipment to do the proper switching / timing of the experiment.

1.4 Directions for performing a correct measurement

In general the TP02 has proven to be suitable for laboratory use.

It has shown to be sufficiently robust to survive common handling. In our samples the probe could be inserted without help. Some sample types, like hard soil might require pre-drilling or use of guiding tube (see appendices on pre-drilling and guiding tubes).

Field use is possible but is unlikely to reach the same level of accuracy as use in the laboratory.

Consult the product manual to see if a NSSP can be applied in the intended experiment. Most common foodstuff, soils, powders, slurry's and gels are suitable for analysis with a NSSP, so-called "suitable media". In case of doubt, consult Hukseflux engineers.

The NSSP measurement technique can be used with TP02 to measure in the thermal conductivity range of 0.1 to 6 W/mK. The expected accuracy for carefully made measurements of suitable media at 20 degrees is $\pm (3\% + 0.02)$ W/mK.

At temperatures different from 20 degrees, the temperature dependence of the sensor must be taken into account.

Nevertheless after correction there is an additional ± 0.02 %/K uncertainty.

Measurement and control equipment must be selected with care: in particular the thermocouple output measurement and the timing are critical.



The measurement must be performed with care; measurement results must be critically reviewed before acceptance. The measurement technique introduces a heat flow into the material. It is assumed that this heat is transported by conduction and that there are no temperature changes caused by other sources. The probe is supposed to be static during the experiment. If these requirements are not met the measurement can lead to false results.

Examples of possible error sources are: local moisture transport/evaporation by excessive heating (this is the reason why there is low level heating in moist soils), movement of the probe, change of contact resistance by phase changes, local melting by excessive heating and thermal convection when used in fluids.

The thermal conductivity measurement does not suffer from bad contact. What happens in case of a high contact resistance is that the transition time (time to reach a straight line) becomes longer. This can be seen in the measurement curve.

The part of the graph that is analysed must be selected by the user. When analysing a known material or a material that closely resembles a known material, assuming that the contact resistance is not variable, the previously used interval can be used again. When there is no experience with the particular material, or the contact resistance varies, the detailed behaviour must be studied, and a suitable interval must be selected. The transient period varies from one material to the other and also from one probe to the other. For one and the same probe, the transient period is inversely proportional to the volumetric heat capacity of the material. In case of a larger contact resistance, the transient period will be longer. For probes of similar geometry and composition the transient period is proportional to the square of the diameter of the needle.

When working with small sample sizes, smaller than about 15 times the needle radius (for TP02 around 20 mm) around the sensor, the effects of boundary conditions must be analysed. Please consult Hukseflux.

To avoid problems with local evaporation of moisture, the heating power must be kept as low as possible, the heating time as short as possible. In exceptional cases a fluid, grease or gel can be used to reduce the contact resistance.

The sample must be as thermally stable as possible. Ideally not exceeding more than 1% of the temperature rise generated by the heater during the interval that is analysed. If the sample is





not stable, the resulting signal will be added or subtracted from the real heater signal, leading to measurement errors. If the existing temperature change is linear in time, this can be compensated for by subtraction from the sensor signal.

The procedures as recommended in the ASTM standards offer a good guideline, but should not be followed under all conditions. In particular in the ASTM standards no attention is paid to the reduction of heating power and temperature rise during the experiment. This is leading to large errors in moist materials. Also the compensation for existing temperature gradients in the material and temperature dependence of the sensor are not considered. These omissions can lead to mistakes.



2 Specifications of TP02

TP02 Non-Steady-State Probe is used for determining the thermal conductivity of the medium in which it is inserted. It can only be used in combination with a suitable measurement and control system, typically using a spreadsheet for data analysis.

GENERAL SPECIFICATIONS of TP02	
Measurement method	Absolute measurement, according to the standards ASTM D5334-14, ASTM D5930-97 and IEEE Std 442-1981.
Specified measurements	Thermal conductivity of media as specified under suitable media. Directions from the manual should be followed.
Suitable media	Granular materials (grain size smaller than 0.5 mm), powders, soils, gels, pastes, slurries, soft rock, and foodstuff and similar substances in the thermal conductivity (λ) range of 0.1 to 6 W/m.K. Some fluids are suitable; see appendix.
Sample requirements	Preferably the medium is all around the TP02 needle. It must be all around the heated part. It preferably has a radius of > 100 mm, at least 20 mm. The sample may be pre-drilled. Contact fluid may be used. Guiding tubes may be used; see appendix. The sample must be thermally stable ($dT/dt < 1$ % of the heater induced change typically, 5 % max) and reasonably homogeneous.
Duration of the heating cycle h	h must be as low as medium and readout allow. 100 seconds (typical). Empirically verified for each medium type.
Heating Power / m Q	Q must be as low as the medium and readout allow. Typically Q is adjustable so that the total temperature rise is no more than 1 degree. Typically lower than 5 W/m.
Temperature range	-55 to +180 °C (base and cable) to +250 °C (needle)
Protection class	IP68 (needle and base) IP67 (entire probe)
ISO requirements	TP02 is suitable for use by ISO certified laboratories. Use of CRC calibration cylinders should be considered; see appendix.
CE requirements	TP02 complies with CE directives

Table 2.1 List of TP02 specifications (continued on next page)

MEASUREMENT SPECIFICATIONS	
Data analysis	Preferably in a spreadsheet (MS-Excel) or a dedicated mathematical program, alternatively in the MCU.
Expected accuracy	$\pm (3\% + 0.02 \text{ W/mK})$ for homogeneous media with good contact to the needle.
Repeatability	$\pm 1 \%$
Temperature dependence	$< \pm 0.02\%/^{\circ}\text{C}$ after correction of tc temp. dependence
SENSOR SPECIFICATIONS	
Thermocouple joints	1NiCr-NiAl type K, according to ANSI MC96.1-1982
Temperature sensor	Pt 1000, Class B, IEC 751:1983
Required readout	2 differential voltage, 1 current, 1 Pt1000 channel, storage of data points versus time.
Voltage input	3 VDC (maximum), switched, typically adjustable to the medium properties, 1 VA max.
Thermocouple R_e	50 ohm (nominal)
Heater R_e , length L	85 Ohm/m (nominal), specified for each individual sensor within $\pm 0.25\%$, 100 mm \pm 2 mm, total 11 Ohm (nominal)
Probe dimensions	Needle: 150 by 1.5 mm, Base: 50 by 10 mm
Cable length, type	2.5 meters, PTFE, diameter 5 mm, 6.5 mm max.
Weight	0.3 kg (including 2.5 m cable) 0.8 kg including packaging (6 by 32 by 30 cm) and manual
CALIBRATION	
Calibration traceability	Thermal Conductivity to NPL National Physical Laboratory of the UK. Thermocouple according to ANSI MC96.1-1982
Recalibration interval	Every 2 years using either CRC calibration reference cylinders or alternatively glycerol or agar gel.

Table 2.1 *List of TP02 specifications (started on previous page)*



3 Short user guide

Preferably one should read the introduction and the section on theory.

If TP02 is supplied without a MCU, additional equipment is required:

A voltage source delivering power to the heater, and a MCU capable of switching, readout of voltages, a current, Pt1000 and timing with sufficient accuracy. If possible but not necessarily this system also should have the capability to perform calculations based on the measurement. Typically the end result is calculated from raw data during post processing, so that also the measurement graph can be visually reviewed. Specifications for this equipment can be found in the appendix. If this is available, it is recommended to use Campbell Scientific CR10X or CR1000 Measurement and Control Module.

The connection of TP02 to the MCU differs from case to case. Usually this is done using clamp connectors or high quality connectors. The wiring scheme can be found in the chapter on electrical connection.

The following chapters contain information on installation, how to put the system into operation and how a normal measurement should be performed and how the end result can be calculated.

3.1 Installation of TP02

TP02 is generally installed in a laboratory environment. The surrounding should be thermally quiet. For an optimal measurement, doors should be closed to avoid unnecessary convection. TP02 preferably works in equilibrium with ambient air temperature.

Working at higher or lower than ambient temperatures is possible by putting TP02 in a climate chamber and allowing the medium and probe come to an equilibrium. In general, during a measurement in a climate chamber it is recommended to turn active climate control and ventilation off during the measurement.

TP02 has a very broad working range, and the complete assembly can be used up to 180 degrees C.





3.2 Putting TP02 into operation

It is recommended to start with a simple test the sensor functionality by checking the impedance of the main sensor and the heater, and by checking if the sensor reacts to changing temperatures. After this it is suggested to perform a measurement using a well known medium, such as a Calibration reference cylinder. With these two actions, operation is trained, and proper operation is confirmed.

