TAM Air Calorimeter

Operator's Manual

Revision G
Issued September 2011

159 Lukens Drive
New Castle, DE 19720

Notice

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Notes, Cautions, and Warnings

The following conventions are used throughout this guide to point out items of importance to you as you read through the instructions.

A NOTE highlights important information about equipment or procedures.

A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Safety

Instrument Symbols

The following label is displayed on the TAM Air instrument for your protection:

Symbol	Explanation
	This symbol indicates that you must unplug the instrument before doing any maintenance or repair work; AC mains power voltage is present in this system. High voltages are present in this instrument. If you are not trained in electrical procedures, do not remove the covers unless specifically instructed to do so in the manual. Maintenance and repair of internal parts must be performed only by TA Instruments qualified service personnel.

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *TAM Air Calorimeter Operator's Manual* contains cautions and warnings that **must** be followed for your own safety.

Electrical Safety

Always disconnect TAM Air from the mains electricity supply before attempting to change the fuse and before opening the back panel.

WARNING: For protection against fire hazard, ensure that the correct fuse is always used. Always use a 4A Slow for 100/130V, or 2A Slow for 200/250V. Note the tag on the back side of the instrument that indicates the required voltage.

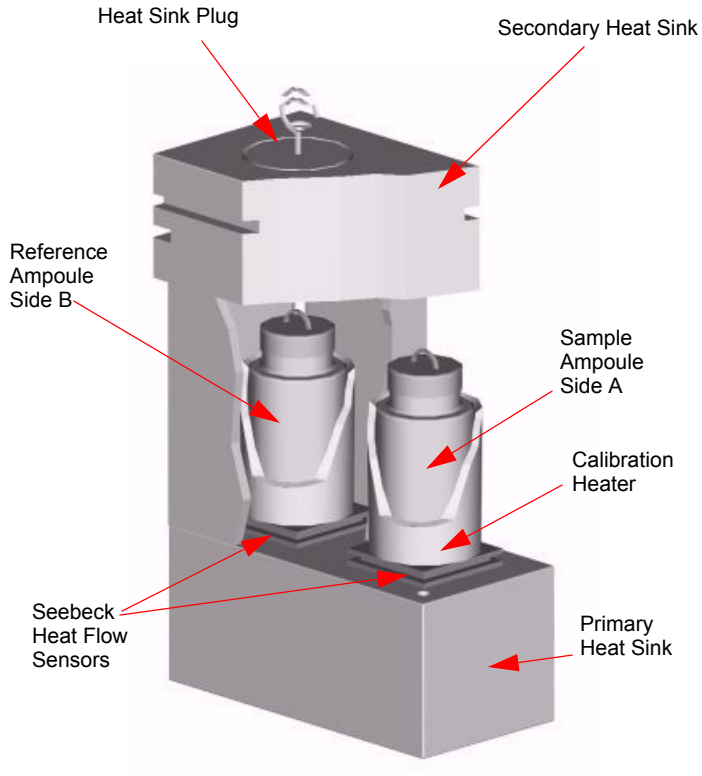
Chapter 1

Introducing the TAM Air Isothermal Calorimeter

Overview

TAM Air is an eight-channel, isothermal, heat conduction calorimeter operating in the milliwatt range. All eight calorimetric channels are mounted together to form a calorimeter block (single heat-sink block) housed in a temperature controlled air thermostat.

Each calorimetric channel is constructed in twin configuration with one side for the sample and the other side for a reference. The figure below illustrates this twin configuration.



Cutaway View
Twin Configuration
One of Eight Calorimetric Channels

TAM Air Isothermal Calorimeter

During measurement, the sample and reference materials are held in 20-mL ampoules. Solids, liquids or slurries may be studied.

The calorimeter block, as well as the ampoule holders, are made of aluminium, which has been anodized to withstand corrosion.

Measurements are recorded continuously and in real time through an eight-channel data logger, which is connected to a computer.

Heat Flow Measurement Principle

Each calorimetric channel is constructed in twin configuration with one side for the sample and the other side for a static reference. Each calorimeter operates using the *heat flow principle*. Heat created by any physical or chemical reaction in the sample will flow rapidly to its surroundings.

Within each calorimetric channel, there are two Seebeck heat flow sensors–one under the sample and one under the reference. The main route for heat exchange between the sample and its surrounding is through the heat flow sensor. The flow of heat, caused by the temperature gradient across the sensor, creates a voltage signal proportional to the heat flow.

The twin configuration of sample and reference within a channel allows the heat flow from the active sample to be compared directly with the heat flow from the inert reference. The voltage difference is a quantitative expression of the overall rate of heat production in the sample. This twin measuring principle enhances the heat flow stability and reduces noise within the system.

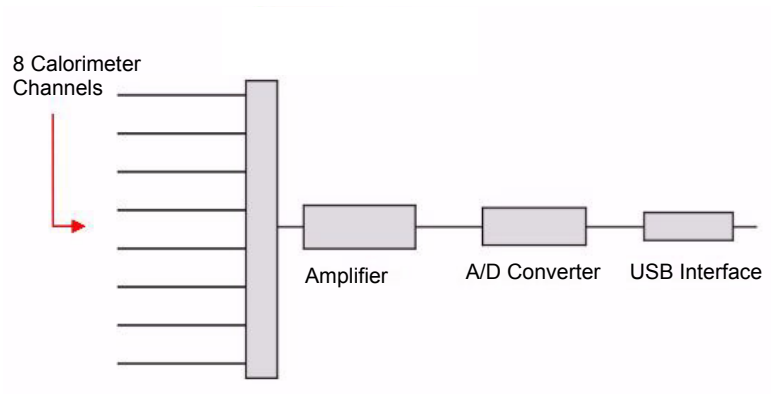
Calorimeter Block

The eight twin calorimetric channels are held together in the calorimeter block. The sample and the reference calorimeters of each channel are accessed from above for loading through inlets marked A and B on the insulated thermostat lid. The ampoule holding the sample is placed in side A, and the reference ampoule into side B. The difference in heat flow between the sample and the reference is monitored, *i.e.*, side A minus side B. Thus, an exothermic process in side A (the sample) results in positive heat flow values.

Each side of the calorimetric channel is sealed with a removable cylindrical metal heat-sink plug to prevent thermal disturbance from the circulating air.

Samples are held in glass, plastic (HDPE), or, for special applications, in stainless steel sealed ampoules.

A connector box mounted on the calorimeter block is used for connection to the data logger via a 25-pin Dsub connector. The data logger includes a 24-bit data logger. The data logger is located in the electronic compartment and connected to the USB port of a computer from the backside of TAM Air. The data logger accepts, amplifies, and converts signals from all eight twin calorimetric channels.



TAM Air Signal Pathway

NOTE: Exothermic reactions result in positive heat flow values (*i.e.*, exotherm is up).

Calibration

The objective of electrical calibration is to calculate an individual calibration constant for each of the eight calorimetric channels. Each channel has a precision calibration heater permanently mounted on the A-side (the sample side). Each has a resistance of 100 ± 0.1 Ω and a very low temperature coefficient.

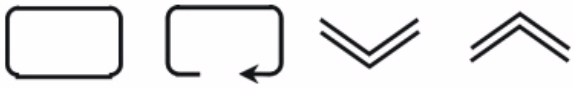
The calibration constant, which TAM Assitant calculates, is called *Gain factor*. The Gain factor is calculated relative to a calibration performed at TA Instruments before the delivery of a new instrument. Hence, the Gain factor is unitless and the deviation is expressed relative to unity. For example, a Gain factor of 0.99 expresses a 1% deviation from the factory calibration.

A full description on how to calibrate is given in Chapter 3.

Operating Temperature Control Panel

The temperature of the air thermostat, *i.e.*, the operating temperature, can also be set from the TAM Air Assistant or the temperature control panel located on the front of the instrument. This set temperature is recalled in the event of a power failure.

The control panel has two digital indicators, one with red digits showing the actual temperature of the air thermostat and the other with green digits showing the set temperature. The amber indicator light in the top right-hand corner of the control panel will normally flash, indicating that the heater is on. The amber light will remain off if the thermostat is cooling.



Temperature Control Panel Operating Buttons

The two arrow buttons are for increasing and for decreasing the set temperature. The two buttons to the left have no function in normal operation. They are used in the factory together with a special code for programming the control panel. Note that settings for the calorimetric temperature also can be made directly in the software, see the section, "TAM Air Assistant Software Overview" for details.

Air Thermostat Temperature Regulation

The operating temperature range of the TAM Air thermostat is 5 °C to 90 °C, with a stability of ± 0.02 °C.

> NOTE: when TAM Air is operated at temperatures below ambient care must be taken to avoid condensation. See the next section, "Operating Below Ambient."

The temperature of the air thermostat is controlled by a large Peltier module operating in conjunction with a temperature-sensing probe. The operating temperature of the air thermostat is set either in the software or on the front control panel. The Peltier module can either heat or cool to maintain the set temperature.

The air thermostat has an insulated outer cabinet with the Peltier module mounted on the bottom. An inner aluminium chamber is located on the Peltier module. At the bottom of the inner chamber there is a raised aluminium plate (calorimetric board) on which the calorimeter block should be positioned.

The circulation of air at a pre-set temperature controls the temperature of the calorimeter block. An internal fan, which is an integral part of the Peltier module, is mounted at the bottom of the inner chamber, beneath the aluminium support plate. The fan continually circulates air around the block and back through the space between the inner chamber and outer cabinet. The inner chamber lid has holes to allow the air to circulate through it. A second fan is mounted on the Peltier module (outside the temperature controlled environment of the air thermostat) to remove the heat produced when TAM Air is operated at low temperatures.A temperature probe is mounted above the large Peltier module, adjacent to the fan and under the aluminium support plate. This probe monitors the temperature of the air circulating within the thermostat.

The signal from the temperature probe is compared with the set operating temperature as selected by the operator. The difference between the two values is fed to a PID regulator and used to control the voltage to the Peltier module.

The power supplies for the Peltier module are located under the air thermostat and are attached to cooling vanes visible through the back panel of the instrument. An external fan, mounted just below the Peltier element, draws cool air from the room via a filter for circulation around the power components and cooling vanes. Air outflow occurs through two vents in the back panel. The external fan is provided with an interchangeable inlet filter to remove dust from the incoming cooling air.

> NOTE: Although the circulating air temperature will reach the set temperature within minutes, the large mass of the calorimeter block will take several hours to stabilize to the set temperature.

Operating Below Ambient

TAM Air can be operated down to 5 °C as long as the dew point is not reached inside the air thermostat or the calorimeters. Before placing samples and ampoules into the calorimeter, we recommend that you thermostat the samples.

The dew point is the temperature at which the relative humidity reaches 100 % and condensation of moisture begins. The dew point depends on the temperature and the relative humidity of the room. For instance, a relative humidity of 65 % at a room temperature of 20, 25, 30, and 35°C results in a dew point of 13, 18, 23, and 27°C respectively.

If the TAM Air is to be operated at a temperature that is lower than the calculated dew point, precautions must be taken to avoid condensation. Recommendations are listed below:

1. Use TAM Air in a cold room at a temperature close to the operating temperature.

2. Flush the calorimeter with a dry gas using the gas inlet on the backside of TAM Air. A small constant flow (approximately 200 mL/min) is generally recommended throughout the measurements to ensure a dry atmosphere. During ampoule loading the gas flow should be increased to avoid humid air to enter. The gas flow should be large enough (approximately 1.0 L/min) to prevent humid air from the surroundings to enter.

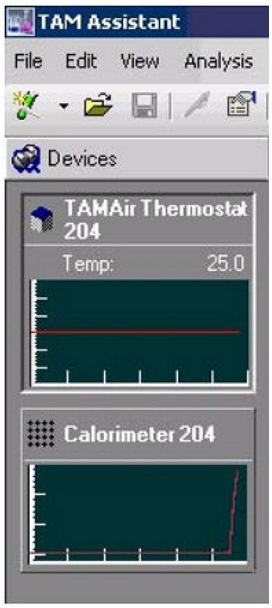
3. If possible, lower the relative humidity of the room.

TAM Air Assistant Software

TAM Air uses *TAM Air Assistant* software for controlling experiments and devices, visualization of results and data analysis.

Overview

TAM Air Assistant is graphical, self instructive software for controlling devices, displaying results, running and analyzing experiments. The software is modular, meaning that different device controllers, experiment types and analysis packages can be added.



TAM Air Assistant Device Manager

- **Device Control** can be accomplished using the *Devices Manager*. The thermostat and the calorimeter block are considered as separate devices in TAM Air. Examples of control features are setting the calorimetric temperature through the thermostat and turning on/off inbuilt calibration heaters in the calorimetric channels.

- **Experiments** can be run using customized wizards to guide you through the whole measurement from start to end. The software divides the calorimetric signal into sections of baselines, pause and main in accordance with the user input. Click **New experiment** in the file menu to choose experiment type and initiate a new experiment.

- The **results file** is created when the experiment wizard has been finalized. The results file is a binary file with the file extension .rstl and can be exported to a text file for analysis with other software such as Microsoft Excel. The data in the TAM Air Assistant results file can be baseline corrected by a single click. The data is normally presented graphically as heat flow versus time, but other plot types can easily be shown, *e.g.*, integrated heat versus time and the quantities normalized against mass.

- The **report function** allows the user to create a report with information about experiment details and various plot types. The report can be saved as a file with the extension .larpt. Plots created from the report file may be easily copied and pasted into alternate programs such as Microsoft Word and Powerpoint.

Technical Specifications

Number of Calorimetric Channels	8
Operating Temperature Range	5 – 90 °C NOTE: For operating temperatures below ambient care must be taken to avoid condensation.
Temperature Accuracy	± 1 °C
Air Thermostat Stability	± 0.02 °C
Maximum Sample Volume	20 mL
Measuring Range	± 600 mW full scale
Time Constant (20 mL water)	< 500 seconds
Limit of Detectability	4 µW
Precision	± 20 µW
Baseline Stability Over 24 Hours: Drift Deviation Error	 < 40 µW < ± 10 µW < ± 23 µW
Short Term Noise	± 4 µW
Overall Dimensions (Width x Depth x Height)	462 mm (18 in.) x 397 mm (15.6 in.) x 900 mm (35.4 in.) (Allow 3 to 4 inches of air space between the instrument and the back and side walls.)
Total Weight	40 kg (88 lbs) approx.
Operating Voltage	110 Vac/220 Vac ±10%, 50/60 Hz
Power Consumption	300 VA

All specifications are valid at an operating temperature of 25 °C, under the recommended external operating conditions of TAM Air. For definitions of specifications, see the next page.

