TAM Assistant Software

Operator's Manual

Revision A
Issued March 2008

Notice

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WARNING:

If this instrument is used in a manner not intended or specified in this manual, the protection provided by the instrument may be impaired.

Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

NOTE: A NOTE highlights important information about equipment or procedures.

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

WARNING:

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

CAUTION: The instrument is not intended for use with combustible materials or corrosive substances without extra precautions.

Regulatory Compliance

Safety Standards

WARNING:

If this instrument is used in a manner not intended or specified in this manual, the protection provided by the instrument may be impaired.

The instrument is built with the intention to fulfill the demands described in the LVD Directive 73/23/EEC.

The EN 61010-1 (IEC 1010-1) standard specifies safety requirements for laboratory instruments.

Nanocalorimeter:

For compliance with requirements according to the electromagnetic immunity standard EN 61 000-4-3 + A1:

If TAM III is used in an environment with radiated electromagnet fields up to 3V/m, signal disturbances up to 250nW are possible. Avoid this by not placing a mobile phone or any other transmitter on top of TAM III, or in its proximity.

Minicalorimeter:

For compliance with requirements according to the electromagnetic immunity standard EN 61 000-4-3 + A1 and EN 61 000-4-6 + A1:

If the TAM III is used in an environment with radiated electromagnet fields up to 3V/m, signal disturbances up to 1uW are possible. Avoid this by not placing a mobile phone or any other transmitter on top of TAM III, or in its proximity.

Safety

Instrument Symbols

The following labels are displayed on the TAM III instrument for your protection:

CAUTION: Do not place a mobile phone or any other transmitter on top of TAM III, or in its proximity. This may cause signal disturbances. See page 7.

Symbol	Explanation
	The exclamation point within the triangle is a warning sign alerting you of important instructions accompanying the product.
	This symbol indicates that a hot surface may be present. Take care not to touch the ampoules, small aluminum lids over the ampoule holders, or interior of the instrument when running the calorimeter at temperatures higher than ambient. Do not allow any material that may melt or burn to come in contact with these hot surfaces.

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

Electrical Safety

For your safety, please observe the following precautions:

- Always disconnect the instrument from the mains electrical supply before attempting to change a fuse, opening the back panel, or in any other way try to access the interior of the instrument.
- Protective earth must be connected when the instrument is in use.
- A defective instrument must not be used and must be disconnected from the mains supply.
- The instrument must be connected to earth at all times.
- Be aware that the instrument can automatically restart.

> **WARNING:**
>
> **Install the instrument only in an environment free from conductive contaminates, moisture, flammable liquids or gases, or corrosive substances. If conductive liquid spills into the instrument, turn off the instrument at once by pulling out the rear power connector.**

- If there is a risk of spilling flammable liquids into the instrument, install an emergency breaker at a secure distance from the instrument. (All spillage inside the instrument must be removed before turning the instrument on again, to avoid damage or danger).
- Do not prevent fan ventilation (at least 30 cm (12 in) distance from any wall, desk panels etc. is recommended.).
- Do not remove or insert any electronic modules without before turning of the instrument.
- A failure has occurred if the instrument has automatically turned itself off and the front LED has changed color to red.

Chapter 1:

Introducing TAM Assistant

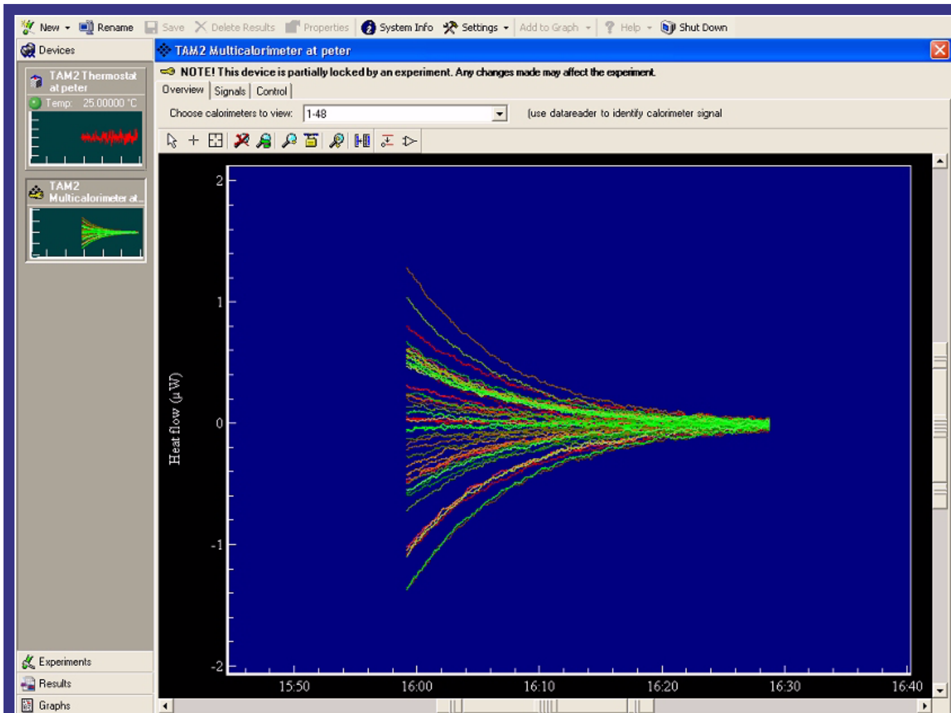
Overview

In this manual, TAM Assistant, the dedicated software to TAM III, the third generation TAM, and the TAM 48-channel calorimeter, is described in detail.

Two versions of the software for TAM III exist:

- TAM Assistant Embedded
- TAM Assistant (external version)

Both versions are essential for running the TAM III. The two versions are similar in appearance and many features are common, but the external version has more functionality with report functions, data analysis, etc. TAM Assistant Embedded is pre-installed on the TAM III system when delivered, while the TAM Assistant is intended for an external computer connected to the TAM III and/or a local network (*e.g*. an office computer).

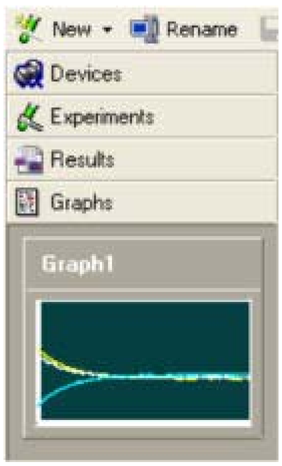


TAM Assistant Embedded Software

A view of the program interface of TAM Assistant Embedded is shown above.

The *Group field* in the left panel of the program main window contains a number of fields that are somewhat different for TAM Assistant as compared to TAM Assistant Embedded, see the table below.

<table>
<tr><th>TAM Assistant Embedded</th><th>TAM Assistant</th></tr>
<tr><td>Devices explorer</td><td>Devices explorer</td></tr>
<tr><td>Experiment explorer</td><td>Experiment explorer</td></tr>
<tr><td>Results explorer</td><td>Results and data files</td></tr>
<tr><td>Graph</td><td>Report</td></tr>
<tr><td>Group Field

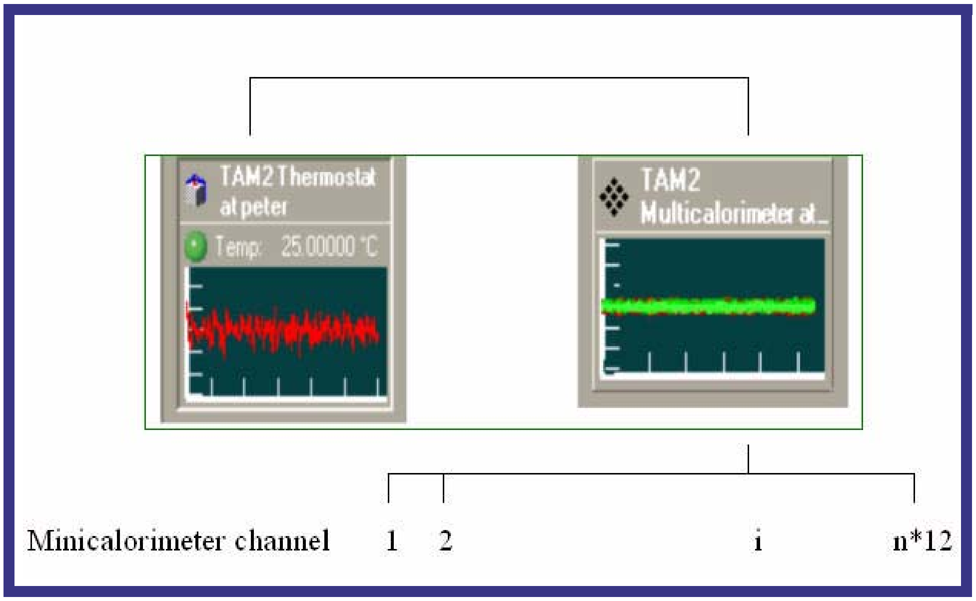


</td><td>Group Field

</td></tr>
</table>

By clicking a group header, a list of *mini-view buttons* is shown that is specific for each group. For instance, clicking the **Device** group displays a list of the connected devices (e.g. the thermostat and calorimeters in the TAM III) or selecting the **Results** group lists saved results files.

The device structure for TAM 48 Channel is shown in the diagram on the next page.

