

Accu
instruments



thw-01L

**A Transient Hot-Wire Instrument (2 Wires)
for the absolute, low-uncertainty measurement
of the Thermal Conductivity of liquids**

In a few words...

The instrument that you have acquired is suitable for the measurement of the thermal conductivity of non-electrically conductive liquids over a range of 0.1 to 0.7 Wm⁻¹K⁻¹, with very low absolute uncertainty (1%). The technique employed is the 2-wires, Transient Hot-Wire (THW) technique. The main characteristics of the instrument are

- it employs a full theoretical model. No approximations!
- it is an absolute method, i.e. no calibration or reference sample is required,
- it gives reliable results with low absolute uncertainty of 1%,

The instrument is suitable for users in industry and academia, research and development institutions.

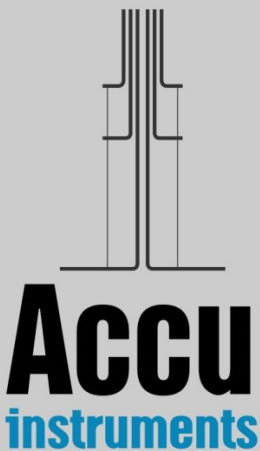
Our experts have more than 30 years of experience with the method and they have been improving and extending the application of this technique. For the first time now, you are holding **a portable instrument based on the 2-wires Transient Hot-Wire technique that is characterized by an absolute low uncertainty of 1%**. This absolute uncertainty is confirmed by measuring the thermal conductivity of toluene (a liquid thermal conductivity reference material of 0.5% uncertainty).

The instrument is composed of

- a) a sensor composed of two-Ta wires of 25 μm diameter inserted in the fluid.
- b) An automatic electronic bridge that employs an ARM architecture CPU to control output voltage and data processing. It has the ability to begin measurements from 1 ms after the initiation of heating and to obtain a large number of data points (usually 400 in one run).
- c) A laptop that controls the experiment setup. It includes
 - the **AccuMeasurement F1.2** custom designed software that performs the measurements, and
 - the **AccuAnalysis F1.2** custom designed software for analyzing the results and obtaining the thermal conductivity.

The Operating Manual has 3 sections:

- Section 1. The theory behind...
- Section 2. Installation.
- Section 3. Instrument Operation.



Section 1

The theory behind...

In the transient hot-wire technique, the thermal conductivity of the liquid is determined by observing the temporal temperature rise of a thin vertical wire, immersed in the fluid (Fig. 1). The wire is initially at equilibrium and a step voltage is applied to it. In this way, electrical current flows through the wire and heats it up, thus creating in the fluid a line source of essentially uniform heat flux per unit length that is constant in time. This has the effect of producing a temperature field throughout the fluid which increases with time.

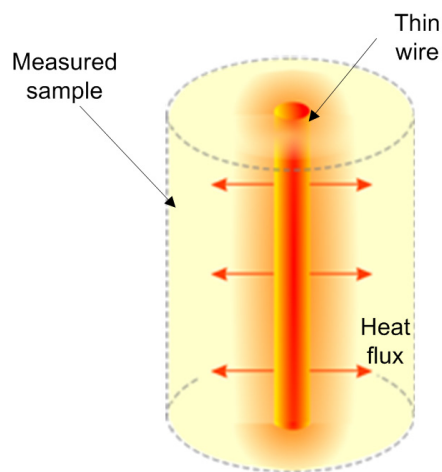


Figure 1. Schematic illustration of the transient hot-wire principle

We note that the wire acts in a double role,

- a) of a line source of constant heat flux per unit length, and
- b) of a temperature resistance thermometer, as its material is usually pure (tantalum in this case).

The evolution of the wire's temperature depends on the thermal conductivity of the test fluid. Thus, the thermal conductivity of the test fluid can be calculated by monitoring the temperature rise of the wire and taking into account the properties of the wire and of the surrounding fluid, as well as the geometry of the wire's enclosure. Finally, to avoid end effects, two wires identical except for their length, are employed. Thus, if arrangements are made to measure the difference of the resistance of the two wires as a function of time, the measurement corresponds to the resistance change of a finite section of an infinite wire (as the end effects being very similar, are subtracted), from which the temperature rise can be determined. **This way absolute measurements can be performed (i.e. no calibration or reference sample is required).**

1.1. Working equations

The actual sensor is composed of two thin tantalum wires spot welded on 1-mm thick tantalum wire supports (Fig. 2). Heat from the wire, is transmitted from the wires to the liquid.

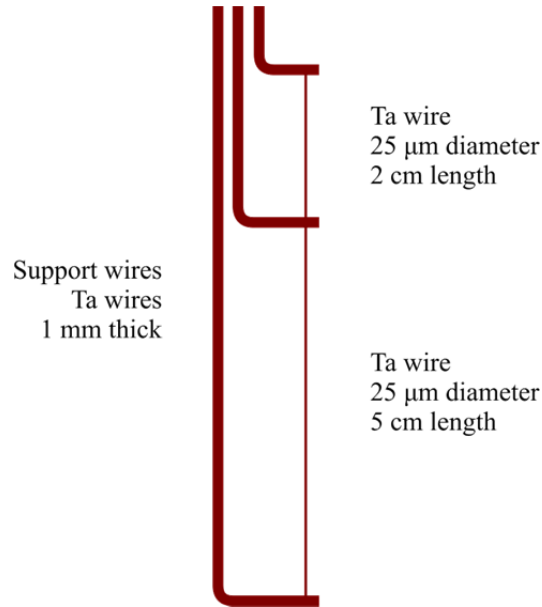
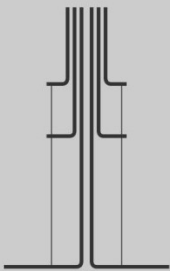


