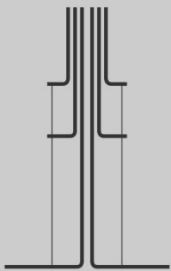# thw-01S

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

In a few words…

The instrument that you have acquired is suitable for the measurement of the thermal conductivity of solids over a range of 0.1 to 4 $Wm^{-1}K^{-1}$, with very low absolute uncertainty (2%). 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 2%,

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 2%**. As there is no solid thermal conductivity reference material to assure this uncertainty, the great advantage of the present instrument is that at its early stage of construction of the sensor, the operation of the instrument can be checked 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 with two-Pt wires of 12 μm diameter embedded in a 1.2-mm thick soft silicone paste that rests on the one side on a plastic support. On its other side, the solid sample to be examined is placed. A thin polyimide film protects the silicone paste from the solid.

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 500 in one run).

c) A laptop that controls the experiment setup. It includes

- the **AccuMeasurement S1.2** custom designed software that performs the measurements, and
- the **AccuAnalysis S1.2** custom designed FEM software for analyzing the results and obtaining the thermal conductivity.
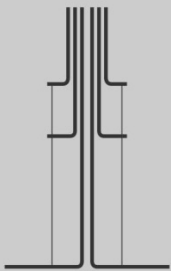
The Operating Manual has 3 sections:

- 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 medium is determined by observing the temporal temperature rise of a thin vertical wire embedded (or immersed in the case of fluids) in the test material (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 medium 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 medium which increases with time.
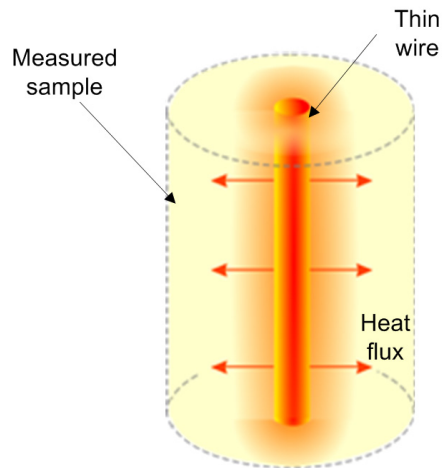

Figure 1. Schematic illustration of the transient hot-wire principle

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 platinum.

The evolution of the wire's temperature depends on the thermal conductivity of the test material. Thus, the thermal conductivity of the test material can be calculated by monitoring the temperature rise of the wire and taking into account the properties of the wire and of the surrounding material, 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 a thin cylindrical platinum wire embedded in a 1.2 mm thick soft silicone-paste layer, that rests on the one side on a plastic support. On its other side, the solid sample to be examined is placed (Fig. 2). A thin polyimide film protects the silicone paste from the solid. Thus heat from the wire, is transmitted through the silicone paste to the solid on the one side and the plastic support on the other side.

Plastic Support

Silicone layer
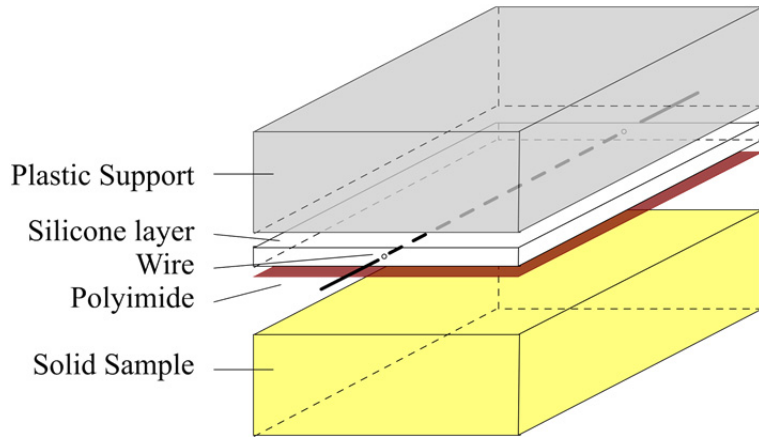
Wire

Polyimide

Solid Sample

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

For each of the materials, the appropriate partial differential equation needs to be solved. The problem is solved in two dimensions (as the wire is considered infinite in the z-direction i.e. its axis), where the x-axis is parallel to the silicone paste and the y-axis is perpendicular to it. Hence, if λ denotes the thermal conductivity [W m^{-1} K^{-1}], T the absolute temperature [K], t the time [s], ρ the density [kg m^{-3}], c_p is the heat capacity at constant pressure [J kg^{-1} K^{-1}], and $a = \lambda / (\rho\, c_p)$ is the constant thermal diffusivity,[1]

a) For the platinum **wire** (W) for $t \geq 0$ and $0 \leq r \leq \alpha$, the equation in two dimensions is:

$$\rho_w c_{pw} \frac{\partial T_w}{\partial t} = \lambda_w \left[\frac{\partial^2 T_w}{\partial x^2} + \frac{\partial^2 T_w}{\partial y^2} \right] + \frac{q}{\alpha^2} \tag{1}$$

b) For the **silicone** paste (and the protective polyimide), the equation is

$$\rho_m c_{pm} \frac{\partial T_m}{\partial t} = \lambda_m \left[\frac{\partial^2 T_m}{\partial x^2} + \frac{\partial^2 T_m}{\partial y^2} \right] \tag{2}$$

c) For the **sample** to be measured and the plastic support, the equation is:

$$\rho_s c_{ps} \frac{\partial T_s}{\partial t} = \lambda_s \left[\frac{\partial^2 T_s}{\partial x^2} + \frac{\partial^2 T_s}{\partial y^2} \right] \tag{3}$$

We do note that
- Eq.(3) is applied on top to the plastic support and underneath to the solid sample. Only the variables (ρ_s, c_{ps} and λ_s) change according to the corresponding solid's properties in that position.
- Also the protective polyimide film (25 microns thick) is in essence incorporated as part of the silicone since its properties are very near the silicone paste properties. This was found to be possible after carefull optimization of the mesh.