<p>Check the heater impedance. Use a multimeter at the 200 Ohms range. Measure between two wires 1 and 2, (black) that are connected at opposite ends of the heater.</p>	<p>This should be between 9 and 11 Ohms. Infinite indicates a broken circuit, zero indicates a short circuit.</p>
<p>Check the impedance of the main sensor. Use a multimeter at the 100 Ohms range. Measure at the sensor output wires 3 and 5, white and yellow. Warning: during this part of the test, please put the sensor in a thermally quiet surrounding, holding the sensor in still air.</p>	<p>A typical sensor impedance should be between 60 and 80 Ohms. (at 2.5 m cable. If more cable is used, add 0.1 Ohm/m back and forth, so 0.2 Ohm/m) Infinite indicates a broken circuit, zero indicates a short circuit.</p>
<p>Check the Pt1000. Use a multimeter in the 2000 Ohms range. Check opposite wires 6 and 7, white and red. The two red wires 7 and 8 are equivalent.</p>	<p>The result at room temperature should be between 1000 and 1200 Ohms when the probe is close to room temperature. Infinite indicates a broken circuit, zero indicates a short circuit.</p>
<p>Check the Pt1000. Use a multimeter in the 10 Ohms range. Check between the two wires at one end of the sensor, 7 and 8, both red.</p>	<p>The result should be in the 0.1 to 1 Ohms range. For each meter of wire, the impedance is around 0.1 Ohm. The total for 2.5 m is 0.5 Ohms.</p>

Table 3.2.1 *Checking the functionality of the TP02. The procedure offers a simple test to get a better feeling how TP02 works, and a check if the system is OK. (continued on the next page)*





<p>Check if the main sensor reacts to differential temperatures. Measure at the sensor output wires 3 and 5, white and yellow.</p> <p>Use a multimeter at the millivolt range. Measure at the sensor output. The signal initially should be close to zero. Generate a signal by touching the hot joint at one third of the needle length, or the cold joint at the tip with your hand. The reaction at the hot joint should be opposite in sign, at the cold joint negative.</p> <p>If a 1.5 Volt battery or power source is available, it is also possible to connect the power source to the heater and see if a signal is generated.</p>	<p>The thermocouple should react by generating a millivolt output signal.</p>
<p>Check the absolute temperature signal from the tip. Use a multimeter at the millivolt range. Measure at wires 3 and 4, the two wires connected to the cold junction at the tip. Generate a signal by touching the probe tip with your hand. Use the Pt1000 temperature signal as cold junction compensation, check the end result.</p>	<p>The thermocouple should react by generating a millivolt output signal.</p>

Table 3.2.1 *Checking the functionality of the TP02. The procedure offers a simple test to get a better feeling how TP02 works, and a check if the system is OK. (started on the previous page)*





	Total resistance between wires	Value TP02
1	1 – 2 (heater)	9-11 Ohm
2	3 – 4 (cold joint)	40- 50 Ohm
3	4 – 5 (hot joint)	20- 26 Ohm
4	3 – shield	0,1- 1 Ohm
5	3 – 5 (main sensor)	60 to 80 Ohm
6	1- shield	Infinite
7	7 – 8 (Pt1000)	0,1- 1 Ohm
8	6 - 7 and 6 – 8 (Pt1000)	1000-1200 Ohm
9	7 - shield and 8 - shield	Infinite
10	Information on the cable should be the same as engraving on the probe.	

Table 3.2.2 *Checklist for resistances.*

The next step is to perform a measurement using either a Calibration Reference Cylinder, or a reference medium such as agar gel or glycerol.

Put the probe in the reference material	Directions how to prepare agar gel or glycerol can be found in the appendix.
Perform a measurement	Directions from the chapter on performing a normal measurement can be followed.
Calculate the end result	Directions from the chapter on calculation of the measurement result can be followed.
Compare the measurement results with the data of the sheet	The difference between the data and the measured result should not exceed $\pm 3\%$

Table 3.2.3 *Checking the instrument performance by making a first measurement using a reference material.*



3.3 Performing a normal measurement and calculating the end result

An example spreadsheet can be obtained at Hukseflux.

Having prepared all the necessary equipment, and having tested the system, performing a normal measurement is easy.

<p>Perform a measurement keeping in mind the recommendations of chapter 1.</p> <p>In general the total heating cycle time is called h. It is recommended to wait for a period of h before the measurement to verify if the thermal environment is stable. Any significant thermal gradient that occurs over this period, can be subtracted from the thermal gradient during the measurement. The total measurement takes $2h$ seconds.</p>
<p>Retrieve the measured data, preferably with a 2 second resolution or better to a spreadsheet.</p>
<p>Verify that the measurement reached a linear dependence on $\ln(t)$ by visual inspection.</p>
<p>Select a suitable portion of the graph, and calculate the result, based on formula 1.1.2.</p>
<p>Perform quality assurance. Suggestions can be found in the appendix.</p>

Table 3.3.1 *Performing a normal measurement.*

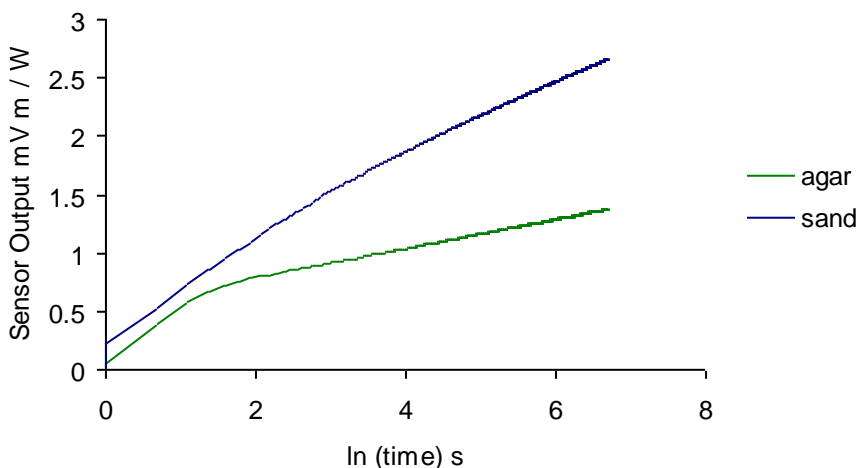


Figure 3.3.1 *Typical measurement results in agar gel and dry sand. The thermal conductivity's are 0.6 and 0.3 respectively. The linear portions of the graph have to be selected by the user. The graph illustrates that the portion that is suitable for analysis changes from one medium to the other.*

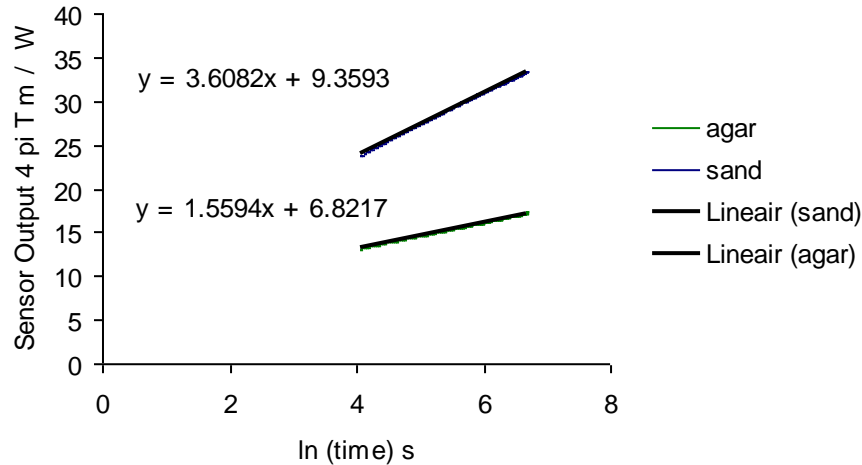


Figure 3.3.2 Example of data analysis in Excel. See also formula 1.1. A linear portion of the graph of the figure 3.3.1 has been selected. The temperature T has been multiplied by 4π and divided by the heating power per meter. The Excel program can automatically calculate the best linear fit. The end result for the thermal conductivity's is $1/3.608$ and $1/1.559$, which is 0.27 and 0.64 respectively.

The following formulas have been used are 1.1.1 and 1.1.2 and (see the list of symbols for explanation of the units):

For the calculation of Q , the power per meter:

$$Q = (U_{\text{current}} / R_{\text{e current}})^2 R_{\text{em heat}} \quad 3.3.1$$

This formula is valid if a current sensing resistor is used. Please mind that R_{em} is in Ohm per meter.

$$\Delta T = U_{\text{sen}} / (E_{\text{sen}} (1 + (dE_{\text{sen}} / dT) T)) \quad 3.3.2$$

$(dE_{\text{sen}} / dT) T$ is the temperature correction of the thermocouple temperature dependence.



For TP02 the following formulas are used:

The main sensor signal is ΔT , the differential temperature, measured between the hot- and the cold joint:

$$\Delta T = U_{\text{sen}} / 10^{-6} (39.40 + 0.050 T - 0.0003 T^2) \quad 3.3.3$$

With the T the tip (or cold joint) absolute temperature before the start of the measurement.

With $T = T_{\text{cold}}$ (before the measurement), measured using T_{pt1000} as cold junction compensation.

