THASYS
Thin Heater Apparatus Measurement System Manual

THASYS manual v%+1&

Edited & Copyright by:

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Hukseflux
Thermal Sensors

Warning:

THA01 is specified for use up to 120 degrees C.

When working at higher temperatures than 100 degrees C contact with water should be avoided. Familiarity with the glycerol safety sheets is assumed.

During transport and storage the slot of THA01 should be closed, using moderate force. For transport in which the horizontality of THA01 cannot be guaranteed, the glycerol in the THA01 should be removed.

In case that a pressure cell is included (optional item), heavy transport and shocks should be avoided. During heavy transport there is an internal transport bolt that should be secured inside the THA01.

Under no condition more than 30 VDC should be put across the THA01 heater.
Under no condition more than 16 VDC should be put to the MCU input.
Putting a voltage over the Pt100 and thermopile sensors possibly will result in permanent damage.

THA01 as well as MCU are operated from 15 VDC. This DC voltage is delivered by an adapter that is supplied with the instrument. Both instruments are safe to use in a laboratory environment.

Contents

List of symbols

Thermal conductivity	λ	W/mK
Heat flux	Φ	W/m^2
Differential temperature across the samples	ΔT_{amp}	K
Effective sample thickness	H_{eff}	m

Acronyms

ASTM	American Society for Testing and Materials
MCU	Measurement and Control Unit
(V)DC	(Volt) Direct Current
THA/THA01	Thin Heater Apparatus
THASYS	Thin Heater Apparatus System
(V)AC	(Volt) Alternative Current
USB	Universal Serial Bus

Introduction

THASYS offers measurement of the thermal conductivity or total thermal resistance of plastics, composites and electrical insulation / interface materials. THASYS works according to the ASTM 1114-98 standard; providing an accurate, fast, uncomplicated and absolute measurement. It consists of a Thin Heater Apparatus (THA01) and a Measurement and Control Unit (MCU). Employing a specially designed high accuracy thermopile sensor, THA01 can handle thin sample materials (typically 0.1 to 6 mm; 0.1 mm samples must be stacked). The method is considered superior to procedures according to ASTM D5470. Using a climate chamber, measurements over a large temperature range can be covered, performing measurements at regular intervals. THASYS is fully PC controlled. For use with high thermal conductivity thin foil materials a different model, type THISYS, is available.

The THA01 is part of the THASYS delivery. Whenever the THA01 is mentioned in this manual it is meant to denote the THA01 sensor and not the MCU.

For determining the thermal conductivity of materials various types of measurement equipment can be used. Proven methods are described in standards of the American Society for Testing and Materials (ASTM).

ASTM C 1114-98 " Standard Test Method" for "Thin Heater Apparatus" (THA) is a relatively new standard (1998) that offers the possibility to perform fast measurements with high accuracy, across a fairly large temperature range.
The THA principle relies on the fact that a thin heater has negligible lateral heat flow. With a combination of a very thin heater, two relatively thin samples of similar thickness and two heat sinks it is possible to generate a homogeneous thermal field with known heat flux through the samples.

By measuring the flux φ (derived from heater power), the differential temperature across the samples ΔT_{amp}, and the effective sample thickness H_{eff}, it is straightforward to calculate the thermal conductivity λ:

$$\lambda = \varphi \, H_{eff} / \Delta T_{amp}$$

The measurements of φ, H_{eff} and ΔT_{amp} are all direct measurements of power, dimensions and temperature. This is

contrary to methods like the " guarded hot plate", that require reference materials or calibrated heat flux sensors. The THA provides an absolute instrument.

The technological novelties are a thin thermocouple thermopile (proprietary Hukseflux design) performing an accurate and ultra-sensitive differential ΔT measurement, and the fact that the measurement is performed in a bath of glycerol. The latter reduces contact resistance and facilitates the THA operation. Commonly used methods like ASTM D 5470 – 01 have shown to be highly sensitive to contact resistance. THASYS offers a solution to this problem.
The THA01 can handle samples of 0.1 to 6 mm thickness. Samples typically are sheet materials with a size of 70 by 110 mm.
The measurement accuracy depends on the total thermal resistance of the samples. In case of very thin samples (0.01mm), measurement accuracy is attained by "stacking", i.e. using four or more samples instead of the usual two.
The measurement essentially is done at the temperature of the THA01. If necessary the temperature can be changed by putting the whole THA01 in a climate chamber, performing measurements while the instrument temperature changes across the required range. The thermopile is attached with its cold joints to one of the heat sinks. Its hot joints are incorporated in the thin heater. The samples are put in between the heat sinks and the heater. Errors due to contact resistance are minimized by using glycerol fluid. In a simplified description, the measurement consists of a heating cycle and a measurement to establish ΔT.
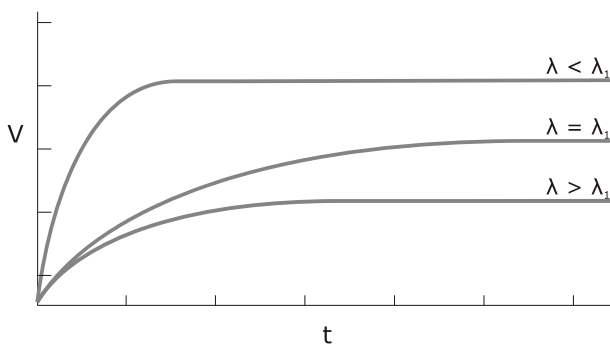
$V \sim 1/\lambda$

Figure 0.1 *Analysis of several samples of filled plastics using the THASYS. The graphs represent the signals when putting the heater on. The signal amplitude is inversely proportional to the thermal conductivity λ.*

The MCU performs the functions of measurement and control as well as data storage. It is PC operated. Software in a Windows environment is part of the delivery. The parameters cycle time, sample thickness and heater area are entered and the experiment is started from the screen.

Verification of the stability can be done by repeated (yearly) testing of Pyrex 7740 samples that are included in the delivery. The calibration is traceable to NPL. THASYS is suitable for use by ISO certified laboratories.

1 General overview

1.1 System description

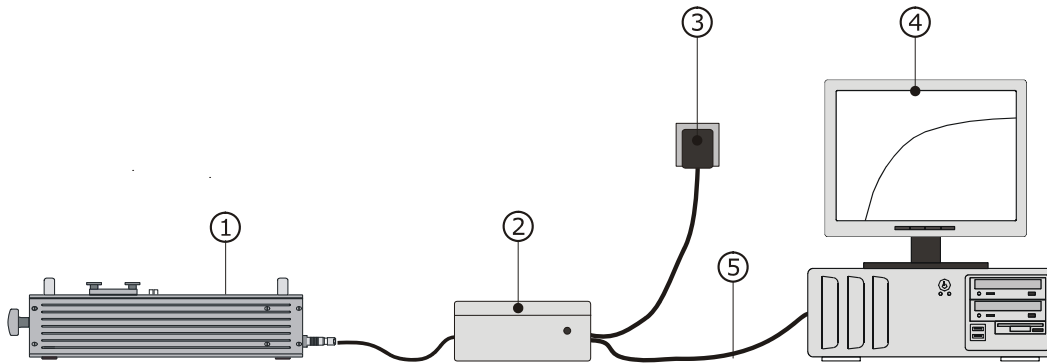Figure 1.1.1 Schematic drawing of a typical measurement setup. *1 is the Measurement apparatus (THA01), 2 the Measurement and Control Unit (MCU), 3 mains plug and 4 as PC (not part of the delivery).*
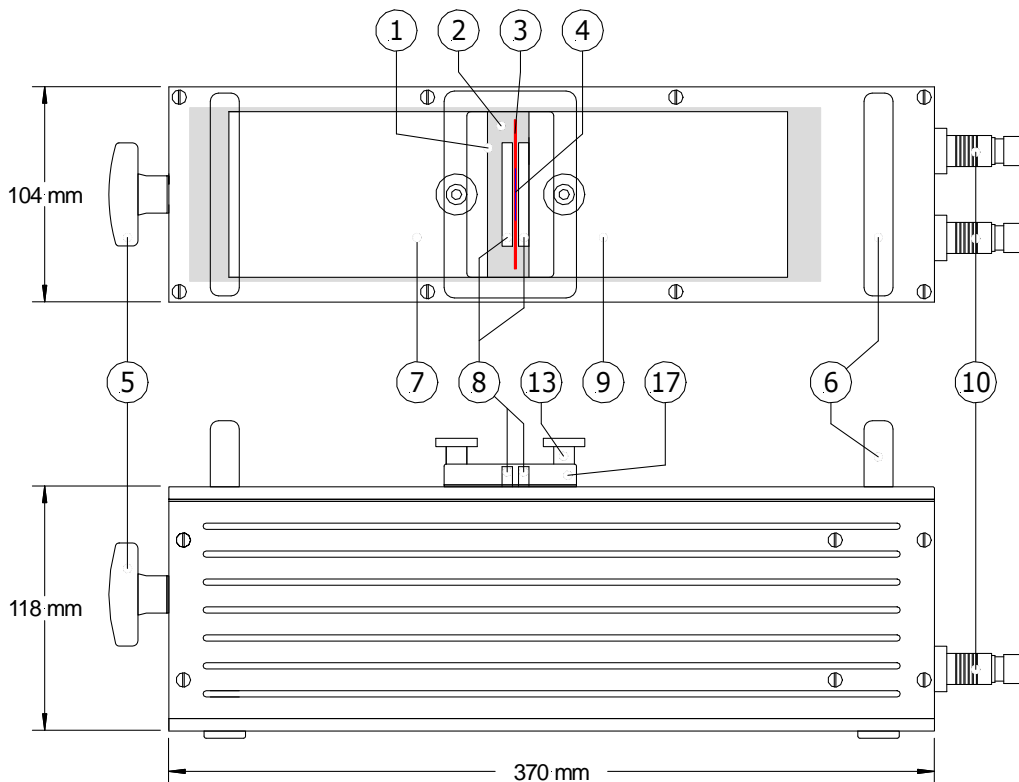