Figure 2. Schematic illustration of the transient hot-wire sensor

In the ideal model, an infinitely long, vertical thin wire with zero heat capacity and infinite thermal conductivity is immersed in an infinite isotropic material of temperature-dependent thermal conductivity λ [$\text{Wm}^{-1}\text{K}^{-1}$], which at time $t = 0$ is at thermodynamic equilibrium with the wire at temperature T_0 . The application of a step voltage across the wire generates heat flux per unit length q [Wm^{-1}]. Note that the heat is transferred through conduction only, since radiation can be considered negligible (as the temperature rise is about 2-5 K), and the effect of convection is rendered minimum by the operation of the instrument. Thus, the heat transfer phenomenon can be described by Fourier's partial differential equation,

$$\rho c_p \frac{\partial T}{\partial t} = \lambda \left[\frac{1}{r} \frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial z^2} \right] \quad (1)$$

where T is the absolute temperature [K], t the time [s], ρ the density [kg m^{-3}], c_p the heat capacity at constant pressure [$\text{J kg}^{-1}\text{K}^{-1}$], and r the wire radius [m].



At the wire radius, the solution of Eq. (1), taking into account the terms that are not negligible, is:

$$\Delta T(r,t) = \frac{q}{4\pi\lambda} \left[\ln \left(\frac{4\alpha t}{r^2 C} \right) \right] \quad (2)$$

where $C = e^\gamma$, and γ is Euler's constant. **Eq.(2) implies that plotting the temperature rise vs the logarithm of the time, will produce a straight line from whose slope the thermal conductivity can be obtained.**

For measurements in liquids, Eq. (2) is the fundamental working equation according to the transient hot-wire technique.

The experimental setup is designed in a way that it approaches the above ideal model, but in practice the actual setup leads to a number of deviations from the ideal solution. Thus, a number of corrections need to be made to the experimentally acquired data, so that they can be adjusted to the ideal model.

$$\Delta T(r,t) = \Delta T_{\text{exp}}(r,t) + \sum_i \delta T_i \quad (3)$$

The applied corrections may be negligible or not, depending on how important their effect is on the actual temporal rise (effect less than 0.01% on ΔT).^{1,2} Due to the design and construction of the setup, the effect of convection and radiation is negligible. On the other hand, three contributions remain that cannot be neglected

- i) the outer boundary,
- ii) the finite wire's properties, and
- iii) the test material's properties.

i) The outer boundary correction

In the ideal model, there is no boundary surrounding the heat source. Thus, an infinite outer boundary to the heat wave is ideally considered. However, this is not the case in practice, as in an actual experimental setup, there is always a vessel around the wire.

There is however an analytic correction to the temperature rise.¹ In practice, the design of the sensor (the wires) is such that this correction is never larger than 1% of the temperature rise.

ii) The finite wire's properties

This correction takes into account the actual properties of the tantalum wire. The wire employed in the apparatus has a finite radius, a finite thermal conductivity, and a heat capacity per unit volume.

Also in this case, there is an analytic solution to the temperature rise.¹ In practice, this correction never exceeds 1% of the temperature rise.

iii) The fluid's properties

The variation of the thermal conductivity and the heat capacity per unit volume of the liquid to be measured with temperature, produces a correction that should be applied to the ideal solution. Since the temperature rise is of the order of 2-5 K, this variation is small. Nevertheless, there is an analytic correction¹ and this is applied to the acquired experimental data.

The temperature rise is obtained by employing an automatic bridge. This will be discussed in the following section.

1.2. The electronic bridge

As already discussed, the purpose of employing an electronic bridge in the transient hot-wire instrument is twofold: first, to measure the evolution of the resistance change of a finite segment of infinite wire (by automatically compensating for axial heat conduction from the wire ends), and second, to ensure that a known constant heat flux is generated in the hot wires.

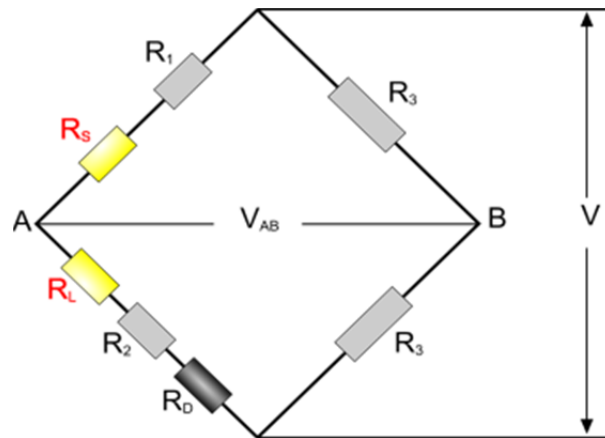


Figure 3. Schematic diagram of the electronic bridge

This is achieved by placing two identical wires of different length and resistance (R_L and R_s) in the two arms of a Wheatstone-type bridge circuit (Fig. 3). In this way the bridge becomes sensitive to the difference in resistance of the two wires, which is equivalent to the resistance of a segment of an infinite wire. The temperature rise, which is required to calculate the thermal conductivity, can then be calculated from the temperature-resistance characteristics of the hot wires.