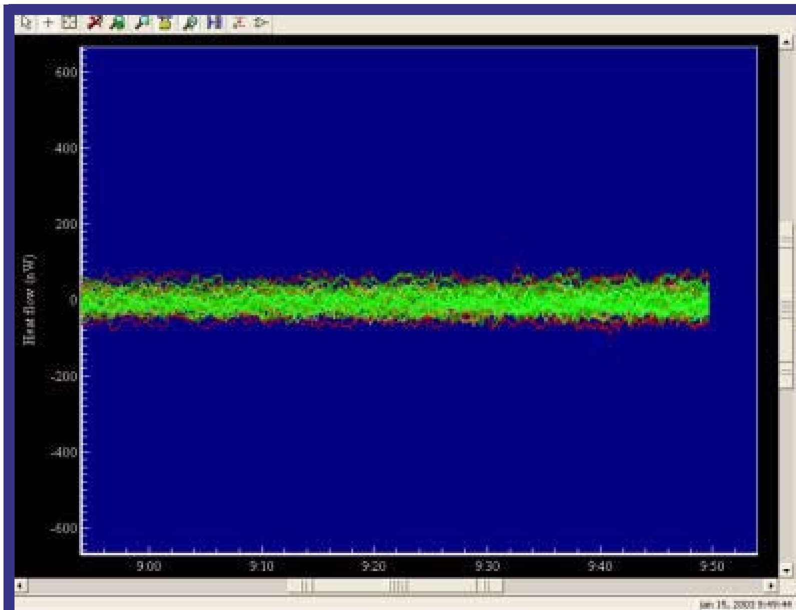


The calorimeters for the TAM 48 Channel or the multicalorimeter of the TAM III may be regarded as *sub* devices under the multicalorimeter *main* device, and a software control interface exists for each of them. Device windows for the individual calorimetric channels are accessed through the Multicalorimeter main device button (*i.e.*, the mini-view button). A unique device interface exists for the thermostat. The number of connected calorimeter sub devices is always a multiple of 6 for the Multicalorimeter TAM III or 12 for TAM 48 Channel (with n=1, 2, 3 or 4).

TAM Assistant Plot Windows

Plot windows in TAM Assistant can be viewed in a variety of situations. For example, a plot can be viewed under the **Overview** tab associated with a connected device under the **Devices** group, or when showing the results in a saved experiment file accessed from the **Results** group in TAM Assistant Embedded or from the **Results and data Files** group in TAM Assistant external version. Interactive graphs appear also in TAM Assistant report files.

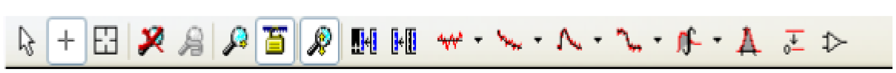
An example of the plot window for a multicalorimeter device is shown in the figure below.



Plot Window

The **scale bars** on the horizontal and vertical directions of the plot window is used to change the scale of the x and y axes and also move along an axes in the x or y direction without changing the scale.

For example, mouse clicking on the left side of the horizontal scale bar (x-axis) and dragging to the left will scale down the time axis towards earlier time values. Up scaling from low time values is achieved when dragging to the right. Mouse clicking on the middle part of the scale bar and dragging to the left or right simply moves the time axis toward lower or higher time values with the time scale preserved.



Plot Toolbar

The toolbar above the plot contains a number of tools with which one can control the plot window. See the table on the next page for an explanation of each button.

Toolbar Buttons

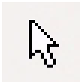
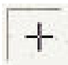
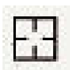
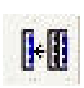
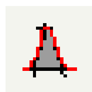
Button	Description
	Use the selection tool to choose a curve when creating a report or graph.
	Activating the **plus** sign (enter Zoom Mode) allows you to manually zoom in on events on the graph by clicking and dragging.
	Press the **sight** sign (enter Data Reader Mode) to show the value of the x and y axis in a pop up text box when the cursor is within the bounds of a curve. It is the position of the cursor along the x axis that determines the chosen x,y point. The color of the curve turns white when sighted. If the left mouse button is pressed down while moving the cursor, the change in heatflow and time between marked points will be seen.
	The **reset zoom** button (Scale to show all) scales the plot to a predefined scale. The predefined scale may be considered as 100 % magnification.
	Use the **undo zoom** button to recapture the last magnification. Press this button several times to recapture subsequent plots.
	**Auto scale right side x** (left icon) and **lock x-axis range** (right icon) work as check boxes. Select one of the two functions.
	The **auto scale y** button auto scales the y-axis according to a defined magnification, which is dependent on the min/max range of the graph.
	The **Multiple Offset** button changes the offset of each individual curve so that it is positioned approximately in the middle of the screen. This is useful for comparison of curves positioned far from each other. The absolute values of the curves are the same, but the offset differs, *i.e.*, it will not have a value, only a plus sign below on the x-axis.
	Normally the scale is the same for several graphs in common plot. Press the **Multiple Scales** button to create individual scales for each graph. The magnification will hence be 100 % for each graph. Use this function when comparing the signal quality between graphs as generated by signals of different absolute values.

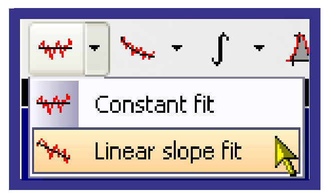
table continued on next page

Button	Description
	Use the **curve fitting** buttons to fit a mathematical model to a chosen part of a curve. Use the first button to choose between linear fit and a constant fit (*i.e.*, zero slope). Use the second button to choose between a number of exponential equations. Use the third button to fit dual exponentials. Use the fouth to fit different sigmodials , see below for more details.
	Select a curve then use the **Integrate** button to perform a peak integration analysis. You can click the arrow to the right of the button to integrate in different ways, see below for more details.
	Select a curve then use the **Analyze Peak** button to obtain the peak's range, integral, width, height, inflection, half-height, amplitue, etc.
	Use the **set signal offset** and **define signal gain** buttons to define the signal offset as well as the calorimeter gain factor. These operations can be used to perform a calibration manually.

NOTE: A single plot can be chosen among several plots in the graph.

Curve Fitting Functions

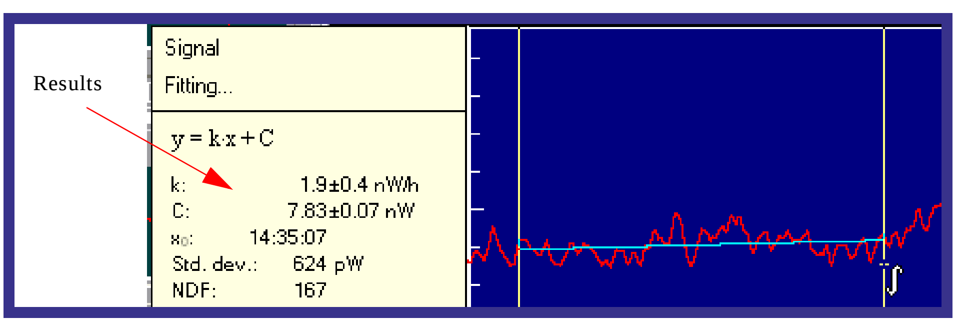
The curve fitting buttons can be used in the graphs of the **Devices** group, in a Results file, or in a Report.



Click on the down arrow and choose the model of interest, *e.g*., **Linear slope fit**, as seen in the figure to the right.

Move the cursor to the plot in the graph that is of interest.

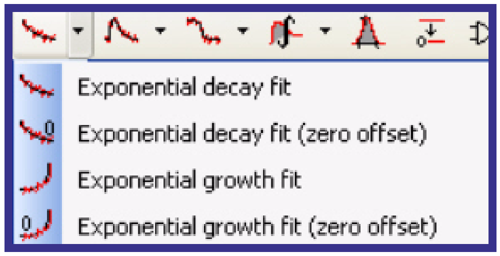
The broken lines appear on both sides of the curve, click the left mouse button to mark the curve. To make fitting move the cursor to the initial time, click and drag the cursor over along the curve over the time interval that is to be fitted.



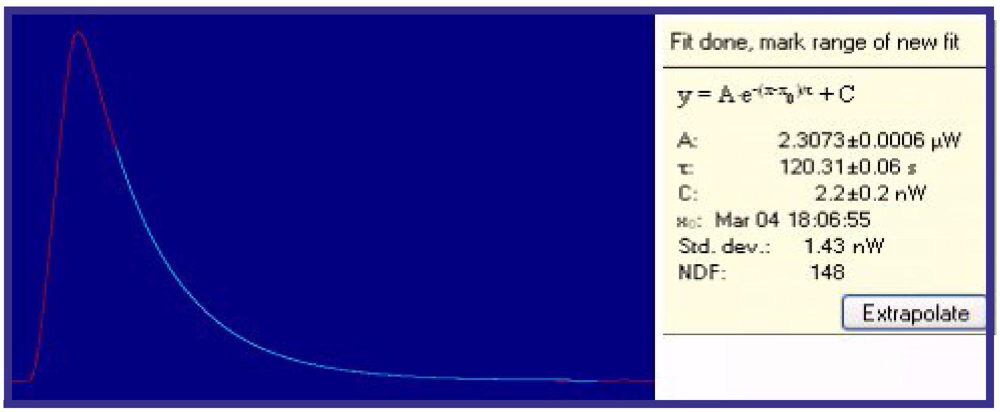
The results of the regression are displayed in the text field on the upper left side of the graph. Beside the estimated fitting parameters (slope, k and intercept, C in the example above) the initial time, $\mathbf{x_0}$, is given. This time corresponds to x = 0 in the equation. The assignment **Std. dev** is the standard deviation of the residuals, *i.e.* the difference between the calculated values with the estimated model parameters and the experimental points. **NDF** is the number of data points used in the regression.

Exponential Decay Fits

There are four different exponential fits available for either exponential decay or exponential growth. In the options with zero offset, the fitting function assumes the decay to go to zero heat flow or the growth to origin from zero heat flow.



A fit could look like the figure below (blue line) and the results are given in the small window appearing when clicking the fit button. The function fitted is shown in the results box .