NOTE: The specification varies slightly with temperature and may differ up to 20 percent at an operating temperature of 90 °C.

Recommended External Operating Conditions

Ambient Temperature	20 – 30 °C: for operating temperatures between 15 – 90 °C 5 – 20°C: for operating temperatures 5 – 15 °C
Ambient Temperature Stability	± 1 °C
Relative Humidity	< 65 % RH
Computer Specification	PC running Windows 2000 or higher USB port (Notebook computers may not work due to power drain from USB ports on such computers.)

Definitions

The following terms are defined as applicable to the TAM Air Calorimeter.

Time Constant (20 mL water): The time needed for the heat sensor to detect a heat flow corresponding to 63 % of a heat pulse.

Detectability: The minimum heat flow that can be detected.

Precision: Precision is reported as twice the standard deviation for the results of a series of exactly repeated experiments. Here reported as the baseline variations on ten repeated loadings of a 20 mL disposable glass ampoule.

Base Line Stability Over 24 Hours: Calculated from a linear fit of baseline data, during 24 hours, using the method of least squares.

Drift: Defined as the slope of the linear fit

Deviation: Defined as twice the standard deviation of the linear fit of baseline data.

Error: The maximum error after 24 hours calculated from the following formula.

$$Error = \pm\sqrt{\left(\frac{Drift}{2}\right)^2 + Deviation^2}$$

Short-Term Noise: Defined as the noise of the heat flow signal during 60 sec (peak to peak values).

Chapter 2

Installing the TAM Air Isothermal Calorimeter

Unpacking and Installing the TAM Air

Before shipment, the instrument is inspected both electrically and mechanically so that it is ready for operation upon proper installation. Only limited instructions are given in this manual, consult the online documentation for additional information. Installation involves the following procedures:

- Inspecting the system for shipping damage and missing parts
- Connecting cables and lines

It is recommended that you have your instrument installed by a TA Instruments Service Representative, call for an installation appointment when you receive your instrument.

CAUTION: To avoid mistakes, read this entire chapter before you begin installation.

Inspecting the System

When you receive your TAM Air, look over the instrument and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

- If the instrument is damaged, notify the carrier and TA Instruments immediately.
- If the instrument is intact but parts are missing, contact TA Instruments.

Choosing a Location

It is important to choose a location for the instrument using the following guidelines. The TAM Air should be:

In

... a temperature-and humidity-controlled area. The temperature of the room should be within the range 20 – 30 °C with a stability of ±1 °C. The relative humidity should be less than 65%.

... a clean, vibration-free environment.

... an area with ample working and ventilation space. The minimum floor space required is 47 cm (22 in) wide x 50 cm (24 in) deep. This space allows sufficient clearance around and below the entire instrument to provide for the required free flow of cooling air. Ensure that the air inlet under the instrument is never blocked.

Near

... a power outlet (120 VAC, 50 or 60 Hz, 15 amps, or 230 VAC, 50 or 60 Hz).
... compressed lab air and purge gas supplies with suitable regulators and flowmeters, if working at temperatures below ambient.
... a bench with sufficient space available for the computer system. Additional space will be required for sample preparation.

Away from

... dusty environments.
... exposure to direct sunlight.
... direct air drafts (fans, room air ducts).
... poorly ventilated areas.
... noisy or mechanical vibrations.
... all sources of heat or drafts.

NOTE: For operating temperatures below ambient care must be taken to avoid condensation. See the section "Operating Below Ambient" on page 12.

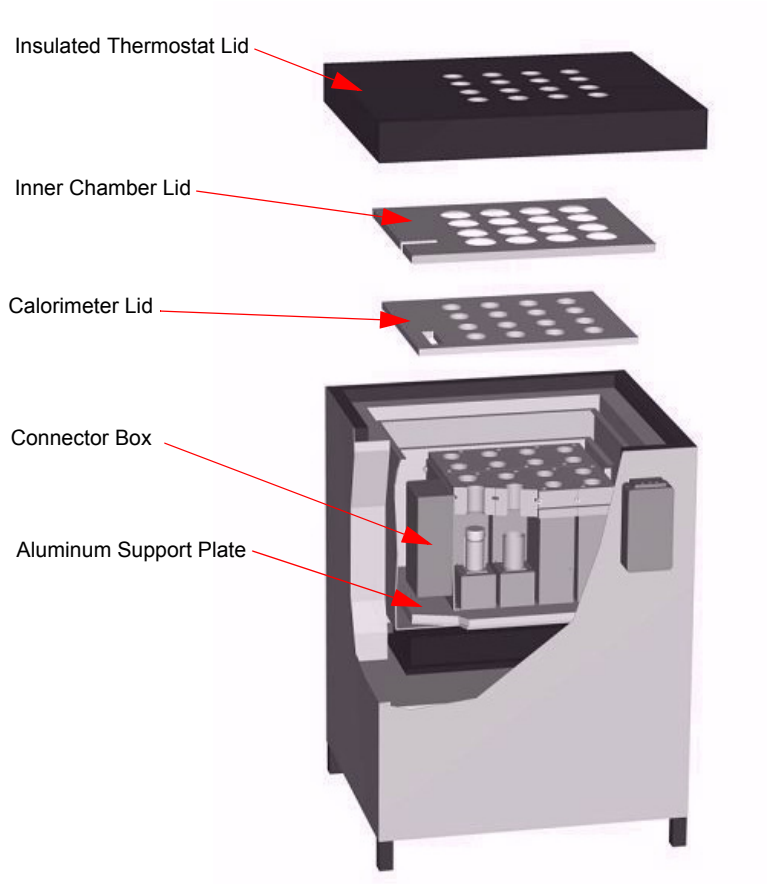
Installing the TAM Air

Refer to the figure below right when installing the TAM Air instrument.

1. During shipment of TAM Air the instrument is fixed to a wooden pallet by two fixtures. Remove the fixtures and mount the screw adjustments included in the package, one at each leg, to allow a horizontal positioning of the instrument.

2. Place the TAM Air thermostat in the position where it is to be used. Leave enough space (2 to 3 inches) around all sides of the thermostat to allow for free circulation of air. The outlets from the external fan are on the back panel and must never be obstructed. Do not position the thermostat close to a wall.

3. To compensate for an uneven floor, use the screw adjustments on the legs of the thermostat stand to level the instrument.



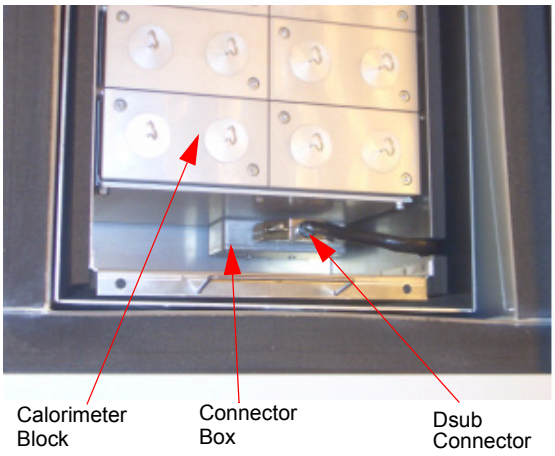
Assembling TAM Air

4. Remove the thermostat lid from TAM Air. The inner chamber containing a 25-pin Dsub connector at the bottom can be observed. See the figure to the right.

5. Lift the Dsub connector outside the inner chamber when positioning the calorimeter block (don't apply stress). Use the two hand brackets on each side of the calorimeter block and carefully lower it into the calorimeter insulation box with the connector box to your left-hand side. Make sure the Dsub connector is not damaged.

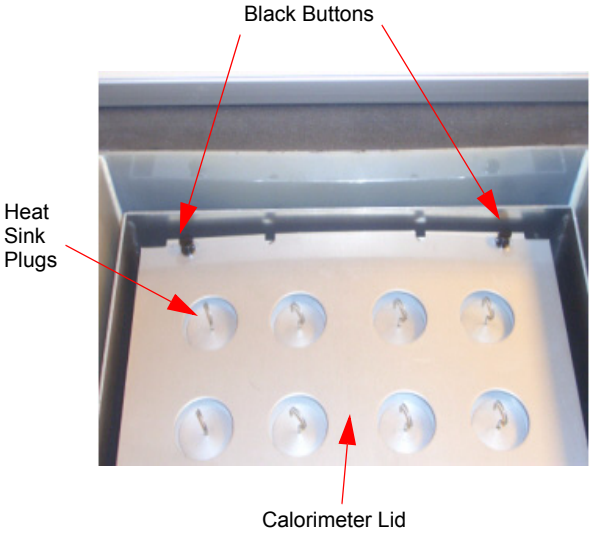
6. Position the calorimeter block and its insulation box centrally in the chamber to ensure a free flow of air around all sides. The alignment of the sample and reference tubes will determine the final position of the block. You may need to perform further fine adjustment.

7. Connect the Dsub connector to the connector box on the left side of the calorimeter block. See the figure below.



8. Fit the heat sink plugs into the 16 holes of the calorimetric channels.

9. Fit the calorimeter lid to the calorimeter block and lock it in the position by pressing the black buttons in each corner.



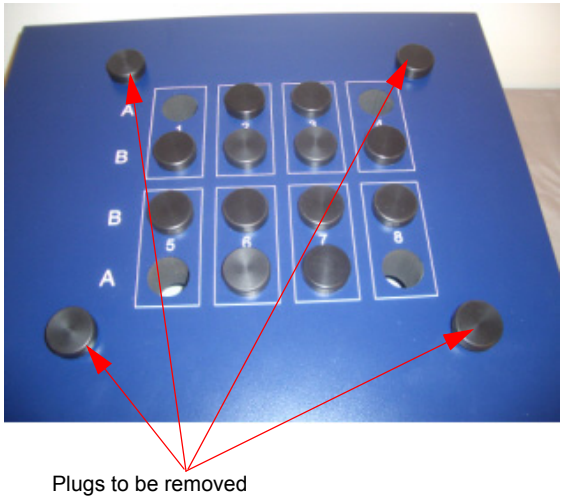
10. Fit the inner chamber lid as seen below.

11. Fit the thermostat lid, shown in the figure below.

12. Remove the four plugs in the corners. Use the lifting tool to lift out and refit the heat sink plugs at each corner to check that they are in direct alignment with the sample and reference tubes. It may be necessary to bring them into line by removing the thermostat lid and inner chamber lid and then slightly repositioning the calorimeter block. See the figure to the right.

13. Place the black plugs onto the 16 positions in the thermostat lid. See the figure to the right.

Thermostat Lid



Plugs to be removed

14. Install TAM Air Assistant on the dedicated computer **before** connecting the USB cable. This is important because the drivers have to be installed on the computer for the communication to work, see the section regarding installation of the TAM Air Assistant software (on the next page) before proceeding to point 15.

15. Connect the USB cable from the port on the back of the thermostat to a USB port on the computer.

NOTE: Install the TAM Air Assistant software before connecting the TAM Air USB to the computer.

Making the Electrical Connections

Follow these directions to connect the TAM Air to the necessary electrical source.

1. Check the voltage indicated on the label on the backside of TAM Air to ensure that it is the same as that used in the laboratory.

2. Check that the mains fuse on the back panel of the thermostat is correct for the mains voltage in the laboratory. For 100-130V, use 4A T (slow) fuse For 200-250V, use 2A T (slow) fuse. Never use a fuse of the wrong type or value.

CAUTION: Never use the TAM Air with a different voltage than indicated.

3. If necessary, change the mains cable delivered with the instrument to a cable of the local type.

4. Fit the mains cable and connect it to the mains power socket. Use the switch on the back panel of TAM Air to switch on. The two air circulating fans will start and the digital indicator on the temperature control panel will light up.

Computer Settings Before Installation

Before installing the TAM Air Assistant software as directed in the next section, make sure you have checked the following computer settings.

1. Make sure that the energy saving functions are disabled on the computer, as experiments are rather long it may be long times with inactivity on the computer, the computer must always be on, this applies to hardrives also as the software access it often.

2. Make sure that the Microsoft Windows Update feature is set to NOT install updates automatically. Sometimes the automatic update will restart the computer, which is not desirable. You can try setting this option to download updates, but let you decide when to install them. This would allow you to be notified of an update.

Installing the TAM Air Assistant Software

Hardware and Software Requirements

TAM Air Assistant is intended to be installed on a PC running under Windows 2000/XP/7 and a minimum of 512 Mb RAM. The PC must have a USB port V1.0 or 2.0.

The installation CD, included with a newly delivered TAM Air instrument, is intended for a new installation of TAM Air Assistant. Running the installation program on the CD includes the installation of the Windows software package ".NET Framework 2.0" (English version), in addition to the installation of TAM Air Assistant.

In case of future upgrades of ".NET Framework 2.0" it might be necessary to get an upgraded installation CD and perform another complete installation of TAM Air Assistant.

The installation of an upgraded version of TAM Air Assistant requires only one installation file, *e.g.*, "TAMAssistantInstaller v0.9.988.0.msi."

NOTE: The figures in this section refer to the installation under Windows XP and 2000. For examples of screen shots during installation under Windows 7, refer to the document "Installing TAM and TAM Air Assistant".

NOTE: When upgrading from versions earlier than 1.1.14.6, all FTDI drivers must first be uninstalled in the Add/Remove Programs option within the Windows Control Panel.

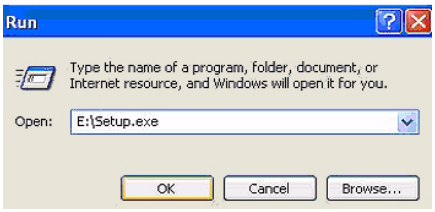
The installation CD required for a new installation as well as for the installation of an upgraded version of TAM Air Assistant can be obtained from TA Instruments.

Installation

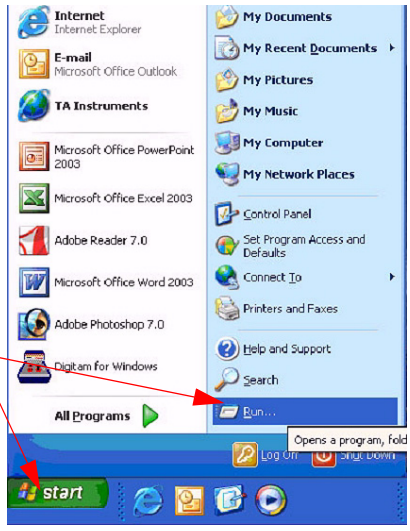
NOTE: Installation of the software must be made **before** connecting the TAM Air unit to the computer for the instrument drivers to be installed correctly.