For the current measurement often a 10 ohm precision resistor is used, so that 3.3.1 becomes:

$$Q = (U_{\text{current}}^2 / 100) R_{\text{em heat}} \quad 3.3.4$$

Alternatively, if automatic cold junction compensation using the T_{pt1000} is not possible:

$$T_{\text{cold}} = T_{\text{pt1000}} + U_{\text{cold}} / 40.35 \cdot 10^{-6} \quad 3.3.5$$

The calculation of the thermal conductivity is done as follows:

$$\lambda = (Q / 4 \pi \Delta T) \ln(t_2 / t_1) \quad 3.3.6$$

The straight part of the curve should be selected only. The nice thing is that

1 if the line is straight, automatically the measurement is good

2 if it is not straight it is an indication that something is wrong.

Visual review is always recommended.



3.4 Quality assurance

The straight part of the curve should be selected only. The nice thing is that

- 1 if the line is straight, automatically the measurement is good
- 2 if it is not straight it is an indication that something is wrong.

Visual review is always recommended.

During the measurement a number of checks can be done to verify if the measurement conditions were good.

The suggested checks are as follows (units as in the list of symbols):

$ABS\{(Q_h - Q_{2h}) / Q_{2h}\} < 0.5\%$ Check of the power stability.

$ABS(\Delta T_{0.5h} - \Delta T_h) < 0.05$ $ABS(\Delta T_{1.5h} - \Delta T_{2h})$ Check if the temperature gradient before the experiment is not too large compared to the temperature gradient during the measurement.

$T_{2h} > T_{1.8h} > T_{1.7h} \dots > T_h$ Check if the signal is monotonously rising.

$0.25 < T_{2h} - T_h < 2.5$ Check if the total temperature rise is not too large or small.

$0.1 < \lambda < 6$ This is a range check of the thermal conductivity.

$100 < h < 1000$ Check if the heating time is not too short or too long

Check on the input parameters like sample number, operator number etc



4 Electrical connection of TP02

In order to operate, TP02 should be connected to a measurement and control system as described in the appendix. The wiring schedule is shown in figures 4.1 and 4.2.

For the purpose of making a correct measurement of the heater power, Q_{heat} , there is a 2-wire connection to the heater. Two wires carry the current. The current should be measured. Typically this is done using a shunt resistance.

Typically 3 voltage levels are supplied, for instance: 2VDC, 1VDC and 0.7 VDC. These can be switched by relays and adding a series resistor to every relay.

The Pt1000 also has a 3-wire connection.

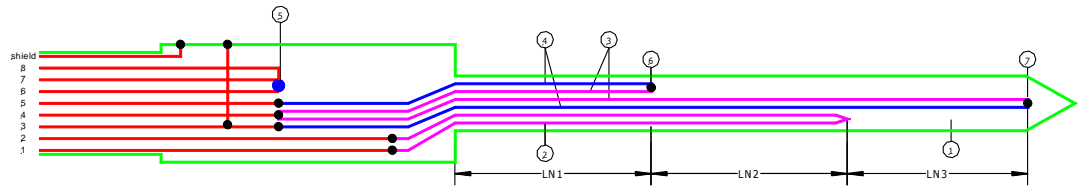
The thermocouple cold joint at the tip produces a small voltage signal. In this configuration the Pt1000 serves as a "cold junction" compensation for the cold joint thermocouple at the tip. The main differential temperature sensor (between hot- and cold joint) only needs a voltage readout, and conversion to a differential temperature by division by the sensitivity.

For formula's see the previous chapter.

Warning:

putting more than 3 Volt across the heater may result in permanent damage to the sensor.





Number	Group	Name	Colour
1	1	heater +	Black
2	1	heater -	Black
3	2	main sensor - tip thermocouple +	White
4	2	sensor central tip thermocouple -	Yellow with red sleeve
5	2	main sensor +	Yellow
6	3	Pt1000 current -	White
7	3	Pt1000 current +	Red
8	3	Pt1000 sensing	Red
Shield			Grey
Base		connected to number 3 and shield	

Figure 4.1 Wiring schedule of TP02.

1	Measurement of heater power per meter by combining the heater current measurement through wires 1 and 2 (here using a voltage measurement across a 10 Ohm resistor as an example) with the heater resistance per meter (given with each probe). Formula 3.3.1.
2	Measurement of the main sensor signal, done by measuring a voltage V_1 across wires 3 and 5, and later determining the temperature difference ΔT using the thermocouple sensitivity E_{sen} and the approximate needle/medium temperature T_{cold} . Formula 3.3.3
3	Estimate of the needle/medium temperature. This is done by using the Pt1000 temperature T_{pt1000} as an "external" cold junction compensation for the thermocouple signal of the cold junction. Cold junction voltage V_2 is measured across wires 3 and 4.

Table 4.1 The main measurements summarised



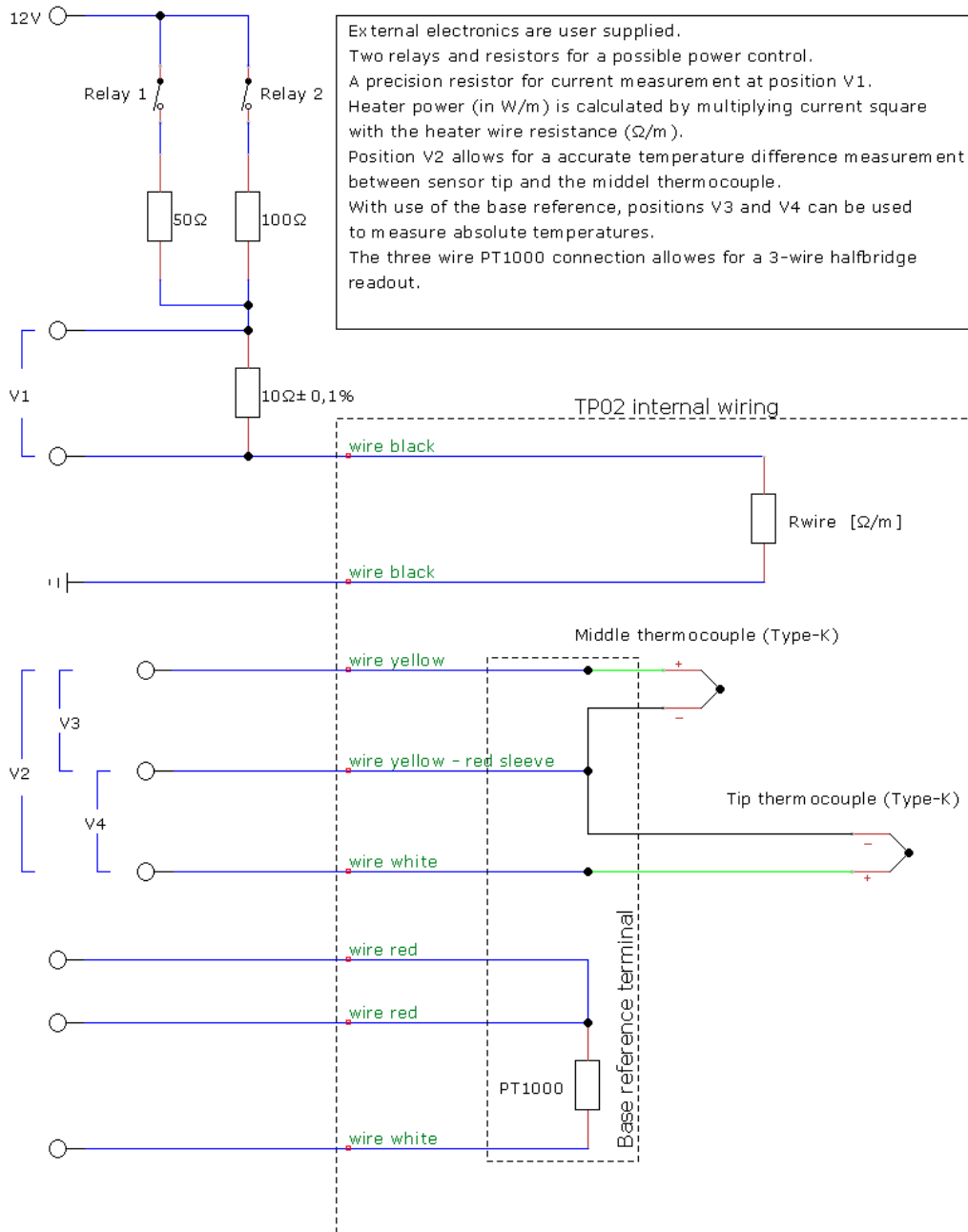


Figure 4.2 Typical wiring diagram of TP02 to a measurement and control system. Two main functions are the measurement of heater power per meter, the measurement of the differential temperature between hot and cold junction. For correction of the temperature dependence, the medium/needle temperature must be roughly estimated. The upper left shows a possible solution for switching different power levels and for performing current measurements. The 100 and 50 Ohm resistors are meant to offer variable heating power, and reduce the heater voltage to less than 3 VDC, starting at 12 VDC. This solution is not the only correct possibility.



5 Requirements for Measurement and Control

The MCU performs the measurement and control of the TP02. The evaluation is done in a spreadsheet like Excel, or in a mathematical program like Matlab.