The above equations, subjected to initial and boundary conditions, are solved by finite elements method (FEM), for the exact geometry of the sensor.

Therefore for a given heat input, the temperature rise of the sample is calculated by the FEM and compared with the experimental one. The two curves coincide for the correct value of the thermal conductivity of the sample solid. The properties of the silicone paste and the plastic support required are also obtained in the process, as it is discussed in Section 1.3.

Before we proceed with this comparison, which is the innovative core of the technique, we ought first to discuss how the experimental temperature rise is obtained. For this task a novel electronic bridge is available.

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.
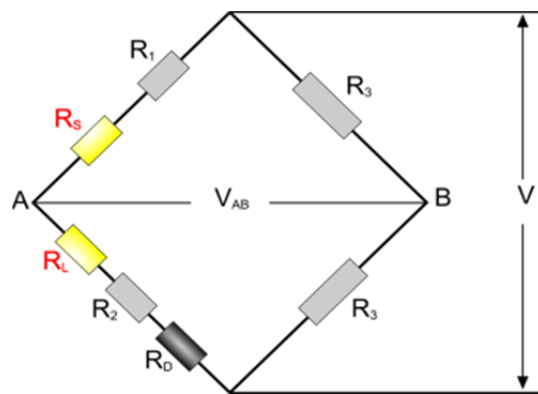

Figure 3. Schematic diagram of the electronic bridge

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,[1] 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. This is compared with the temperature rise obtained by the FEM solution of the theoretical equations for the exact geometry employed.

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 500 in one run).

c) Times from 0.001 s to 10 s, up to 500 points, with a temperature resolution of 5 mK and time resolution of 1 μs.

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.
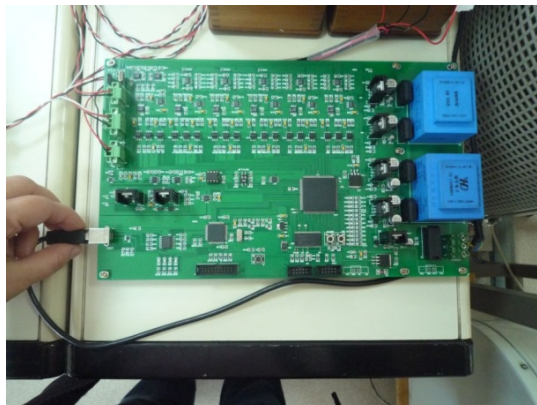

Figure 4. Bridge circuit

1.3. FEM analysis – Results

Eqs. (1)-(3) can not be solved analytically. Instead, it is preferably to solve them by a Finite Elements Method (FEM), over the exact geometry of the sensor. FEM is a numerical analysis technique applied to a wide variety of problems and scientific fields. The basic idea is that a certain given domain can be represented by a mesh of small, interconnected geometric shapes, the finite elements. One of the advantages of the method is the ability to use elements of different shapes and sizes, allowing for more detailed discretization at areas of interest, such as interfaces.

The FEM analysis software, AccuAnalysis S1.2, is a custom built suite that performs two tasks (Fig. 5)

1) For a given thermal conductivity of the solid, and for the sensor geometry, it calculates the temporal temperature-rise curve at the predetermined time intervals of the experiment.