To obtain the absolute temporal resistance change, upon initiation of the voltage applied by the user, the actual steady state resistance values of the two wires are also required (resistance values at zero voltage, i.e. at room temperature). These are obtained automatically by a steady-state measurement before the transient one.

Hence the bridge performs two tasks

- **Steady-state measurements** of the resistance of the wires before the start of the experiment, and
- **Transient measurements** of the resistance change of the wires during heating.

Solving the bridge equations,³ the absolute resistance change of the central portion of the long wire is obtained and from that, the actual temperature rise at the surface of the wire.

The principal characteristics of the new bridge circuit are:

- a) It is a compact, portable, printed electronic board that employs an ARM architecture CPU to control output voltage and data processing.
- b) It has the ability to begin measurements from 1 ms after the initiation of heating and to obtain a large number of data points (usually 400 in one run).
- c) Times from 0.001 s to 1 s, up to 400 points, with a temperature resolution of 5 mK and time resolution of 1 μ s
- d) All required bridge resistances are mounted on this new board.
- e) Change of the wires' resistances over time is obtained by recording all voltages at the left arm and the off-equilibrium signal of the bridge, at predetermined times.
- f) Steady-state values required are acquired through voltage ratios employing a known standard resistance of high accuracy.

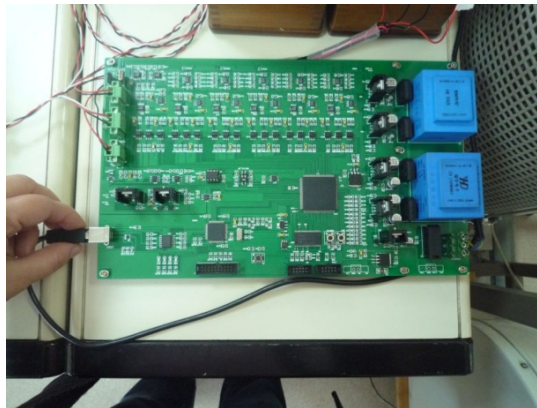


Figure 4. Bridge circuit

1.3. Analysis – Results

AccuAnalysis F1.2 reads the voltages obtained by the electronic bridge (with AccuMeasurement F1.2), and converts them into resistance changes and consequently, temperature rise at the wire surface, as a function of time (Fig. 5). These temperature rises are corrected for the outer boundary correction, and the finite wire's properties.

The corrected temperature rises are plotted vs the logarithm of the time. The result is a straight line. From its slope, according to Eq. (2), the thermal conductivity is obtained. A typical graph is shown in Fig. 6.

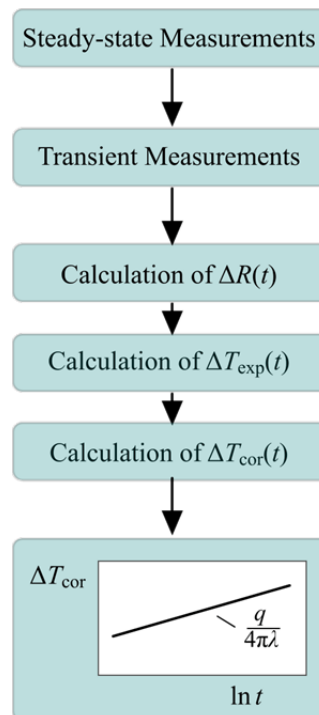


Figure 5. Procedure to obtain the thermal conductivity

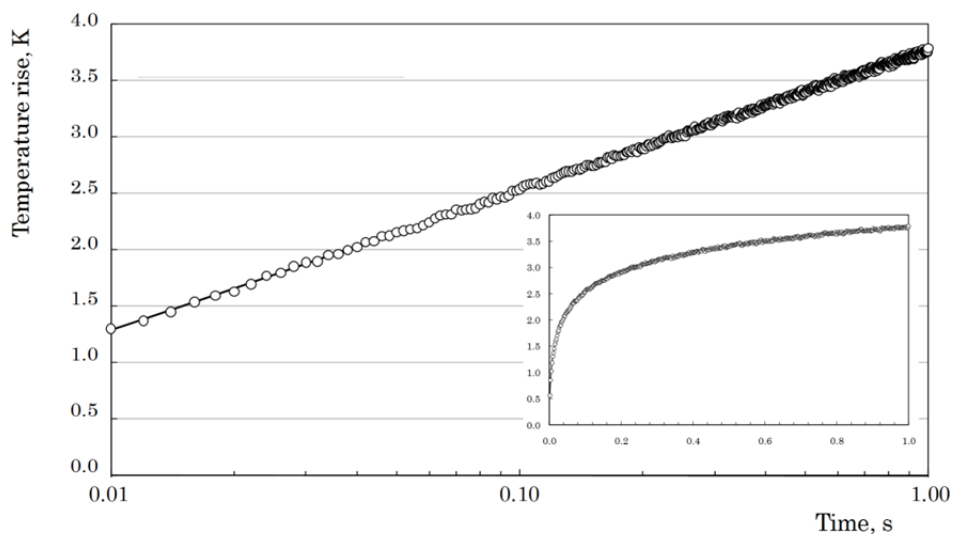
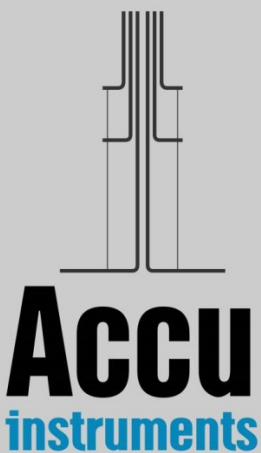


Figure 6. Corrected temperature rise as a function of time



1.4. Discussion of uncertainty

It is already discussed in the previous section that prior of use, the instrument is tested by measuring the thermal conductivity of toluene. As the thermal conductivity of toluene is known⁴ with an uncertainty of better than 0.5%, this comparison shows that the absolute uncertainty of the present instrument is 1%.