A : the signal amplitude at time x

τ: the time constant for the decay (growth)

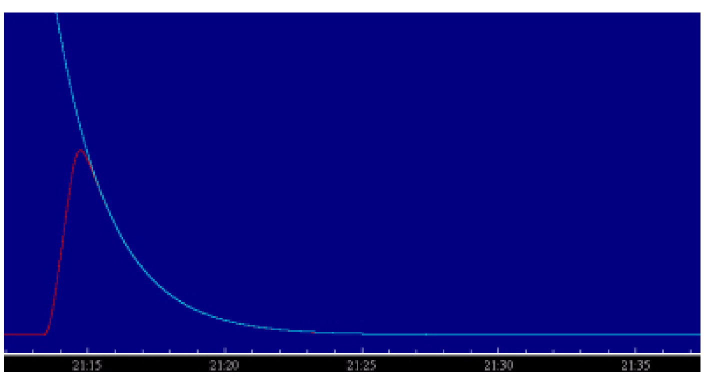
C: the offset. It will not be shown if the zero offset function is used.

x: the start time of the fit.

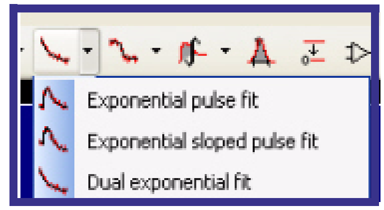
Std.dev: the standard deviation of the residuals i.e. the difference between the calculated values with the estimated model parameters and the experimental points. This is also referred to as the signal noise level

NDF: the number of data points used in the regression excluded the number of unknowns

If the **Extrapolate** button is pressed, the exponential fit will be extended in both ends.

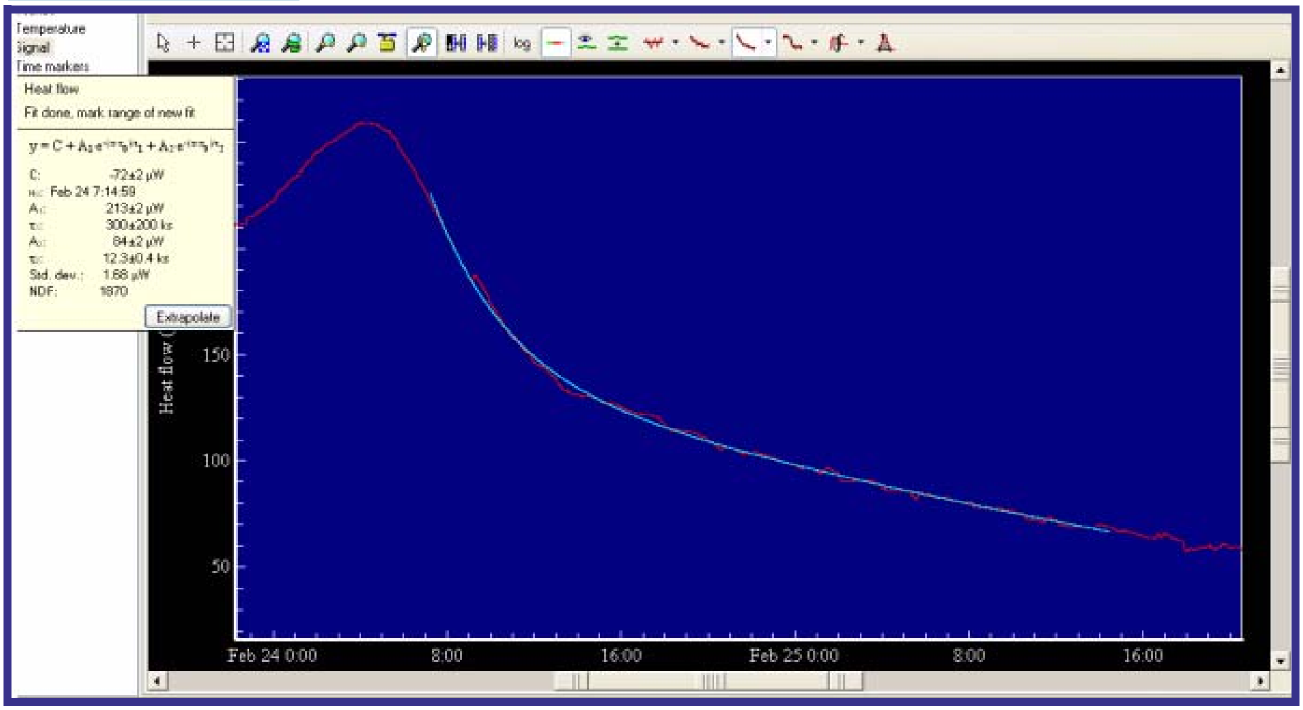


Dual Exponential Fits

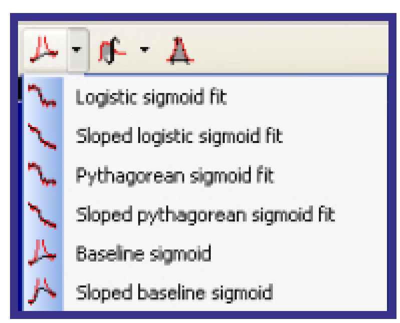


When using the **Dual exponential fit** button there are three options. In all these fits two time constants are returned.

In the example below there are two decays in the heat flow, one that is much faster than the other. The returned results will contain the two different time constants.

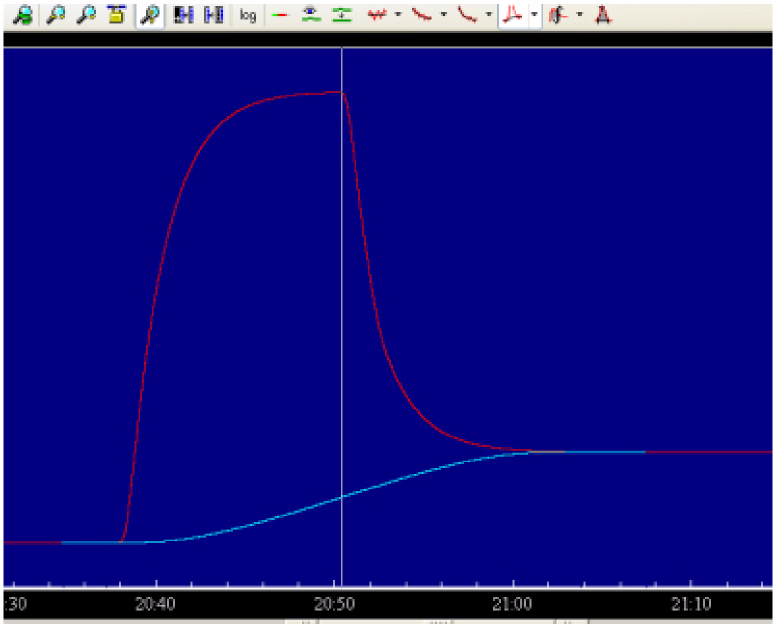


Sigmoid Baseline Fitting Functions

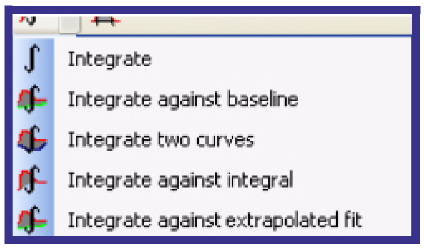


There are three different types of sigmoid baseline fits—Logistics, Pythagorean and Baseline. The Logistics and the Pythagorean sigmoids are calculated in two different ways. The function is shown in the results window. You will define the baseline sigmoid. Each of these models allows constant or sloping baseline sections to be fitted.

To use the baseline sigmoid mark a section before the peak and then a section after the peak. Now a sigmoid baseline will be drawn between the two sections and a white line defining sigmoid midpoint. This line can be moved to change the midpoint. See the figure on the next page.

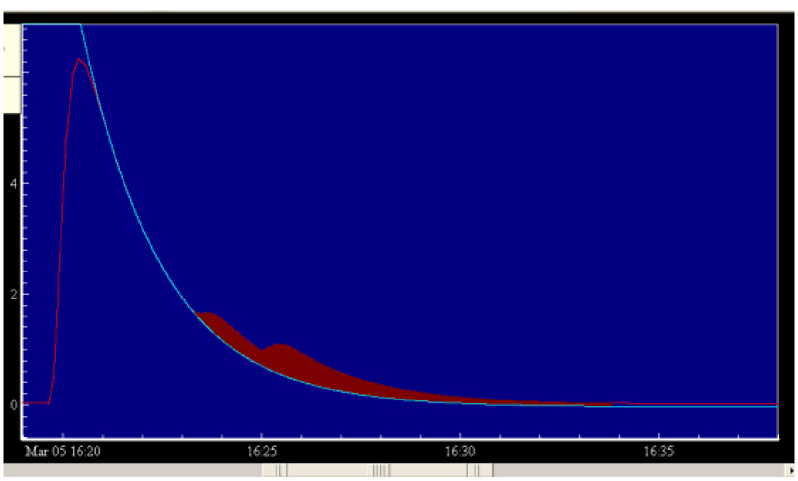


Integrations



There are five different integration functions.

- **Integrate** - Integration between the user defined start and stop in the curve.
- **Integrate against baseline** - Integration between user defined start and stop taking into account the baseline defined by the experiment or if no experimental baselines are available to a zero offset.
- **Integrate two curves** - Integration of an area between two curves.
- **Integrate against integral** - Integration based on the assumption that the rate of baseline shift is proportional to the magnitude of the measured signal. This will give a sigmoid baseline that is not operator dependent.
- **Integration against extrapolated fit** - The integration is made from an extrapolated baseline fit.