Follow these steps to install the TAM Air Assistant:

1. Close all windows programs before installing TAM Air Assistant.

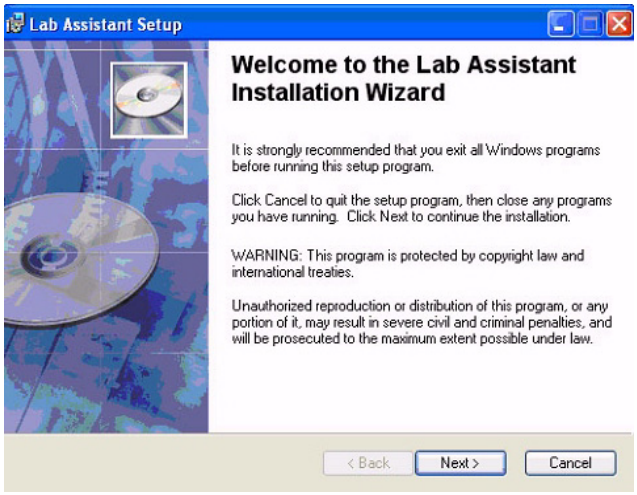


Start/Run

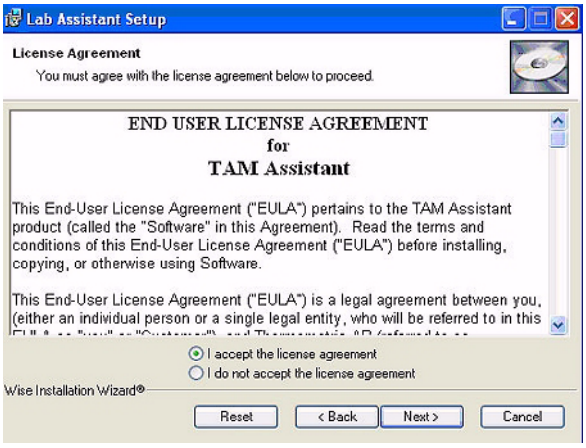


4. Click **OK** to start the Setup program. The installation wizard starts.

5. Step through the Wizard according to the instructions given.



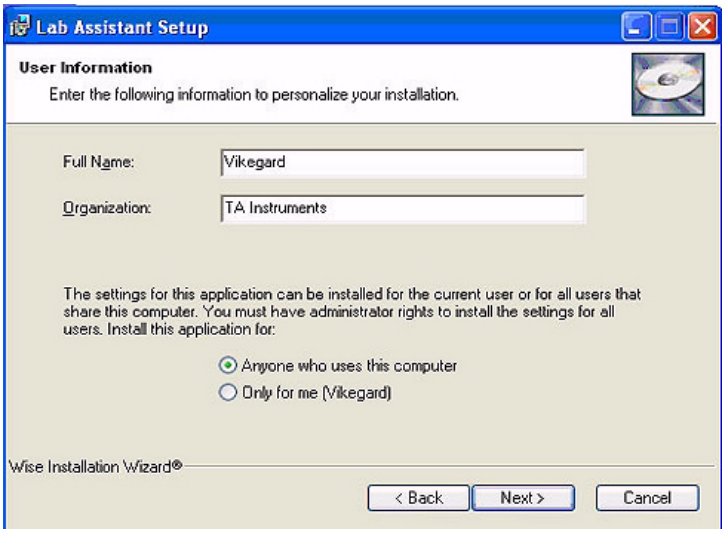
6. Click Next to continue. Read the end user license agreement carefully before proceeding.

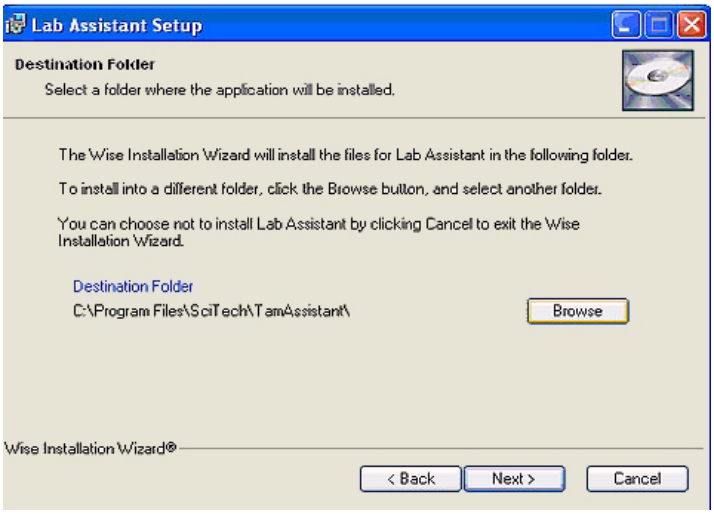


7. Click the radio button 'I accept the licence agreement' to continue.

8. Click **Next**. Enter the licensee's name and organization. Choose who will be using the software— all users of the computer or only for the name given. Administrator rights are needed to give rights to other user accounts of the computer.

9. Click **Next** when the settings have been made.

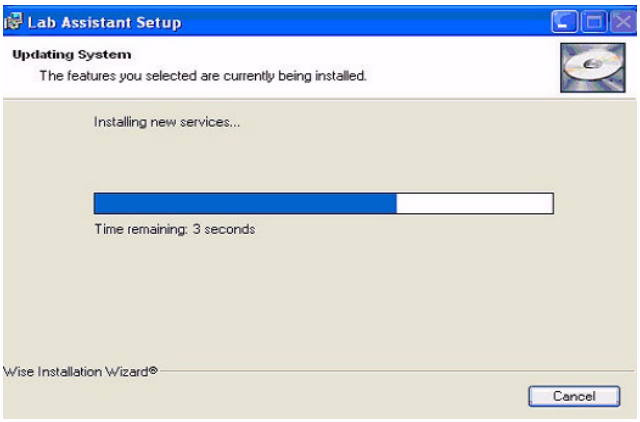


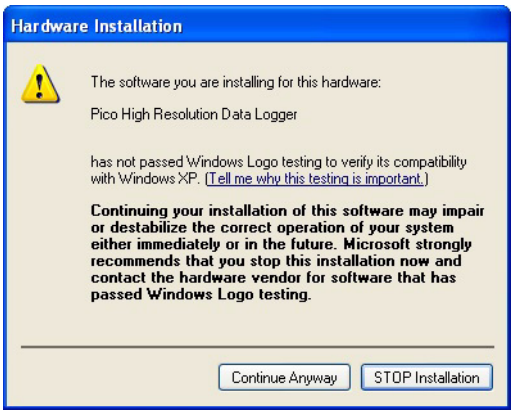


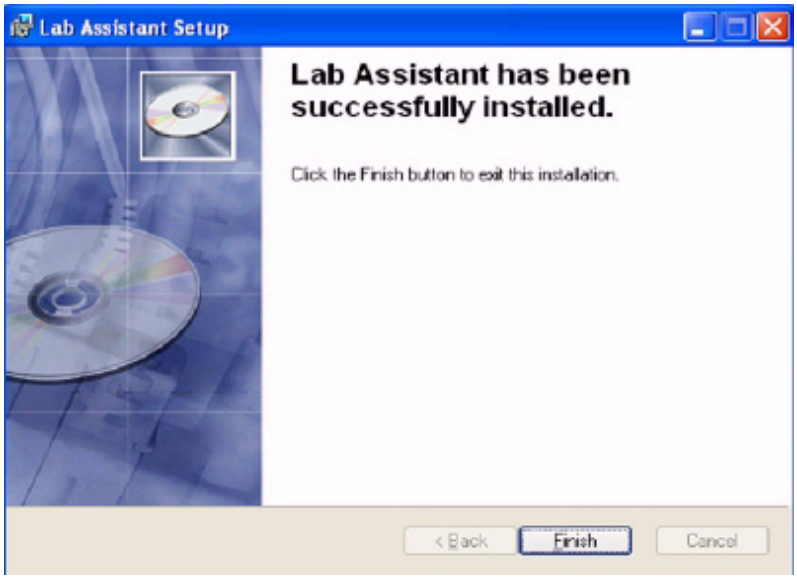
10. Browse to the destination folder for the software files. If the default destination folder is acceptable, just click **Next**.

11. Click **Next**. The software is now being installed. This is a quick process that normally takes less than 30 seconds. (If the computer doesn't have .net Framework installed already the installation takes a bit longer).

NOTE: At this stage, the following warning may appear. Click **Continue Anyway** to continue the installation.







12. Click **Finish** to exit the Setup program.The TAM Air Assistant software has now been installed. Before starting the program, the drivers for the Calorimeter Data-Logger and Thermostat must be installed. This is only necessary when TAM Air Assistant is installed for the first time, or if the USB port is changed, or if the drivers from an earlier installation has been uninstalled on the computer.

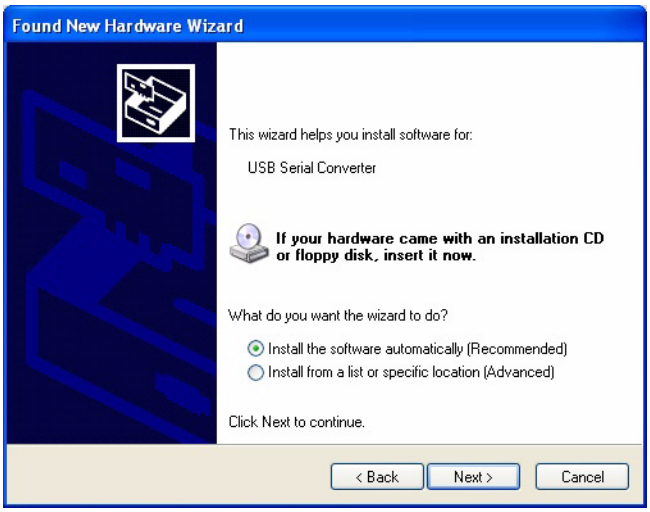
Connecting TAM Air with the Computer

This section will tell you how to connect the TAM Air with your computer. Follow the instructions below:

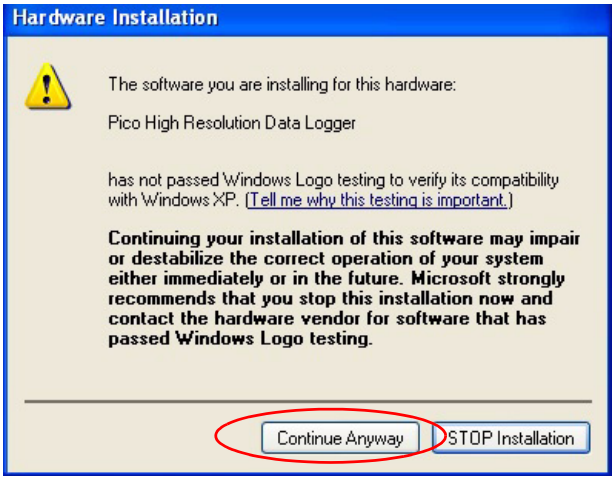
1. Make sure that the instrument is powered on.

2. Plug the USB connector into the computer and the back of the TAM Air instrument.

   The operating system Windows recognizes that new hardware has been connected to the computer. The window shown in the figure to the right is displayed (in Windows XP).



NOTE: If this dialog does not appear, Windows might have found and installed the wrong driver. This might be due to an erroneous setting in Windows. To correct the settings in Windows XP, see "Windows Update Settings for Drivers" on page 28.



A message is displayed that will request connection to Windows Update (the internet) to search for software (drivers for the connected hardware).

3. IMPORTANT: Select **No, not this time**. Click **Next** to continue.

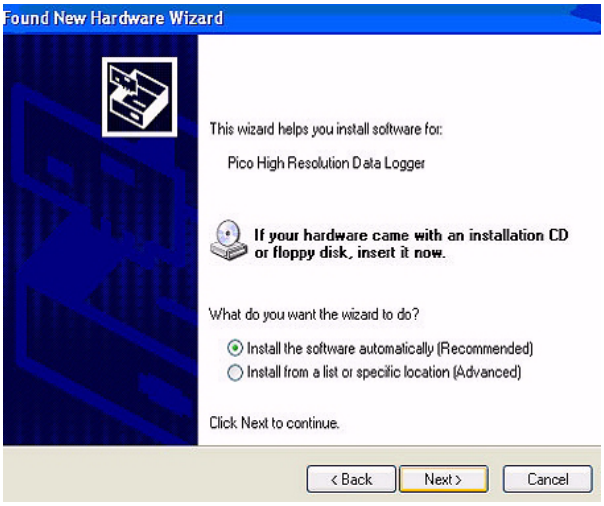
   Windows recognizes two devices, USB Serial Converter and the Pico high resolution data logger. Two consecutive installations for the two devices will be performed, usually starting with the drivers for the USB Serial Converter device. A form like the one shown the figure to the right will be displayed.