Figure 1.1.2 *THA01 dimensions and functional items. 1 slot, 2 glycerol, 3 heater, 4 temperature sensor, 4 knob for moving heat sink, 6 handles, 7 movable heat sink, 8 samples, 9 fixed heat sink, 10 cables for power and signal.*

1.2 Delivery

The following list shows what's included in the delivery:

MCU, THA01, PC cable (USB or RS232)	The MCU, a CR1000 measurement and control module, and a SC32B RS-232 interface. The PC cable has a 9-pin female connector at one end. The user is supposed to have a COM port with a 9 pin male connector. Do not confuse the RS-232 PC connection with the CS I/O connection. Cable length PC to MCU: about 1 meter. Cable length of THA01 about 2 meters.
Optional USB Driver	In case of USB connection: USB driver on CD-ROM
Adapter 12 VDC	This adapter can be used with any mains supply from 100 to 240 VAC, 50 or 60 Hz. If possible it is recommended to use this adapter. It can be connected to the MCU directly using the plug that is connected to its cable. Cable length to MCU: about 1 meter. Note: please make sure that the adapter has the right output level. If available, 13.8 volts should be chosen, otherwise 12 VDC is sufficient.
LoggerNet Software	To be installed on the PC for communication with the MCU. CD-ROM
THASYS Software	A program that is specially developed for use with THASYS, on USB flash drive
Pyrex 7740 samples	Set of two

Table 1.2.1 *Delivery: parts and their function.*

1.3 General operation

During every measurement, the THASYS program creates the following:

1. A file containing raw data file. This which contains all relevant measurement data as the are collected by the datalogger.
2. A file containing the result of calculations performed by the program, such a thermal conductivity, sample temperature, etc.
3. Messages regarding the dynamics of the measurement, for example thermal stability of the sample and apparatus, and if all signal from the sensors behave as expected.

The THASYS has several modes of operation. A normal measurement at a fixed apparatus temperature is most commonly used. It is also possible to put the THASYS in a climate chamber and vary the temperature of the THASYS as well as the sample up to 120°C while performing the measurements. In addition there is a function that allows the user to check the accuracy of the measurements using a reference sample to assure optimal functioning at all times.

General rules to ensure reliable operation
Check if the software version is the same as described by this manual (chapter 4).
Read the comment in the THASYS vxxxx.CR1 file. This can be done using the dedicated CRBasic compiler or any text editor that reads the ASCII file format.
Chose a proper measurement time for the sample that will be measured. The measured temperature difference should be constant near the end of the measurement. This may be determined by trial and error (chapter 10.6).
Always pay attention to warning messages. Try resolve the cause of the message or make sure you know the influence on the final result of the measurement (chapter 8).

Table 1.3.1 *General rules for working with a THASYS.*

Although it is, in principle, possible to run an autonomous measurement with the THASYS once it has been started with a PC, the system will normally always stay connected to a PC trough RS-232 or a USB port. This allows the user to display the measurement data in real time. A typical way of monitoring a measurement is using a 'Graph window' in the LoggerNet

software (figure 1.3.1). It provides an easy way to do a visual quality assurance, see chapter 5.5 for more detailed information.
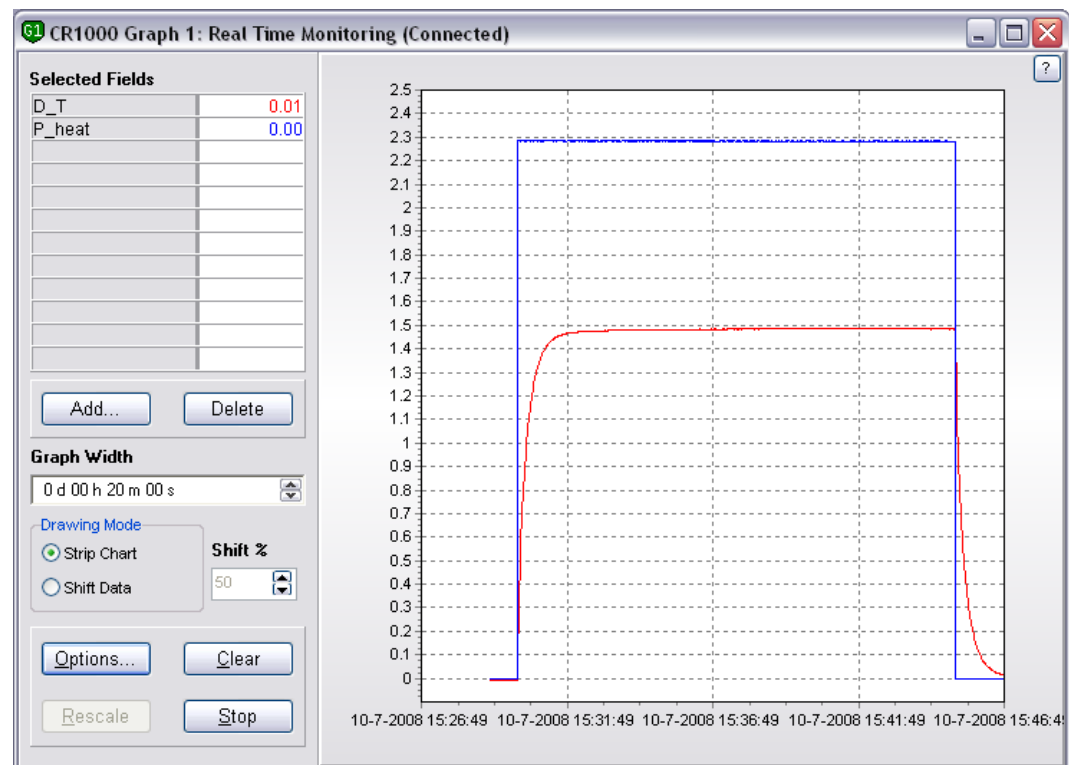

Figure 1.3.1 *Signals from the THASYS sensor can be displayed in real time during a measurement to ease quality assurance. The data shown has was collected with Pyrex 7740 reference samples with a thickness of 5 millimetre each and a thermal conductivity of 1.09 W/m.K.*

The value of the signal, when the signal has a constant high value, is determined by the thickness of the samples and the thermal conductivity of the sample. The power supplied trough the heater also plays a role, but this will be the same for all measurements.

The figure below shows a typical screen that can be used by the user for numeric in- and output. Variables can be displayed in the screen by clicking add and choose variables from the 'Public' data table. The third and fourth column display all variables from the 'Rawdata' and 'Results' data table respectively. Variables can be deleted from the screen by selecting the field a pressing delete. Public variables can either be changed by double clicking, entering the value and pressing enter, or using the menu that appears when right-clicking a cell.