The Devices Explorer

In the **Device** group, the connected devices are listed in the group area. Different auxiliary devices like pumps, mass flow controllers, etc. may be included, depending on the TAM configuration.

Click on a mini-view button under the Device group to open a new window in the view area, which is specific for the chosen device. If the chosen device is of a type that generates an output signal, the signal is shown in a plot window with a graph of the signal evolution as a function of time.

The Thermostat Device

In normal operator mode, the thermostat device interface consists of three views accessed through the **Overview, Control,** and **Settings** tabs.

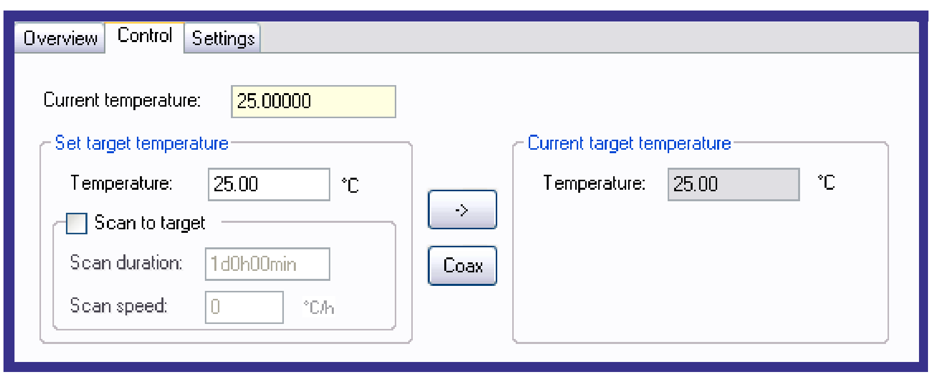
Under the Control tab, the signal evolution of the thermostat temperature, as a function of time, is shown in a plot window. See the previous section in this chapter, “TAM Assistant Plot Windows” for a description of the plot window.

Changing Temperature Using the Control Tab

Follow these steps to change the temperature setting of the thermostat:

1. Access the temperature settings of the thermostat using the **Control** tab, see the figure below. The temperature of the thermostat can be set to any value within 15 to 150 °C for the oil based thermostat. When using the water thermostat, the range is 15 to 90 °C.

2. Enter the new value in the text box marked **Temperature** in the **Set target temperature** box to change the temperature.
3. Click the arrow button. The new temperature will appear in the corresponding



text box under the **Current target temperature** box, and the thermostat will start to adjust itself to the chosen temperature.

4. To decrease the time for the calorimeters to come to thermal equilibrium at a

new temperature, press the **Coax** button instead of the arrow. By doing this the temperature of the thermostat will be overset for a short period of time, then decreased to the set temperature

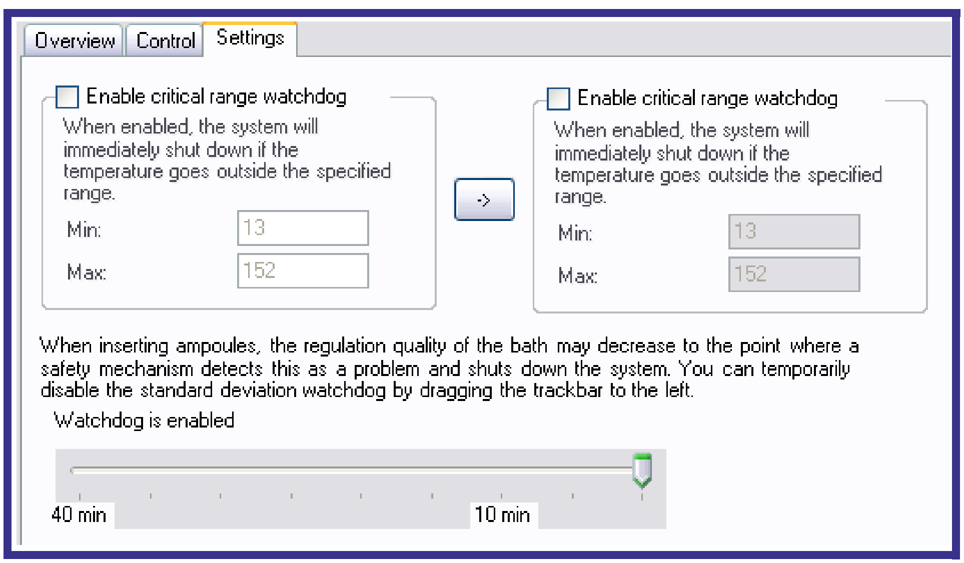
5. To change the temperature in a controlled way, check the box **Scan to target**. There is now an option to set the change rate or the time to reach a new temperature.

The target temperature will be reached within 10 to 60 minutes, depending on the magnitude of the temperature jump. The rate of the temperature change is between 2 to 10 K h^{-1}, depending somewhat on the absolute value of the temperature.

If you attempt to change the temperature from the current value to an arbitrary value outside the specified interval, an error message will appear and the command will not be implemented.

Setting a Safety Range Using the Settings Tab

Select the **Settings** tab to set a safety range of the thermostat temperature. If the **Enable critical watchdog** box is checked, the thermostat will shut itself off, providing the temperature is outside this range. TAM III will not restart after that. If the thermostat is shut down by the critical watchdog, it needs to be reactivated when TAM III is manually started again.



The 3228 20-mL Multicalorimeter Device

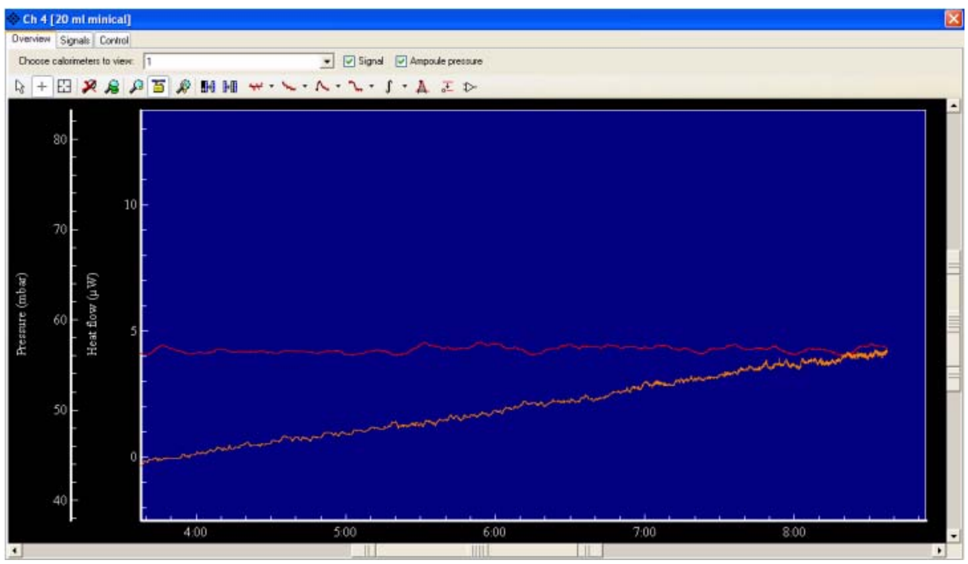
When the 20-mL Multicalorimeter device is selected in the **Devices** group, a window opens that let you choose between a numbers of views associated with the 3226 20-mL Minicalorimeters.

To view and control features of the 3228 Multicalorimeter device in TAM Assistant, enter the Devices explorer to the left of the TAM Assistant window and click the Device Miniview button.

There are three main views that can be accesses through the tabs: **Overview**, **Signals** and **Control**.

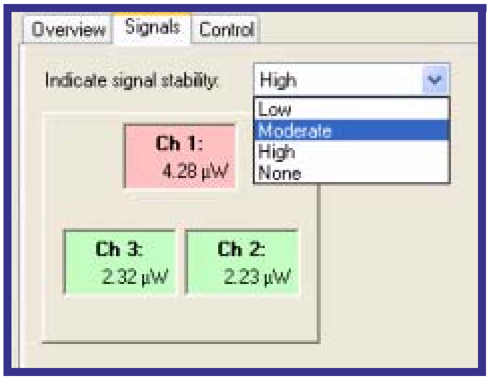
The Overview Tab

Under the **Overview** tab the signal evolution of the heat flow from the calorimeters can be viewed continuously as a function of time. When a pressure sensor is connected, the ampoule pressure can also be displayed, see the *Pressure Ampoule Operator's Manual* for details. A more detailed description of the of the functionalities associated with the plot window is found earlier in this chapter.



The Signals Tab

Click on the **Signals** tab. The figure to the right appears for the Minicalorimeters. A similar view appears for the 3208 Multicalorimeter of TAM III or the TAM III 48 Channel instrument.



Each rectangle in the figure represents a Minicalorimeter. In this case the three indicators represent the calorimeters of a 20-mL Multicalorimeter and the current signal is displayed for each channel.

A red rectangle indicates that the signal from the Minicalorimeter is not stable according to a specified stability criterion. When the signal becomes stable according to the given criterion the rectangle changes color to green. The criterion can be defined in the drop-down menu named **Indicate signal stability**. The signal stability conditions include **Low**, **Moderate**, and **High**. The default value is Moderate. This is a criterion that should be fulfilled by all calorimeters in a TAM III system. The values for the different stability criteria are given in the table below.