Other errors that can affect the measurement of the thermal conductivity of the liquid are

a) Supply voltage

The voltage applied to the bridge, and its evolution during a transient experimental run, is monitored digitally with an uncertainty of about $\pm 1 \mu\text{V}$. As it was shown, the recorded voltage differences are transformed into resistances changes and consequently on temperature differences of a wire with no ends. The effect of this parameter on the temperature rise of the wire is estimated to be of the order of $10^{-4} \%$.

b) Experimental time

The experimental time is measured and registered through the electronic board with an uncertainty of $\pm 1 \mu\text{s}$. In the calculation of thermal conductivity value, the logarithm of time is used and therefore its influence on the uncertainty of the obtained value is lower than $\pm 10^{-3} \%$.

c) Temperature coefficients of resistance

This was obtained by employing a Class I platinum resistance thermometer, with an uncertainty of $\pm 1 \text{ mK}$. This has no measurable effect in the temperature rise of the wire.

d) Other parameters

There are no other variables with a measurable effect on the measurement. According to the Joint Committee for Guides in Metrology,⁵ the combined uncertainty $u_c(y)$ of the quantity $Y(x)$, is the positive square root of the combined variance $u_c^2(y)$ obtained from:

$$u_c^2(y) = \sum_{i=1}^N \left(\frac{\partial Y(x)}{\partial x_i} \right)^2 u^2(x_i) \quad (4)$$

where $u^2(x_i)$ are the variances of the input quantity x_i . The partial derivatives of $Y(x)$ are called sensitivity coefficients and describe how the output estimate y varies with change in the values of the input estimates x_1, x_2, \dots, x_N . Applying Eq.(4) in the aforementioned errors, and taking into account the ability of checking with toluene, it is believed that **the instrument is able to measure the thermal conductivity with an absolute uncertainty of less than 1%.**

Section 2

Installation

2.1. Preinstalled Software

The software is already pre-installed in your computer. In your desktop there should be two icons

- AccuMeasurement F1.2
- AccuAnalysis F1.2

AccuMeasurements performs the measurements, while AccuAnalysis performs the analysis of the measurements.

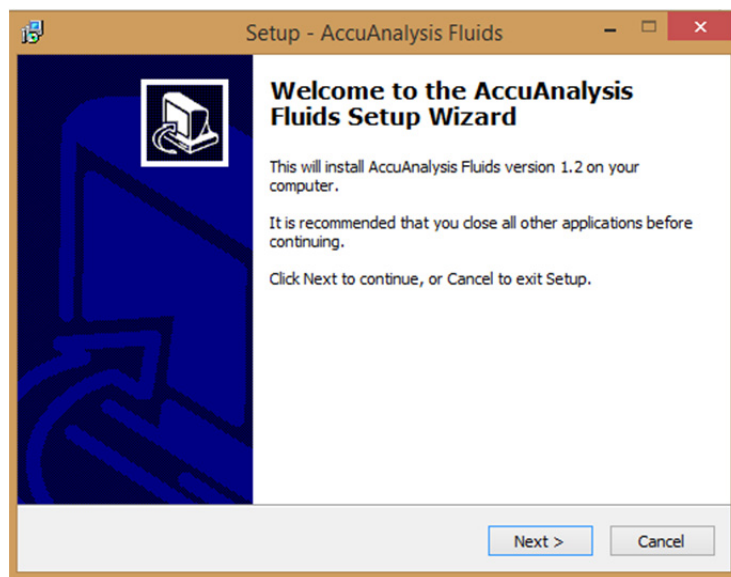
In this case, no further installation is necessary. Please proceed to Section 3.