4. Choose the option **Install the software automatically** and Windows will find the correct drivers on the hard drive.

5. Click **Next**.The warning to the left may be displayed.

6. Click **Continue Anyway** to install the drivers.The figure shown to the right appears.

7. Click **Finish** to exit the Windows Wizard. After exiting the Wizard for the first device, a similar

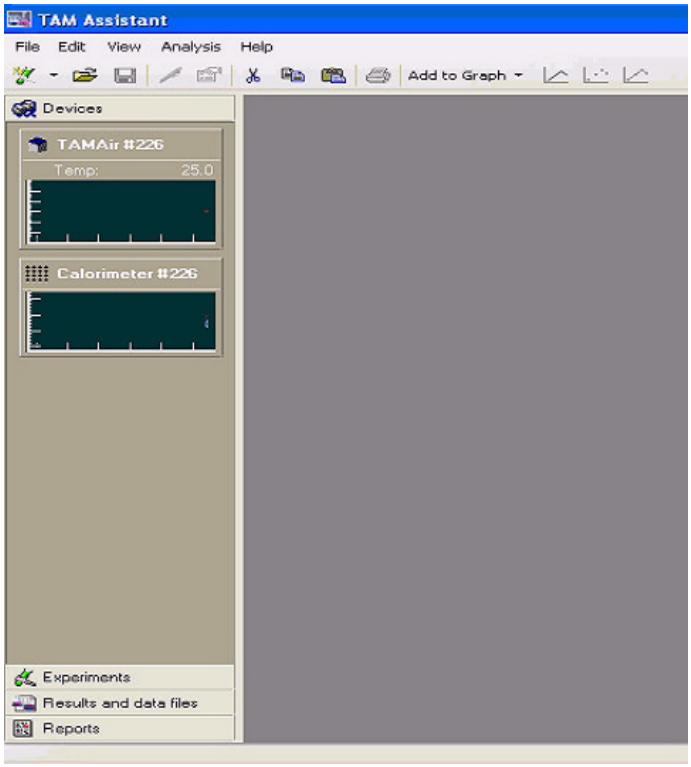


f

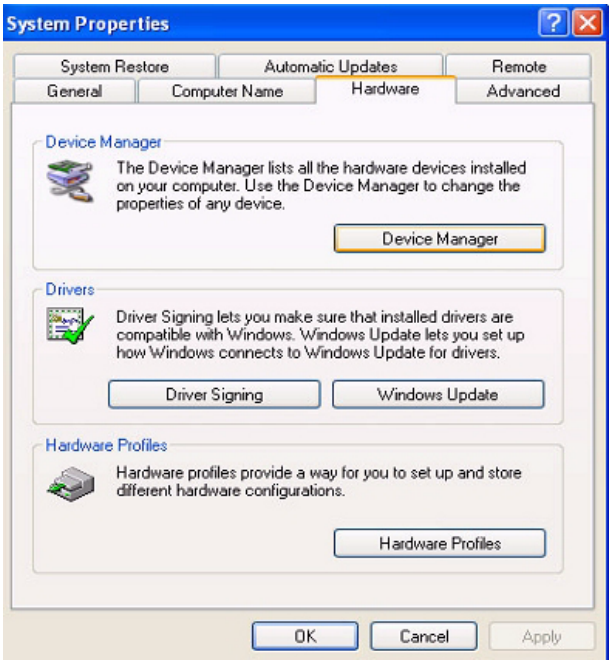
or the second device (Pico High Resolution Data Logger in this example) according to the form below. Step through the Wizard as directed on the previous page.

8. Open the TAM Air Assistant software from the **Start** menu after finishing the second installation of the drivers. The Thermostat and Calorimeter Devices will be displayed under the Devices manager to upper left of the Window as shown in the figure below.

The installation is now complete and the instrument is ready for running experiments.

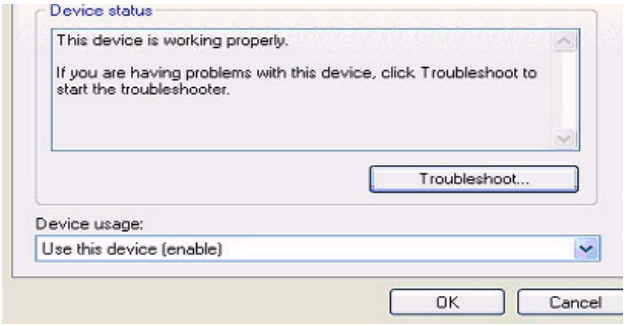


NOTE: If the thermostat and calorimeter devices do not appear, verify the device installation (see page 27).



.Close the open windows.

NOTE: If both drivers for the TAM Air are not present, contact TA Instruments for the instructions on proper driver installation procedures.



Checking Device Installation

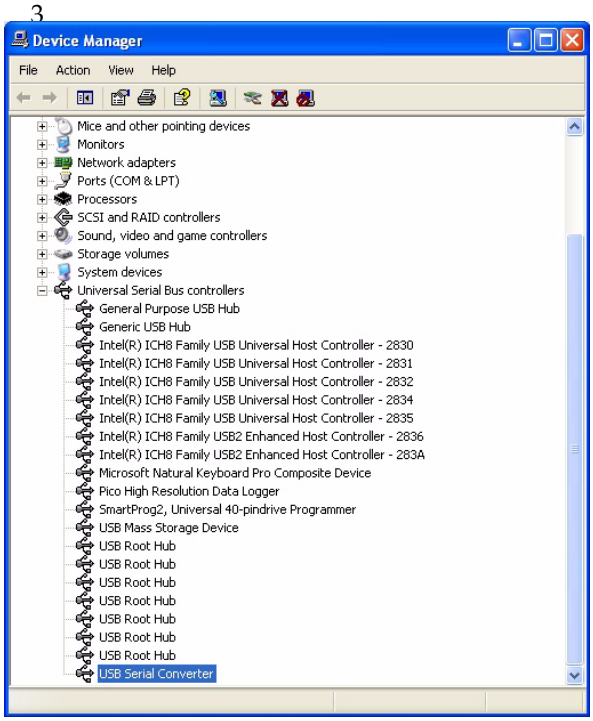
To check that the software for the devices USB Serial Converter and Pico high resolution data logger open the **Control Panel** in the **Start** menu and chose System.

The figure shown to the right should appear.

1.Click on **Device Manager** under the **Hardware** tab. The following figure appears.
Under Universal Serial Bus Controllers the controllers for the two TAM Air devices should appear. They are USB Serial Converter and Pico High Resolution Data Logger. This confirms that the Device controllers are installed.

2.Right click on the Device controller and check Device status. If it says "This device is working properly" as shown in the figure to the right, the installation is successful.

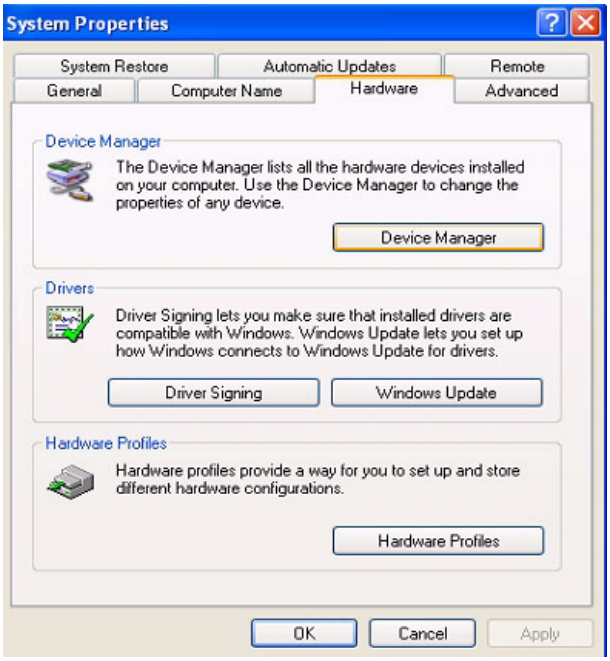
3



Windows Update Settings for Drivers

This section describes how the Windows Update Settings related to driver installations must be set for proper installation of TAM Air Assistant.

1. Open **System** in the **Control Panel**. The window shown in the figure to the right should be displayed.



2. Click the **Windows Update** button. The following window is shown.

3. IMPORTANT: Select "**Ask me to search Windows Update every time I connect a new device**." DO NOT select the option, "**If my device needs a driver, go to Windows Update without asking me**." It is peferable to use the device drivers installed for your instrument and prevent accidental file overwrites.

Uninstalling the Drivers

If the drivers are not working properly, or you have installed the wrong device, uninstall the devices using the instructions in this section. Then reinstall the drivers following the instructions in the previous sections.

To uninstall a device, right click on the Device control and choose **Uninstall** from the drop-down menu. One example of a device that must be removed is the "Virtual Comport Driver."

If you continue to have problems, please contact TA Instruments for service.

Installing Multiple TAM Air Instruments

Several TAM Air instruments can be run on the same computer. The CDs supplied with TAM Air Assistant software must contain the device files for all the TAM Air instruments. Contact TA Instruments if you need assistance.

IMPORTANT! Follow the instructions for the software installation. When the instruments are to be plugged into the computer for the first time, plug them in one at the time and perform the installation of the device controllers separately.

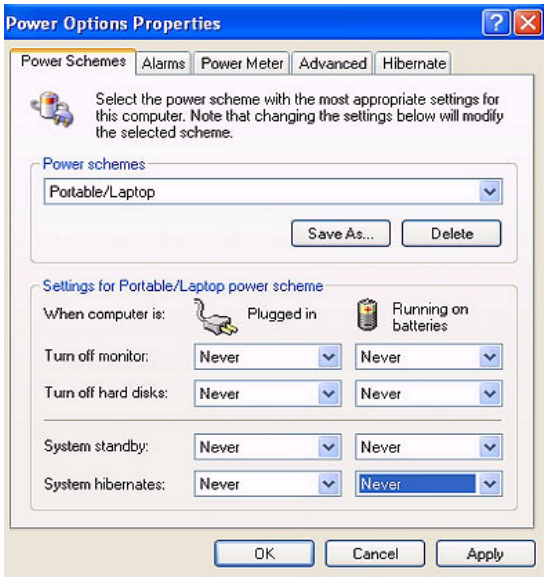
Computer Settings

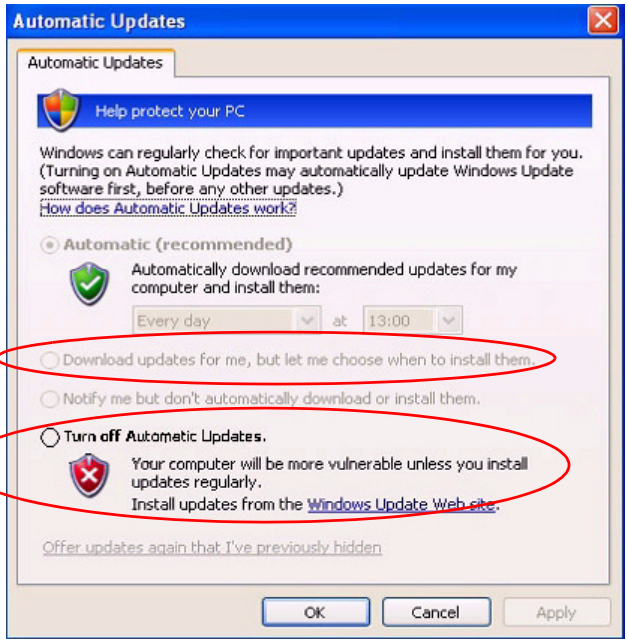
TAM Air measurements are usually long, running for several days. It is important that the data collection is not interrupted or cancelled during the time of the measurement. To decrease the likelihood of unwanted interruptions, set up your computer as directed below:

1. Choose the **Settings/Control Panel/Power Options** from the **Start** menu. Select the **Power Schemes** tab as shown in the figure to the right.

2. Select **Never** from both of the lists to the right of **System Standby** and **System Hibernates**. See the figure to the right.

3. Choose the **Settings/Control Panel/Automatic Updates** from the **Start** menu. See the figure below.





4. Choose one of these settings: "**Turn off Automatic Updates**" or "**Download updates for me, but let me chose when to install them**." See the options circled in red on the figure to the left.

Important Notes

Please read the following notes about the software and its set up.

- The CD-ROM is unique for the TAM Air thermostat and calorimeter as it contains device files holding calibration constants for one particular TAM Air instrument. Once installed, the files will not be overwritten when upgrading the software.

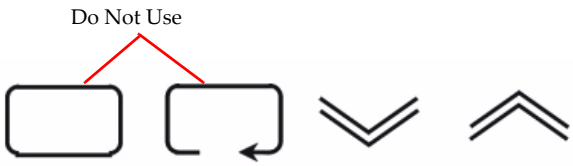
- TA Instruments recommends that the disc be placed in a safe place and/or backup the device files containing the factory calibration data of the instrument. The files are located in the directory Device Files on the disc.

- If the system is recalibrated in the future, the device file located on the local computer will be valid. If device file on the computer is correct, TA Instruments recommends that you backup the file from the HDD. The files are located in the directory:

  C:\Documents and Settings\All Users\Application Data\SciTech\LabAssistant\Router\Devices.

Setting the Operating Temperature

The temperature of the air thermostat, *i.e.*, the operating temperature, can also be set from the TAM Air Assistant or the temperature control panel located on the front of the instrument. This set temperature is recalled in the event of a power failure.



Temperature Control Panel Operating Buttons

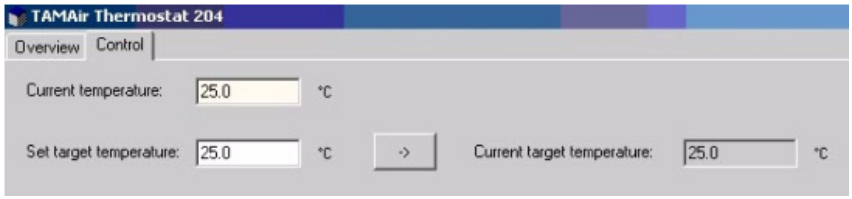
The control panel has two digital indicators, one with red digits showing the actual temperature of the air thermostat and the other with green digits showing the set temperature. The amber indicator light in the top right-hand corner of the control panel will normally flash, indicating that the heater is on. The amber light will remain off if the thermostat is cooling.

The two arrow buttons are for increasing and for decreasing the set temperature. The two buttons to the left have no function in normal operation. They are used in the factory together with a special code for programming the control panel. If you enter the menu by accident, press the left-most key once to exit. Note that settings for the calorimetric temperature also can be made directly in the software, see the section, "TAM Air Assistant Software Overview" for details.

WARNING: The two buttons on the left of the control pad should only be used by authorized personnel.

1. To set the operating temperature use one of two methods:

Using the TAM Air Assistant:



a. Choose the Thermostat in the TAM Air Assistant devices explorer and click the **Control** tab.

b. Enter the new temperature in the text box named **Set target temperature** and click the arrow (pointing to the right) to enter the temperature. The new value is seen in the **Current target temperature** text box.

Using the Control Pad:

NOTE: This option should only be used in situation when the TAM Air Assistant software is not in use.

a. Use the up and down arrow buttons until the required set temperature is displayed by the green digital indicator.

2. Allow the temperature to stabilize for at least **eight** hours. However, to ensure that the system is stable, verify that the calorimeter signal is at a steady state close to zero. It is recommended that you perform the gain calibration for all eight calorimeter channels, if the thermostat temperature is changed.

NOTE: Although the circulating air reaches the set temperature very quickly, the calorimeter block will take several hours to reach the temperature because of its large mass, high heat capacity and surrounding insulation.

NOTE: If at any time it is necessary to check the actual temperature of the calorimeter block, please see Chapter 3 for details.

Completing the Installation

Once you have performed all of the steps in this chapter, follow the next few steps to complete the installation of the TAM Air.

1. Make sure the operating temperature has stabilized. Then perform a gain calibration. (See Chapter 3 for information).

2. After the Gain calibration the TAM Air is ready to use.

Chapter 3

Use, Maintenance, & Diagnostics

Using the TAM Air

All of your experiments will have the following general outline. In some cases, not all of these steps will be performed. The majority of these steps are performed using the TAM Air Assistant software.