CR1000 Numeric Display 1: Real Time Monitoring (Connected)

Countdown	0.00	exp count	9.00
exp no	10	Warning	0.00
sam id	XXX	Lambda0 ref	1.06
lambda	0.32	tc ref	0.00
H time	900.00	Lambda ref	0.00
T	22.56	HS ref	5.00
P heat	0.00	A heat	0.00
phi mom	0.00	C 1	1.00
D T	-0.01	C 2	463.00
R cont	0.00	C 3	0.96
R cont n	0.00		
R total	0.00		
R th	0.00		
P N	0.00		
H time Rc	600.00		
T step	10.00		
HS 1	3.27		
HS_2	3.58		

Buttons: Add..., Delete, Delete All, Options..., Stop, Help

Figure 1.3.2 *A typical 'Numeric Display' that can be used for user in- and output.*

2 Preparation before use

In addition to the delivered parts, it is recommended to have the following items available:

Mains power or low voltage DC power source	Preferably mains power for the Adapter. This can be 100-240 VAC, 50/60 Hz. If this is not available, a power source of 10 to 13 VDC, at 5 Watts is sufficient. The absolute value of the power supply is not important as long as it is constant within 1% during 5 minutes. A normal battery will be able to meet this specification. The power supply must be available within 1 meter from the point of measurement.
PC	PC, running Windows XP or higher, with CD drive and with one free 9 pin RS 232 port (male connector on the PC) or a USB port.
Glycerol	As the transport of fluids is not allowed by most carriers without special permission, the user must purchase glycerol locally. Glycerol or glycol can be purchased at most pharmacies. Glycerol Fluid, consumer quality of at least 96% purity, 1 litre. The glycerol is used to fill the measurement cavity of the THA01 to reduce contact resistances between the sample and the heat sinks.

Table 2.1 *Items that need to be available.*

3 Step 1: Receiving the equipment

When the system arrives, it is recommended to check if all is there. For a general check: see table 1.2.1. It is possible that additional items were ordered (extension cables, reference samples, etc.). It is recommended to compare to the original order.

- After rough (air)transport with pressure cell (optional), open the THA01 and release the transport nut.

- Purchase glycerol 1 litre (also called glycerine or glycol). This fluid is available in any pharmacy. It is safe for hands, eyes etc. Fill the THA01 with glycerol. Open the slot. Put in the Pyrex 7740 reference samples. Close the slot using corded knob at the side (part 5 in figure 1.1.2).

4 Step 2: Software

The latest version of the software is always shipped with the system. Always make a backup of all received files before using them.

First, install the Campbell Scientific LoggerNet software, this is straightforward. If help is needed the information on the LoggerNet CD-ROM is sufficient in most situations.
A separate USB flash drive contains a version of the THASYS software. The file, thasys vxxxx.CR1 contains a description of the program and a wiring diagram. Any text editor or the dedicated CRBasic compiled in the LoggerNet package can be used to view (and edit) the file. It can be loaded into the MCU using the Connection screen of LoggerNet.

When operating with a USB cable, the user also needs to install the USB driver on the PC. This driver will install an extra COM port which can be used by LoggerNet to communicate with the MCU (CR1000). During the installation the installer will ask you to connect the system to a USB-port. Make sure chose the same port as will be used during the measurements, because in some situations connection problems may occur when a different port is used.

5 Step 3: Installation and Testing

Before the system is ready to do a first measurement, a few things have to be set up. If the MCU is always used in combination with the same PC this procedure has to be performed only once. Next, a first instructive measurement can be made by performing a calibration procedure using the Pyrex 7740 reference samples.

5.1 First power up

Use the power supply delivered with the system and make sure the supply is set to 12 VDC. In case the system is equipped with a battery pack, the charging voltage should typically be between 13.8 and 18 VDC. Please consult the battery manual about this. After plugging in the power supply the red LED on the MCU should light up.

5.2 Connecting THA01 to the MCU

The THA01 unit can now be connected to the MCU. Both cables should be plugged in: one side in the MCU, other side in the THA01. Both cables have different plugs, so user should take care to insert each plug into the corresponding socket.

5.3 Connecting THASYS to the PC

The THASYS can now be connected to the PC using the LoggerNet software. First make sure the software is installed as described in chapter 4.

- Make sure the system is powered up.
- Connect the MCU to on the PC, either with the USB cable or with the RS-232 cable.
- Start the LoggerNet software.

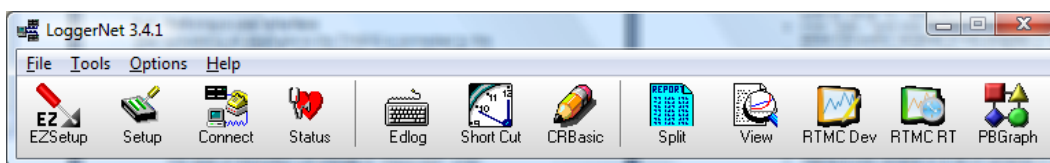The following screen will appear:

Figure 5.3.1 *The main menu of the LoggerNet software (the version number may be different).*

To configure the system for normal use, click on the "Setup" button. A screen similar to the following figure will appear:
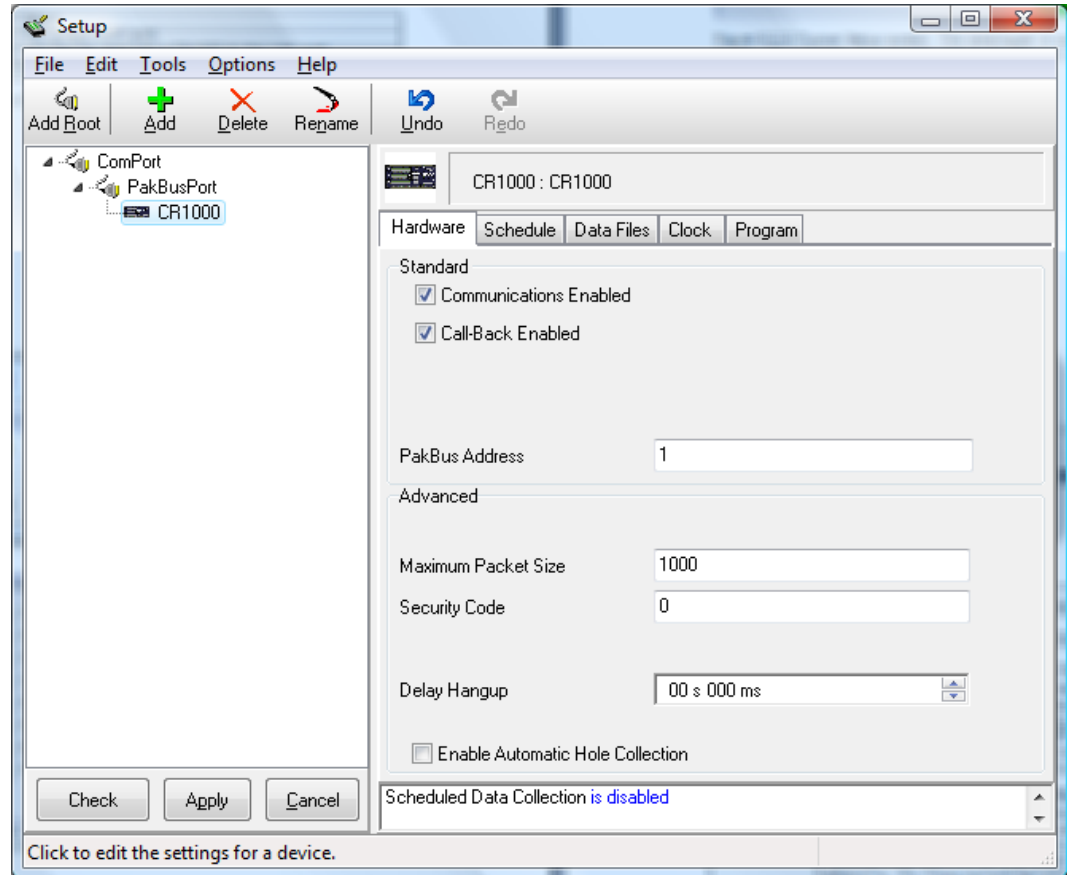

Figure 5.3.2 *The setup screen is only used to configure the system when it is used for the first time or case the LoggerNet software is re-installed.*

To install the THASYS MCU
- Select the ComPort entry in the left list. Choose "COM4" in the "Com Port Connection" pull down menu. It may be the case that the driver assigned a different com port to the USB connection. A general rule that works in most cases is to chose the highest com port number.
- Click the "Add" button in the top menu. Select "PakBusPort (Other Loggers)", then select CR1000 and click "Close".
- Close the Setup screen and answer "Yes" when asked to apply the changes.