The requirements for the MCU are summarised in the following table.

Capability to measure voltages (U_{sen} , U_{cold})	approx. -2.5 to +2.5 mV, with 0.001 mV or better accuracy. A zero offset does not influence the measurement as long as it is constant.
Capability of measuring currents I_{heat}	Approx. 0.3 A max, 0.1% accuracy, typically done with a 10 ohm shunt resistor.
Capability of switching	Max 3 volt at 0.3 A approx. For the heater, preferably with 2 relays offering 3 heating levels at 0.5 and 0.25 of the maximum.
Capability of measuring Pt1000	± 3 degrees accuracy
Capability of using external cold junction compensation	Use the Pt1000 value as cold junction compensation for the cold joint temperature measurement.
Capability of timing the measurement	The measurement timing and timing of the switching should be accurate to within 0.01 seconds.
Requirements for power supply of the heater	Capability to supply 3 Volt DC stable within 0.2%, at 0.3 A max. This can be achieved with any normal 12 V adapter, provided that the mains power supply is stable.
Capability for the MCU or the software.	To store the entire data set, including the time of measurement. Possibly performing calculations.

Table 8.3.1 *Requirements for measurement and control.*



6 Programming for TP02

The TP02 measurement protocol and the necessary calculations are outlined in earlier chapters. Here follows a summary of the general program

Setting up the system	Allow entering the default values	The parameters of E_{sen} (as a function of medium temperature), $R_{\text{e heat}}$, and h are entered as default values. It should be noted that these can change and should be user definable.
Before every experiment	Allow entering parameters for every measurement	Like sample number, experiment number etc
	Allow choosing the heating cycle time	The cycle time can vary between 100 and 1000 seconds.
	Allow selection of the heater power	Typically there is a choice of 3 levels. With one voltage supply, these can be derived using 2 resistors and 2 relays (see electrical connection).
$t = -h$	Wait for a period h to allow the probe to adjust to the medium temperature. Measure $U_{\text{sen } 0}$. Store these data at a certain interval. Determine T . Use T to correct E_{sen}	

Table 5.1 *Typical ingredients of a program for measurement and control of THA01. (continued on next page)*



After a period h	Measure $U_{sen\ h}$ and $I_{heat\ h}$	If the temperature gradients through the medium are zero and the electronics are perfect, this signal will be equal to zero. In practice, it will have a value different from zero.
	Store $U_{sen\ h}$ and $I_{heat\ h}$ and T_h (t = h seconds)	
t = 0	Switch heater on. This must be accurately timed.	At t = 0, the zero reading is taken. After this, the heater is switched on.
At least every 10 seconds	Measure the current $I_{heat\ t}$ and $U_{sen\ t}$	Optional quality assurance: see if the signal rises continuously.
	Calculate the heater power using $R_{e\ heat}$	
t = h	Switch off the heater	
	If the MCU allows this, the end result can be calculated, and quality checks can be made, and even the measurement result can be calculated.	See the appendix on this subject for quality assurance. For analysis of the measurement data a common approach is to calculate the signal gradient over a series of intervals. This can be from 0.5h to h, from 0.6h to h, from 0.7h to h. The standard deviation of these gradients offers an automatic way to see if the transient period is over or not. The gradient before the measurement and the temperature are used for correction.

Table 5.1 *Typical ingredients of a program for measurement and control of THA01. (started on previous page)*



7 Maintenance of TP02

TP02 does not need a lot of maintenance. After use the equipment can be cleaned using water. Usually errors in functionality will appear as unreasonably large or small measured values. As a general rule, this means that a critical review of the measured data is the best form of maintenance. At regular intervals the quality of the cables can be checked. On a yearly interval the calibration can be checked.

Every 1 year	Perform on site calibration of TP02 using a Calibration Reference Cylinder, or low cost alternative such as agar gel or glycerol
Every 2 years	Perform calibration of the MCU.

Table 6.1 *recommended schedule for calibration of TP02 and MCU*



8 Delivery and spare parts

TP02 delivery includes the following items:

TP02
Protection tube

Manual TP02
Calibration form for TP02

The TP02 delivery does NOT include the MCU.

TP02 can be delivered with extended cable. However, the delivery time of units with cable extension is significantly longer than that of standard sensors.



9 Appendices

9.1 Appendix on modelling TP02 behaviour

Modelling a finite line source is the subject of many scientific publications. Various efforts have been made to estimate errors and to improve the model such that a more accurate measurement can be attained.

The analytical solution to the problem is known. This involves the fact that the probe has a certain geometry, no significant conduction along the probe itself and has different thermal properties than the medium. Also it assumes that there is a certain constant contact resistance between the probe and medium. The equation is given in Kosky and McVey.

On the other hand in most applications the ideal model, without considering probe thermal properties and contact resistance is used:

$$\Delta T = (Q / 4 \pi \lambda) (\ln t + B) \quad 8.1.1$$

It turns out that also if contact resistance and different thermal parameters are involved, the long time solution of the analytical model and the ideal model lead to the same result for λ . The only difference is that B is larger in the analytical model because of the contact resistance. In other words, the effects of the probe thermal properties and contact resistance are no longer visible some time after the heating has started. Because B cancels from the equation for determination of λ , 1.2 is still applicable.

$$\lambda = (Q / 4 \pi \Delta T) \ln(t_2 / t_1) \quad 8.1.2$$

The normal transient period under ideal conditions is:

$$t_{\text{transient}} = 10 D^2 / a \quad 8.1.3$$

Under ideal conditions this means that the transient time is proportional to the medium thermal diffusivity a , and the probe cross section (D is the diameter). The only consequence of the addition of contact resistance and probe thermal properties, is that the transient period will take longer.



The remaining measurement error has been analysed by various authors. Conclusions are that the main parameters determining the transient period are R_{cont} , the contact resistance, and $C_{v, \text{needle}}/C_{v, \text{med}}$, the ratio of the volumetric heat capacities. The higher the contact resistance and the lower the medium volumetric heat capacity, the longer the transient time.

Theoretically, it would be best to take the measurement time of more than 10 minutes. This is not possible because of three reasons:

1. the temperature rise becomes too small relative to the temperature changes induced by outside sources.
2. there is a risk that the boundary conditions of the sample start playing a role.
3. there is a risk that the total energy that is released into the medium becomes so large that the condition of the medium is affected.

Various attempts have been made to simplify the measurement procedure. There has been limited success. The general recipe now is: take a look at the contact resistance. If a large contact resistance is expected, and the medium allows the use of contact fluid or grease (like toothpaste, agar gel, glycerol or Vaseline) can be considered. This is often applied when analysing rock samples.

After this, consider the medium volumetric heat capacity (see the table on thermal properties). This table clearly shows that the materials that have a very low volumetric heat capacity are those with a large porosity, containing air. In practise these materials also have a very low thermal conductivity, as well as a fairly normal thermal diffusivity. It is the heat capacity that forces to use very long measurement times. However, the thermal diffusivity is such that we would also need very large samples. This combination, and not the low thermal conductivity as such, makes these materials unsuitable for analysis by NSSP methods.

In practise, the limitation of the measurement range from 0.1 to 6 W/mK prevents us from making serious measurement errors.

This means that for all practical purposes for the measurement of thermal conductivity's the model of formula 1.2 can be used.

Regarding the accuracy of the measurement; the estimate of $\pm (3\%+0.02)$ W/mK is derived from various literature sources. For more details, see the list of references.





In case of measurements at low thermal conductivities (that is in the range of 0.1 W/mK or lower) one must realise that the measurement accuracy is not very high. The accuracy specification is $\pm (3\% + 0.02 \text{ W/mK})$. In case of 0.1 W/mK, this already is $\pm 23\%$. This has to do with the fact that the heat capacity of such materials typically is low as well, which affects the measurement.

9.2 Appendix on the accuracy of TP02 over its entire temperature range

Note: symbols used here do not comply with the list of symbols. Explanations are found in the accompanying text. The probe properties vary with the probe temperature. There are two factors of importance:

1 the temperature dependence of the sensitivity of the thermocouples. As the temperature dependence of type K thermocouples is well known, this can be compensated for. Using the Pt1000 and the reading of the cold joint, we have sufficient information, and the error can be limited to within $\pm 0.02\%/K$ (leading to an absolute error of about $\pm 2\%$ at 100 degrees C. If a probe would have no real temperature reading, the measurement accuracy will be reduced by about $0.25\%/K$ for an average thermocouple, leading to an error of around 20% under similar conditions.

2 the noise and range errors of the amplifier. When measuring a sensor signal, the accuracy can be expressed as a zero-offset, Z , (describing an offset at the output for zero input), a noise, N , (describing fluctuations of the output at constant input, often called resolution because it also describes the smallest change that can be detected with significance) and a range error, R , (describing the error in amplification, often expressed as a percentage of the reading). All these can be temperature dependent, but we assume that the amplifier is in a stable environment, and only the probe is subjected to extreme temperatures.