- Setting the operating temperature
- Calibrating the instrument
- Performing preliminary checks
- Preparing the sample and reference
- Starting the experiment
- Loading the sample and reference.

To obtain accurate results, follow procedures carefully.

Before You Begin

Before you set up an experiment, ensure that the TAM Air and the TAM Air Assistant software have been installed properly. Make sure you have:

- Made all necessary electrical connections
- Powered up the unit
- Become familiar with software and instrument operations
- Calibrated the TAM Air. See the next section.

Calibrating the TAM Air

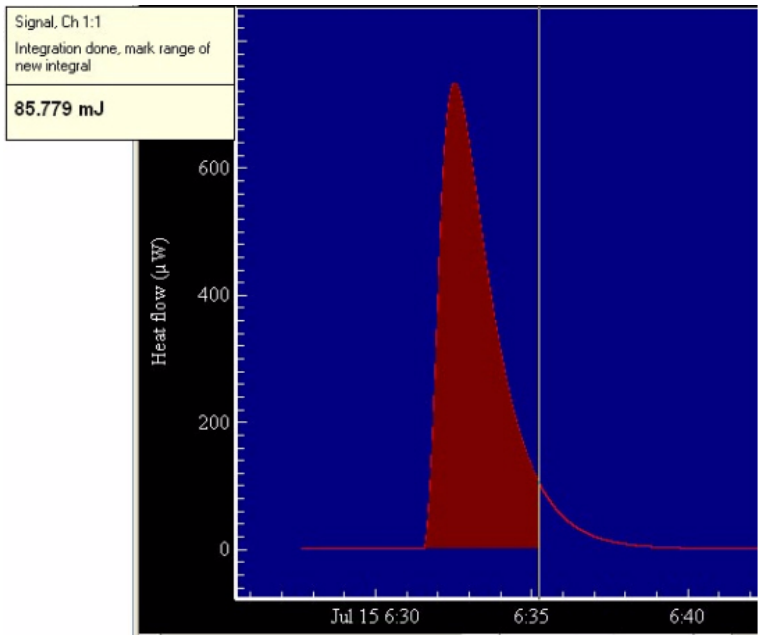
The objective of electrical calibration is to calculate the calibration constant for each individual twin calorimetric channel. The calibration constant is called the *Gain factor* in TAM Air Assistant and is a relative value with respect to a calibration performed at the factory, see page 11. The calibration is fully automated and the Gain constant is entered in TAM Air Assistant as soon as a new calibration has been performed.

Each channel has a permanent precision calibration heater on side A, the sample side. Side B, used for the inert reference, does not need to be calibrated. The heater has a resistance of 100 ± 0.1 Ω and a very low temperature coefficient. The eight calibration heaters are connected in series together with a reference calibration resistor of 25 ± 0.1 Ω.

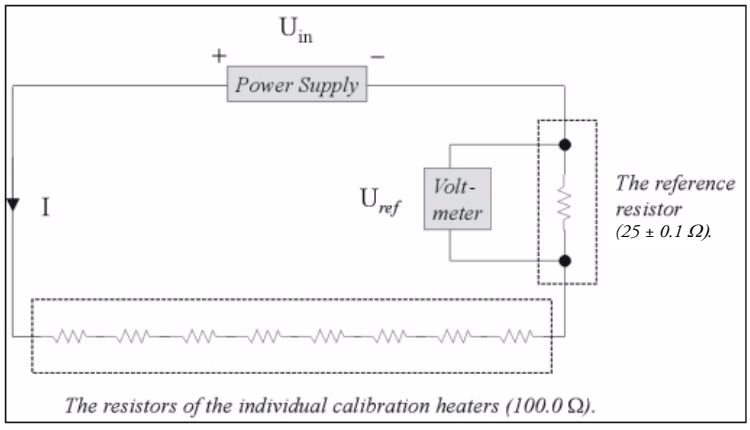
Normally, calibration is performed with empty calorimetric channels. Once a stable baseline has been recorded, a known voltage is applied over the calibration heaters during a predefined time period. The calibration constant is calculated as the measured integrated area divided by the heat released in the electrical heaters.

The obtained calibration constant is a value close to unity, normally within the interval $0.9 < 1 < 1.1$.

The resistors of the individual calibration heaters and the reference resistor are connected in series. Thus, the voltage measured across the reference resistor will be the same as that across each individual calibration resistor. See the figure below.



Calibration Plot



Schematic Diagram of Calibration Circuit

When to Calibrate

Calibration should be performed regularly, four times per year for example. Each channel in use must be independently calibrated, which can be done manually or automatically through the TAM Air Assistant software. It is recommended that the calibration constants are determined for each operating temperature; however the differences in values will not be large.

Preparing for Calibration

Before beginning the calibration, perform the following steps:

1. Stop any experiment that is currently running and remove all experimental sample and reference ampoules. After removing the ampoules, it will take about 40 minutes for the calorimetric signals to become stable (moderate stability condition) and close to zero.

2. Check that the TAM Air thermostat is set at the correct temperature. Refer to page 40, "Changing the Set Temperature," if necessary. If the temperature must be changed, wait at least eight hours before performing the calibration.

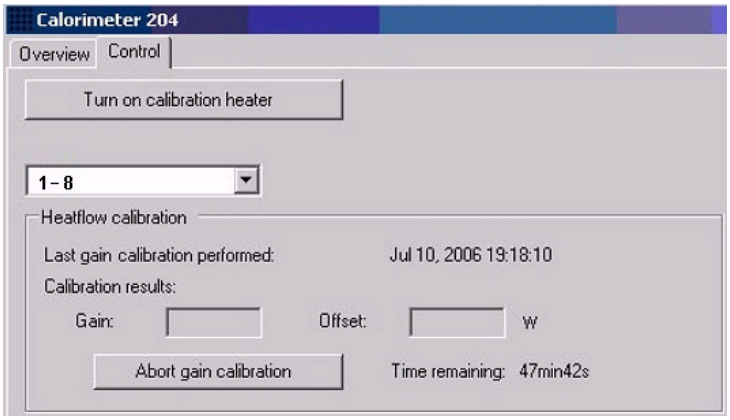
Performing the Calibration

Observe that all channels are calibrated simultaneously when starting the calibration. The calibration heaters for the individual channels are connected in series and it is not possible to select only individual channels.

1. Select the **Overview** tab for the calorimeter in the Devices manager.

2. Wait until the calorimetric signal is stable within the stability criteria, see the section called "Baseline Stability" above for details on stability conditions.

3. Choose all calorimeters from the drop-down menu under the **Control** tab. (See the figure to the right.) Click the button **Perform gain calibration** to begin the procedure. The text on the button changes to **Abort gain calibration**.



NOTE: You will be able to select individual calorimeters for the calibration, but since the calibration heaters are connected in series, all channels will be subjected to the calibration heat. For the channels that are not selected, the old calibration data will remain.

The whole procedure takes approximately 55 minutes to complete. A countdown of the remaining time needed for the procedure will be displayed. After completion, the Gain factor for each calorimeter is calculated and stored by the software. The Gain factor should be close to 1.0.

Once the calibration is complete, the calibration data (Gain constant and Offset) is displayed in the text boxes with the same name, and the signal will be shifted down to zero. Note that the Calibration data for each particular channel is shown only if that channel has been individually chosen from the drop-down menu. We recommend that you record the Gain and Offset values for future calibration comparison.

Operating the TAM Air Calorimeter

Preliminary Checks

Before beginning to use the TAM Air, follow the instructions below:

1. Check that the TAM Air thermostat is set at the correct temperature for the experiment. (See Chapter 2 "Setting the Operating Temperature.")

   NOTE: For operating temperatures below ambient care must be taken to avoid condensation.

2. Take care when measuring large exothermic or endothermic processes. Continuous inputs of large heats are likely to change the temperature of the calorimetric channels significantly. To overcome this situation, it is recommended that the sample size be reduced as a precaution. After the calibration, the signal is shifted to zero in accordance with the offset calculation. The Gain constant should be close to 1 (0.9<Gain<1.1).

   NOTE: When measuring samples with large heat production rates, the measured heat flow values will be accurate; however, the temperature of the sample may deviate considerably from the operating temperature. For example, at a continuous rate of heat production of 550 mW, the temperature of the sample has been found to increase by approximately 3 °C. Refer to Chapter 3 for details on the independent measurement of calorimeter block temperature.

3. Ensure that the calibration constants for each channel are valid as defined in the section, "Performing Calibration" found in this chapter.

4. Check your results. The drift of the recorded baseline should be less than 0.2 % per hour of the expected maximum values during measurement.

   For example, for an expected signal of 20 mW, the baseline before and after measurement as well as the heat flow during measurement should be considered stable if the drift is less than 0.002*20 mW/hr = 40 μW/hour. However, if the monitored heat flow during a measurement is in the range of 1 mW, it should be considered stable when the monitored heat flow does not change more than 0.002*1 mW/hr = 2 μW/hr.

Preparing Samples

Follow the instructions below to prepare your samples for use with the TAM Air:

1. Select the type of ampoule to be used. 20 mL disposable glass ampoules or 20 mL plastic ampoules (HDPE) are normally used. Alternatively, 20 mL stainless steel ampoules are available for special applications.

2. Weigh the ampoule.

3. Load the sample into the ampoule. Take care not to spill any material on the neck or over the outer wall of the ampoule.

4. Weigh the loaded ampoule. Subtract the weight of the empty ampoule and record the sample weight.

5. For 20 mL disposable glass ampoules, seal the lid onto the ampoule using the cap crimping tool.

6. Use the centering tool to make an indentation in the center of the aluminium crimp cap. Screw a lifting eyelet into the indentation. For plastic ampoules the lifting eyelet clips to the neck of the ampoule.

NOTE: Do not dispose of the lifting eyelets. Keep them for future experiments.

Selecting the Reference

The sample and the reference ampoules should be balanced, *i.e.*, having the same thermal response (time constants) in order to ensure a stable baseline and reduced background noise.

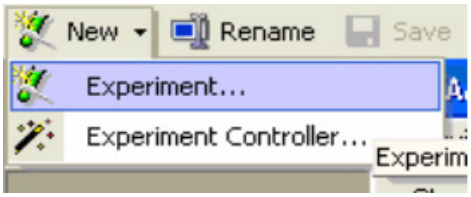
This is achieved by using the same type of ampoule for the reference as used for the sample and by using an inert material with approximately the same heat capacity as the sample in the reference ampoule.

An example of how to calculate a reference is outlined below:

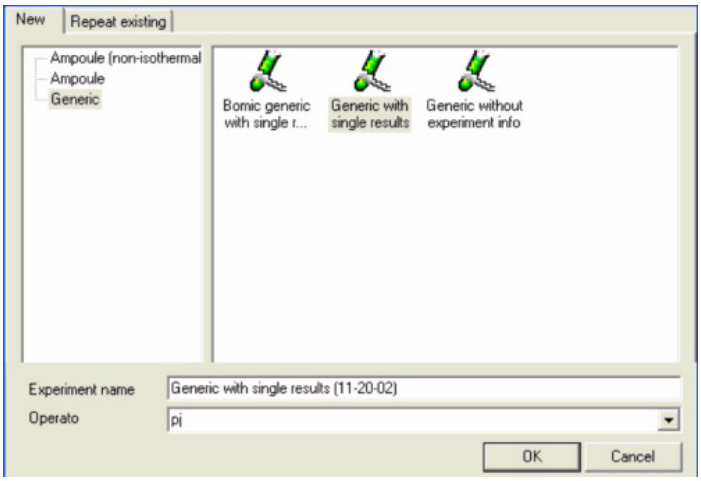
- Mix 5 g of cement powder with 2.5 g water to give a 0.5 water-to-cement ratio.
- Balance the cured cement with 5.3 g sand.
- If balance wet mix 19.26 g sand:
  Cp cement ~ 0.8 Jg^{-1} K^{-1}
  Cp water ~4.18 Jg^{-1} K^{-1}
  Cp sand (quartz) ~0.75 Jg^{-1} K^{-1}

New Experiment

When starting a new experiment, independent of what type, click on **Experiment** in the **New** menu as shown in the figure to the right.



A dialog box appears for you to choose from a number of experiment types. (See the figure below.) You will need to enter an **Operator** name or initials. The **Experiment name** is the file name where the result and additional information is stored. As the new experiment type is being chosen, a default name stating the type of experiment and date will automatically appear as the experiment name. You can enter any name desired.



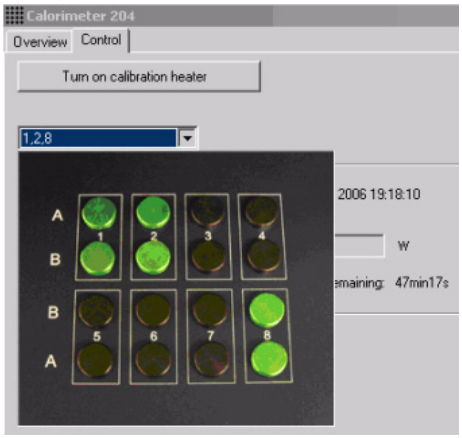
There are three basic experiment types included in TAM Air Assistant: **Ampoule** experiments, **Generic** experiments, and a few **Validation tests** (Gain validation and General Performance Test [GPT]).

Generic is the simplest type of experiment where data is collected without any division into pause, baseline and main sections.

The Ampoule Experiment Wizard

The ***Ampoule*** *Experiment Wizard* guides you through a number of steps that provide instructions on operational details and allow you to enter information about the experiment.

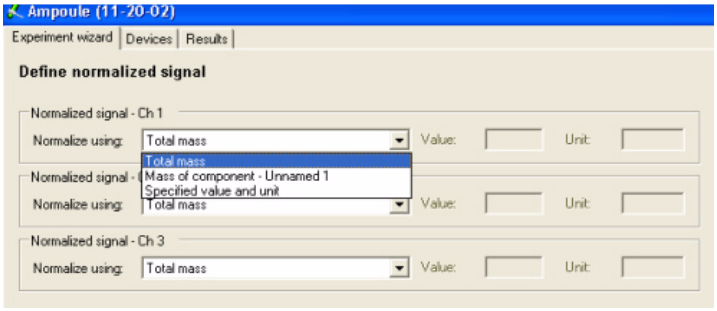
1. Choose **Ampoule experiment** from the experiment types in the file menu then click **OK** to initiate the *Ampoule Wizard*.The first dialog form appears, together with an icon under the experiment manager. The icon shows the experimental name and the current status of the experiment.