5.4 Location of measurement results

The results of a measurement will be written to a data table and stored in the MCU. Even when the power is disconnected the data will be kept. Data is received from the MCU when the steps described in chapter 7 are performed. In the following steps the location of the data files is defined:

- Open the setup screen by clicking on the "Setup" button in the main menu of LoggerNet.
- Select the datalogger the is just installed, CR1000 normally.
- Make sure the logger is connected and powered up and click on the button "Get Table Definitions" in the tab "Data Files".
- Select "RawData" in the list in the centre of the screen to make the setting for the data table "RawData".
- Click on the "…" button next to the field that shows something similar to "c:\Campbellsci\LoggerNet\CR1000_RawData.dat" and select the directory (folder) to store the data in.
- Repeat the last step for the data table "Results".

5.5 Defining a user interface

After completing all steps above the THASYS is connected to the PC and ready to define a user interface. Loading new software on the MCU, controlling measurements and displaying results is all done in the "Connect Screen". First we ensure that the right software program is loaded in the THASYS MCU, called CR1000 in the setup screen.

- Click "Connect" in the LoggerNet main menu. A screen as in figure 5.5.1 appears.
- Select "CR1000" in the list on the left and click "Connect". The label of the button will change to "Disconnect" when the connection is made.
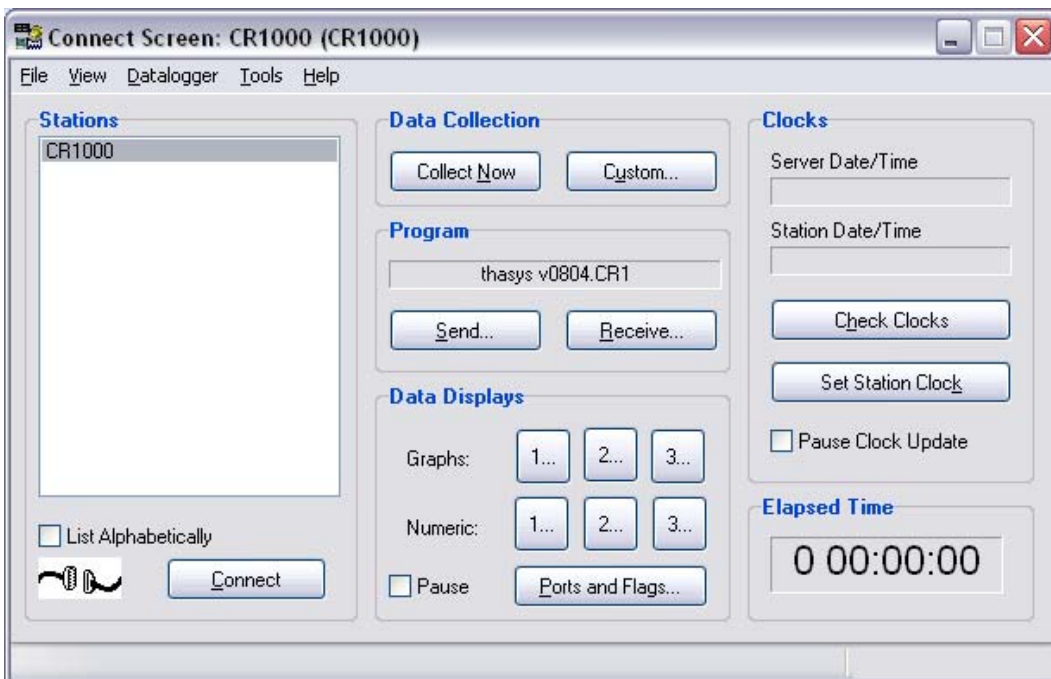
Figure 5.5.1 *Connection screen of the LoggerNet software while a system is connected. This screen is used to load a program in the MCU, to receive data and to perform numeric and graphical in- and output communication while a program is running.*

The "program" frame should show "thasys vxxxx.CR1" where the x's represent the version number. If this is not the case then click "Send" and select the program delivered with the system. This file should have the extension .CR1.

WARNING: when uploading a program to the logger, all data will be erased, as well as the modified values of the variables. Before uploading a new program, user should write down these values and save all measurement data to the PC.

The numbered buttons behind "Numeric:" are used to for numeric in- and output. To define a typical numeric user in- and output screen:

- Click "1…" and a screen similar to figure 5.5.2 will appear. It is possible to define three different numeric screens in total by using "2…" and "3…".
- Click "Add…" and click "Public" in the list on the left to select the public variables of the program in the right list.
- Select the variable "Countdown" go down to the variable "C_3" and select it while pressing the shift key on the keyboard. Then click "Paste" and close the screen.
- The most commonly used variable are now listed in the numeric screen. They can be rearranged at will by clicking the variable name, drag to the desired cell while holding the mouse button and release the button.
- Pressing "Clear All" will make the screen blank again.
- Adding a one variable or a list of variables in a particular cell can be done by clicking an empty cell and then click "Add" and repeat the procedure as above.

After pressing the 'Start' button, the variables visible in the screen show the current value while the MCU is connected.

Figure 5.5.2 *The Numeric Screen as typically used when viewing the measurement on line.*

The program control is done using flags. A flag is a Boolean operator that can take on a high or low value (on or off respectively). Perform the following steps to setup the "Ports and Flags" screen.
- Open the "Ports and Flags" screen by clicking the button in the "Connect Screen".
- Click on the top left label and click "Add…".
- Select public and click "Paste" to add all Boolean variables and close the screen.

Figure 5.5.3 Shows the screen while a measurement is running. A flag can be activated by clicking on the button. Activated flags show up light green.

Figure 5.5.3 *The control screen after variables have been added. The buttons can be pressed to activate programmed procedures and to read of the status of the MCU.*

5.6 Using the interface

The "Numeric Screen" is used for numeric in- and output. Values of variables can be changed as follows:

- Right-click on the field that has to be modified and select "View/Modify Value…" (figure 5.6.1).
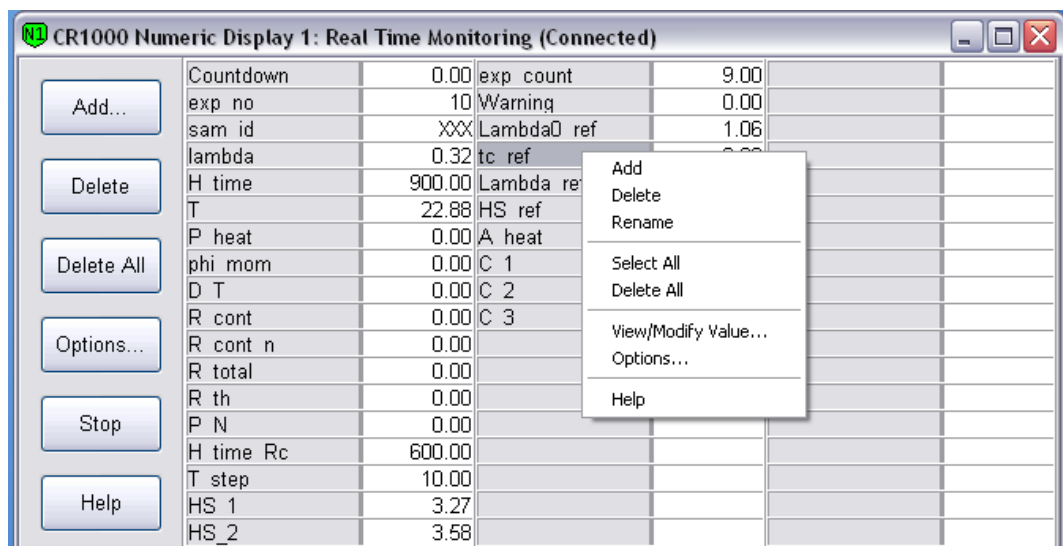- Key in the desired value (figure 5.6.2) and click "Apply".

Figure 5.6.1 *Changing values of numeric variables in using the right-click menu.*

An alternative way is to double-click in the cells, the colour of the cell changes to yellow. Now, key in the desired value and press enter to confirm.