Stability Criteria

	Slope$_{min}$ /nW h^{-1}	**Slope$_{max}$** /nW h^{-1}	**Deviation** /nW	**Window** /min
High	50	500	100	30
Moderate	250	3000	200	20
Low	1000	5000	500	15

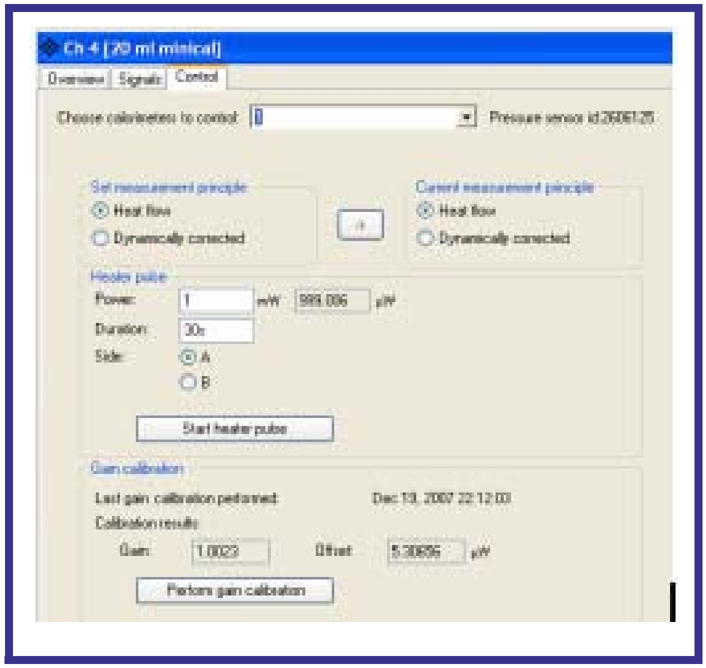
For a detailed explanation and definition of the stability criteria see “The 3208 Multicalorimeter Device” in this chapter.

The Control Tab - Control of Calibration Heaters

Under the **Control** tab, a form is displayed that can be used for manually controlling the actions of calibration heaters and calibrations according to a defined protocol. However, the easiest way of performing calibrations is through the Perform gain calibration push button, see Chapter 2 for details on how to calibrate the calorimeters.

The channels to be included are chosen in the drop down picture by pressing the *down arrow* on the right side of the text field on top of the main frame.

There are two calibration heaters for each Minicalorimeter, one on the sample side (A) and the other on the reference side (B) of the Minicalorimeter. A prefix, *e.g.*, m, µ, n etc., is entered automatically according to the magnitude of the given heat effect. The magnitude and duration of the heat pulse is entered in the text boxes in the frame labeled **Heater pulse**.



Pressing the **Perform gain calibration** button in the **Gain calibration** group initiates an electrical calibration. The calibration will run according to a defined protocol, see Chapter 2 for a more detailed description.

The values of the Gain and Offset from the latest performed calibration are shown in the corresponding text boxes only when one Minicalorimeter channel is chosen in the drop-down list. The time for the last calibration is also displayed.

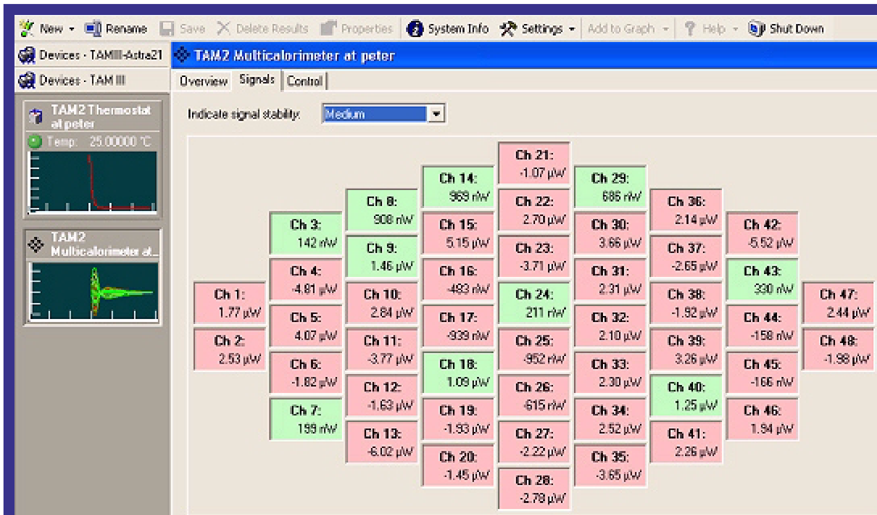
The 3208 Multicalorimeter Device

When you select the Multicalorimeter device in the **Devices** group, a view opens that lets you chose between a number of views associated with the 3206 calorimeters.

In normal mode you can access three main views through the tabs—**Overview**, **Signals,** and **Control**. Under the **Overview** tab the signal evolution can be followed as a function of time in a plot. See the previous section in this chapter, “TAM Assistant Plot Windows” for a description of the plot window.

The Signals Tab

Click on the **Signals** tab to display the following view for the calorimeters of TAM 48. A similar view appears for the 3208 Multicalorimeter of TAM III.



Signals View

Each rectangle in the figure represents a calorimeter. In the figure above there are 48 calorimeter channels installed on the system, and the current signal is displayed for each channel.

A red rectangle indicates that the signal from the calorimeter is not stable according to a specified stability criterion. When the signal becomes stable according to the given criterion, the rectangle changes color to green.
The criterion can be defined in the drop-down menu named **Indicate signal stability**. The signal stability conditions include **Low**, **Moderate**, and **High**. The default value is **Moderate**. This is a criterion that should be fulfilled by all calorimeters in a TAM III system. Stability criteria are given in the table on the next page.

NOTE: The criterion chosen here acts locally, together with the diagram, and will not affect an ongoing experiment since baseline criterions, etc. are chosen uniquely for each experiment during the start-up phase. The purpose of this diagram is for information only.

Stability Criteria

	Slope$_{min}$ /nW h^{-1}	**Slope$_{max}$** /nW h^{-1}	**Deviation** /nW	**Window** /min
High	50	500	100	30
Moderate	250	3000	200	20
Low	1000	5000	500	15

A stability criterion is composed of the three parameters shown in the table.

- Slope is a fitted straight line (polynomial of order 1) by a minimization procedure.
- Deviation is the standard deviation of the residuals with respect to a fitted straight line.
- Window is the time interval for which the slope and deviation are calculated. The window is moving stepwise with an interval of one second.

When the value for the slope is found to be below the Slope$_{min}$, the calorimetric signal is considered *stable*. After the value for Slope$_{min}$ and Deviation have been met, the signal is considered *unstable* whenever the slope is higher than Slope$_{max}$. In order for the signal to be considered stable, after it has become unstable, the criterion for Slope$_{min}$ has to be met again.

In addition to the Slope, the criterion for the Deviation has to be fulfilled at all times in order for the signal to be considered stable.

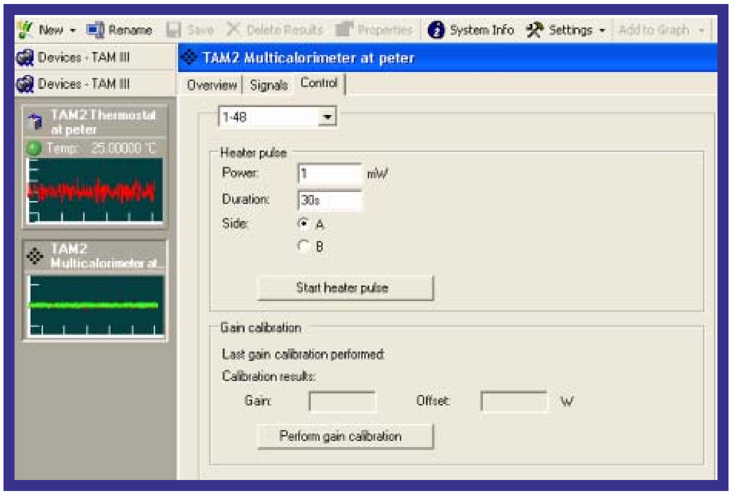
When **None** is chosen all rectangles changes color to white.

Controlling the Calorimetric Calibration Heaters

Under the **Control** tab of a calorimeter device, a form representing a user interface from which manual control actions of calibration heaters and calibrations according to a defined protocol can be made.

The channels to be included are chosen in the drop-down picture by pressing the *down arrow* on the right side of the text field on top of the main frame.

There are two calibration heaters for each calorimeter, one on the sample side (A) and the other on the reference side (B). A prefix, *e.g.*, m, µ, n, etc., is entered automatically according to the magnitude of the given heat effect, or it can also be inserted manually. The magnitude and duration of the heat pulse is entered in the text boxes in the frame labeled **Heater pulse**.



Press the button **Perform gain calibration** in the box named **Gain calibration** to initiate an electrical calibration. The calibration will run according to a defined protocol, see Chapter 2 for a detailed description.

The values of **Gain** and **Offset,** from the latest performed calibration, are shown in the corresponding text boxes only when one calorimeter channel is chosen in the drop-down figure.