Assuming that the signal before the heating phase is U_0 , at some time t during the heating phase it is U_t , all expressed in mV. The accuracy A with which the difference between U_t and U_0 can be determined is:

$$A = 0.5 (U_t + U_0) R\% + 2N \qquad 8.12.1$$

The zero offset is included in the measurement signal before the measurement and automatically cancels. This assumption is valid as long as Z is stable and a relatively small compared to U_t and U_0 (which it usually is).

The table below shows a comparison of two sensors. The conclusion is that by using a design like TP02, the signal stays closer to zero, and the measurement accuracy will be much higher.

	design with one temperature sensor like TP08	design with a differential measurement (2 sensors) like TP02
Medium temperature	100 °C	100 °C
Measurement signal $U_t - U_0$	0.1 mV	0.1 mV
Signal before measurement U_0	3.2 mV	0 mV
Amplifier range	10 mV	2.5 mV
Range error $0.5 (U_t + U_0) 0.05\%$	0.002 mV	0.00005 mV
Noise (varies with range) (2N)	0.001 mV	0.0003 mV
Resulting error by amplification	3%	0.35%

Table 8.13.1 *calculation of the error caused by amplification when working at extreme temperatures. Comparison between a probe with one joint and a probe with two thermocouple joints. In general the two-thermocouple joint probe is significantly more accurate at temperatures from -10 outside the range of +40 degrees C.*

In case of dynamic temperature changes (not static as above), the two junction design has the advantage that the effects of changes in temperature are suppressed. In the ideal situation the error that would occur with one junction designs is perfectly compensated; Both junctions are equally affected, and the resulting differential signal is zero.





9.3 Appendix on thermal diffusivity and heat capacity measurement

Concerning thermal diffusivity (or heat capacity) measurement:

Short answer:

Hukseflux recommend not to use needles for thermal diffusivity measurement. The result will be quite unreliable. (estimated +/- 50% in lab conditions, worse in the field). If required it is possible to put some extra lines in the software for estimation. Hukseflux recommend to use calculations based on literature values and moisture content measurement.

Long answer:

All these probes are primarily designed for the measurement of soil thermal conductivity. The measurement of soil heat capacity (or thermal diffusivity, which can be calculated from capacity and thermal conductivity) is extremely difficult. Although some probe publications claim successful high accuracy heat capacity measurements, at Hukseflux we have not been able to see substantial evidence of these claims. In case of TP01 the accuracy of this measurement is specified, but with a very low accuracy (+/-20%). It is expected that in steady installation, the relative accuracy is better than this, so that it is possible to follow trends. For more details on this measurement, see the TP01 manual. In case of TP01, which is typically installed in the field, in long term monitoring, the information of the trend of the thermal conductivity is useful anyway, because this offers information on the soil water content.

The determination of soil heat capacity with TP02 and TP08 has been tried by several scientists, but, in our personal opinion, can only be measured when great care is taken. In soils, in particular in field experiments the contact resistance can vary and can be quite high. This contact resistance however is of very large significance to the estimate of the soil heat capacity. In fact it ruins the measurement (which is based on the analysis of the rise time of the signal) and the end result will be a mix of the loose soil around the probe and the solid soil further away. (for the thermal conductivity measurement this is no problem as this is based upon the measurement after longer times).

The accuracy claims for the volumetric heat capacity measurements (and in fact also for the lower part of the thermal conductivity measurement range) are typically based upon low contact resistance values, in other words on "perfect lab experiments with perfectly and homogeneously compacted soils".





Although Hukseflux can put the same mathematical procedure in the analysis software, it prefers not to do this, unless the customer specifically asks for this.

It also is possible, using the stored measurement data to perform the same determination of heat capacity or thermal diffusivity during post-processing of the data.

Hukseflux recommendation is to estimate heat capacity by using textbook values for the dry soil, and by adding to that the heat capacity of the water in the soil. The water content can be estimated by weighing the sample before and after drying out.

In case the measurement of thermal diffusivity or heat capacity is absolutely necessary, we recommend to use TP01 for this purpose. Please read the TP01 manual before purchasing TP01.

9.4 Appendix on the use of TP02 in fluids

Regarding the application of TP02/ TP08 in liquids: This can be found out only by trial and error. The general idea is that with fluids heating must be as low as possible, otherwise there will be thermally induced flow. The TP02 in combination with the CR1000 (also employed in TPSYS) is a good choice in that respect; the CR1000 has a 0.3 microvolt accuracy, so you do not have to heat a lot to get a good signal. Whether or not a measurement is OK fortunately is visible in the graph of the signal versus the natural logarithm of time. Any convection will be apparent by non-linear behaviour (log time graph). On a more detailed level: the important parameters are coefficient of thermal expansion and viscosity (actually the ratio of these two). To give an idea: measurement in water cannot be carried out successfully. Hukseflux applies a trick to do it anyhow, turning the water into a gel (using agar gel) thus immobilising the fluid (no more convection). By using only a little gel (about 0.5 to 2 percent by weight, it can safely be assumed that the thermal conductivity is not influenced by the addition of the gel. Successful measurements have been performed (without adding any gel) in paints, oils and viscous fluids like glycerol.

In case there is hesitation to follow the trial and error approach, a trial sample can be analysed free of charge at Hukseflux.





9.5 Appendix on soil thermal conductivity versus water content

In case the soil thermal conductivity of a soil type must be measured as a function of water content: The main points of interest will be the completely dry state and the saturated state (at the field density).

Typically the procedure is as follows:

- 1 measurement in the field, establishing a in-situ thermal conductivity.
- 2 taking a sample in the field. Establishing a semi-in situ density. Note: the volume should be taken down as well as the weight.
- 3 taking the field sample to the laboratory.
- 4 recommended: inserting a guiding tube (obligatory with cements and bentonites).
- 5 re-compacting to the field density. (verification of weight). IMPORTANT NOTE: if possible mix the soil before compacting. The risk of having inhomogeneous samples is typically high.
- 6 taking a thermal conductivity measurement.
- 7 drying out in an oven (at 120 degrees C, typically 2 hours)
- 8 weighing and determining the water content in the field
- 9 taking a thermal conductivity measurement. (100% dry)
- 10 saturating the sample (keeping soil density intact)
- 11 taking a thermal conductivity measurement (saturated)
- 12 plotting a curve
- 13 comparing the curve to expected values.

It typically is quite difficult to measure at moisture content levels between the dry and saturated state. The reason for this is that the homogeneity of the moisture content is both critical and hard to achieve.

Please note that it is assumed that samples are homogeneous and grain sizes are in the order of magnitude of millimetres, and that samples are delivered in holders of around 150mm deep and at least 80 mm diameter, with a guiding tube inserted. In case of saturation: in case this is not possible at the end of the experiment (for practical reasons, for example with pure bentonites this takes too long), it is suggested to supply samples in a saturated state. Guiding tubes can be obtained at Hukseflux free of charge.

In case a comparison to other soils is necessary it is suggested to include reference samples in the experiment, and not to rely on literature values.





9.6 Appendix on insertion of the needle into hard media

During the measurement, the TP02 needle should be completely in contact with the medium. It should be in a perfectly static position, with no possibility of movement. Usually this is achieved by fixation of the cable.

In case of soft media the needle often can be inserted without any particular trouble. In case of hard media like hard soil, a hole may be pre-drilled.

There are several possibilities for pre-drilling:

<p>Use so-called guiding tubes. See separate documentation on the GT series. The heating cycle time must be increased. The measurement accuracy can be maintained using careful visual checks of the data.</p>	
<p>In case of modestly hard media: 1.5 mm diameter pins can be used to prepare the path for the TP02 needle. Normally the heating cycle time can remain unchanged. The measurement accuracy will not be affected.</p>	<p>To improve contact between needle and medium, the medium around the TP02 needle is typically compressed manually. The degree of compression should be no more than that of the undisturbed medium.</p>
<p>In case of hard media: Pre-drilling using a small diameter drill or metal pin is possible. The smaller the drill or pin diameter, the better. A diameter of up to 6 mm is recommended, up to 1 cm is allowable in extreme cases. The heating cycle time must be increased. The measurement accuracy can be maintained using careful visual checks of the data.</p>	<p>In this case a hole is created that has a significantly larger diameter than that of the TP02 needle. In this case the use of contact material is necessary.</p>

Table 8.5.1 *Possibilities for working in hard media: guiding tubes and pre-drilling*





Using a large diameter metal pin or a drill essentially increases the TP02 needle diameter. As the normal TP02 diameter is very small, this is not a fundamental problem. However, in order to make sure that the contents of the hole do not play a role in the measurement, three measures need to be taken

<p>After inserting the TP02 needle the hole must be filled with contact material. This can either be the original medium, now loose, or a gel (solidified agar) or paste (any white toothpaste or silicone glue). In any case the presence of fluid materials that might have thermal convection, in the hole must be avoided.</p>	<p>Loose medium can be compressed as much as possible around the TP02 probe by hand. Agar gel can only be used in saturated soils, because it typically loses some water. For dry or semi-moist media, toothpaste is preferred over silicone glue because of its relatively high thermal conductivity.</p>
<p>The heating cycle duration is increased from the usual 100 with the square of the diameter to a maximum of approximately 300 s.</p>	<p>This is to make sure that the heat generated by the TP02 extends well beyond the diameter of the hole</p>