2) It compares the calculated curve with the experimental one, and iterates for the thermal conductivity of the solid sample, until the two curves coincide. When they coincide, then that value of the thermal conductivity is the correct one.
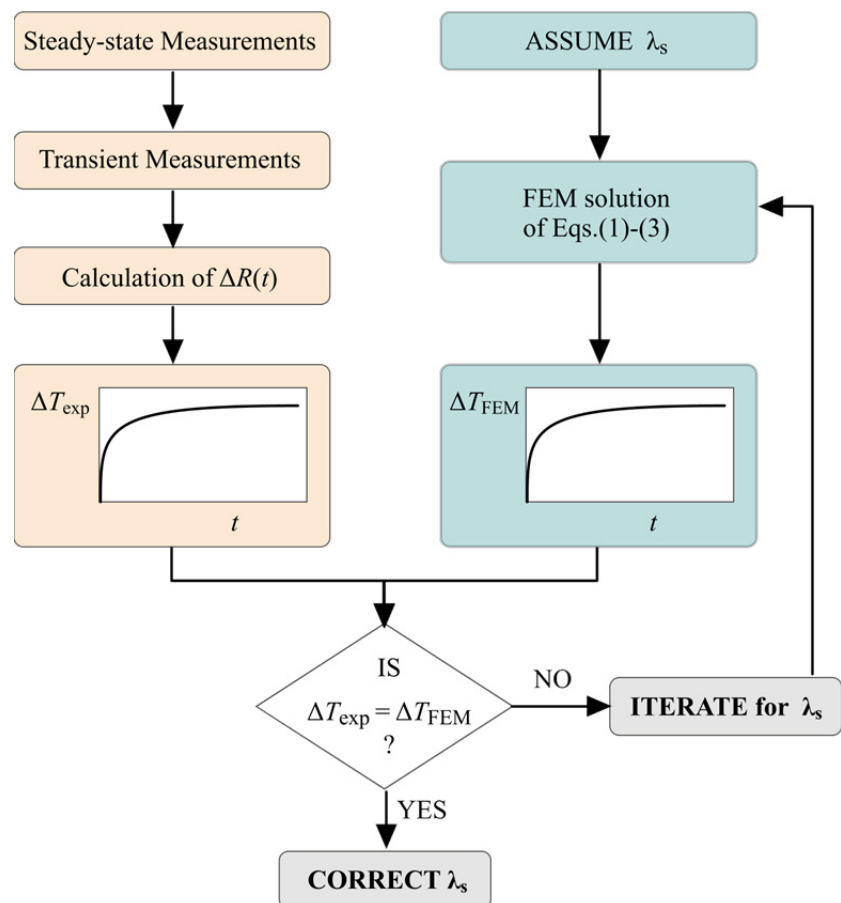

Figure 5. Procedure to obtain the thermal conductivity

AccuAnalysis S1.2 employs the open-source FiPy library developed by NIST (USA), for pre-processing and solving the finite elements. It is written in Python (SciPy, NumPy) and it is very flexible and reliable. The exact geometry of the sensor has been imported in 2D Mesh from the Gmsh CAD application.
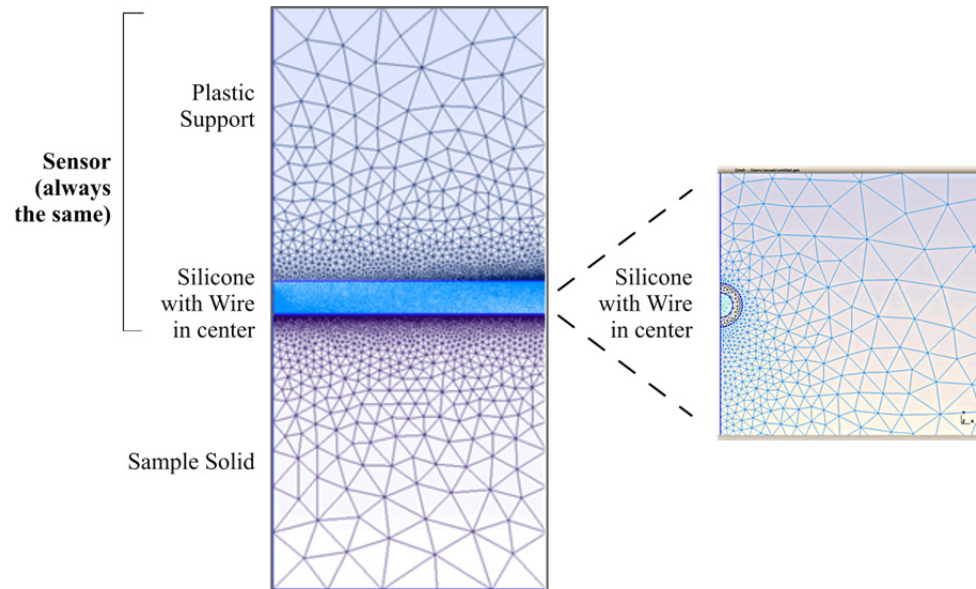

Figure 6. Mesh details

The construction of the mesh is crucial as the geometry changes from the order of cm size in the support and sample, to micron sizes in the wire. For this reason the 2D Frontal Triangulation algorithm is employed, and triangle sizes were optimized adding 'latent' points.

To iterate for the correct thermal conductivity, Machine Learning and the method of **Bayesian Optimization (BO)** technique is used. This is a state-of-the-art technique used in the optimization of black-box functions (derivative free). It actually employs Gaussian Processes to fantasize the function, and an acquisition function to guide exploration/exploitation tradeoff. As a result it usually converge in 5-6 simulations!!!

Two more tools are incorporated in AccuAnalysis S1.2.

- Time steps are optimized. An experimental run usually employs 500 values (1 value every 0.02 s). FiPy showed that 280 values is a good number to use. These are selected as exponentially growing time steps, as more accuracy is required in the first 0.1 s.
- Reusability of run start. Until 0.1 s, the heat has not reached the measured material. Hence, all calculated states until 0.1 s are saved and reused.