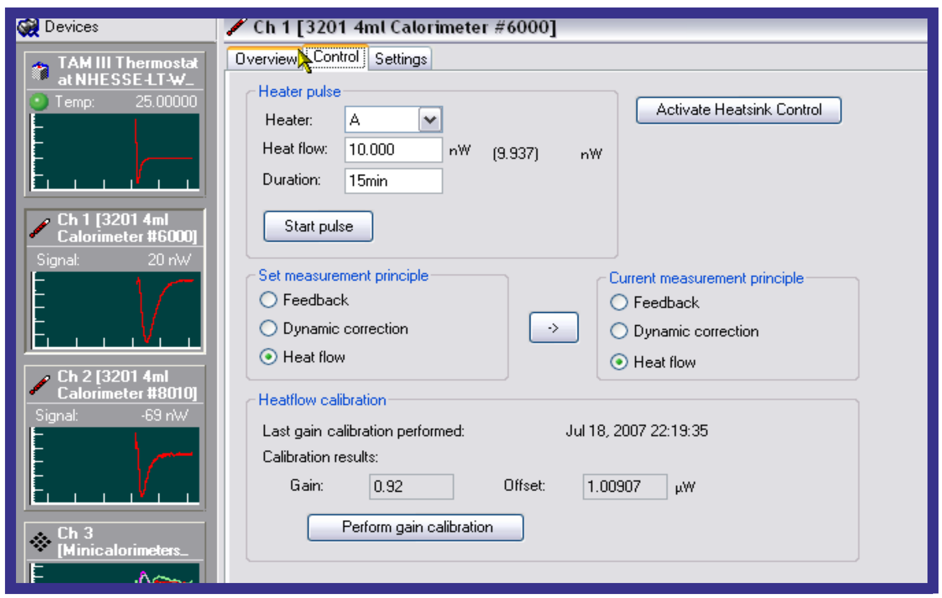
The 3201 Nanocalorimeter / 3220 Microcalorimeter Device

Select the calorimeter device in the **Devices** group to chose between a numbers of views associated with the 3201 Nanocalorimeters (or the 3220 20-mL Microcalorimeter device).

There are three main views that can be accessed through the tabs—**Overview, Control,** and **Settings**.

The Overview tab allows you to follow the signal evolution as a function of time in a plot. See the previous section in this chapter, “TAM Assistant Plot Windows” for a description of the plot window. The Control tab is explained in the next section.

The Control Tab

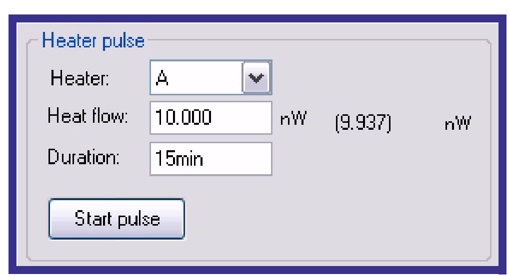


The Control Tab

The **Control** tab of the 3201 Nanocalorimeter Device (shown in the figure above) is used for manual control of the calibration heaters, and for performing calibrations according to a defined protocol. Other functions include controls of **Measuring principle** and the **Peltier regulator** (or active heat sink control), which are described below.

Heat Pulse

Refer to Chapter 2: Calibrating the TAM III Calorimeter for information on the **Heater pulse** functions.

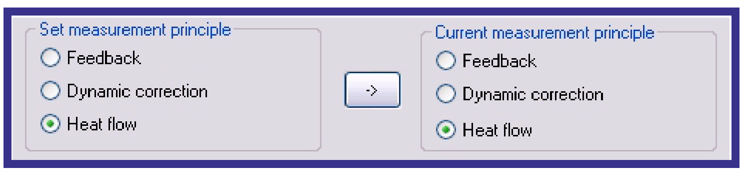


Activate Heat Sink Control

The Peltier regulator consists of a *thermopile* and a *feedback mechanism*. The thermopile thermally connects the heat sink of the 3201 Nanocalorimeter and the 3220 Microcalorimeter to the thermostat liquid. The thermopile senses whenever there is a measurable temperature difference between the surface of the heat sink and the liquid in the thermostat. For instance, the thermopile will sense a temperature change of the thermostat. The feedback mechanism then sets in. The thermopile then works as a Peltier element that "pumps" thermal energy to or from the heat sink to even out the temperature differences and reach thermal equilibrium between the thermostat and the calorimeters faster. This function significantly speeds up the time needed for the calorimetric signal to stabilize after a temperature change.

When changing the temperature of TAM III, the button **Activate Heat Sink Control** should be pressed. The button name changes to **Deactivate Active Heat Sink Control**. Once the calorimeter has reached the set temperature, press the button again to deactivate.

Measurement Principles



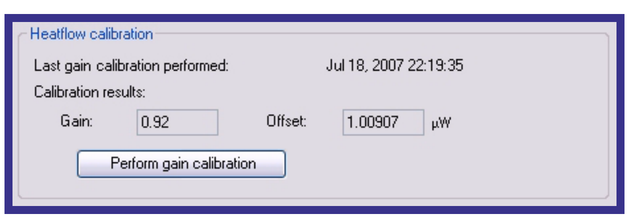
The 3201 Nanocalorimeter can be operated in accordance with three different measurement principles—**Heat flow** (most common), **Dynamic correction**, and **Feedback**.

Use the **Heat flow** mode whenever processes have slow conversion rates, or when it is the thermodynamics of the reaction that is of interest rather than the rate. If you are using the calorimeter as a titration calorimeter (or ITC, isothermal titration calorimetry), this mode should not be used.

In ITC experiments, you can operate the instrument in **Dynamic correction** mode to gain speed. Both the Dynamic correction and Feedback modes use a mathematical procedure to correct for the thermal inertia of the calorimeter. These modes, therefore, reflect the true heat production rate in the sample closer than the heat flow mode.

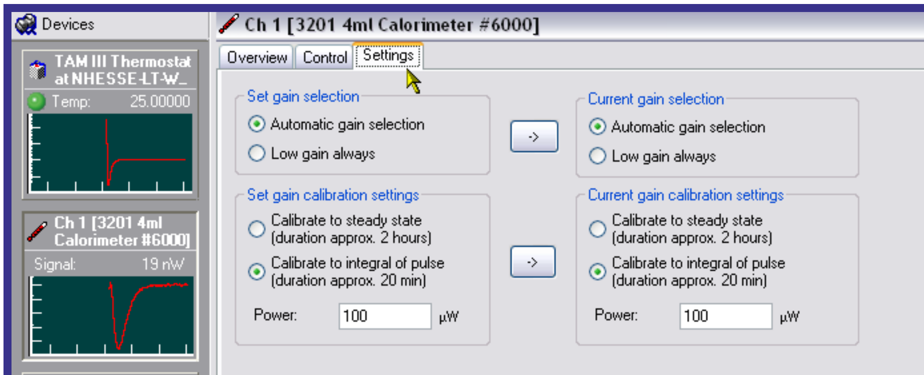
You will need to perform a dynamic calibration when using the Dynamic correction mode to get time constants for the instrument. This is not necessary for the Feedback mode, which uses counter power of the calibration heaters. All measuring modes may use the same calibration, or gain factors. See below on how to calibrate the heat flow.

Heatflow Calibration



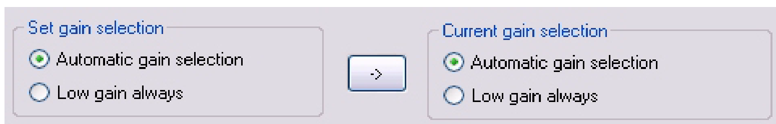
Refer to Chapter 2: Calibrating the TAM III Calorimeter for information on the **Heatflow calibration** and **Heater pulse** functions.

The Settings Tab



Settings Tab

Gain Selection



Gain selection has two options as seen in the figure below—**Automatic gain selection** and **Low gain always**.

Automatic gain selection is used to help regulate the heat flow values. When the heat flow value, in absolute terms, becomes larger than approximately 500 µW (or 480-580 µW depending on the calorimetric temperature), the software will automatically turn to lower gain (*i.e.*, lower resolution). When the absolute heat flow decreases and becomes lower than 500 µW, the software automatically turns to a higher gain. This option ensures that the highest resolution is given when measuring in the nanoWatt range. The resolution as given from the A/D converter is then approximately 0.2 nW.

When the software changes the Gain from high to low or vice versa, a minor disturbance in the heat flow signal can occur. In order to avoid this, you can select **Low gain always** so that the low-resolution mode is always on. However, this is not suitable for highly sensitive measurements, and should be used only when signals higher than 2 µW are of interest. For most applications with the 3201 Nanocalorimeter, **Automatic gain selection** is the preferred option.

Set Gain Calibration Settings

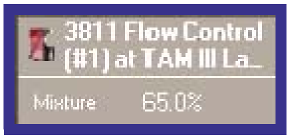
Refer to Chapter 2: Calibrating the TAM III Calorimeter for information on gain calibration.

The 3811 Flow Control Device

The Flow Control device controls the mass flow controllers, which are used for gases. The system can be configured for flow-through using one gas, or it can be used for flow-mix systems with which gases are mixed. In addition to the software module, the system consists of mass flow controllers connected to a specially-designed electronic interface.

In the case of flow-mix, the mixing can take place either outside the calorimeter or *in situ* in the measuring ampoule depending on the ampoule design. The controlled RH perfusion ampoule is a special case. This type of ampoule controls two mass flow controllers to set a mixture of a dry flow line, and a flow line saturated with a vapor, most commonly water.

Click the Flow Control Mini-view button to display a view that is used for various settings and controls. To set the value for the mixture and mass flow, enter the preferred value in the text box in **Set wanted mixture**.



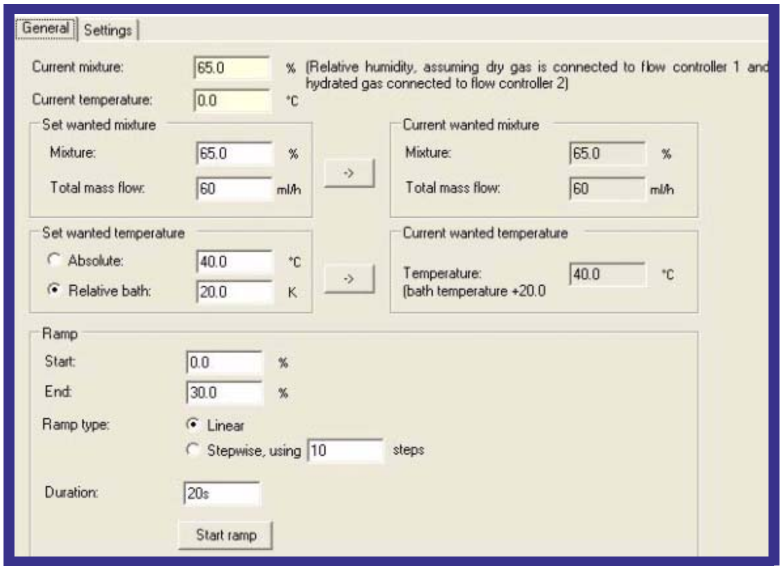
The total mass flow should not be higher than 200 mL h^{-1}.