Table 8.5.2 *Measures taken after pre-drilling with a small diameter drill or with a metal pin with a larger diameter than the probe.*





9.7 Appendix on guiding tubes

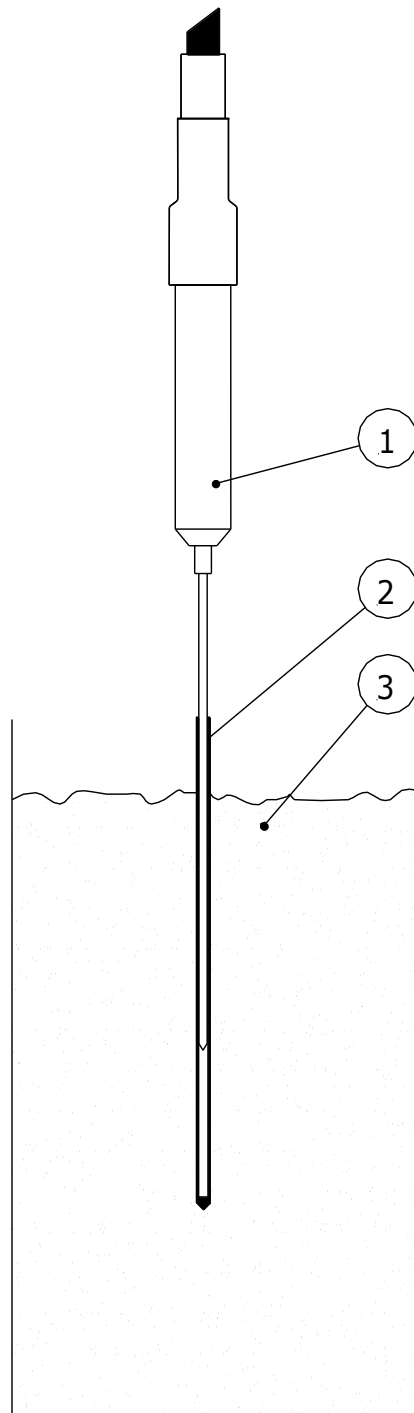


Figure 8.14.1 *Non-Steady-State Probe. The needle (1) is inserted into a guiding tube (2), typically in a solid sample (3).*

The GT series guiding tubes are accessories for Non-Steady-State Needle probes. They are intended to facilitate measurements in hard soils, concrete, cement and bentonite.





Also in case of measurements on large quantities of samples one can use several guiding tubes and one single needle.

A recurring problem is that insertion into hard soils, bentonites and cements is quite difficult.

The main problem is that it is quite difficult to insert needles into these hard materials.

The use of guiding tubes in many cases can solve this problem; The guiding tube should be inserted into the medium, and the needle can be inserted at a later stage.

Hukseflux is a leading supplier of Non-Steady-State Probes, and is the inventor of this guiding tube technology.

In case of bentonites and cement the guiding tubes are cast into the material and experiments can be carried out later

In case of hard soils, a typical approach is to pre-drill a hole, insert the guiding tube, compact the soil again, and perform a measurement.

In case of dry out experiments guiding tubes are inserted into the samples, dry out can then take place at high temperature (the needle is not inserted).





It has been verified that the addition of a guiding tube does not have a significant impact on the measurement accuracy; when inserting a needle into a guiding tube, essentially a larger diameter needle is created. This results in a larger transient time; one has to wait longer before the desired linear behaviour (of temperature versus the logarithm of time) is occurring. This is normal behaviour for thicker needles.

In case it is possible, it is recommended to add a small quantity of glycerol into the guiding tube (before inserting the needle) to minimise the contact resistance. This improves the repeatability of the measurement. One should however be careful not to pollute the soil with the glycerol.

Type	Dimensions	Suitability for needle types
GT01	L = 90 OD = 2	TP08
GT02	L = 170 OD = 2.38	TP02
GT03	L = 150 OD = 4.50	TP07

Table 8.14.1: *GT guiding tube types and their application.*

Directions for use:

- 1 put the GT into the medium
- 2 cast or compact as much as possible
- 3 remove the cap
- 4 (optional) put a small quantity of glycerol into the GT. Make sure not to spill glycerol on the soil.
- 5 put the needle into the GT
- 6 perform an experiment however increase the heating time by 50% relative to the normal situation
- 7 remove the needle
- 8 put the cap onto the GT
- 9 if applicable: remove the GT from the sample (in many cases the GT can be re-used).





9.8 Appendix on ASTM and IEEE standards

With TP02 it is possible to perform measurements in accordance with the ASTM and IEEE standards. These standards are:

ASTM D5334-14 Standard Test Method for Determination of Thermal Conductivity of Soil and Soft Rock by Thermal Needle Probe Procedure and ASTM D5930-97 Standard Test Method for Thermal Conductivity of Plastics by Means of a Transient Line Source Technique.

Both standards can be obtained from ASTM. For information see <http://www.astm.org>.

IEEE Std 442-1981, IEEE Guide for soil Thermal Resistivity Measurements.

Resistivity is the inverse of the conductivity. This parameter is not used in the text of this manual.

The IEEE standard can be obtained from IEEE. For information see <http://www.ieee.org>.

For full compliance with ASTM, it is necessary to follow the procedures described in the respective standards. Hukseflux suggests allowing slightly modified specifications. The described deviations are positively affecting the quality and practicality of the measurement, and altogether compliance with the standards can be stated when following the recommendations in this manual in addition to those in the standards.

Based on practical grounds, the ASTM D 5930-97 calibration against fused silica is replaced by calibration against a Calibration Reference Cylinder (basically a specially prepared amorphous plastic) or low cost alternatives such as agar gel or glycerol. On physical grounds most importantly, the application is not limited to soil, soft rock and plastics.

Hukseflux objects to the IEEE requirement of extension of the experiment to 1000 seconds (around 15 minutes) and recommends heating times of 100 to 300 seconds. In a lot of cases, the 1000 s requirement will actually lead to unreliable measurement results.





The reasons are summarised below:

1 for an accurate measurement, one needs to heat as little as possible; heating at a level of more than 10 W/m will soon lead to local evaporation of water

2 for an accurate measurement, one needs to heat as short as possible; heating for more than 300 seconds will soon lead to local evaporation of water.

3 for an accurate measurement, one needs to heat as short as possible; heating for more than 300 seconds will soon lead to local evaporation of water. The longer the time the higher the eventual needle temperature

4 for an accurate measurement, one needs to heat as short as possible; heating for more than 300 seconds will soon lead to undetectable small changes in temperature; the effects of ambient temperature changes will grow larger, the more time passes by.

4 for an accurate measurement, one needs to heat as short as possible; heating for more than 300 seconds will soon lead to errors due to the heat wave hitting the sample outer dimensions. As a rule of thumb, the sample radius to the power of two must be smaller than 10 times the heating time times the thermal diffusivity. For a 1000 second heating time, this means that a dry sand sample needs a radius of 50 mm, a wet sand sample of 100 mm.

At Hukseflux the measurement time of 1000 s is replaced by a heating cycle time of 100 seconds (measurement time of 200s) and the temperature range is extended from "20 to 100" to "-55 to +180". In comparison to ASTM D5334-14, again the temperature range is different. Also the thermal conductivity range is not applicable.





Again the calibration medium is changed on practical grounds. The recommended sample radius of ASTM D5334-14 is judged to be too small and could lead to serious errors.

Conclusion: For compliance with ASTM standards, the procedure of the respective standard must be followed with a few, well motivated exceptions.

The IEEE standard is specifically written for application in the power industry, for estimating thermal budgets of electrical cables. There is a clear distinction between field measurements and laboratory measurements. In general TP02 will be used in laboratory situations. Hukseflux can offer alternative heavy duty needle designs for field use.