2.2. Software not-Installed

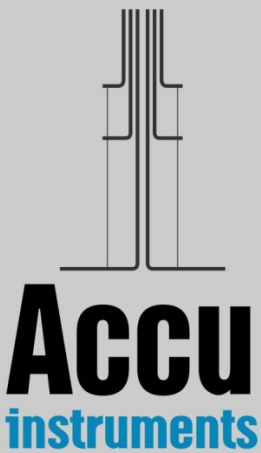
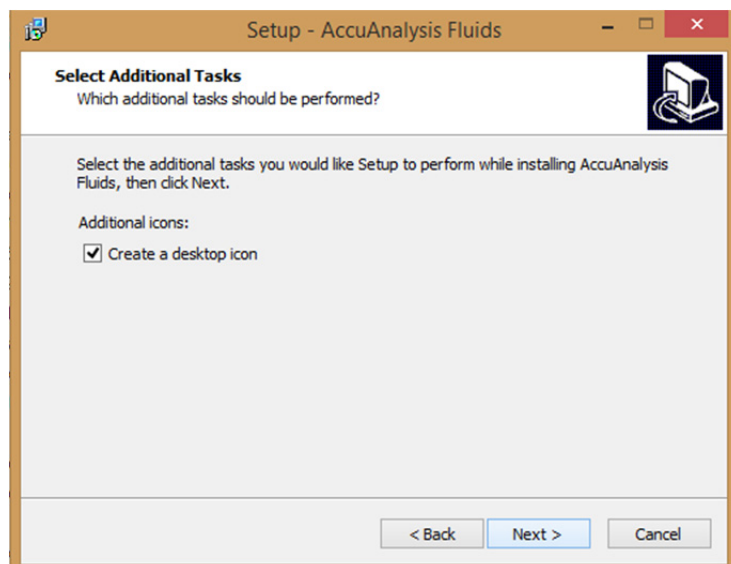
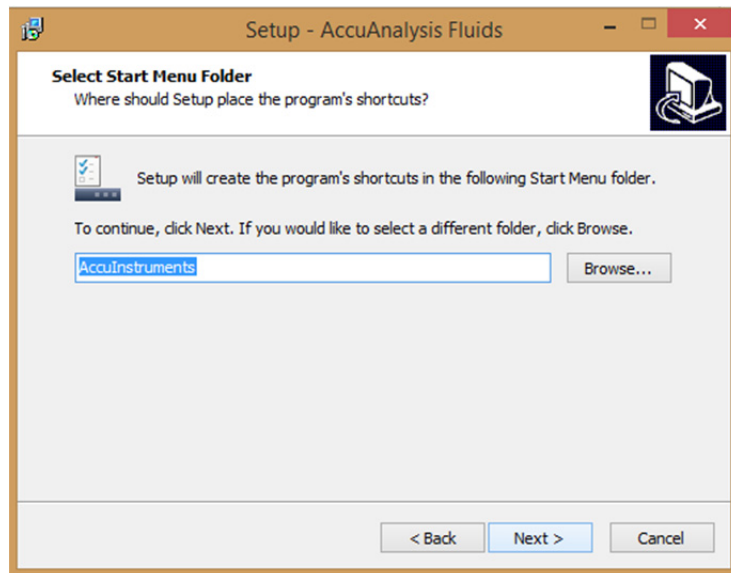
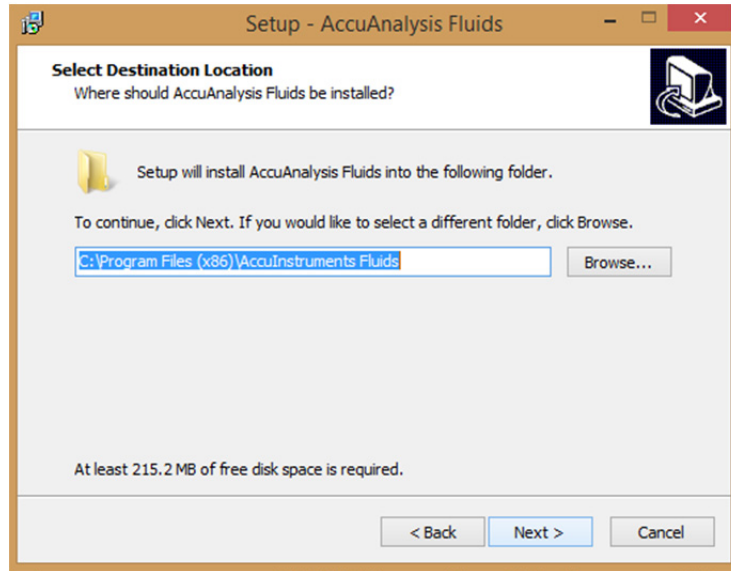
If the software is not installed (the two icons are not present), or a more recent version is supplied, then please follow the next steps.

Step 1. Double click on '**setup_fluids_1.2.exe**'. If the message 'Do you want to allow the following program from an unknown publisher to make changes to this computer' appears, select 'Yes'.

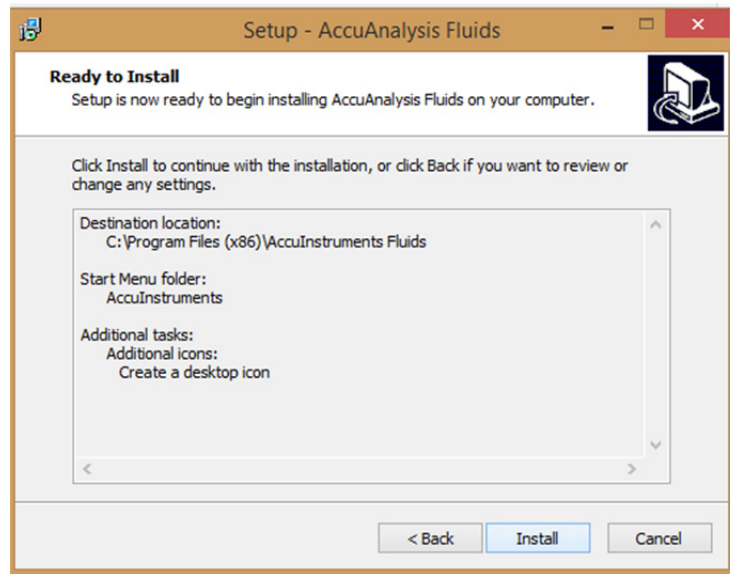
The following screen appears. Select 'Next'