Click the arrow to apply the new settings, it will be displayed in the frame **Current wanted mixture** on the right-hand side of the window.

Setting Up a Ramp

The most convenient way to control mass flow controllers is to do it within an experiment. Using this method, you can coordinate different events with respect to baseline and main sections.

You can use a Generic experiment or an Ampoule experiment, for example, to capture data while controlling devices directly from the Devices manager. Select the **General** tab as shown in the figure below.



Follow these steps to set up a ramp:

1. Use the text boxes in the **Ramp** frame to set up a ramp from the Device control. You can program both linear continuous ramps and stepwise ramps.

2. Enter the duration, in seconds, for the overall ramp in the text box **Duration**. This value is independent of the **Linear** or **Stepwise** ramp selection.

   Example: Set up a stepwise ramp consisting of 4 steps where each step is 5% and the initial value is 10%. Let's assume that the calorimeter signal needs 30 minutes to reach stability after each step change and it is stabilized before the ramp is initiated.

   (a) Enter 10.0 % in the text box Start and 30.0 % as the end value.

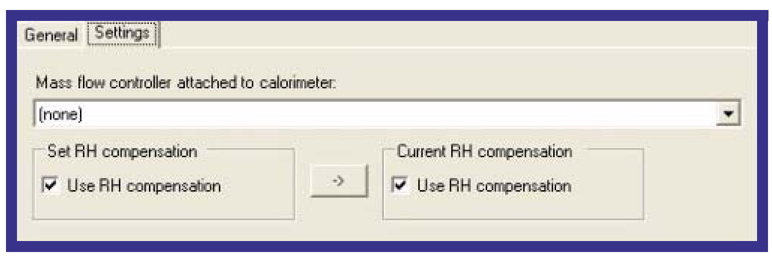
   (b) Enter 1440 seconds (240 min) in the text box Duration. This time will dispersed among the different mixture values as follows: 30 min (10%) - 60 min (15%) - 60 min (20%) - 60 min (25%) - 30 min (30%).

3. Click the **Start ramp** button to initiate the programmed ramp.

Controlling the RH Perfusion Ampoule

As already mentioned, a special case appears when using the RH perfusion ampoule. Since the mass flow of the wet flow line increases when the incoming dry gas picks up vapor from the humidifier cups, the mixture between the wet and the dry flow line doesn't correspond exactly to the relative flow rates as set by the mass flow controllers. The software compensates for this automatically, if the checkbox, **Use RH compensation,** has been chosen as shown in the figure below.

Choose the RH compensation when running the RH perfusion ampoule with water as the vapor.



Leave this option blank when running other types of flow mix experiments (*e.g.*, liquid mixing) . If you use vapors other than water, the compensation will be erroneous since that liquid has a vapor pressure different from that of water. In such cases, the correction must be made manually after the experiment.

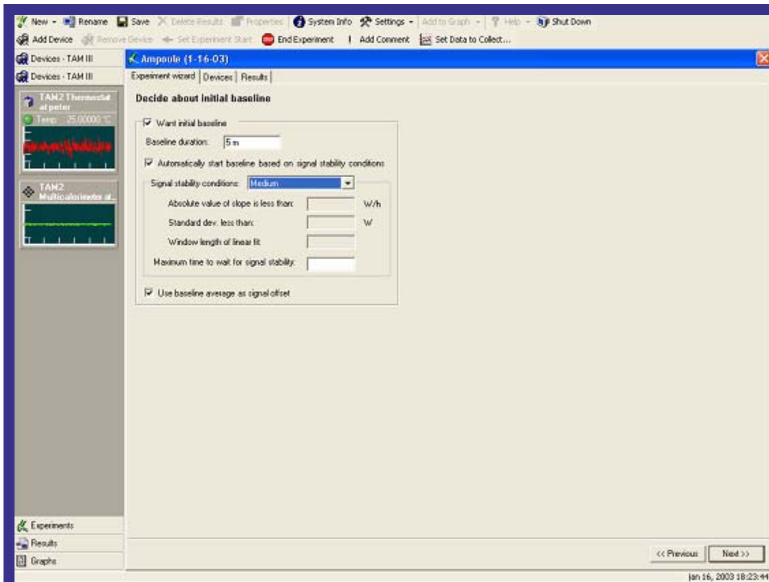
The setting in the frame, **Set wanted temperature,** applies only to RH perfusion experiments. This refers to an electrical heater mounted at the top of the RH perfusion ampoule. The function of this heater is to warm the outgoing wet gas to a temperature above the dew point to avoid condensation inside the ampoule. Set the temperature of the heater to the recommended value of 20°C above the TAM thermostat temperature. If you chose the option **Relative bath**, the heater temperature will automatically be set to the chosen value above the calorimetric temperature.

The procedure for setting up a ramp for an RH perfusion experiment is the same as described in the previous section.

The Experiment Explorer

TAM Assistant provides customized, step-by-step wizards that can be used to guide you through the process of running an experiment. Experimental wizards, specific to the type of experiment to be run, are available. For example, you can use a wizard to set up a static ampoule experiment in isothermal or non-isothermal mode.

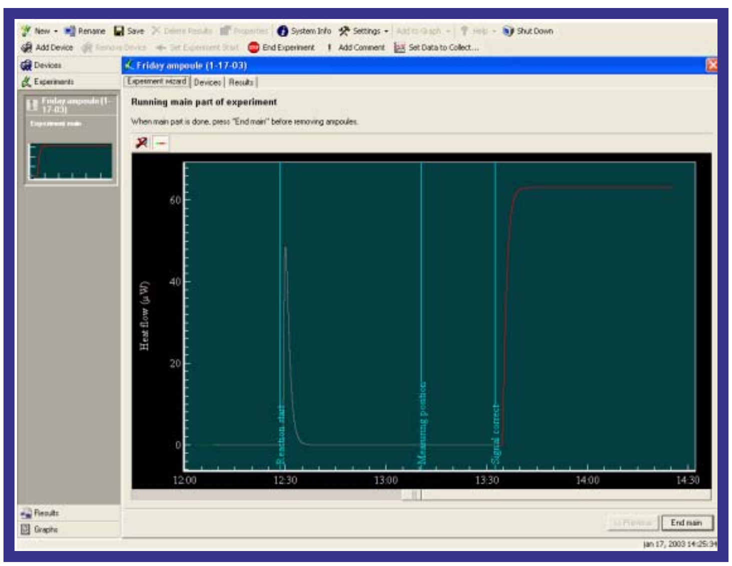
A wizard consists of a number of dialog boxes, instruction views, etc., which appear consecutively in the view area of the TAM Assistant main window, and prompt you to enter information about the experiment or perform specified tasks. The wizard divides the experiment into different parts of pauses, baselines, and main sections that are identified by the various analysis programs for performing automatic calculations or model fitting to data.



Experiment Wizard - Initial Baseline

The figure above is an example of a view appearing in the ampoule wizard where decisions about the initial baseline are entered.

When the experiment group is shown, the names of the experiments that are currently running on the TAM III system are displayed. Click a Mini-view button for a desired experiment to display its current status. The signal evolution can then be viewed



NOTE: Exothermic data is positive and curves in the upward direction.

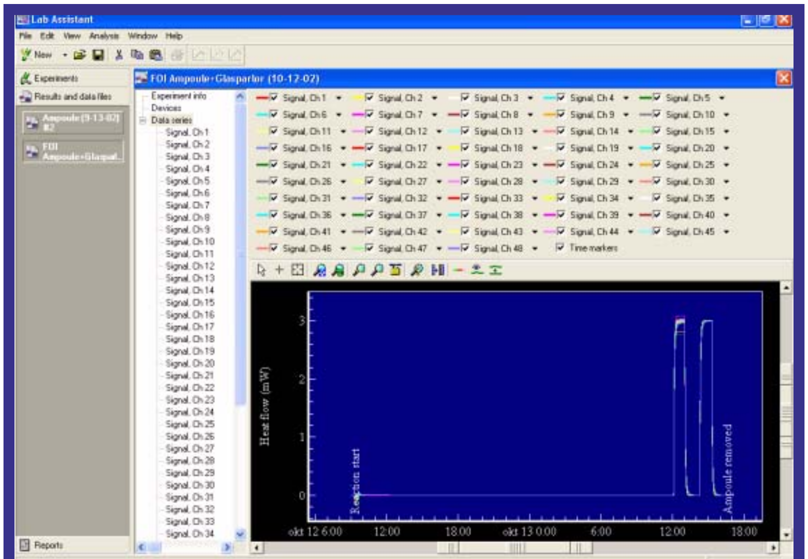
as a function of time. For a fast assessment of the current status for an ongoing experiment, the Mini- view buttons contain information such as signal evolution and current experiment section (initializing, main, baseline etc.). See the example on the previous page.

Detailed descriptions of the experimental wizards and how to run different types of experiments are given in Chapter 3.

The Results Explorer

When an experiment is finalized and saved, the results file will automatically appear under the **Results** group in TAM Assistant Embedded. The filenames of earlier finalized, saved, and open experiments are also listed.