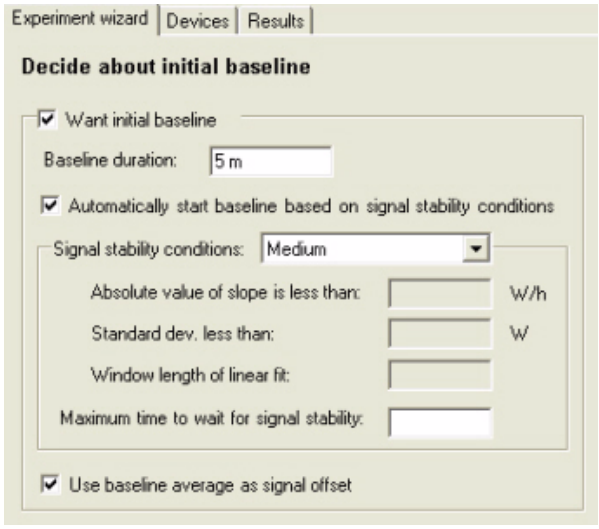
2. Click **Next** to continue.

3. Choose the channels to use in the experiment by clicking on the channel in the figure. Several minicalorimeters can be chosen by pressing the Ctrl-key while clicking on the channel(s) (or, alternatively, by clicking on one channel and drag the cursor over the other channels to be used). The color on the top of the channel lids turn green as they are chosen. See the figure to the right.

4. Click **Next** to continue. The window shown below is displayed.



5. Use this window (above) to enter information on how the signal is to be normalized. The normalization information cannot be changed after the experiment has been initiated. However, you are able to export and/or view alternative normalized signals after the experiment is finalized.



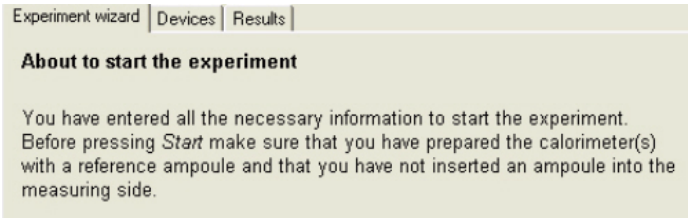
6. Click **Next** in the Experimental wizard to continue. The next window (shown to the right) lets you decide about initial baseline for the experiment.

7. Enter the time for recording the baseline in the **Baseline duration** field. The default value suggested by the software is 30 minutes. (The letter "m" is used to abbreviate minutes and "s" is used for seconds.) The baseline can be started manually by the operator or automatically when the signal is stable according to a specified baseline criterion. The default value for the stability criterion as entered by the software is

medium signal stability condition. This corresponds to a slope of the calorimetric signal 2 µW/h and a standard deviation of 4 µW. These parameters are calculated over a time period of 20 minutes. The other options are Low or Tight which corresponds to 4 µW/h, 50 µW over 15 minutes and 1 µW/h, 2 µW over 30 minutes, respectively.

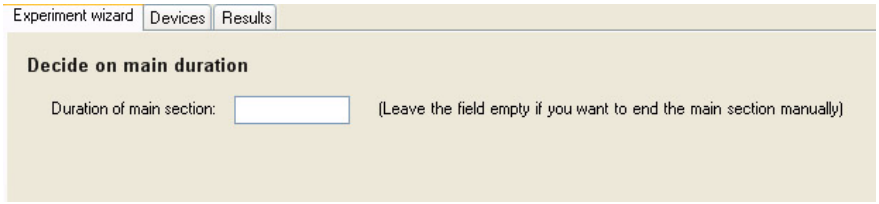
If the checkbox **Use baseline average as signal offset** is marked, TAM Air Assistant automatically uses the calculated mean value of the initial baseline to correct the signal in the main part of the experiment. After the experiment has been finalized one can use more advanced baseline correction with initial/final baselines using linear slope corrections, polynomial correction, etc.

8. Click **Next** to continue.



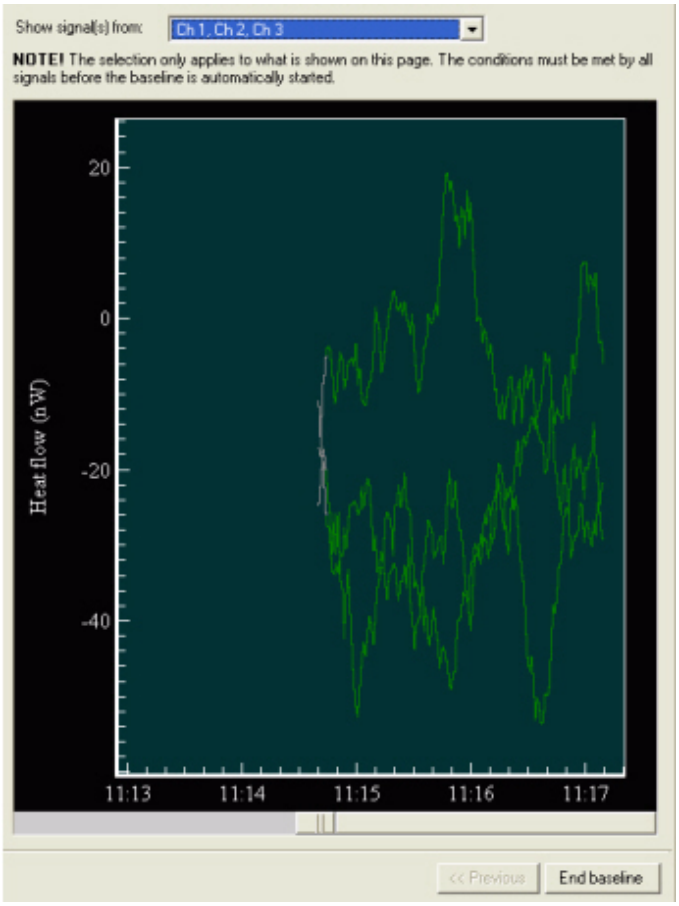
9. Baselines are usually run without an ampoule in the measuring position in order to be sure that no offset due to any thermal activity is recorded. Ensure that the calorimeter is empty before you start the experiment.

10. Choose the duration of the main section of the experiment when the window shown to the right is displayed. It is recommended that you leave the field empty to allow the experiment automatically run until you choose to end it by clicking **End Main** (see the next page).



If desired, you can enter a time instead. Make sure that you enter a value, along with the associated units (s, min, h).

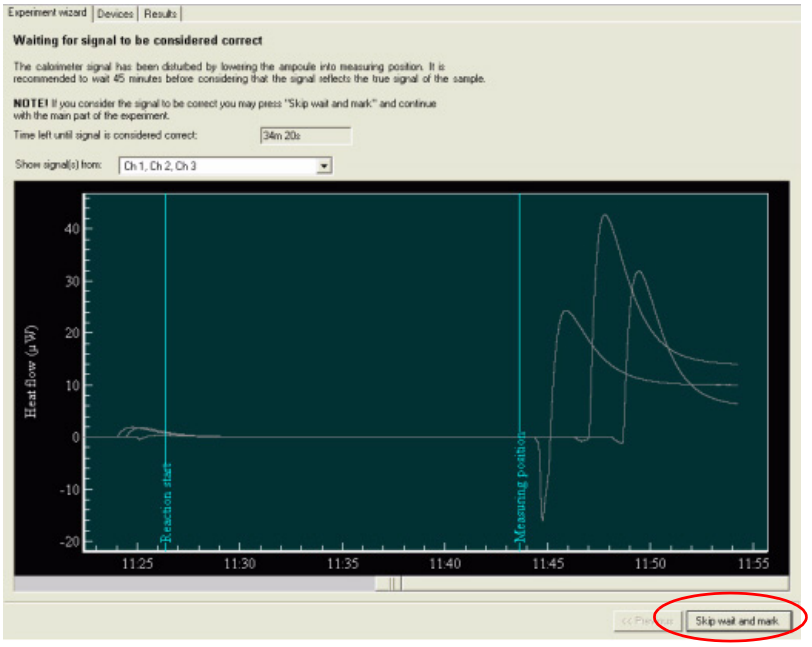
11. Click **Start**. The window shown in the figure to the right appears. The green curve indicates the recorded baseline section, while the gray part of the curve indicates a pause section prior to the baseline.



The baseline section starts as soon as the chosen **Signal stability condition** is met. The program counts down the time specified for the **Baseline duration** and the remaining time is specified in the text box labelled **Time left**. When the baseline recording is finalized, you will be instructed to introduce the sample and reference ampoule into the calorimeter.

12. Insert the sample ampoules into the calorimeter(s). See "Loading the Sample and Reference" in this chapter for more details.

13. Click the button labeled **Skip wait and Mark,** and lower the sample slowly into the measuring position. This introduces an initial disturbance of the signal, mainly caused by the temperature difference between the calorimeter and the ampoules.The software waits until the signal is considered correct, that is free from the disturbances generated because of the introduction of the sample ampoule. The default time is 45 minutes but can be lengthened or shortened at the end of wizard.

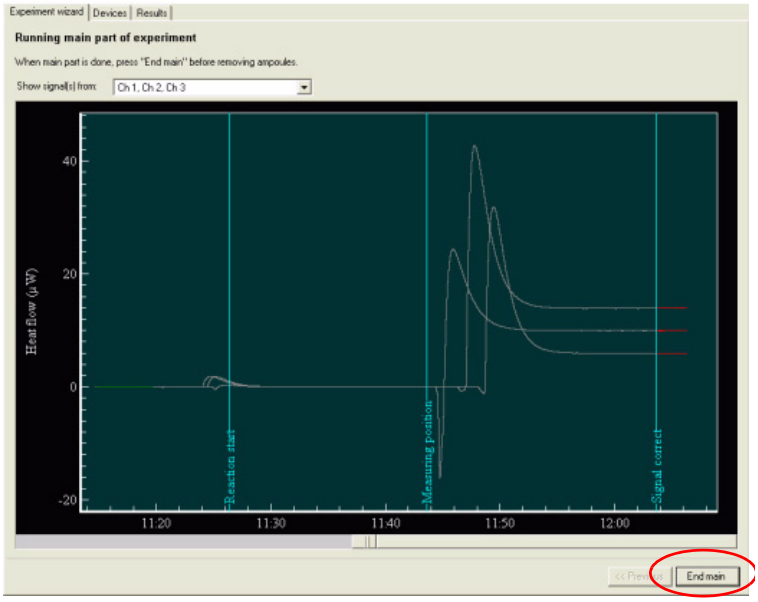


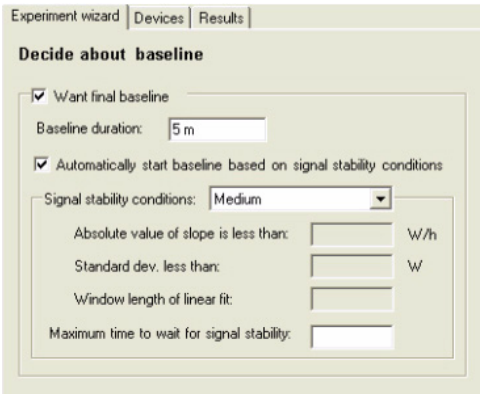
You may also want to press the **Skip wait and mark** button, in case the signal known to be correct before the default time. The graph appearing after the mark is red indicating the main part of the experiment.

14. When the time taken to reach a stable signal has passed, a time marker will appear labeled **Signal correct**.

At this point the signal is considered correct from the instrument's point of view. Unwanted effects in the sample, *e.g.*, redistribution of solvent in a solid sample or different types of sorption phenomena etc., that might occur when the sample is exposed to sudden temperature changes are not included. To avoid such unwanted effects the sample should be well conditioned before the experiment.

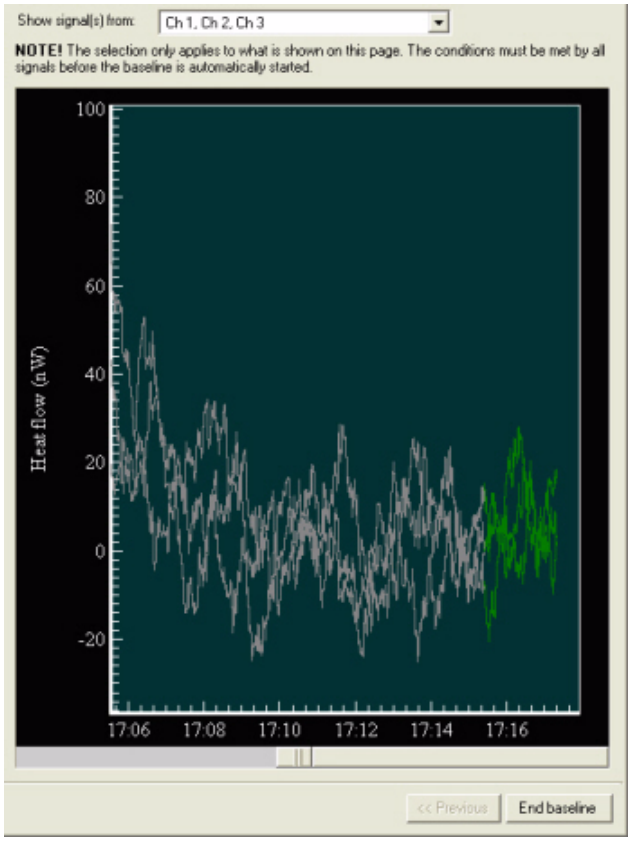
15. Press the **End main** button (shown in the figure to the right) when the experiment's main part is complete. A dialog is displayed to allow you to decide about the final baseline.





16. If the **Want final baseline** check box is marked, the software will wait for the signal to become stable according to the chosen signal stability conditions.

17. Decide about the final baseline and click **Next**. You are now prompted to remove the ampoule (or ampoules) from the calorimeter. After removal, place the Aluminium lid and the black cover into its position.



18. Click **Next** when the ampoules have been removed.

    The software now waits until the chosen stability criterion is met before starting the final baseline.The current slope and standard deviation can be followed for any channel in the experiment by choosing the channel in the drop-down menu above the plot window.

    It is possible to manually start the baseline before the stability criterion has been reached by pressing the **Manual baseline start** button.When the signal is stable in accordance with the set stability criteria the final baseline section will start. The plots changes color from gray to green when the baseline starts.

19. When the final baseline recording is finished a new window in the wizard automatically appears that lets you adjust the time settings. This may be done if the previous time settings are incorrect, *e.g.*, the duration before the signal was considered correct.