Therefore, AccuAnalysis S1.2 succeeds in a very short time to converge to the correct thermal conductivity, with an absolute uncertainty of better than 2%.
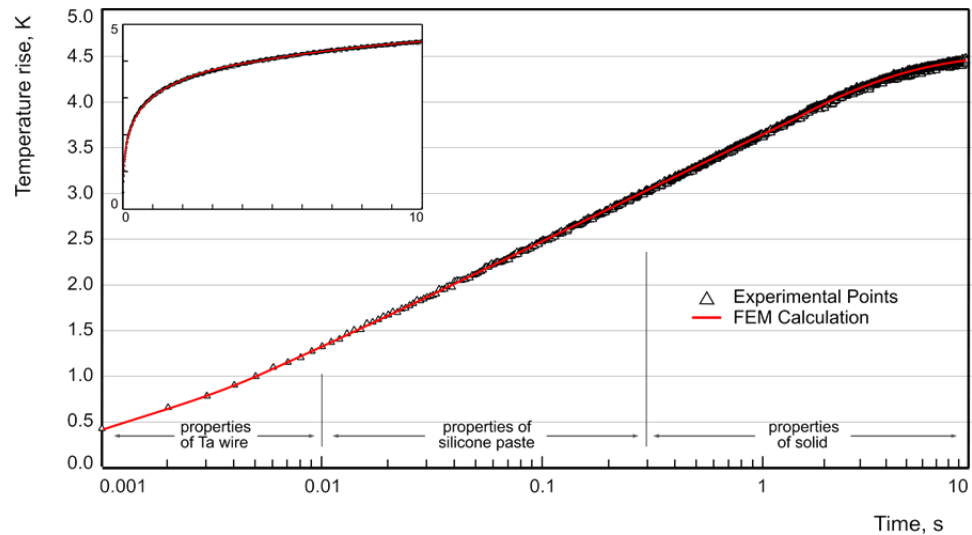
Figure 7. Temperature rise as a function of time, during a measurement

Figure 7 shows a typical experimental temperature rise as a function of the time, during a measurement. In the same figure the calculated temperature rise from AccuAnalysis S1.2 is also shown. Full agreement is observed.

It should be noted that AccuAnalysis S1.2, is also employed for two additional tasks (not available to the end user), during the construction phase of each sensor.

a) Before placing the 2 wires in the soft silicone paste layer, they are inserted in toluene and its thermal conductivity is measured. In this case as it is a single medium all properties of the other three layers (silicone, solid support, sample solid) are set equal. Hence the thermal conductivity of toluene can easily be measured. Toluene is a standard thermal-conductivity reference liquid, and its thermal conductivity is known with an uncertainty of 0.5%.[2] Agreement with experimental value is always within this uncertainty. Hence this comparison, allows us to check the excellent operation of a) all electronic components and bridge operation, b) our measuring procedure, c) the resistance change measurements, d) the temperature rise calculation, and e) the FEM thermal conductivity (for single fluid) calculations. This is a **unique advantage of the present sensor!**

b) The sensor you have in your hand, as already discussed, is composed of two wires embedded in a soft silicone layer that rests on one side on a plastic support. Its other side, protected by a thin polyimide film, comes in contact with the sample solid. Thus the silicone paste and the solid support are always the same. Thus running the experiment

- for very low times (heat wave still in silicone), we can obtain the properties, $(\rho_m c_{pm})$ and λ_m, of the silicone paste.

- Employing as the sample solid, another piece of the plastic support, the only unknowns now are the properties of the plastic support, $(\rho_s c_{ps})$ and λ_s, since the properties of silicone have been obtained. Thus these properties can also be obtained. Therefore the only unknown remaining to be obtained during a run is the thermal conductivity of the sample solid (since $(\rho\, c_p)$ will be given).
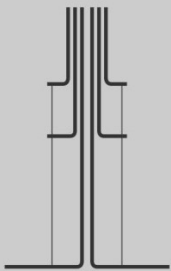
1.4. Discussion of uncertainty

It is already discussed in the previous section that a unique advantage of this instrument is that it can be tested before being embedded in the silicone layer, by measuring the thermal conductivity of toluene. As the thermal conductivity of toluene is known[2] 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 solid sample 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 ±1 µV. 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 ±1 µs. 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 ±1 mK. This has no measurable effect in the temperature rise of the wire.

d) Sensor thickness

The experimental sensor is constructed so that the thickness of the silicone paste film is defined and equal exactly to 1.2 mm. Thus, it does not affect the uncertainty of the technique.

e) Other parameters

There are no other variables with a measurable effect on the measurement. According to the Joint Committee for Guides in Metrology,[3] 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:

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

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, ..., 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 2%.**