9.9 Appendix on typical thermal properties

	Thermal conductivity @20°C W/mK	Density @20°C Kg/m ³	Volumetric heat capacity @20°C 10 ⁶ J/m ³	Thermal diffusivity @20°C 10 ⁻⁸ m ² /s
Air	0.025	1.29	0.001	1938
Glycerol	0.29	1260	3.073	9
Water	0.6	1000	4.180	14
Ice	2.1	917	2.017	104
Olive oil	0.17	920	1.650	10
Gasoline	0.15	720	2.100	7
Methanol	0.21	790	2.500	8
Silicone oil	0.1	760	1.370	7
Alcohol	0.17	800	2.430	7
Aluminium	237	2700	2.376	9975
Copper	390	8960	3.494	11161
Stainless Steel	16	7900	3.950	405
Aluminium Oxide	30	3900	3.413	879
Quartz	3	2600	2.130	141
Concrete	1.28	2200	1.940	66
Marble	3	2700	2.376	126
Glass	0.93	2600	2.184	43
Pyrex 7740	1.005	2230	1.681	60
PVC	0.16	1300	1.950	8
PE	0.25	2200	2.046	12
PTFE	0.25	2200	2.200	11
Nylon 6	0.25	1140	1.938	13
Corian (ceramic filled)	1.06	1800	2.307	46

Table 8.6.1 A list of typical values of thermal properties of various materials. This list is only indicative and can serve for estimating the medium thermal conductivity. (continued on the next page)



	Thermal conductivity @20°C	Density @20°C	Volumetric heat capacity @20°C	Thermal diffusivity @20°C
	W/mK	Kg/m ³	10 ⁶ J/m ³	10 ⁻⁸ m ² /s
Wood	0.4	780	0.187	214
Cotton	0.03		0.001	
Leather	0.14		0.001	59
Cork	0.07	200	0.047	150
Foam glass	0.045	120	0.092	49
Mineral insulation materials	0.04	100	0.090	44
Plastic insulation materials	0.03	50	0.100	30
Sand (dry)	0.35	1600	1.270	28
Sand (saturated)	2.7	2100	2.640	102
Glass pearls (dry)	0.18	1800	1.140	16
Glass pearls (saturated)	0.76	2100	2.710	28

Table 8.6.1 A list of typical values of thermal properties of various materials. This list is only indicative and can serve for estimating the medium thermal conductivity. (started on the previous page)

Range of all reported values for soil	0.15 to 4
Saturated soil	0.6 to 4
Sand perfectly dry	0.15 to 0.25
Sand moist	0.25 to 2
Sand saturated	2 to 4
Clay dry to moist	0.15 to 1.8
Clay saturated	0.6 to 2.5
Soil with organic matter	0.15 to 2
Solid Rocks	2 to 7
Tuff (porous volcanic rock)	0.5 to 2.5

Table 8.6.2 Reported values, as known to the author, of thermal conductivity in different soil types in W/mK.





9.10 Appendix on TP02 calibration

TP02 provides an absolute measurement. What is meant by this is that the measurements done in a TP02 in order to derive the thermal conductivity (Q , T , and ΔT) are all direct measurements of temperature and power. This is contrary to relative methods that require reference materials. The instrument traceability is to length, voltage and temperature. The traceability to temperature is via ANSI MC96.1-1882.

The TP02 characteristics essentially are determined by the characteristics of the thermocouples, the heater and the Pt1000. These are considered to be known for every TP02. Apart from the heater resistance per meter, they are the same for every TP02. During production proper system performance is checked by measuring the thermal properties of agar gel at 20 degrees C.

Calibration of TP02 can be done in any laboratory that has the necessary electronic equipment. The requirements for power supply and readout can be found in the chapter on MCU requirements.

The procedure for calibration is as follows:

There should be perfect contact between the needle and the gel. One can perform a calibration by doing a normal measurement, in agar gel. Knowing the thermal properties of the gel, the deviation from perfect behaviour can be calculated.

When deviating within ($6\%+0.04$ W/mK), twice the accuracy, (plus if performed at temperatures different from 20 degrees C), it is suggested not to change the calibration information. If the deviation of the measurement is outside the range mentioned above, it is recommended to adapt the calibration of the heater resistance accordingly, and leave the thermocouple sensitivity unchanged.

If the calculated thermal conductivity is lower than expected by a certain percentage, the apparent output is too high relative to the apparent heating power. This can be corrected by assuming that a higher power has been applied, which is achieved by increasing the heater resistance in Ohm/m by the same percentage.

If possible, the calibration can be performed at different temperatures.



9.11 Appendix on CRC series calibration reference cylinders

Calibration Reference Cylinders or CRC's contain traceable thermal conductivity reference materials for calibration of Non-Steady-State Probes. There are several different CRC types. The geometry of the different types is adapted to fit needles with a certain diameter.

The hart of the CRC cylinders is a specially manufactured amorphous plastic reference material (5). Contrary to other plastics, this material does not have any directionally dependent thermal properties.

In the plastic material a hole is drilled (4), with the approximate outer dimensions (ND Needle Diameter and IL Insertion Length) of the needle that needs to be calibrated.

The plastic material is incorporated in a metal protection casing (2). A plug (7) is provided for sealing purposes.

In order to guarantee perfect contact between needle (1) and reference material (5), the metal casing is filled with a fluid, glycerol (3).

An additional hole (6) is provided in order to be able to perform checks of material stability with thin needle probes.

The user is recommended to send the CRC to Hukseflux for a check of material stability every 5 years.

Hukseflux is a leading supplier of Non-Steady-State Probes, and is the inventor of the CRC calibration method.

NOTE: As a low-cost (lower accuracy) alternative reference material Hukseflux promotes use of glycerol filled with plastic fibre material. This is delivered as a standard with most Hukseflux systems.

Test method:	ASTM D5334-14 and D5930-97, IEEE Std 442-1981
Temperature range:	0 to 60 degrees C
Thermal conductivity of reference:	0.2 W/mK (nominal value)
Traceability:	to NPL National Physical Laboratory, United Kingdom

Table: 8.16.1 *specifications of the CRC series*

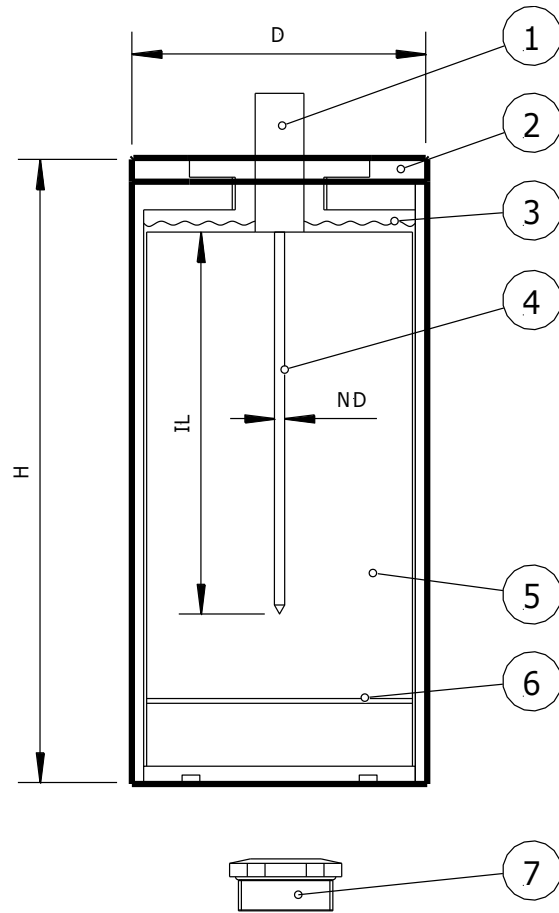


Figure 8.16.1: generalised picture of CRC Calibration Reference Cylinder. Explanation can be found in the text.

Type	Dimensions	Suitability for needle types
CRC01	H = 260 D = 100 IL = 185 - 30 ND = 1.6	TP02, TP08
CRC04	H = 206 D = 100 IL = 137 - 6 ND = 3.6	TP07
CRC05	H = 260 D = 100 IL = 190 - 12 ND = 6.6	TP09

Table 8.16.2: CRC cylinder types and their application





9.12 Appendix on agar gel reference medium

The procedure for calibration relies on the use of agar gel. This is a water based gel, of which the ingredients can be bought in every pharmacy.

The agar gel is often use for growing bacteria.

The agar itself does not significantly influence the thermal properties of water, but eliminates the effects of convection.

The properties of agar gel at 0.4 mass % closely resemble those of water:

Thermal conductivity: 0.60 W/mK, at 20 degrees C.

Thermal conductivity: 0.57 W/mK, at 0 degrees C.

Thermal diffusivity: $0.14 \cdot 10^{-6} \text{ m}^2 / \text{s}$

Temperature dependence of the thermal conductivity: 0.0015 W/mK/°C.

Temperature range that agar gel can be used: 5 to 80 degrees C.

Generally preparation of agar gel can be done by cooking about 4 grams of agar in 1 litre of water, for about 20 minutes, stirring regularly. The solution can be put in a pot, and be allowed to cool down and solidify. This typically takes some hours. Once at room temperature, the TP02 can be inserted into the agar gel.

9.13 Appendix on glycerol reference medium

Glycerol also called Glycerin or Glycerine or Glycol is a reasonably safe fluid. It can be obtained at any pharmacy or chemical trading company.

Glycerol can be used for calibration purposes, provided that it is combined with fibrous material to stop it from flowing (by thermal convection because of heating of the needle, or by mechanically induced convection).

Use of glycerol has advantages over use of agar gel:

- low preparation time
- no degradation of the material

Use of glycerol has the disadvantages that:

- glycerol is not a standardised material
- glycerol with fibres is still less recognised as a standard material





The suggested recipe is to use a 1.5 litre bottle, fill it with fibre material (say 40 grams), and then fill it up with glycerol. The recommended fibre material is Polyester (PETP or PET) fibre, which is often used as a staple filler for upholstery and pillows. Brand names are Dacron, Terylene and Trevira. The thermal conductivity of glycerol is 0.29 W/mK, that of polyester fibres 0.24 W/mK, so that it is feasible that even if the fibres are hollow, the bulk thermal conductivity of the glycerol is not significantly influenced. (40 grams fibre on 1.5 litre glycerol, would amount to around 2% by mass).