Several variables should be displayed with more than the standard 2 decimals. To change the number of decimals shown for a variable, right click on the cell and choose "Options…" in the pulldown menu. A screen as shown in figure 5.6.2 will appear. R_cont should be displayed with 5 decimals, C_1 and C_3 should be displayed with 3 decimals.
There is also a way to set an alarm for a low or high value as well as some other options that may be convenient to use.
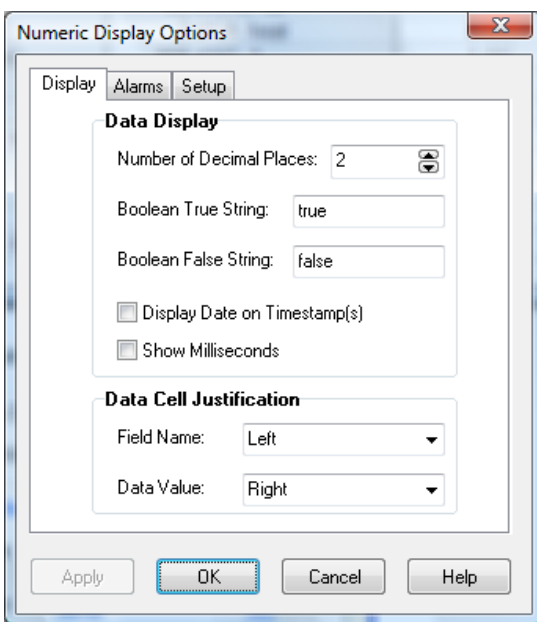
Figure 5.6.2 *Options to change the way the variables are displayed.*

Next to the numeric screen it is also possible to display a graph of the parameters. This may be useful to check the trend of a parameter. Three different graph screen can be created, just as the numeric screens. A graph screen can be opened by clicking on one of the numbers behind "Graphs:" in the LoggerNet Connect Screen and selecting parameters to be displayed. A screen similar to the following appears.
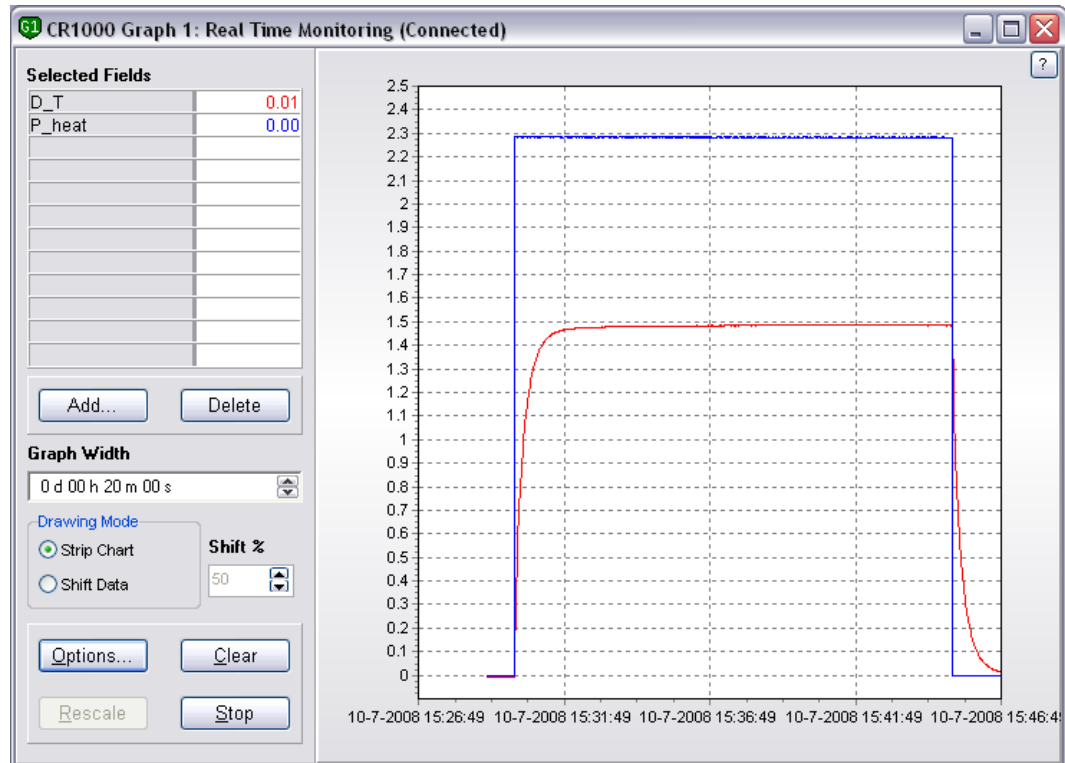
Figure 5.6.3 *An example of a graph screen.*

In the "Options…" it is possible to set several visualisation options, the vertical scale and the time base.

5.7   Calibration and testing

Before testing, user should make sure the correct calibration constants are entered in the system.
Values for C_1, C_2, C_3, A_heat and R_cont should match the form shipped with the system. If not, user should adapt these in the Numerical Screen.

After completing all foregoing steps (1, 2 and 3) the system is ready for a first test. This test is both instructive for a non-experienced user and serves as the check if the system is working properly.
In this test the Pyrex 7740 reference samples, that are part of the standard delivery, will be measured. These samples have a very well defined thickness, are stable in almost all environments and have very accurately known thermal properties.

- Make that the values of the following parameters is set to the proper value for the Pyrex 7740 reference samples:

variable	value
Lambda0_ref	1.058
tc_ref	0.0014
HS_ref	5.00

H_time_Rc	600.00
H_time	800.00

- The first three variables define the temperature dependent thermal conductivity of Pyrex 7740 of 5 mm thickness. When a reference sample with a different thickness or thermal properties is used, these values should be changed. For this test "H_time" should be set to 800 s.

Every time a new program is loaded into the MCU the so-called contact resistance of the connected THA01 should be determined and set in the program. This variable is used to improve the accuracy of the measurement. Please consult the THA01 manual for more information regarding the physical model used to determine the thermal conductivity.

Determining the contact resistance
This is done without any samples in the sample space.
- Close the measurement slots completely and give at least two extra turns after the sinks tough each other.
- Activate the measurement by clicking on the flag "DetermineContRes". The measurement start running, flags "Running" and "Det_R_C" will light up and the parameter "Countdown" displays the time in seconds left to the completion of the measurement.
- After the measurement the value of the variable "R_cont_n" should be copied to the variable "R_cont".

In case the newly found value of the thermal conductivity deviates by more than 2%, it is suggested to adapt C_3 accordingly.

Calibrating the system:
- Put the two Pyrex reference samples in the sample space, one both sides of the foil heater and close the sinks.
- Wait until the value of the variable "D_T" is smaller than ±0.01 indicating thermal stability of the sample and sensor.
- Activate the calibration procedure by clicking the variable "StartCalibration". The system will now automatically perform a test measurement.
- After the procedure is completed, "Countdown" shows 0.00 and the flag "Running" is off, the result is compared with the expected value.
- Check the value of lambda (the thermal conductivity of the sample) this should be around 1.1 at room temperature.

When the flag "Cal_OK" lights up the value of lambda is within 2% of the expected value.

- In case "Cal_OK" doesn't light up repeat the procedure. In case there is still no successful completion also repeat the measurement of the contact resistance or contact the manufacturer Hukseflux Thermal Sensors.

For reliable results according to the specifications this procedure should be repeated yearly.

At this point, after completing all tests and procedure above, the system is ready for normal use and will give reliable results, when operated in a normal manner.

6 Performing measurements

This chapter describes how to use the system in standard operation as well as a procedure that can be used for measuring thick samples.

6.1 Standard samples

The procedure to measure standard samples can be used when the measurement is performed at environmental temperature (room temperature) and the thickness and thermal conductivity is within the specifications of the THASYS.

- Measure the sample thickness within 1% accuracy. The thickness of two samples that are used for a measurement must be as similar as possible in terms of thickness and thermal properties.
- Write down the sample identification and the two thicknesses, time and date.
- Make sure the samples are clean and dry.
- Open the slot of the THA01.
- Insert a sample on each side of the heater/sensor, make sure the samples are located on the central axis of the THA01.
- Close the slot using moderate force or in case of the optional pressure cell, to the specified pressure has been reached.
- If the sample is between 3 mm and 6 mm activate flag "ThickSample".
- Wait until the D_T reaches a value between ±0.01.
- Key in the thickness of the samples and optionally an experiment ID in "exp_id" and a sample ID in "sam_id".
- Activate the flag "Start" to start the measurement.

6.2 Thin samples

When very thin samples are measured it is often difficult to determine an accurate absolute thermal conductivity. The THASYS offers the possibility to measure the total thermal resistance without any corrections. This is a good way for a relative comparison of two thin samples.