ACCU
instruments

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 S1.2
- AccuAnalysis S1.2

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_solids_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'.
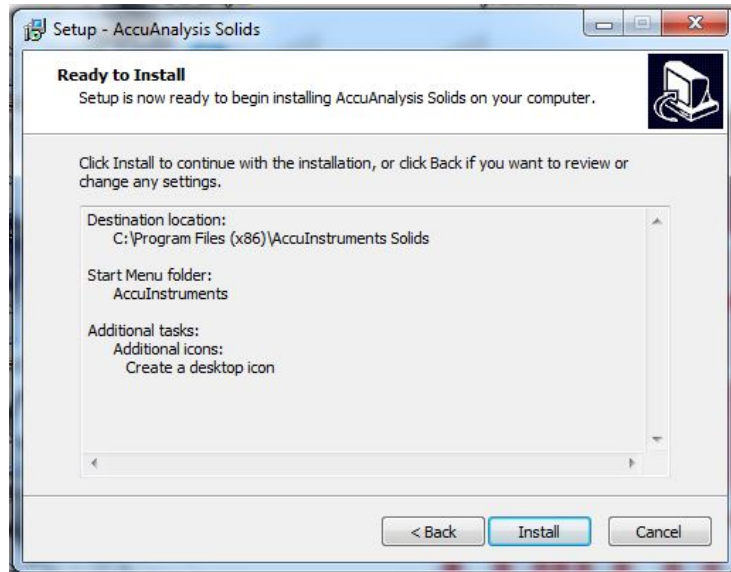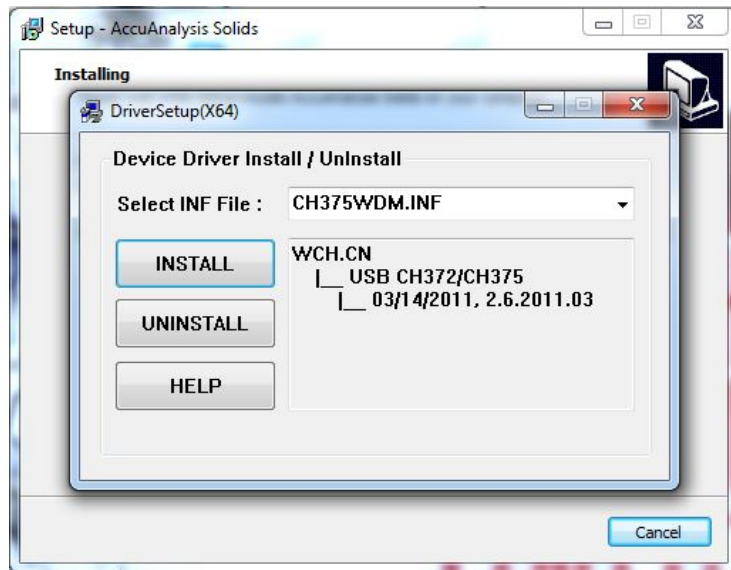The following screen appears. Select 'Next'