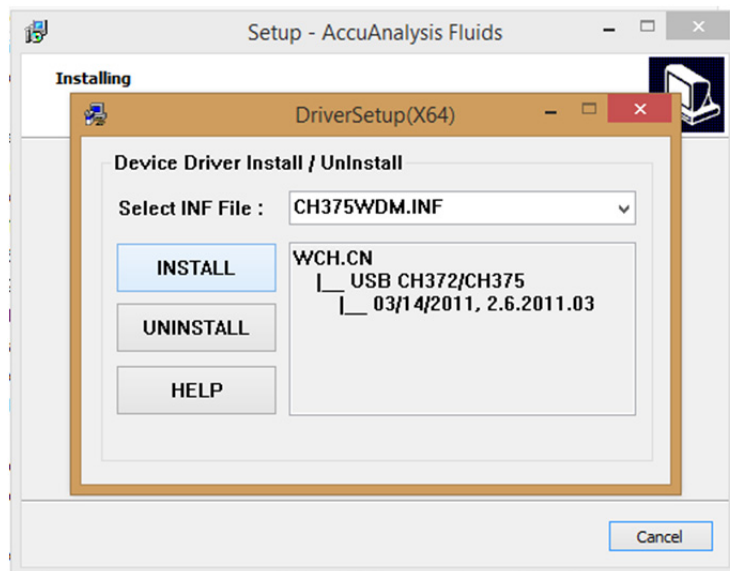
Step 2. Select as destination directory, start-menu folder and desktop icon (to create the two shortcuts), as shown.



Step 3. Select 'Install'.



Step 4. Select 'Install' to install the driver.



Once the driver is installed, select 'OK' and close the window by clicking on its top right corner.

Step 5. Select 'Finish' to finish the installation. In your desktop there should be two icons

- AccuMeasurement F1.2
- AccuAnalysis F1.2

Section 3

Instrument Operation

The instrument you have bought for the measurement of the thermal conductivity of liquids is composed of

- a) **the sensor and the vessel** where the liquid is placedd,
- b) **the electronic board** for the acquisition of the measurement, and
- c) **the laptop (I5) with the software** provided for the data acquisition and analysis, through which the user performs the measurement.

Before we proceed with the operation we ought to say a few words about the sensor. The actual sensor (shown in Fig. 7, is composed of two 25 μm -diameter tantalum wires (5 and 2 cm length), spot welded in 1 mm thick tantalum wire supports, all placed in a SS vessel (not a pressure vessel!), shown in Fig. 8. Holes are provided in the vessel for thermocouples or Pt-thermometers. The sensor and the vessel can be used for measurements up to 200 $^{\circ}\text{C}$.

Alternatively a glass vessel for lower temperatures, can also be provided.

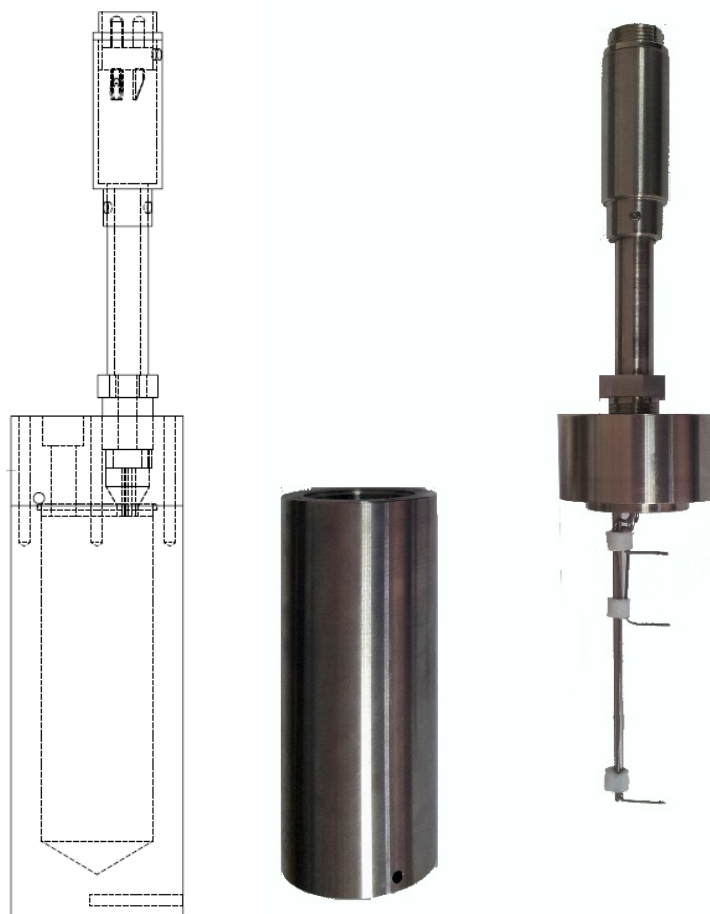
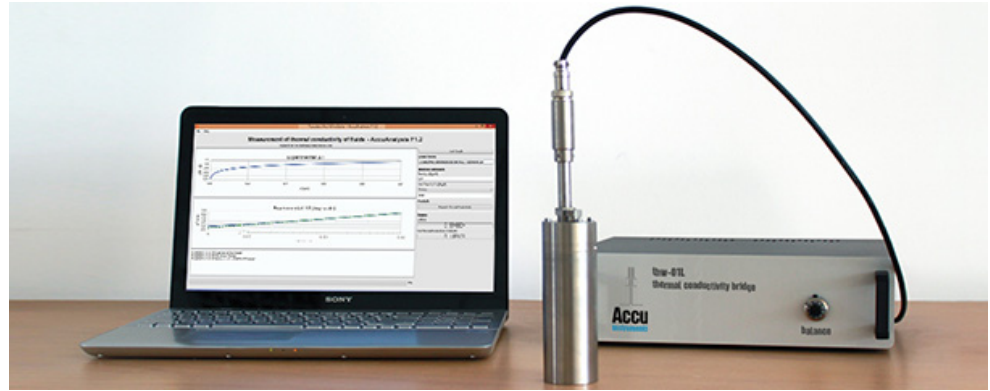


Figure 7. The thermal conductivity sensor

Figure 8. The **thw-01L** instrument

3.1. Step-by-step operation

Your instrument needs no real installation. You simply place the components on a surface with adequate space for working comfortably. You fill the vessel with the liquid and lower carefully the cap with the sensor (or you fill it carefully with the vessel closed). Connect the sensor to the electronic bridge, the electronic bridge to the laptop and you plug the power cable of the electronic bridge and of the laptop to an electrical current outlet (230 V – 50 Hz). Then the laptop is turned on and the user is logged in with the Username/Password provided. Following this, the bridge is activated by turning on the mains switch.