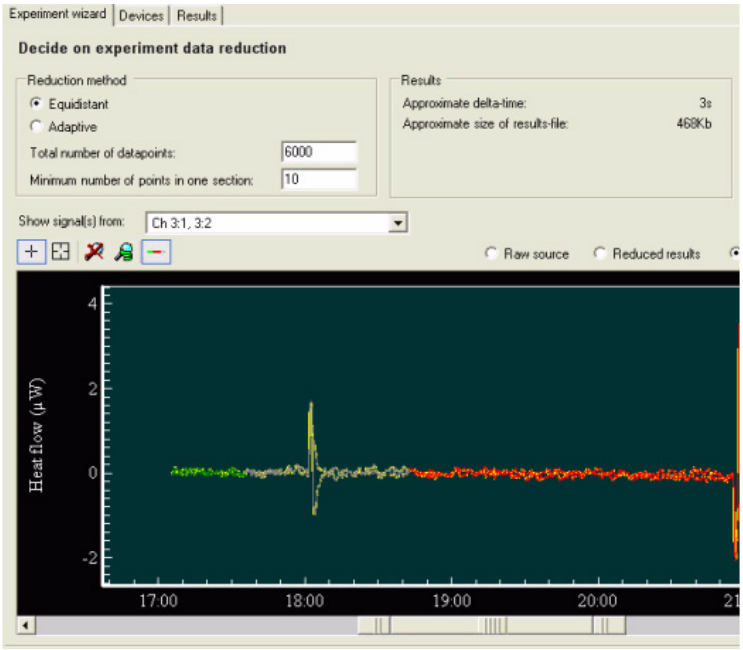
Important: This is the only opportunity to change the time marker.

20. Click and drag a section change time marker to the correct time.

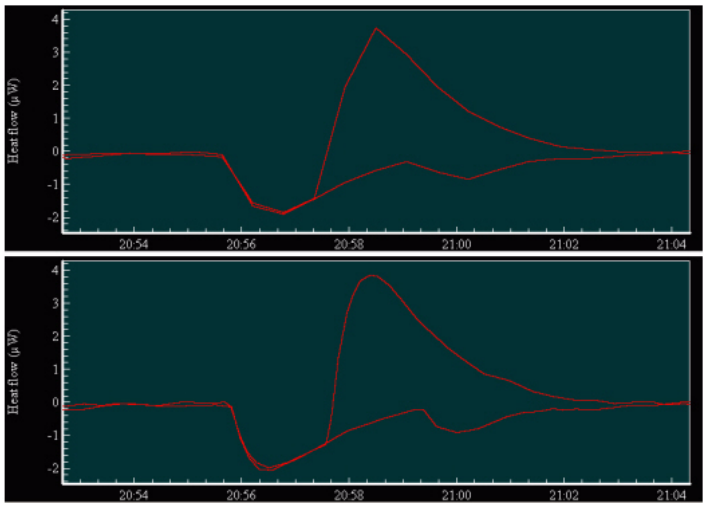
21. Adjust the time setting, if necessary, and click **Next**.

22. Decide on data reduction of the experimental results at this point. In all experiments, data points are collected approximately every second. When running the ampoule wizard, you can select the way to save the data after the experiment. The following form appears when the experiment has been finalized. You can select one of two data reduction methods: *Equidistant* and *Adaptive*.

    The default is always equidistant with 6000 data points independent on the length of the experiment.



The results of the reduction are shown in the **Results** frame with the approximate delta-time (rounded to 1 second) and the approximate size of the results file.



In adaptive mode the chosen number of data points is dispersed to give a higher data point density in part of the experiment with high rate of change in the measured property, *i.e.*, during a phase transition after a period of relative calm.

The upper part of the figure on the left shows results of equidistant data distribution (500 points over approximately 8 hours).

The lower part shows how the data points have been smoothened by use of the adaptive mode for the same total number of data points.

23. Click **Next**. A new form appears that lets you finish the calorimetric experiment.

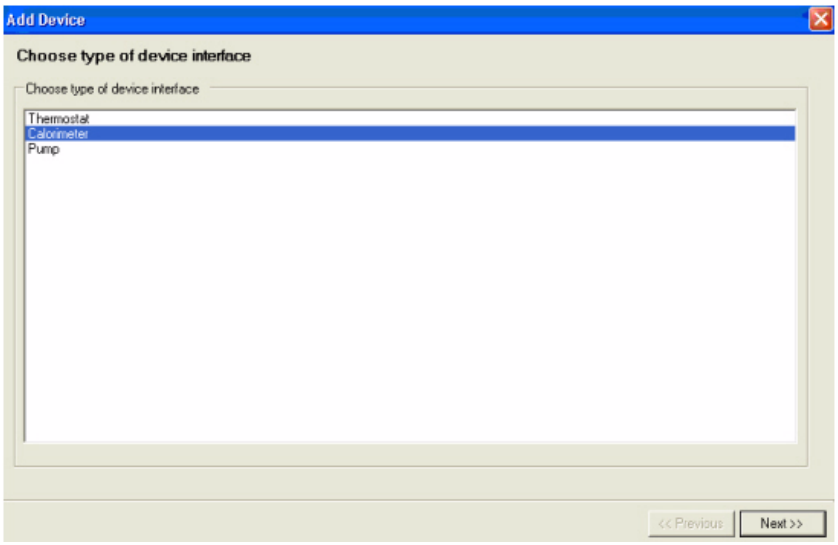
24. If you do not want the results file to appear, mark the check box.

25. Click **Finish**. The results are saved as a results file that can be viewed when the mini-view button representing the file is clicked under the **Results** group.

Generic Experiment

Generic is the simplest type of experiment run by TAM Air Assistant. It may be considered as pure data collection and the collected data are not divided into baseline, pauses or main sections. Hence, there are no means to perform automatic baseline corrections on saved data or perform data analysis using the special analysis functions in TAM Air Assistant.

1. Initialize and start a Generic Experiment. Chose **Generic** among the experiment types in the **New Experiment file** menu. The following view appears letting the operator choose which type of device to be used in the experiment.



2. Choose the calorimeter device, then select the channels that are to be used in the experiment by clicking on the channel in the figure. Several channels can be chosen at once by pressing the Ctrl-key while clicking the respective channel(s) (or alternatively by clicking on one channel and drag the cursor over the remaining channels that are to be used).

   The color on the top of the channels turns green as they are chosen.

NOTE: A thermostat device can also be selected to collect temperature data.

- If the option **Write lock wanted** is checked, the calorimeters are not available for various control actions from other experiments or from the devices, *i.e.*, others cannot write to the devices (default).
- If the option **Read lock wanted** is chosen, it is not possible for any other experiment (and thereby other users) to collect data to another experiment.
- If **No device lock wanted** is checked, full access to the chosen devices is possible from other experiments and users.

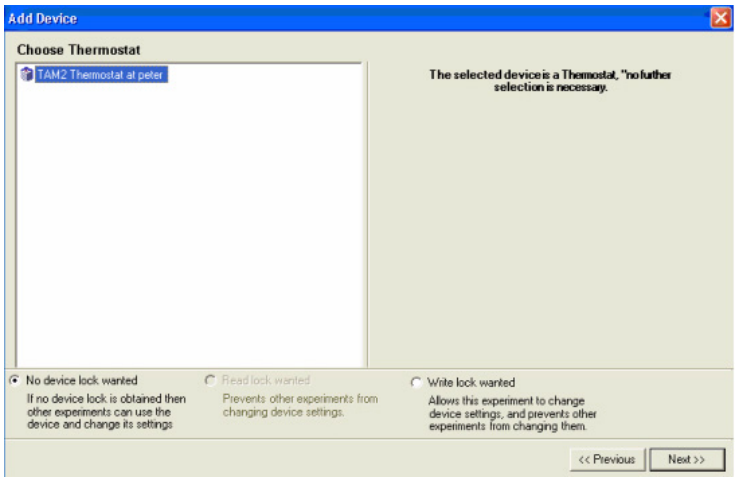
3. Decide which type of experimental information to include in the results file. Choices are made in the two drop-down menus. If **None** is chosen in both drop-down menus, no sample information can be included in the experimental file.

4. Click **Finish** to start the data collection. A button appears in the Experiment group, which can be used to view the progress of the experiment at any time. Experimental information can be entered while the experiment is running.

Adding Devices

At this stage it is possible to add more devices than you had chosen before the data collection was initiated. For instance, thermostat signals or additional calorimeter channels can be added.

To add a new device follow these steps:

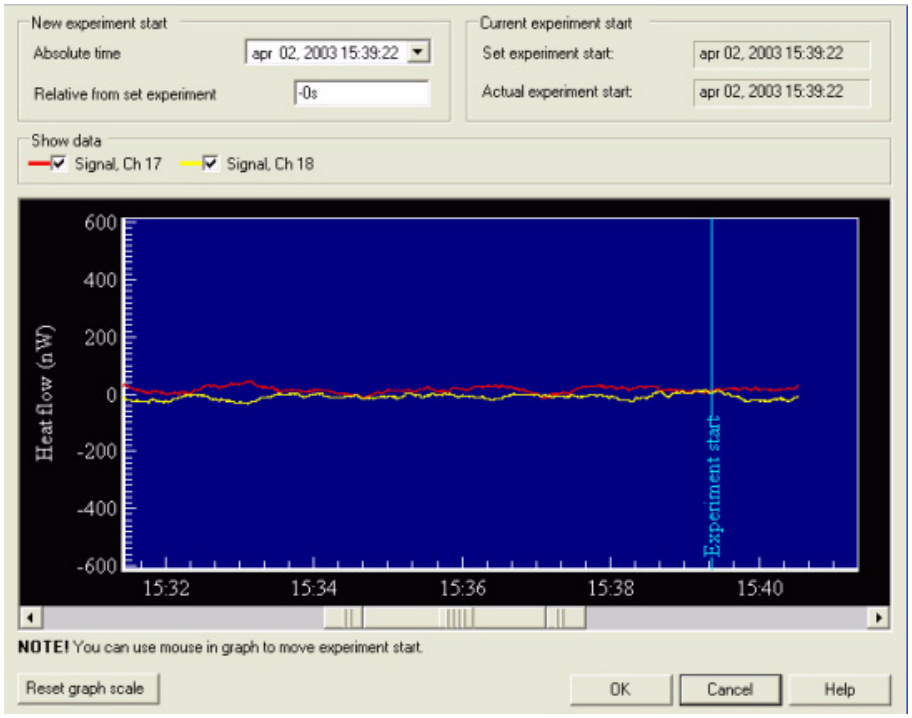


1. Click the **Add device** button in the top menu. The dialog box shown to the right is displayed.

2. Mark the device to be added and click **Next**. A dialog is displayed that allows you to select experimental information.

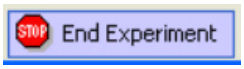
3. Click **Finish** to start the data collection from the newly added device.

6. After the collection of data has been initiated it is possible to set the start time for the data collection. Just click **Set Experiment Start** on the top menu of TAM Air Assistant.



To change the experiment start time, move the cursor to the bar marked **Experiment start** and drag it to the time of choice. Note that this can be done back or forward in time as long as there is data to be captured. The start time can also be entered in the **Absolute Time** field.

7. Click the **End experiment** button in order to finalize the data collection and save the data in a results file. This is done manually in a Generic experiment. A dialog box appears that allows you to decide whether you really want to end the experiment and, if so, discard the results.



8. Leave the check box empty in order to save the collected data in a results file.

9. Click **OK**. The collected data is saved in a newly created results file under the Results group.

Loading the Sample and Reference

Practical Details

NOTE: When you run a series of similar experiments, it is not always necessary to remove or change the reference ampoule between measurements.

1. Clean the outer surface of the ampoules with a new piece of dry, dust-free paper. There is only a very small air gap between the ampoule and the measuring tube, so it is essential to keep the outer surface of the ampoule clean so that the measuring tubes remain free from dust or other particles.

2. Remove the top lid from side A of the channel to be used. Channel A is used for the sample ampoule and Channel B for the reference ampoule.

3. Use the lifting tool to lift out the aluminum heat sink plug.

4. Pick up the sample ampoule using the lifting tool and slowly lower the ampoule down into the measuring position. The ampoule should slide easily down into the measuring tube. Do not force it downwards.

5. Unhook the lifting tool from the ampoule and use it to replace the heat sink plug. Refit the top lid.

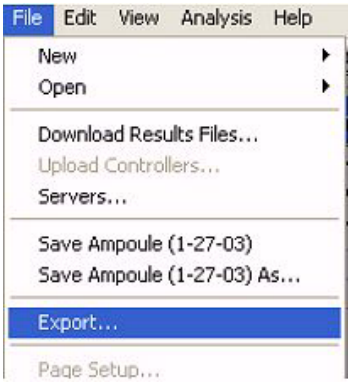
6. Using the lifting tool, load the reference ampoule into side B in the same way. Replace the heat sink plug and the top lid.

   It will take about 30 minutes to equilibrate when both ampoules are in the measuring position. During this time, the thermal disturbance caused by the introduction of the ampoules is eliminated. The true thermal power cannot be measured until equilibration is complete.

   NOTE: When working at higher temperatures in the range, for example 50°C, the time required for equilibration is longer. If required, the equilibration time can be reduced by pre-incubating the prepared ampoules in a dry oven or heating block.

Exporting Data

Data that has been saved in a results file can be exported for use with other software for data treatment, visualization, etc.



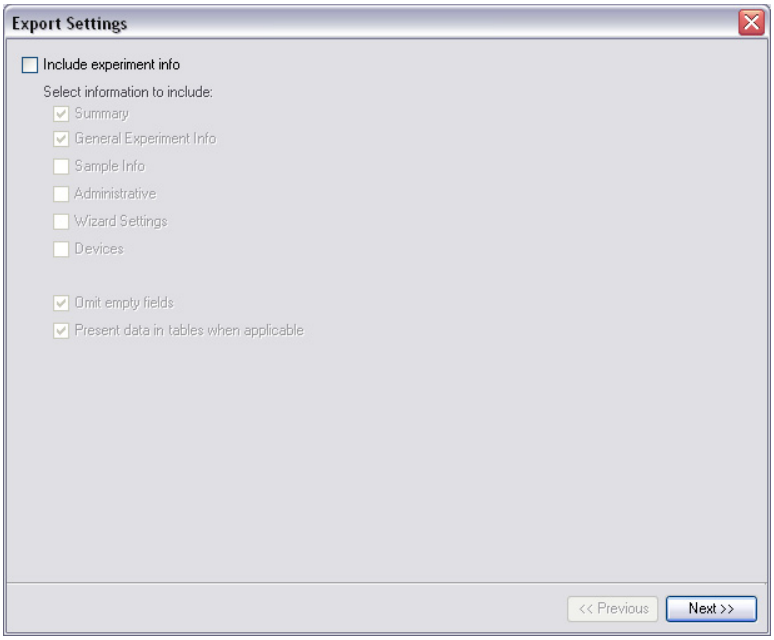
Follow these steps to export results files:

1. Choose **Open/Results File…** under the **File** menu.

2. Select and open the desired results file. Choose **Export…** from the **File** menu.

   A **Save as…** dialog is displayed to allow you to choose the directory and filename.