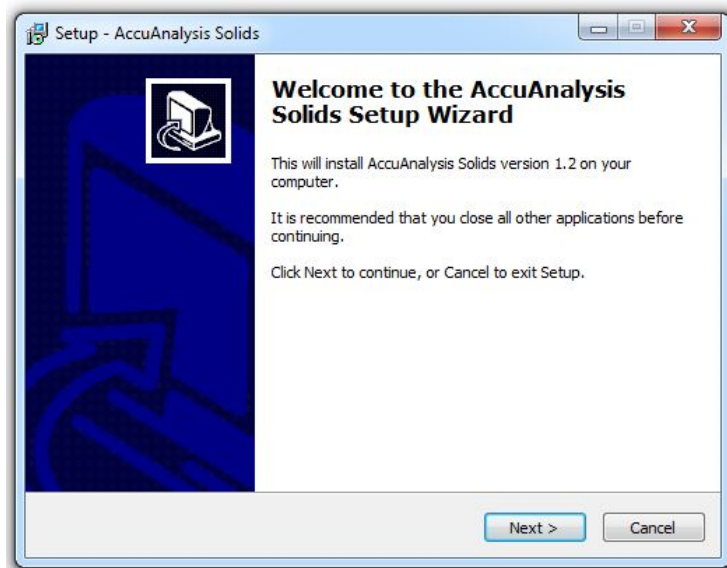
Step 2. Select as destination directory, start-menu folder and desktop icon (to create the two shortcuts), as shown
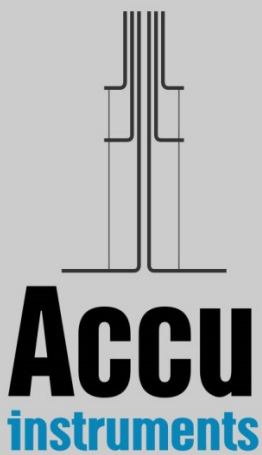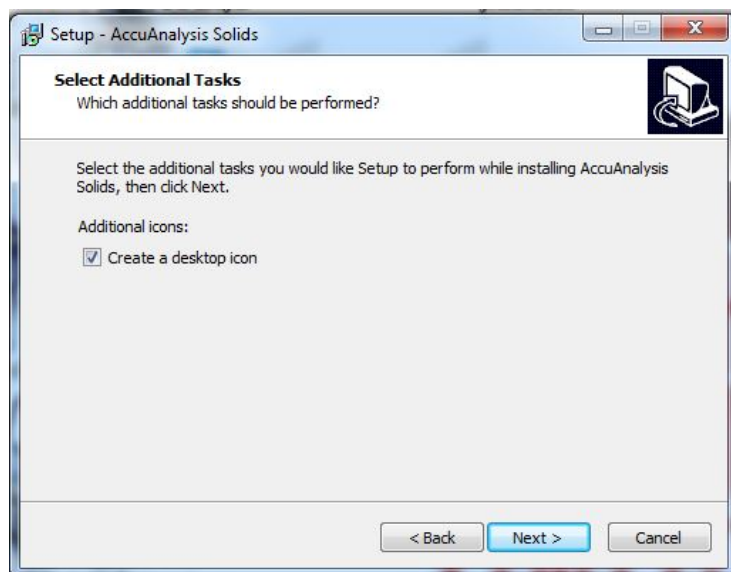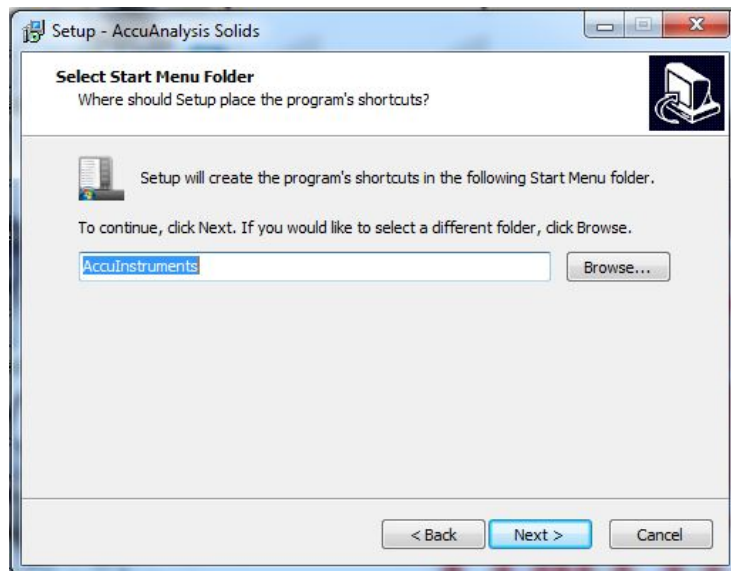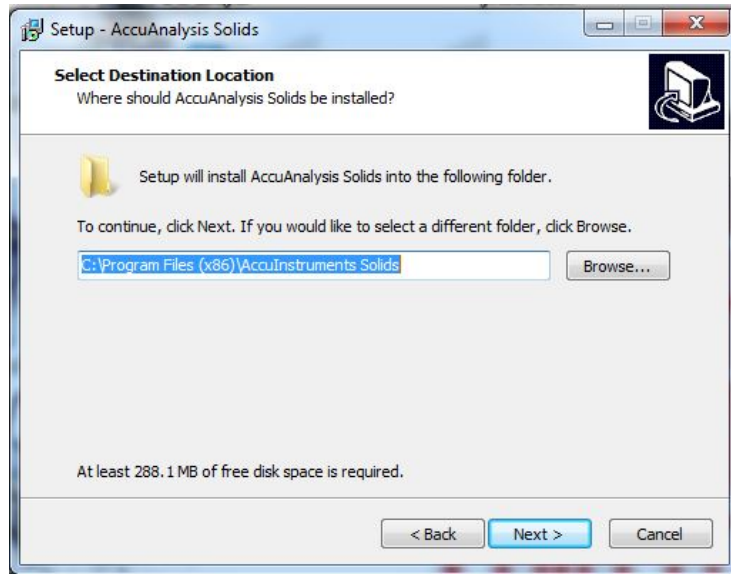
Step 3. Select 'Install'.