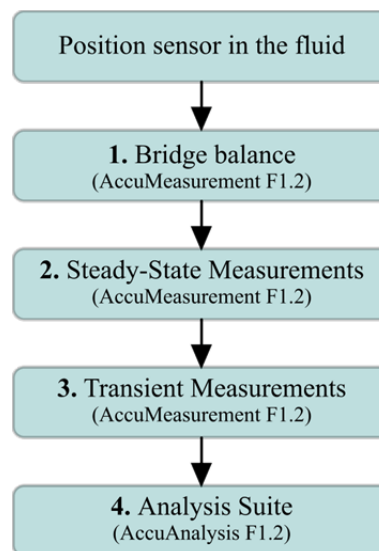


Figure 9. Procedure of measurement

The measurement procedure, shown in Fig. 9, is simple, fast and user-friendly. The liquid to be measured needs to be homogeneous and non-electrically conductive. After filling the liquid, please allow at least 30 minutes for the setup to achieve thermal equilibrium. Measure the ambient temperature, T_0 , of the whole

instrument setup. Avoid moving the liquid (or the vessel) during or before measurements (convection could be initiated).

Step 1. Bridge balance (AccuMeasurement F1.2)

Initiate the data acquisition interface of the electronic board by double-clicking on the 'AccuMeasurement F1.2' icon, located on the laptop desktop. The 'Transient Hot-Wire Control Form', Fig. 10, will appear on the screen.

Before a measurement, the bridge must be balanced. After pressing the top 'Start' button, the user needs to adjust the value of the resistance (R2) located on the front of the electronic box, so that the measured voltage shown next to it is equal or less than 10^{-5} V. This step should be completed rather quickly (a few seconds), because 0.5 V is applied to the bridge, thus heating the sensor's wires. Once this limit is reached press the 'Stop'.

You may need to allow yourself some time to familiarize with the balancing of the bridge. When you have gained confidence in the first step, you can proceed with the measurement of the thermal conductivity. If you are not able to balance within a few seconds, meaning that the initial value of the resistance was far off, do try again after allowing some time for thermal equilibrium. Remember that if you keep on balancing the bridge, you are also heating the liquid, hence convection could appear!

If the bridge has not be connected to mains supply, and the 'Start' button is selected, a warning message appears that reads 'Cannot find a device during enumeration'. In this case 'OK' is selected and the bridge is connected to mains.

Figure 10. Transient Hot-Wire Control Form

Step 2. Steady-state Measurements (AccuMeasurement F1.2)

The next step is the three steady state measurements at 0.5, 1.0 and 1.5 V, that will enable getting the correct temporal rise of the temperature difference of the working wire. This is done automatically by pressing the start button in the Steady-state Measurements section.

Step 3. Transient Measurements (AccuMeasurement F1.2)

Before moving to this step, the user should allow 2 minutes for thermal equilibrium. The transient part is the actual measurement of the temporal rise of the temperature difference of the working wire of your setup. In the case of liquids, this part lasts 1 s. During this step, the voltage you have already selected in the start-up screen ('Voltage' field in Transient Measurement section) is applied, leading to the heating of the wires.

The typical range for the Voltage value selected, is between 3 to 5 V, and depends on the thermal conductivity of the liquid sample. The Voltage selected should have a value so that the calculated maximum temperature rise (Step 4) is in the range of 2.5 to 5 K, i.e. liquids with low thermal conductivity ($0.1-0.3 \text{ Wm}^{-1}\text{K}^{-1}$) require Voltage of about 3.5 V, while liquids with higher thermal conductivity ($0.5-0.7 \text{ Wm}^{-1}\text{K}^{-1}$) require a Voltage of around 5 V.

The transient run starts when the user presses the 'Start' button in the Transient Measurement section. Once the 'Start' button is pressed, transient measurements are recorded until the button is available again; then press 'OK' to close the form. The acquired experimental data are stored by default in the file

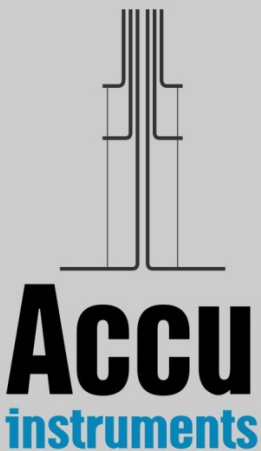
'C:\Program Files\AccuInstruments Fluids\bridge\hotwire.csv'

Step 4. Measurements Analysis (AccuAnalysis F1.2)