3. Choose the export format. There are three formats to choose from: CSV (comma-separated value), txt (text file), and as a Microsoft Excel file.



4. Click **Save**. The dialog shown in the figure to the right is displayed. This dialog lets you choose the content of the exported data. You will be offered the opportunity to include experiment information, various data series, raw data, and/or statistics.

5. Check the desired options then click the **Next** button to proceed.

   In an ampoule experiment the baseline can be subtracted from the exported data. If baseline data before and after the experiment exists, the baseline is an interpolated linear function, taking possible baseline drift into account.

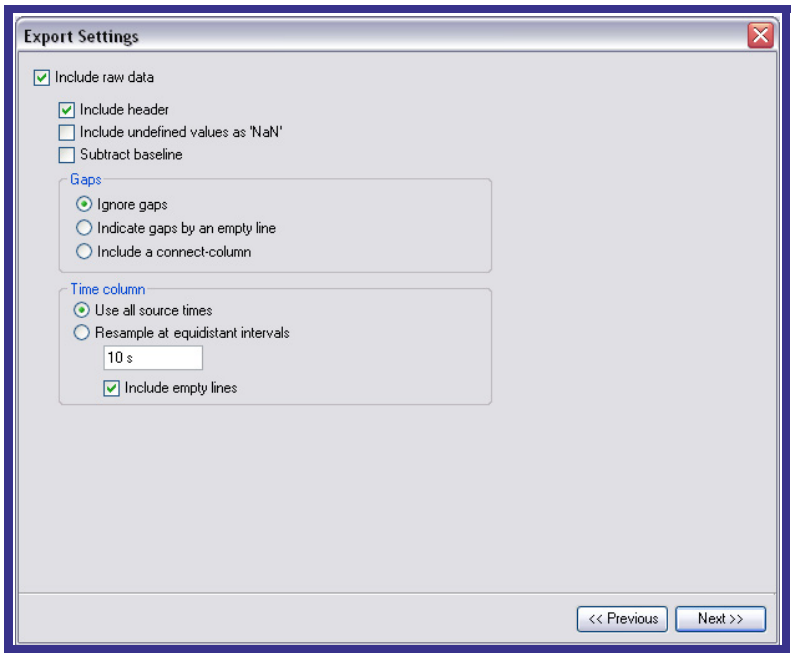
6. Choose the data series (types) for each channel to include in the export file by checking the desired items under **Select columns to include**. The data types are Time column, Heat flow, Heat, Normalized heat flow and Normalized heat. By default all of the data types are marked, so you will need to uncheck the ones that should not be included. Select the desired data series to export by checking the boxes then click **Next** again.

   If the signals from more than one device were collected in the results file, the dialog lets you choose the signals to be exported.

   NOTE: Scroll downward to enter the information for all channels.

7. Select **Next** to proceed.

8. Choose the desired raw data options shown in the figure to the right.



   The following guidelines should be considered:

- If the check box labeled **Include header** is marked when selecting raw data to include, the experimental information and sample information fields entered previously will be included in the among the exported data.

- Use the items in the **Time column** frame to choose how the data should be dispersed in time.

  - Select the **Use all source times** button to set up the exported data to include all collected data points as determined at the end of the experiment. All source time data collected does not need to be equidistant.

  - Select the **Resample at equidistant intervals** button to set a constant time interval between the data points, according to the time chosen in the text box. If the chosen interval is *shorter* than the interval between the raw data points, an interpolation method is used. If the chosen interval is *larger*, data will be removed by use of a filter function, and the time is adjusted to fit the time series starting with time=0.

9. Click **Next** to continue.

10. Check **Include statistics**, if desired, then check the information to include in the exported file. When completed, select the **Finish** button.

Exporting Data During an Ongoing Experiment

In cases when the experiments are long it can be useful to export and present data of an initial part of the experiment. This is possible by following the instructions above.

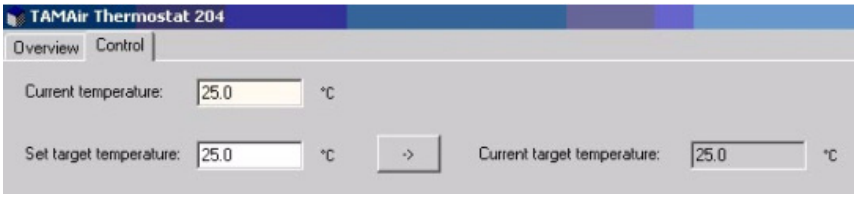
Changing the Set Temperature (Operating Temperature)

This section provides the instructions you need to set the operating temperature for the TAM Air.

Using TAM Air Assistant to Set the Operating Temperature

1. Choose the Thermostat in the devices group, and click the **Control** tab.

2. Write the new temperature in the text box named **Set target temperature**.



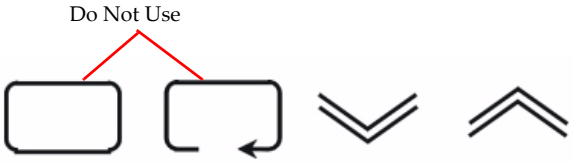
3. Click the arrow pointing to the right to enter the temperature. The new value is seen in the **Current target temperature** text box.

4. After setting the temperature, allow the temperature to stabilize for at least 8 hours. Although the circulating air reaches the set temperature very quickly, the calorimeter block will take several hours because of its large mass, high heat capacity and surrounding insulation.

NOTE: If you need to check the actual temperature of the calorimeter block, see the next section for instructions.

Using the Control Panel to Set the Operating Temperature

The temperature of the air thermostat, *i.e.*, the operating temperature, can also be set from the TAM Air Assistant or the temperature control panel located on the front of the instrument. This set temperature is recalled in the event of a power failure.



Temperature Control Panel Operating Buttons

The control panel has two digital indicators, one with red digits showing the actual temperature of the air thermostat and the other with green digits showing the set temperature. The amber indicator light in the top right-hand corner of the control panel will normally flash, indicating that the heater is on. The amber light will remain off if the thermostat is cooling.

The two arrow buttons are for increasing and for decreasing the set temperature. The two buttons to the left have no function in normal operation. They are used in the factory together with a special code for programming the control panel. If you enter the menu by accident, press the left-most key once to exit. Note that settings for the calorimetric temperature also can be made directly in the software, see the section, "TAM Air Assistant Software Overview" for details.

WARNING: The two buttons on the left of the control pad should only be used by authorized personnel.

NOTE: This option should only be used in situation when the TAM Air Assistant software is not in use.

1. Use the up and down arrow buttons until the required set temperature is displayed in the green digital indicator.

2. After setting the temperature, allow the temperature to stabilize for at least 8 hours. Although the circulating air reaches the set temperature very quickly, the calorimeter block will take several hours because of its large mass, high heat capacity and surrounding insulation.

NOTE: If you need to check the actual temperature of the calorimeter block, see the next section for instructions.

Care and Maintenance

TA Instruments recommends that you create a maintenance list for regular maintenance operations, such as cleaning the measuring cups or maintaining the air filter as described in the following sections.

Cleaning the Measuring and Reference Cups

Since there is only a very small air-gap between the ampoule and the walls of the measuring tube, dust and other particles can get into that gap and create problems. Therefore, it is essential to ensure that the measuring and reference tubes and cups are kept clean. The top lid, the measuring tubes and cups, and the holes in the top part of the calorimeter must be cleaned regularly.

To clean the inlet tubes of a calorimeter, use a vacuum line fitted with a 10-mm wide plastic suction tube. Alternatively, you can use a plastic or wooden rod with the tip wrapped in lint-free cotton.

WARNING: Never introduce cleaning liquids of any kind into the measuring and reference tubes since this may cause damage to the measuring sensor.

After cleaning, check that the clean ampoule slides easily down into the measuring tubes. **Never force an ampoule into a measuring tube.**

Maintaining the Air Inlet Filter

Failure to adequately maintain the air inlet filter is the most common cause for instrument failure. This failure usually results in the inability of the thermostat to regulate the temperature properly.

There is a fiberglass air inlet filter on the external air circulation fan located on the underside of the thermostat cabinet. This must be removed and inspected at regular intervals (at least every month) to ensure that blocking does not occur. In a dusty environment, filter checks should be made at more frequent intervals.

To access the filter, pull off the black square cover plate on the underside of the thermostat cabinet. When refitting the filter holder after cleaning or replacing the filter, note the position of the locating pins on the edge of the two parts of the filter holding frame.

Either clean the filter with a vacuum line or wash it in warm soapy water and then dry it. Ensure that it is thoroughly dry before replacing it. **NEVER** replace the filter while it is still wet.

If the filter cannot be cleaned, replace it with a new one. Approximately ten cut filters are provided with the instrument. See the parts list at the end of this chapter to order more filters.

Checking the Fans

To check the functioning of the fans, just hold a light paper tissue close to the air outlet on the back side of the instrument.

Relocating TAM Air

TAM Air weighs approximately 40 kg (88 lbs). If you wish to move the TAM Air within a meter (approximately three feet) or so of its existing working position, follow these steps:

1. Stop any ongoing experiment and exit the program.
2. Switch off and disconnect the mains.
3. Disconnect the USB signal cable.
4. Lift the instrument as described below, keeping it level during the move.

If TAM Air has to be transported for a greater distance, follow the instructions above. In addition, perform these steps:

5. Remove all sample and reference ampoules.
6. Remove all insulation lids.
7. Disconnect the Dsub cable from the data logger.
8. Remove the calorimeter block from the air thermostat.
9. Use two people to lift the TAM Air from beneath the thermostat cabinet.
10. After repositioning the instrument, check the alignment of the measuring tubes. Use the lifting tool to lift out and refit the heat sink plugs at each corner to check that the holes in the lids are in direct alignment with the measuring tubes. It may be necessary to bring them into line by removing the thermostat lid and inner chamber lid and then slightly repositioning the calorimeter block. You may need to refer to page 17, "Choosing a Location."

Independent Measurement of the Calorimeter Block Temperature

To measure the temperature of the calorimeter block using an independent digital thermometer, follow these steps:

1. Prepare a plug of the same diameter as the hole in the thermostat lid, and fit the probe into the plug. This will prevent a flow of air from the thermostat.
2. If possible, surround the probe of the digital thermometer with a solid metal adapter of the same diameter as the sample ampoule.

3. Remove the stopper and the heat sink plug from any calorimetric channel.

4. Insert the probe into the measuring tube.

5. Ensure that there is no flow of air from the thermostat lid.

6. Wait for a stable reading from the digital thermometer. This will take about five minutes if an adapter is fitted, and about fifteen minutes if the probe hangs loose in the measuring tube.

The temperature reading on the digital thermometer should be within ± 1 °C of the set temperature. Any variation from this could be due to a highly active sample changing the temperature of the calorimeter block.

Troubleshooting

Use the following table to help troubleshoot problems with the TAM Air.

Symptom	Fault	Diagnosis/Action
No indicator lights on the temperature control pad and the two fans are not operating.	TAM Air appears to have no mains supply.	1. Disconnect mains supply. Check that the voltage indicated on the label on the backside of TAM Air is the same as used in the laboratory. Inspect the fuse and replace it if necessary with the correct fuse type and value. For 100-130V, use 4A T (slow) fuse For 200-250V, use 2A T (slow) fuse **Never use a fuse of the wrong type or value.** 2. If the fuse was found to be OK, refit it and leave the instrument for ten minutes or so with the top lid removed. It is possible that the automatic heat sensitive cutout has been activated. This device will cut the regulation heater if the air thermostat temperature exceeds 92°C. Once the temperature drops below 92°C the cutout will automatically reset without operator intervention. Replace the lid and reconnect the mains supply.
The operating temperature is not decreased.		If TAM Air restarts, suspect that the air inlet filter on the air external circulation fan is blocked. Carry out the Cooling Air Inlet Filter Maintenance as describedon page 42. Check that the two fans works properly.
Program cannot find data logger.	This fault might arise if the computer is being used on more than one application.	Close TAM Air Assistant and restart. If necessary, exit Windows and restart. Check that the correct USB-port is used.

(table continued)

Symptom	Fault	Diagnosis/Action
Temperature not controlling normally.	The two left buttons on the temperature control pad have been accidentally pressed and the temperature control system has entered the programming mode.	Press the two left buttons at the same time and hold for three seconds to leave the programming mode. Check the filter. If this does not overcome the problem, contact TA Instruments.
Abnormalities in the measurement values.		Calibrate and update the calibration constants.

Accessories and Spare Parts

This section lists the replacement parts for the TAM Air that are available from TA Instruments. Some parts must be replaced by a service representative. See the table below to order parts.

Part Number	Description
3350-2	Start-up Kit, 20 mL disposable glass ampoules, including: glass ampoules (452) and caps (500), ampoule lifting eyelets (20), crimping tool (1), centering tool
2421-010	Lifting Eyelet, for 20 mL disposable glass ampoules, pkg/1
24 60 2001	20 ml disposable glass ampoules, pkg/113
86 33 2000	Aluminum caps + sealing discs, for 20 mL disposable glass ampoules, pkg/100
3346-5	20 mL HDPE plastic ampoule with screw cap, pkg/1000
334601	Lifting eyelet, 20 mL HDPE plastic ampoules, pkg/1
311407	Top lid, pkg/1
323906	TAM Air ampoule lifting tool, pkg/1
323802	Heat sink plugs pkg/1 (Aluminum)
84 52 2550	Inlet filter material, pkg/5
2602-20	Stainless Steel ampoule, 20 mL, with circlip lid, pkg/1
2602-21	Glass Ampoule, 20 mL, with circlip cap, pkg/1
2277-309	Stainless Steel ampoule, 20 mL, with threaded cap, pkg/1
2461-020	Centering Tool, for 20 mL disposable glass ampoules, pkg/1
2462-000	Crimping Tool, for 20 mL disposable glass ampoules, pkg/1

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