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 S1.2
- AccuAnalysis S1.2

Section 3

Instrument Operation

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

    a) **the sensor** where the specimen is placed,

    b) **the electronic board** for the acquisition of the measurement, and

    c) **the laptop (I5) with the software** provided for the data acquisition and FEM analysis, through which the user performs the measurement.

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

Before we proceed with the operation we ought to say a few words about the sensor. The actual sensor (shown in Fig. 9), is composed of two 12 μm-diameter platinum wires (5 and 2 cm length), connected one after the other, and embedded in a 1.2 mm-thick soft silicone-paste layer, that rests on the one side on a plastic support. On its other side, the solid sample to be examined is placed. A thin polyimide film (yellow color in Fig. 9) protects the silicone paste (white color) from the solid.

Weight (1 kg)

Sensor (wires underneath)

Sample whose thermal conductivity is to be measured

Soft silicon paste with 2 Pt wires embedded along its center axis (one after the other)
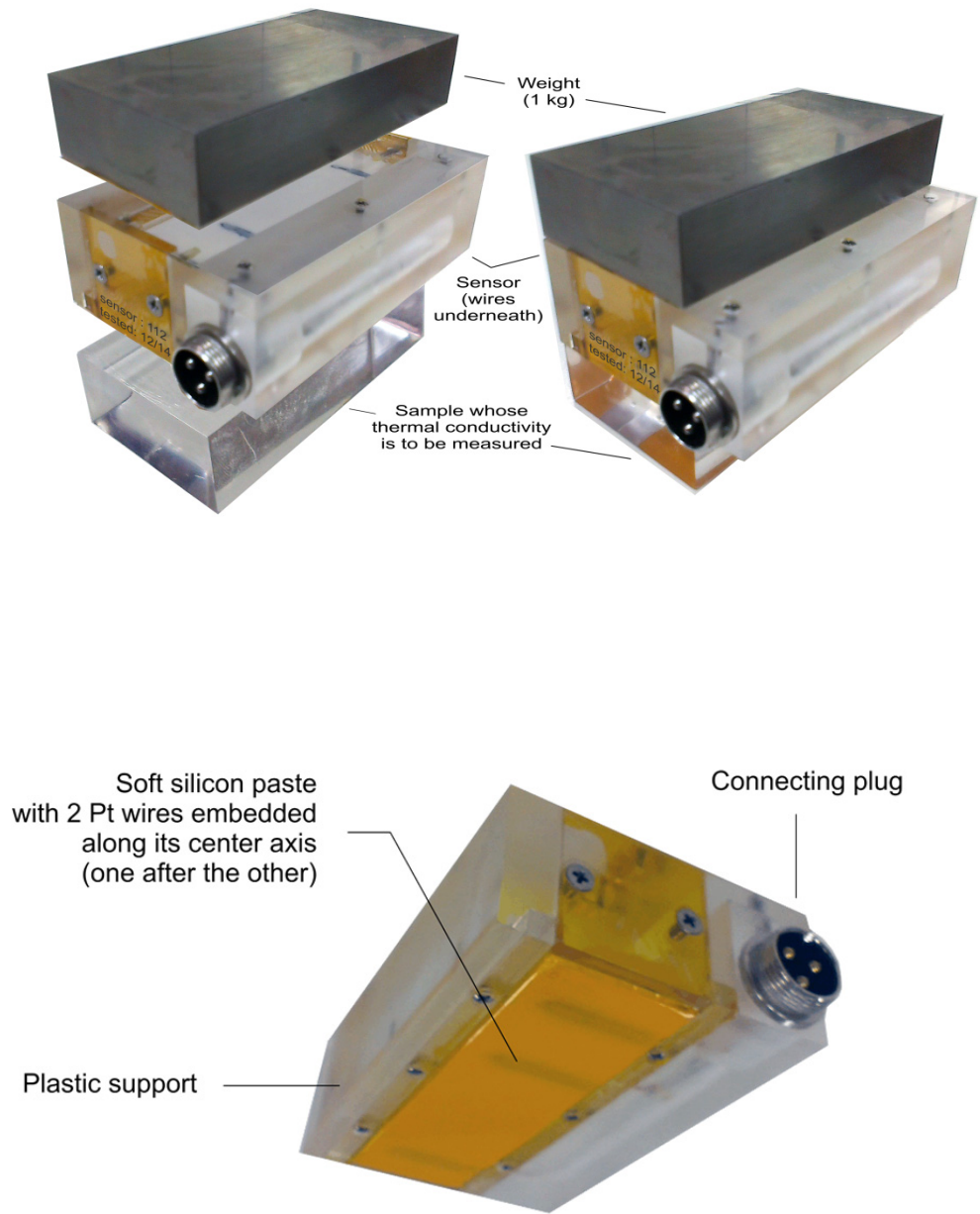
Plastic support

Connecting plug

Figure 9. The thermal conductivity sensor
top: sensor assembly with sample and weight
bottom: sensor view from below.

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 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.
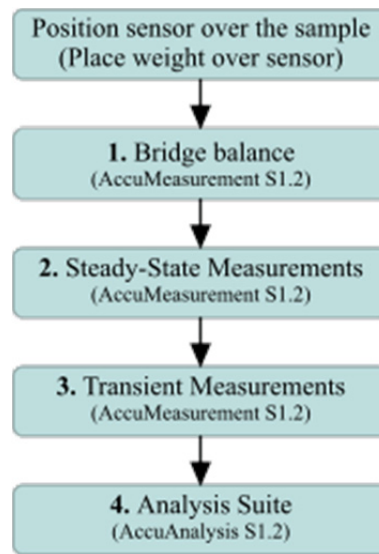
Figure 10. Procedure of measurement

The measurement procedure, shown in Fig. 10, is simple, fast and user-friendly. **The sample solid to be measured needs to be homogeneous**. Firstly, the surface of the sample solid that will be in contact with the apparatus (at least 10 cm x 3 cm) must be very smooth and clean, without bumps or scratches and free from any impurities and dust. Apply a single drop from the oil provided, on the sensor surface to be in contact with the solid. Then, the sensor is placed over the sample solid and the 1 kg metallic weight available with the sensor should be placed over the sensor, to provide a gentle pressure on the surfaces. After placing the sample to be measured, please allow at least 30 minutes for the setup to achieve thermal equilibrium. Measure the ambient temperature, T_0, of the whole instrument setup.

Step 1. Bridge balance (AccuMeasurement S1.2)

Initiate the data acquisition interface of the electronic board by double-clicking on the 'AccuMeasurement S1.2' icon, located on the laptop desktop. The 'Transient Hot-Wire Control Form', Fig. 11, 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, in 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' button.

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.

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 11. Transient Hot-Wire Control Form

Step 2. Steady-state Measurements (AccuMeasurement S1.2)

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 S1.2)

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 solids, this part lasts 10 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 sensor's wires.