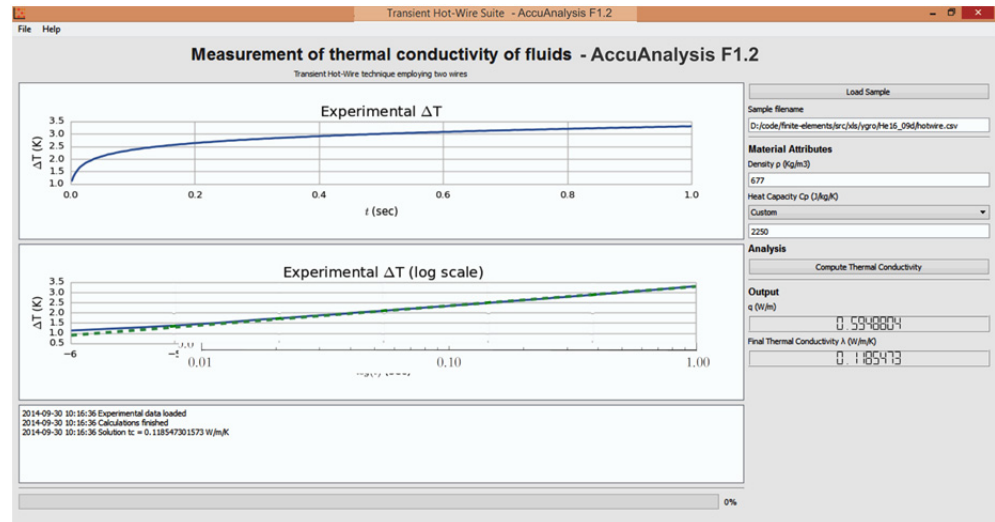
The next step is the actual calculation of the liquid's thermal conductivity employing the user-friendly analysis software AccuAnalysis F1.2. The start-up screen (Fig. 11) appears by double clicking on 'AccuAnalysis F1.2' icon located on the laptop desktop. The user places all the necessary data on the right side of the screen. More analytically:

- The user clicks on the 'Load Sample' button and selects the experimental data file 'Hotwire.csv' created in the previous Step.
- The user needs to provide the following liquid's properties.
 - a) **Type the value for the density** [kg/m^3].
This is usually easily obtained by weighing a liquid sample of specific volume.
 - b) **Type the value for the heat capacity** [$\text{Jkg}^{-1}\text{K}^{-1}$].
You can select Custom and type the value, or select the value from an indicative list for the liquid closest to your sample.

When you are ready, press the 'Compute Thermal Conductivity' button and the value of the thermal conductivity of the liquid will be automatically computed.

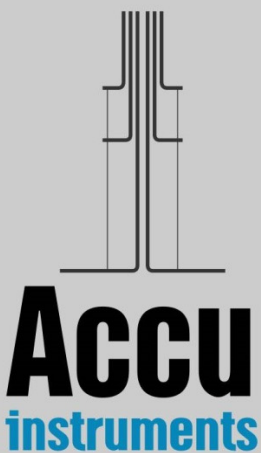


During the calculation of the thermal conductivity of the liquid, the changes of voltage differences are converted to resistance differences and these are subsequently converted to temporal temperature difference. Then, the corrections are applied, and the thermal conductivity is obtained from the slope of the temperature rise vs $\ln t$ plot. In Fig. 11 the experimental temperature difference is plotted against time on the top diagram, and vs the logarithm of the time on the bottom. On your right you can see the heat per unit length, q , generated in the transient part of the measurement.



This is a detailed view of the right-hand menu of the software. It includes a 'Load Sample' button, a 'Sample filename' field with the path 'D:/code/finite-elements/src/xls/ygro/He16_09d/hotwire.csv', and 'Material Attributes' for Density ρ (Kg/m³) = 677 and Heat Capacity C_p (J/kg/K) = 2250. Below these are 'Analysis' and 'Compute Thermal Conductivity' buttons. The 'Output' section displays 'q (W/m) = 0.5948804' and 'Final Thermal Conductivity λ (W/m/K) = 0.1185473'.

Figure 11. (Top) AccuAnalysis F1.2 Form. (Bottom) Detail of right menu of AccuAnalysis F1.2.



References

1. Healy J.J., de Groot J.J., and Kestin J., 'The Theory of the Transient Hot Wire method for measuring the Thermal Conductivity', *Physica C* **82**:392-408 (1976).
2. Kestin J., and Wakeham W.A., 'A Contribution to the Theory of the Transient Hot Wire technique for the Thermal Conductivity Measurements', *Physica A* **92**:102-106 (1978).
3. Assael M.J., Dix M., Gialou K., Vozar L. and Wakeham W.A., 'Application of the Transient Hot-Wire Technique to the Measurement of the Thermal Conductivity of Solids', *Int. J. Thermophys.* **23**:615-633 (2002)
4. Ramires M.L.V., Nieto de Castro C.A., Perkins R.A., Nagasaka Y., Nagashima A., Assael M.J., and Wakeham W.A., 'Reference Data for the Thermal Conductivity of Saturated Liquid Toluene over a wide Range of Temperatures', *J. Phys. Chem. Ref. Data* **29**:133-139 (2000).
5. Joint Committee for Guides in Metrology, 2008, *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement (GUM)*, JCGM-100:2008.



AccuInstruments is a startup company, currently based in Greece, created by people that have been in this field for more than thirty years. Our instruments are a result of the latest technology developed under an excellent cooperation between Laboratories in Greece and China.

We have started with two Transient Hot-Wire Instruments, one for solids and one for liquids, to establish our reputation.

For further information, please contact

- Information: info@accuinstruments.com
- Sales: sales@accuinstruments.com
- Support: support@accuinstruments.com

or visit us at www.accuinstruments.com