The measurement procedure is the same as for the standard procedure, but instead of using the "Start" flag to start the measurement use the "DetermineContRes" flag. By default, the

same measurement time as for the measurement of the contact resistance, "H_time_Rc", will be used. The measurement will run as for standard samples. After finishing the total thermal resistance will be displayed in "R_total".

6.3   Changing the measurement temperature

The THASYS is equipped with a sensor to monitor the temperature in real-time. In the MCU is a procedure programmed that can be used to trigger a measurement upon a temperature change set by the user. By putting the THA01 in a climate chamber or an oven the instrument temperature can be raised/lowered.

Warning: Never use the THA01 at temperatures higher than 120 degrees C as this may cause permanent damage to the apparatus. Avoid contact with water when working at temperatures higher than 100 degrees C. Familiarity with the glycerol safety sheets is assumed.

The procedure below describes how to do a measurement at a temperature different from the lab temperature. Prepare the measurement as described in chapter 6.1, i.e. load the sample and make all settings. Now there are two modes of operation possible:
1. Bring the THA01 to the desired temperature and start a measurement, or
2. do an automatic measurement at a fixed temperature interval as the THA01 warms up / cools down.

In the first case (1), the temperature of the THA01 is displayed in the numeric screen in the variable "T". It is recommended that the temperature is displayed in a graph screen. This enables the user to make sure the right conditions are met to do a measurement, in particular the thermal stability of the system. Once the desired temperature is reached and the system is reasonably stable, the measurement is can be made following the standard procedure (chapter 6.1).
The second case is similar, but instead of waiting until the system reaches a certain temperature, measurement are made automatically as the system temperature changes. The procedure is as follows:
• Set the temperature interval between the measurements with the parameter "T_step".

- Put the system in a climate chamber and set the temperature to the highest (or lowest) temperature of the temperature range.
- Start the procedure by clicking the flag "StartHeating". When the system is running a measurement the flag "Running" will light up as normally and in idle status the flag "WaitForHeating" will be active.
- Because of the high heat capacity of the system the temperature of the THA01 will much more slowly change than the temperature in the climate chamber. Be aware of this and never set the temperature of the climate chamber higher than 120 degrees C as certain parts of the THA01 may be damaged due to overheating.
- In some cases it may happen that the temperature change is such that during the time of a measurement the temperature has changed more than the set "T_step" value. The system will run measurements continuously and not at the desired temperatures. This can be resolved by bringing the THA01 in several smaller steps to the desired highest (or lowest) temperature or by increasing the value of "T_step".

7 Retrieving data

For normal operation the raw data is processed by the MCU and the results are written to a data table. In case extra quality assurance or more advanced data analysis is needed it is possible to write the raw data to a data table.
The location of the files is set during the setup procedure (see chapter 5.3).

7.1 Downloading data

In normal operation the results of a measurement are stored to the data table CR1000_Results.dat, when the datalogger name (MCU) is CR1000. When raw data is needed the table CR1000_Rawdata.dat is filled as well. Normal operation is selected by starting a measurement using the flag "Start" (not the flag "StartCalibration"). The file format is ASCII with comma separated columns. This file format can be imported in virtually any application for data analysis or in Microsoft Word or Microsoft Excel.
Downloading the data from the MCU is done using the button "Collect Now" in the "Connect Screen". All data tables will be copied to the selected directory (chapter 5.3).
Instead of closing the screen that appears by clicking on "Ok", it is also possible to view the collected data by clicking on "View File" (see figure 7.1.1). This screen has, beside viewing of numerical data, the possibility of creating graphs by selecting the data column and then click on the graph icon in the menu bar.

Figure 7.1.1 *Viewing a data table using LoggerNet.*

Alternatively the data can be imported in for example Microsoft Excel. This can be done by starting Excel, chose File, Open…, chose in "Files of type" the option "All Files". Now, go to the directory where the data file is located, select the file and click

Ok. Click on "Next >" in the screen that appears, tick the comma option and click "Finish" to open the file. The Excel sheet that is now created should look similar to figure 7.1.2.

Figure 7.1.2 A typical data file as downloaded from the MCU and opened in Microsoft Excel.

An detailed explanation of the head can be found in the appendix (chapter 10.1) including all variable names and the units in which they are displayed.

7.2 Collecting raw data

When raw measurement data is needed, a measurement should be started using the "StartCalibration" flag instead of the "Start" flag. Raw measurement data, such as the sensor signal and system temperature, will be stored in real time to the data table "Rawdata". All other options can be set in the normal way. If the thermal conductivity matches the set reference sample values (chapter 5.6), the "Cal_Ok" flag which will light up after completion of the measurement. This can be ignored without any complications.
Viewing the file is done in exactly the same way as described in the foregoing chapter 7.1.

8 Warning messages

Warnings are not necessarily errors, but are indicating that the user should pay attention to this aspect of the measurement.

Warning	Significance	Numerical addition to " Warning"
War_lambda	The thermal conductivity is out of range	1
War_H_t	The heating time is out of range	2
War_id	The sample identification is not entered	4
War_P_sta	The power is possibly unstable	10
War_grad	The temperature gradient before the measurement is possibly too large	20
War_monot	The signal is not monotonous, possibly the sample or heater is moving	40
War_heat_H	The heating power is too high	100
War_grad_A	The signal amplitude is possibly not sufficiently stable	200

Table 8.1 *Warnings are given as a summation of separate messages.*

When "Warning" equals for example 327:
    This can be decomposed in:

    Warning = 200 + 100 + 20 + 1 + 2 + 4

The messages can now we looked up in table 8.1.

9 Delivery and spare parts

THA delivery includes the following items:

- MCU01
- THA01
- Set of two Pyrex 7740 samples
- Calibration form for Pyrex samples
- Manual THA01
- Calibration form for THA01
- THA01 cable with connectors 1
- THA01 cable with connectors 2

Spare parts are:

- Thermopile plus heater assembly
- THA01 cable with connectors 1
- THA01 cable with connectors 2

The THA delivery does NOT include: the MCU, glycerol contact fluid and tools to measure the sample thickness.

Options are:

- Force sensor
- Power supply for thin
  sample analysis

Services:

- Recalibration of the Pyrex 7740 samples
- Analysis of samples

10 Appendices

10.1 Relevant variable names and description

variable	description	Units
General system variables		
PTemp	Temperature of CR1000 datalogger in MCU	$^{\circ}C$
Batt_volt	Current power supply voltage	V
General measurement variables		
Countdown	Time to go to the end of measurement	s
Exp_no	Experiment number for user reference	-
Sam_id	Sample ID for user reference	-
lambda	Measured thermal conductivity	$W/k{\cdot}m$
H_time	Heating time (3x heating time is total measurement time)	s
T	THA01 temperature	$^{\circ}C$
P_heat	Power supplied to the THA01 heater	W
Phi_mom	heatflux trough the sample	W/m^2
D_T	Temperature difference over the sample	
R_cont	Contact resistance	$K{\cdot}m^2/W$
R_cont_n	New determined contact resistance	$K{\cdot}m^2/W$
R_total	Total measured thermal resistance without corrections	$10^{-3}\ K{\cdot}m^2/W$
R_th	Sample thermal resistance	$K{\cdot}m^2/W$
P_N	Force on sample (optional)	N
H_time_Rc	Heating time used for determing contact resistance	s
T_step	Temperatures step used in automated measurements	$^{\circ}C$
HS_1	Thickness of sample 1	mm
HS_2	Thickness of sample 2	mm
Exp_count	Number of experiments done in automated measurement	-
Warning	Warning code (chapter 8)	-
Calibration data regarding the reference sample and the temperature difference sensor		
Lambda0_ref	Thermal conductivity of sample at $0\,^{\circ}C$	$W/k{\cdot}m$
Tc_ref	Temperature coefficient of reference sample	$W/k{\cdot}m{\cdot}^{\circ}C$
Lambda_ref	Thermal conductivity of sample at current temperature	$W/k{\cdot}m$
HS_ref	Thickness of reference samples	mm
A_heat	Effective surface area heater	m^2
C_1	Calibration constant sensor	V/K^2
C_2	Calibration constant sensor	V/K
C_3	Calibration constant sensor	-

Table 10.1 *Numeric program variables and their function. The variables have the same order as in the program and numeric screen*