9.14 Appendix on cable extension and connectors for TP02

Warning: It is a general recommendation to keep the distance between MCU and sensor as short as possible and to avoid using connectors or soldered joints whenever possible.

Warning: PTFE cable cannot properly be glued, and therefore connections using shrunk covers cannot be considered waterproof. Also the temperature specification of

The standard sensor has a cable length of 2.5 meters. Cables generally act as a source of distortion by picking up capacitive noise. However, if necessary TP02 cable can be extended. Done properly, the sensor signal, although small, will not degrade, because the sensor impedance is very low.



Cable and connection specifications are summarised below.

Cable	8-wire shielded, copper core
Core resistance	0.1 Ω /m or lower
Outer diameter	(preferred) 5 mm
Outer sheet	(preferred) PTFE. Other materials like PVC and polyurethane are also applicable, depending on the application.
Connection	It is essential that the connection does not generate fluctuations in the sensor signal, and that there is no heater current flowing away. Either solder the new cable core and shield to the original sensor cable, and make a semi-waterproof connection, or use gold plated waterproof connectors. In both cases it is recommended to have significant thermal mass, preferably well-conducting, to make the joints thermally stable. Please mind that zero offsets do not affect the measurement as long as they are stable during the measurement heating cycle.

Table 8.9.1 *Specifications for cable extension of TP02*

The connectors that are used by Hukseflux for TP02 are manufactured by WW Fischer.

In order to protect the TP02 cable, the cable is first covered by a sleeve. The sleeve typically is installed by thermal shrinkage.

Type	Code
Normal male connector to TP02 cable	S 104 A055-130 with internal part E3 104.2/6.7 + B
Normal female chassis part	DEE 104 A055-130 with 105.680 grounding washer (for soldering shield) and possibly with 104.551 spacer 6 mm (for attachment to a thin wall)
Female connector for extension cable	K 104 A055-130 with internal part E3 104.2/6.7 + B

Table 8.9.2 *Connectors for use with TP02. Please note that the TP02 cable must be covered with a sleeve for protection.*



9.15 Appendix on comparing 1-needle to 2-needle techniques

An alternative to the single needle of TP02 are systems using two needles. Both principles / systems have been used in various scientific experiments. Here is a comparison.

	1 needle (TP02)	2 needle
Measurements	Thermal conductivity only	Thermal conductivity and thermal diffusivity
Accuracy	Thermal conductivity: $\pm (3\%+0.02 \text{ W/mK})$ @ 20 °C	Thermal conductivity: unspecified, typically lower than single needle because of sensor base is in the thermal flow field. Thermal diffusivity: unspecified, typically $\pm 30\%$ attainable only with dedicated signal processing
Reliability of the measurement	Insensitive to bending. Reliable with proper data review.	The thermal diffusivity measurement is sensitive to bending because the distance between the needles is a critical parameter. Data review does not provide a fail safe check.
Suitability for repeated insertion into various media	Suitable	Not recommended because of the vulnerability of the needles.
Long term stability	Good provided that the sensor has a sealed construction	Undetermined because of possible bending of the needle.

Table 8.10.1 comparison of single-needle (like TP02) to dual-needle techniques (continued on the next page)



Standardisation	Standardised by ASTM	Not standardised
Measurement range	0.1 to 6 W/mK	Typically 0.1 to 2 W/mK. This is too low for general soil analysis, reasonable for foodstuff.
Data processing	Relatively simple analysis of the slope of the curve versus $\ln(t)$. Can be performed in any spreadsheet.	Thermal diffusivity analysis cannot be expected to be accurate without dedicated processing.

Table 8.10.1 *comparison of single-needle (like TP02) to dual-needle techniques (started on the previous page).*

9.16 Appendix on trouble shooting

This paragraph contains information that can be used to make a diagnosis whenever the TP02 does not function.

It is recommended to start any kind of trouble shooting with a simple check of the sensor and heater impedance, and a check to see if the thermocouples give a signal.

This test is described in the chapter on putting TP02 into operation. We now have confirmed that the connections are OK and that the sensor is still functioning.

If this check does not produce any outcome, proceed to the next table.

No signal from the sensor	Check the main sensor impedance as in the table below.
	Check the MCU by applying an artificially generated voltage to the input. Preferably a millivolt generator is used for this purpose.
	Check the heater impedance (see table below)
	Check the functionality of the heater by putting it on. A simple 1.5 V battery can be used. When it is on, check the sensor output.

Table 8.11.1 *Extensive checklist for trouble shooting. (continued on the next page)*



	Check the sensor connection / wiring measuring all resistance values according to table 8.11.2
Signal too high or too low	Check the MCU by applying an artificially generated voltage to the input. Preferably a millivolt generator is used for this purpose.
	Check the zero level of the data acquisition system by putting a 50 ohm resistor in place of the main sensor. The data acquisition system should read less than 20 microvolt and be stable within 5 microvolt. Now put the heater on with still the resistor and not the sensor connected. The signal should not react to this by more than 10 microvolt. If there is a larger reaction, there is a ground loop from the heater to the sensor. Check the electrical connection.
	Check if the data acquisition system has sufficient sensitivity. This should be in the microvolt range.
Signal shows unexpected variations	Check is there are no large currents in your system which can cause a ground loop. If these are there, switch them off, and see if any of these is causing the disturbance.
	Check if the probe position is fixed.
	Check if the connection between medium and probe is tight.
	Check the surroundings for large sources of electromagnetic radiation. Radar installations, microwave emitters, etc.
	Inspect the sensor itself.

Table 8.11.1 Extensive checklist for trouble shooting. (started on the previous page)



	Total resistance between wires	Value TP02
1	1 - 2 (heater)	9-11 Ohm
2	3 - 4 (cold joint)	40- 50 Ohm
3	4 - 5 (hot joint)	20- 26 Ohm
4	3 - shield	0,1- 1 Ohm
5	3 - 5 (main sensor)	60 to 80 Ohm
6	1- shield	Infinite
7	7 - 8 (Pt1000)	0,1- 1 Ohm
8	6 - 7 and 6 - 8 (Pt1000)	1000-1200 Ohm
9	7 - shield and 8 - shield	Infinite
10	Information on the cable should be the same as engraving on the probe.	

Table 8.11.2 *checklist for resistances.*



9.17 Appendix with measurement form / test report

For an example of a measurement form, see the next page





Thermal Conductivity Test Report

Experiment

Experiment date	D/M/Y
Experiment time	Hour/ min
Check instrument date & time	OK
Experiment number	#
Instrumentation identifier	#

Sample

Sample number	#
Location identifier/ co-ordinates	
Measurement / sample depth	m

Soil physical description (sand, clay, silt, homogeneity)	
Water content	Vol%
Density	Kg/m ³
Dry density	Kg/m ³

Results

Thermal Conductivity (W/mK)	W/mK
Standard deviation (W/mK)	W/mK
Check Standard Deviation < 10% Thermal Conductivity	OK
Filename for storage	

Further analysis required	Yes / No
Signature	
Name	Name
Date	Date





9.18 Appendix on literature references

The following literature gives a good overview of the Non-Steady-State Probe measurement technique.

Application of Parameter Estimation Techniques to Thermal Conductivity Probe Data Reduction, Koski, J. A. , McVey, D. F., Thermal Conductivity 17, Plenum Press New York, 1986, pages 587-600.

Determination of the Thermal Conductivity of Moist Porous Materials near The Freezing Point, Van Haneghem, I. A., Van Loon, W. K. P., Boshoven, H. P. A., High Temperatures-High Pressures, 1991, Volume 23, pages 157-162.

Error Analysis of the Heat Pulse Probe for Measuring Soil Volumetric Heat Capacity, Kluitenberg, G. J., Ham, J. M., Bristow, K. L., 1993, Soil Science Society of America Journal 57:1444-1451

Standard Test Method for Determination of Thermal Conductivity of Soil and Soft Rock by Thermal Needle Probe Procedure, American Society for Testing and Materials, D5334-14, 2014.

Standard Test Method for Thermal Conductivity of Plastics by Means of a Transient Line Source Technique, American Society for Testing and Materials, D5930-97, 1997.



9.19 CE declaration of conformity



According to EC guidelines 89/336/EEC, 73/23/EEC and 93/68/EEC

We: Hukseflux Thermal Sensors

Declare that the product: TP02

Is in conformity with the following standards:

Emissions: Radiated: EN 55022: 1987 Class A
Conducted: EN 55022: 1987 Class B

Immunity: ESD IEC 801-2; 1984 8kV air discharge
RF IEC 808-3; 1984 3 V/m, 27-500 MHz
EFT IEC 801-4; 1988 1 kV mains, 500V other

Delft,
January 2003