The typical range for the Voltage value selected, is between 3 to 5 V, and depends on the thermal conductivity of the solid 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. materials with low thermal conductivity (0.1-0.5 $Wm^{-1}K^{-1}$) require Voltage of about 3.5 V, while materials with higher thermal conductivity (1-4 $Wm^{-1}K^{-1}$) 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

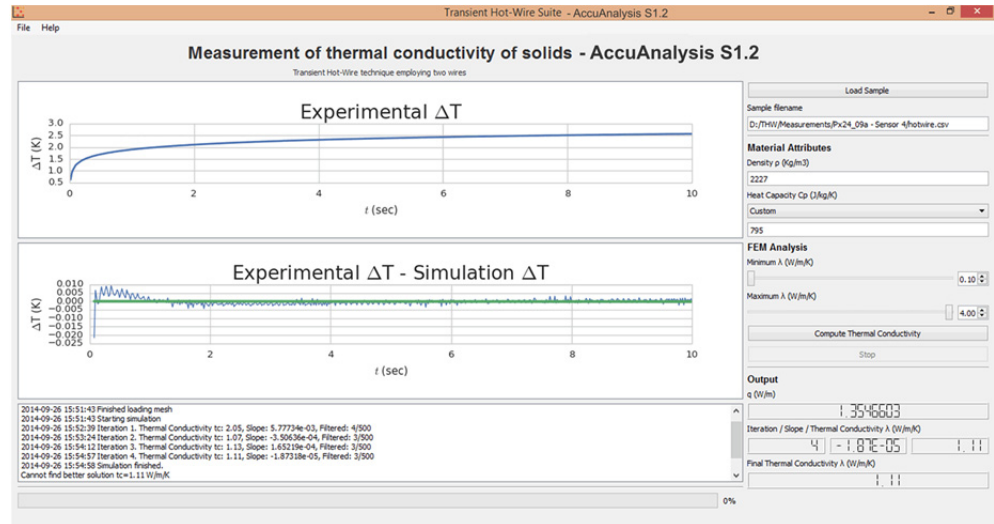**'C:\Program Files\AccuInstruments Solids\bridge\hotwire.csv**

You can move this file to another directory, to avoid overwriting.

Step 4. Measurements Analysis (AccuAnalysis S1.2)

The next step is the actual calculation of the sample's thermal conductivity employing the user-friendly analysis suite AccuAnalysis S1.2. The start-up screen (Fig. 12) appears by double clicking on 'AccuAnalysis S1.2' icon located on the laptop desktop. The user places all the necessary data on the right side of the screen. More analytically:
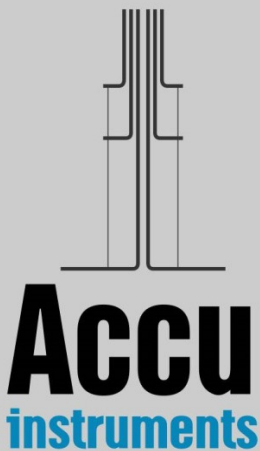
- The user clicks on the 'Load Sample' button and selects the experimental data file 'hotwire.csv' created in the previous Step.
- The user needs to provide the following material's properties.
  a) **Type the value for the density** [kg/m^3].
     This is usually easily obtained by weighing the sample.
  b) **Type the value for the heat capacity** [$Jkg^{-1}K^{-1}$].
     You can select Custom and type the value, or select the value from an indicative list for the material closest to your sample.
- Additionally, for the analysis part, select the minimum and maximum value for the thermal conductivity. If you don't have an estimate for the thermal conductivity, you can leave the values as they are. Otherwise, you can narrow down the interval where the program will look for the value of the thermal conductivity. In this way, you can minimize the iterations and, thus, the time needed for the acquisition of your experimental result.

When you are ready, press the 'Compute Thermal Conductivity' button and the calculation is initiated, which is divided in four stages. During the execution of the calculation, the progress is shown in the bar bellow the aforementioned button. You can choose to abort the procedure at any time by pressing the 'Stop'.
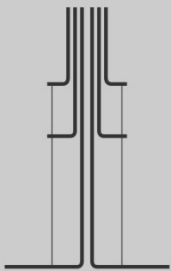

Figure 12. (Top) AccuAnalysis S1.2 Form.
(Bottom) Detail of right menu of AccuAnalysis S1.2.

During these stages the calculation of the thermal conductivity of the sample solid is accomplished. The changes of voltage differences are converted to resistance differences and these are subsequently converted to temporal temperature difference. Then, the finite elements part of the program kicks in. While the program is executed, you can see at your left side two graphs (Fig. 12). On the top, the experimental temperature difference is plotted against time and, on the bottom, the difference between the experimentally obtained temperature difference and the FEM calculated one, is shown.

The trend line at the lower diagram must be a horizontal straight line (the slope is less that 10^{-5}). On your right you can see the heat per unit length, q, generated in the transient part of the measurement. As the solving process using finite elements progresses, you can see the current number of iteration, along with its slope and estimate of the thermal conductivity. When the convergence criterion is met, the final value of the thermal conductivity λ [Wm^{-1}K^{-1}] appears. This is your final experimental result!

<div align="center">

**PLEASE remember that
the thermal conductivity has only meaning
when measuring homogeneous solids.**

</div>

References

1. 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)

2. 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).

3. Joint Committee for Guides in Metrology, 2008, *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement (GUM)*, JCGM-100:2008.
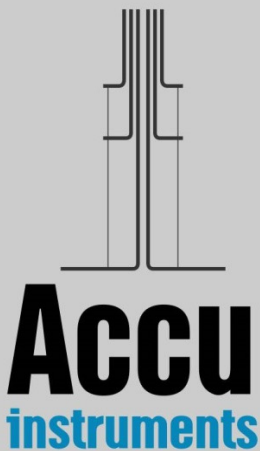
AccuInstruments is a startup company, currently based in Greece, created by people that have been in this field for more than thirty years. Our instruments are a result of the latest technology developed under an excellent cooperation between Laboratories in Greece and China.

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**