Filled in case of repeated automated measurement triggered by a temperature change of the system (chapter 6.3), n is the experiment number set by "Exp_count".		
Temp(n)	System temperature	°C
Lambda_disp(n)	Measured thermal conductivity	W/k·m
R_th_disp(n)	Sample thermal resistance	K·m^2/W
Warning_disp(n)	Warning code (chapter 8)	-

Table 10.1 (continued) *Numeric program variables and their function. The variables have the same order as in the program and numeric screen.*

10.2 Data tables

	A	B	C	D	E	F	G	H	I
1	TOA5	CR1000	CR1000	14834	CR1000.Std.14	CPU:ceast	2951	RawData	
2	TIMESTAMP	RECORD	Exp_Id	Re	time	I_heat	U_sen	U_cold	T
3	TS	RN		Ohm/m	Sec	A	mV	mV	Deg C
4			Smp	Smp	Smp	Smp	Smp	Smp	Smp
5	44:34.5	0	1	84.28	-100	0	-0.00199	0	25.62258
6	8-4-2008 6:44	1	1	84.28	-99.5	0	-0.00132	0	25.62258
7	44:35.5	2	1	84.28	-99	0	-0.00132	0	25.61405

Figure 10.2.1 *A screen shot of the 'Results' data table header.*

The cells in the first row contain the following information from left to right:

- File format, always the same, ASCII comma separated.
- Station name, chosen by the user during installation on PC.
- Logger model, always a CR1000 data logger.
- Serial number of the build-in CR1000 logger.
- Operating system version on the CR1000.
- Program name.
- Program ID
- Data table name.

The second row contains data label of the corresponding column, the third the units in which the data is displayed. The forth column gives information on how the data is collected, this information is not relevant in this context.

The following variables are written to the results table

batt_volt	Minimum power supply voltage in Volt
exp_no	Experiment ID
sam_id	Sample ID
P_N	Force on the sample in N (shows 0 when not installed)
T_measure	THA01 temperature of current measurement
lambda	Thermal conductivity
R_th	Thermal resistance
R_total	Total thermal resistance, without corrections
HS_1	Thickness sample 1
HS_2	Thickness sample 2
Con_ok	Contact resistance within specifications
Cal_ok	Calibration value within specifications

The raw data table is only recorded when using the flag "StartCalibration" as described in chapter 7.2. The figure below shows the raw data table imported in MS Excel.

	A	B	C	D	E	F	G	H	I	J
1	TOA5	CR1000	CR1000	7194	CR1000.St	CPU:thasy		48724	RawData	
2	TIMESTAMP	RECORD	batt_volt_N	exp_no	sam_id	I_heat	U_heat	T	U_sen	P_N
3	TS	RN				A	V	dgrC	mV	N
4			Min	Smp	Smp	Smp	Smp	Smp	Smp	Smp
5	10-7-2008 16:11	0	11.92	10	XXX	0.214	10.66	24.13	0.216531	0
6	10-7-2008 16:11	1	11.92	10	XXX	0.214	10.66	24.16	0.336054	0
7	10-7-2008 16:11	2	11.92	10	XXX	0.214	10.67	24.16	0.419157	0
8	10-7-2008 16:11	3	11.92	10	XXX	0.214	10.67	24.16	0.481733	0

Figure 10.2.2 *A screen shot of the 'Rawdata' data table header.*

The "RawData" table contains the following variables in its columns:

batt_volt	Minimum power supply voltage in Volt
exp_no	Experiment ID
sam_id	Sample ID
I_heat	Current trough the THA01 heater
U_heat	Voltage over the THA01 heater
T	THA01 temperature
U_sen	Sensor signal from the THA01
P_N	Force on the sample (shows 0 when no force is sensor installed)

10.3 MCU compatibility with THISYS

The THASYS and THISYS are systems which are very closely related. THISYS is a sensor that is designed to measure the thermal conductivity of thin good sheet material with a high thermal conductivity, such as for example metal foils. The hardware of the MCU of both systems is identical. By changing the software of an MCU delivered with a THASYS it can also be used with a THISYS and vice versa. Please refer to the THISYS manual for general information and to the THI01 manual for the physical working principles of THISYS. These manuals are available at on request a Hukseflux Thermal Sensors.

10.4 Transport safety optional pressure cell

In case there is an optional pressure cell installed in the THA01, the pressure cell is blocked to prevent damage during transport.

The top cover plate of the THA01 needs to be removed to release the transport safety. This can be don by using an 3 mm Allen key to remove the 8 screws at the top side of the THA01 (see top view figure 1.1.2).
Figure 10.4.1 shows where the pressure cell (5) is situated. The tread end prevents a force to be applied to the pressure cell during transport.
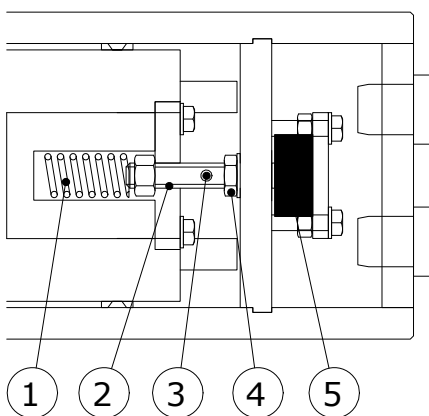

Figure 10.4.1 *Detailed view of the pressure sensor and the transport safety nut (4). The spring is not visible in reality.*

Releasing the safety nut:
- Put a rod of 3 mm in diameter in the hole (3) to block the thread end.
- Release the safety nut (4) by at least two full turns.

After following the steps 1, 2 and 3 of this manual the system is ready to use.

10.5 Appendix on typical thermal properties

A table describing the typical values for the thermal properties of some materials is given below. Note that the list is only indicative, it has no value as a literature source. For good literature values, use for example the "Handbook of Chemistry and Physics", or HBCP as it is often called, by CRC press.

	Thermal conductivity W/mK @ 20°C	Density Kg/m³ @ 20°C	Volumetric heat capacity J/kgK @ 20°C	Thermal diffusivity 10^{-8} m²/s @ 20°C
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	1.087	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
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 10.5.1 *A list of typical values of thermal properties of various materials. This list is only indicative.*

10.6 Appendix on typical heating cycle times

Material	Sample thickness mm	Minimum recommended heating cycle time ($5H^2$ / a) s
Aluminium Oxide*	5.00	14
Quartz*	6.00	128
Concrete	6.00	273
Marble*	3.00	36
Glass	6.00	423
Pyrex 7740	5.00	209
PVC	1.00	61
PE	3.00	368
PTFE	3.00	396
Nylon 6	2.50	242
Corian (ceramic filled)	6.00	392
Wood	4.00	37
Cotton	1.00	< 60
Leather	2.00	34
Cork	6.00	120
Foam glass	4.00	164
Mineral insulation materials	4.00	180
Plastic insulation materials	4.00	267
Sand (dry)	3.00	163
Sand (saturated)	6.00	489
Glass pearls (dry)	3.00	285
Glass pearls (saturated)	5.00	446

Table 10.6.1 *typical orders of magnitude for the minimum recommended heating cycle times h in seconds. It can be seen that 450 seconds is sufficient for a large number of sample materials and sizes. The total measurement time is 3 times h. See also the previous paragraph for corrections. The samples that do not qualify (in this case because corrections become too large) are marked with *

10.7 Glycerol Material Safety Data Sheet (93 / 112 EC)

This safety sheet is a copy of the Material Safety Data Sheet Version according to 93/112 EC of 10-08-2000 of the company Ephuysen Chemie. The THA01 user is responsible for obtaining the latest Version of this sheet from the manufacturer when purchasing glycerol for use with THA01.

1 identification of the substance / preparation and of the company/ undertaking
• Production details -
• Trade Name: Glycerine
• Article Number: 013311
• Manufacturer / Supplier:
Ephuysen Chemie N. V.
Noordeweg 3
3336 LH Zwijndrecht
The Netherlands
Tel: 00-31-78-612702 Fax: 00-78-6121129
e-mail ppelt@ephuysen.com
• Further information available from: environmentally Department
• Information in case of emergency: see above
2 Composition / information on the ingredients
• Chemical characterisation: Cas No Description: 56-81-5 Glycerol
• Identification numbers: -
• EINECS Number: 200-289-5
3 Hazards identification
• Information concerning to particular hazards to man and environment: Not applicable
4 **First-aid measures**
• General information: No special measures required
• After inhalation: Supply fresh air; consult a doctor inn case of complaints
• After skin contact: Generally the product does not irritate the skin
• After eye contact: Rinse opened eye for several minutes under running water
• After swallowing: If symptoms persist consult a doctor
5 Fire fighting measures
• Suitable extinguishing agents: Use fire extinguishing methods suitable to surrounding conditions
• Protective equipment: No special measures required
6 Accidental release measures
• Person-related safety precautions: Not required
• Measures for environmental protection: Dilute with plenty of water
• Measures for cleaning / collecting: Absorb with liquid binding material (sand, diatomite, acid binders, universal binders, sawdust).
• Additional information: No dangerous substances are released

7 Handling and storage
- Handling :-
- Information for safe handling: No special measures required
- Information about fire - and explosion protection: No special measures required

- Storage:
- Requirements to be met by storerooms and receptacles: No special requirements
- Information about storage in one common storage facility: Not required
- Further information about storage conditions: None
- Storage class: -
- Class according to regulation an flammable liquids: void

8 Exposure controls / personal protection
- Additional information about design of technical facilities: No further data; see item 7
- Ingredients with limit values that require monitoring at the workplace: 56-81-5 glycerol OEL: 10 mg/m^3
- Additional Occupational Exposure Limit Values for possible hazards during processing: Glycerine is (almost) odourless. The MAC-value can exceed the limit unnoticed.
- Additional information: The lists valid during the making were used as a basis

- Personal protective equipment: -
- General protective and hygienic measures: The usual precautionary measures are to be adhered to when handling chemicals
- Respiratory protection: not required
- Protection of hands: not required
- Eye protection: Goggles recommended during refilling

9 Physical and chemical properties
- Form: viscous
- Colour: Colourless
- Odour: Odourless
- Change in condition: -
- Melting point / Melting range: 18.2 °C
- Boiling point / Boiling range: 290°C
- Flash point: 177 °C
- Ignition temperature: 400 °C
- Danger of explosion: the product does not present an explosion hazard
- Explosion limits:
- Lower: 0.9 Vol %
- Vapour pressure: at 50 °C 0.003 hPa
- Density: at 20 °C 1.26 g/ cm^3
- Solubility in / Miscibility with water: Fully miscible
- PH value; Not determined
- Viscosity:
- Dynamic: at 20 °C 106.9 mPas

10 Stability and reactivity
- Thermal decomposition / conditions to be avoided: No decomposition if used according to specifications
- Dangerous reactions: No dangerous reactions known
- Dangerous decomposition products: No dangerous decomposition products known

11 Toxicological information
- Acute toxicity:LD/ Lc50 values relevant for classification: 56-81-5 glycerol Oral LD50: 250 mg/kg(mus)
- Primary irritant effect:
- On the skin: It is not irritating
- On the eye: No irritating effect
- Sensitisation: No sensitisation effects known.
- Additional toxicological information: When used and handled according to specifications, the product does not have any harmful effects to our experience and the information provided to us. The substance is not subject to classification according to the latest Version of the EU lists.

12 Ecological information
- Ecotoxical effects:
- Aquatic toxicity: 56-81-5 glycerol LC50/24 h: > 5000 ppm (fish)
- General notes: generally not hazardous for water

13 Disposal considerations
- Product
- Recommendation: Smaller quantities can be disposed with household waste
- Uncleaned packaging
- Recommendation: Disposal must be made according to official regulations
- Recommended cleansing agents: Water, if necessary together with cleansing agents.

14 Transport information
- Land transport ARD / RID (cross border)
- UN-Number: -
- Hazard label: -

15 Regulatory information
- Labelling according to EU Guidelines: The substance is not subject to classification according to EU lists and other sources of literature known to us. Observe the general safety regulations when handling chemicals, The product is not subject to identification regulations under EU Directives and the Ordinance on Hazardous Materials (German GefStoffV).
- National regulations
- Classification according to VbF: Void
- Technical instructions (air): Class Share in %: NK 100.0
- Waterhazard class: Generally not hazardous for water.

16 Other information
This information is based on present knowledge. However, this shall not constitute a guarantee for any specific product features and shall not establish a legally valid contractual relationship.
- Department issuing MSDS: Environmentally Department
- Contact: P. van Pelt

10.8 International Chemical Safety Card for Glycerol

The following pages contain information on the International Chemical Safety Card for glycerol. The THA01 user is responsible for obtaining the latest Version of this card from the glycerol manufacturer when purchasing glycerol for use with THA01.

International Chemical Safety Cards

GLYCEROL			ICSC: 0624

GLYCEROL
Glycerin
1,2,3-Propanetriol
1,2,3-Trihydroxypropane
$C_3H_8O_3$ / CH_2OH-$CHOH$-CH_2OH
Molecular mass: 92.1
CAS # 56-81-5
RTECS # MA8050000
ICSC # 0624

TYPES OF HAZARD/ EXPOSURE	ACUTE HAZARDS/ SYMPTOMS	PREVENTION	FIRST AID/ FIRE FIGHTING
FIRE	Combustible. Gives off irritating or toxic fumes (or gases) in a fire.	NO open flames.	Powder, alcohol-resistant foam, water spray, carbon dioxide.
EXPLOSION			In case of fire: keep drums, etc., cool by spraying with water.
EXPOSURE		PREVENT GENERATION OF MISTS!	
INHALATION	Cough.	Ventilation.	Fresh air, rest.
SKIN	Dry skin.	Protective gloves.	Remove contaminated clothes. Rinse skin with plenty of water or shower.
EYES	Redness. Pain.	Safety goggles.	First rinse with plenty of water for several minutes (remove contact lenses if easily possible), then take to a doctor.
INGESTION	Nausea. Vomiting.	Do not eat, drink, or smoke during work.	Rinse mouth. Give plenty of water to drink. Rest. Refer for medical attention.

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SPILLAGE DISPOSAL	STORAGE	PACKAGING & LABELLING
Ventilation. Collect leaking liquid in covered containers. Absorb remaining liquid in sand or inert absorbent and remove to safe place.	Separated from strong oxidants. Dry.	
SEE IMPORTANT INFORMATION ON BACK		
ICSC: 0624	Prepared in the context of cooperation between the International Programme on Chemical Safety & the Commission of the European Communities © IPCS CEC 1993	

International Chemical Safety Cards		
GLYCEROL		**ICSC: 0624**
I M P O R T A N T D A T A	PHYSICAL STATE; APPEARANCE: HYGROSCOPIC, VISCOUS COLOURLESS LIQUID. PHYSICAL DANGERS: CHEMICAL DANGERS: The substance decomposes on heating producing corrosive fumes of acrolein. Reacts with strong oxidants causing fire and explosion hazard. Reacts violently with acetic anhydrides in the presence of a catalyst. OCCUPATIONAL EXPOSURE LIMITS (OELs): TLV: ppm; 10 mg/m^3 mist (ACGIH 1995-1996).	ROUTES OF EXPOSURE: The substance can be absorbed into the body by inhalation of its mist. INHALATION RISK: Evaporation at 20°C is negligible; a nuisance-causing concentration of airborne particles can, however, be reached quickly on spraying. EFFECTS OF SHORT-TERM EXPOSURE: The substance irritates the eyes, the skin and the respiratory tract. EFFECTS OF LONG-TERM OR REPEATED EXPOSURE:
PHYSICAL PROPERTIES	Decomposes below boiling point at 171°C Melting point: 18.2°C Relative density (water = 1): 1.26 Solubility in water: miscible Vapour pressure, Pa at 20°C: <0.1	Relative vapour density (air = 1): 3.17 Flash point: 177°C. Auto-ignition temperature: 370°C Explosive limits, vol% in air: 0.9-? Octanol/water partition coefficient as log Pow: -1.76
ENVIRONMENTAL DATA		
N O T E S		
	Also consult ICSC # 0090, acrolein.	
	NFPA Code: H1; F1; R0	
ADDITIONAL INFORMATION		
GLYCEROL		
ICSC: 0624		
© IPCS, CEC, 1993		
IMPORTANT LEGAL NOTICE:	Neither the CEC or the IPCS nor any person acting on behalf of the CEC or the IPCS is responsible for the use which might be made of this information. This card contains the collective views of the IPCS Peer Review Committee and may not reflect in all cases all the detailed requirements included in national legislation on the subject. The user should verify compliance of the cards with the relevant legislation in the country of use.	

10.9 CE declaration of conformity

C E

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

We: Hukseflux Thermal Sensors

Declare that the product: THASYS

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
July 2008