Click on a filename to open the view for the chosen experiment in the view area. All information associated with the experiment, *e.g.*, Experiment information, devices, raw data etc., can be accessed from this view.



Results Group

This is a result file where the results from 48 channels have been collected and saved in a single file. You can show the result from all 48 channels in the same plot, or from a selected number of calorimeters by the check boxes above the plot window.

NOTE: Experiments saved on the TAM III embedded computer should be relocated to an external computer at regular intervals to prevent over-burdening the memory capacity on the instrument system. The procedure for moving or copying files to an external computer is described in the section called "Downloading Results Files," found in Chapter 3.

The Report Explorer

TAM Assistant contains a flexible environment for creation of laboratory reports. As mentioned previously, the report function is included in the external version of the TAM Assistant, but not TAM Assistant Embedded.

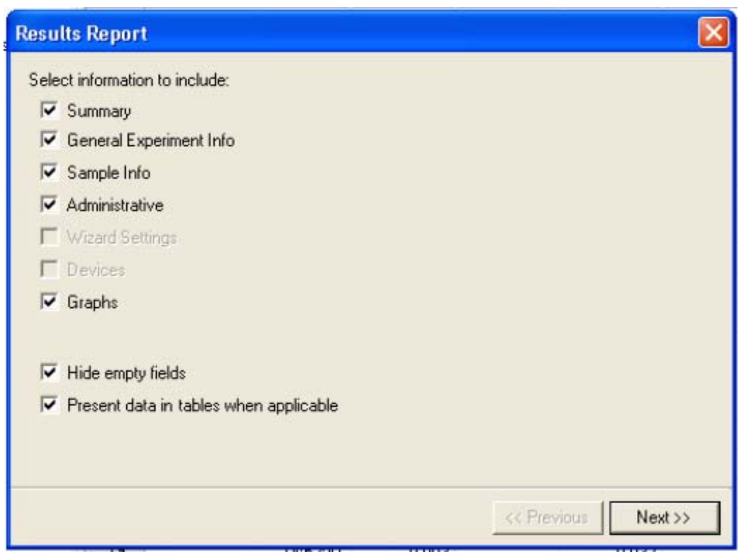
Reports can be created manually, or by use of templates associated with various analysis programs. You can also view data from different sources and perform interactive calculations and data fitting.

Creating a Results File Report

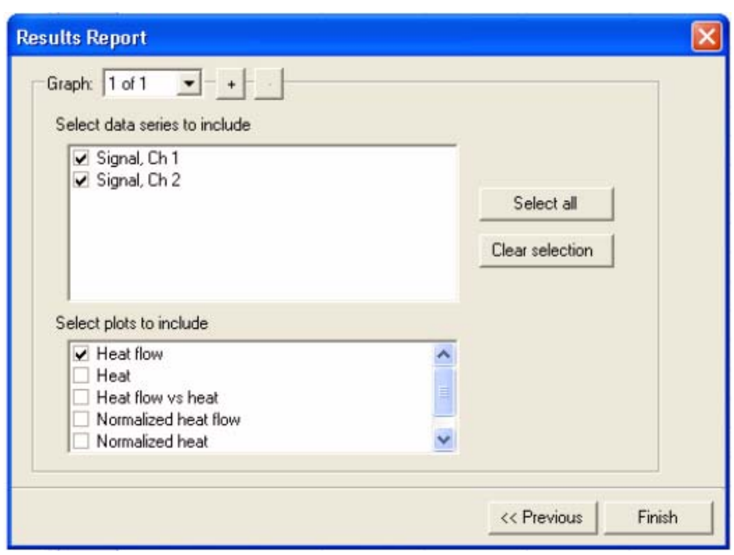
The report function can be accessed under the **Results file report...** in the **File** menu. In order to access the **Results file report** function the results file for which the report is to be written should be open and shown in the view area.

When chosen, a wizard opens that lets you determine which information to be included in the report, see the figure to the right.

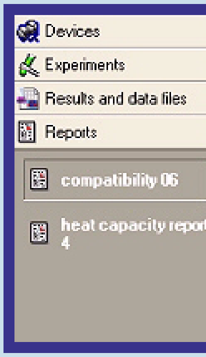
Choose the information and press **Next**.

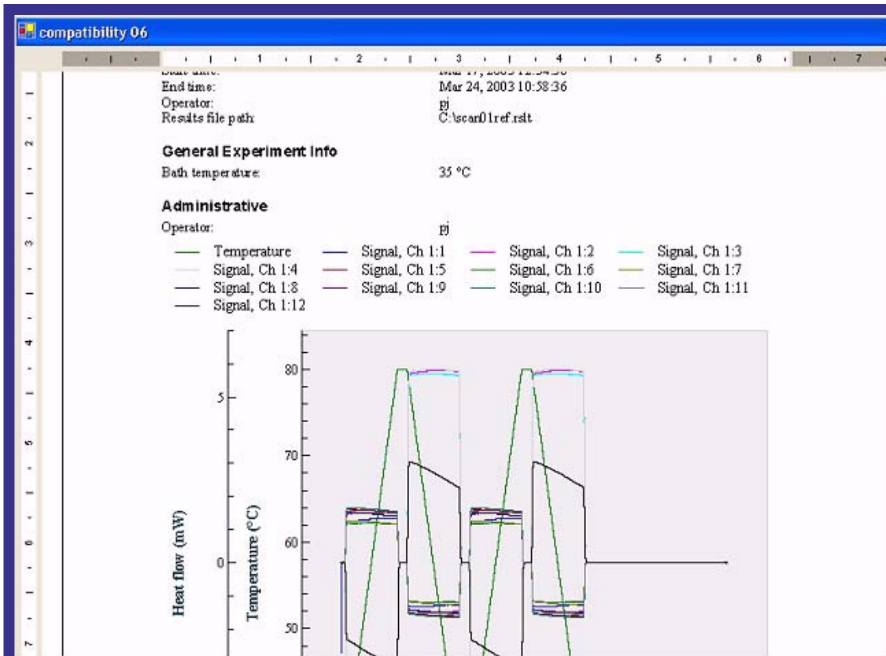


The next dialog lets you select the signals and the type of plots to be included in the report. Many different plots can be defined by pressing the plus sign by the **Graph** text box. See the figure to the right.



When the choices have been made press **Finish** to create the report. See the example below.

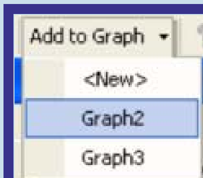




Example Report

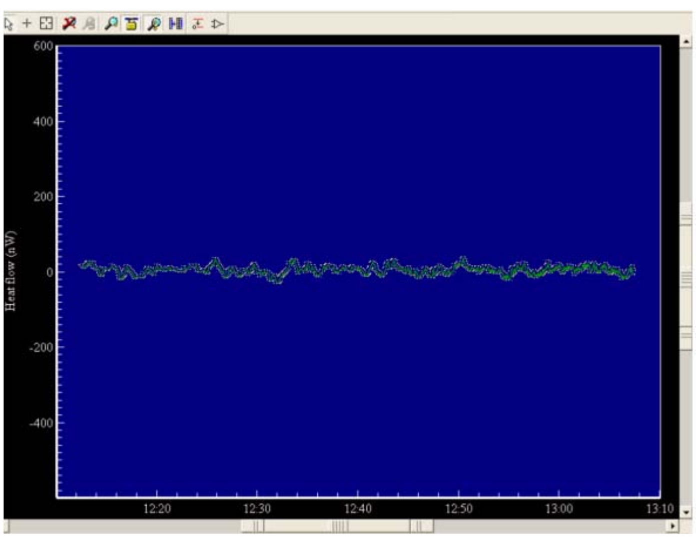
Once the report has been created it can be saved under a name and location of choice with the **Save** *report name* or **Save** *report name* **as...** under the **File** menu.

The Graph Group (TAM Assistant Embedded)



The **Graph** group exists only in TAM Assistant Embedded and can be regarded as a stripped down version of the Reports function in TAM Assistant.

Graphs from various plots in the Device group, Experiment group or the Results group can be copied to a separate plot placed in the Graph group. This may be done in order to compare plots from different experiments.

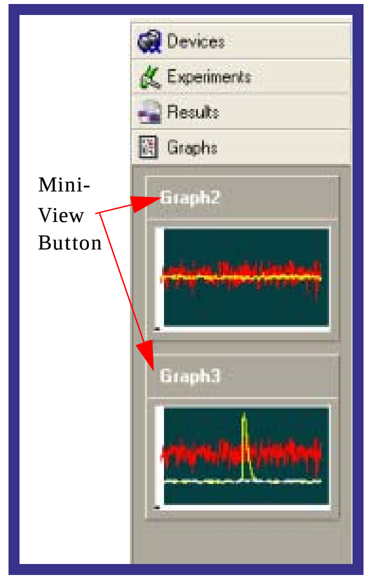


Use the **arrow** and click within the domain of the graph that is to be added to the plot. Broken lines are shown on both sides of the marked graph. Use the **Add to graph** menu to add the marked graph to an already existing plot or to a new plot. You must select the down arrow in order for the option to be active.

The added graph is online when chosen from a Devices group or the Experiment group. This means that the signal evolution, as a function of time, is displayed in the plot of the Graph group.

However, if a graph from the Results group is added, the whole graph will be added to the plot with the time scale preserved.

For each new plot created, a Mini-view button appears under the Graphs group.



TAM Assistant in Network

TAM Assistant Embedded on the TAM III system is mainly used for running experiments and visualization of signals and experimental data. TAM Assistant installed on a desktop computer can, in principle, be used for all of the same operations as the TAM Assistant Embedded software. In addition, TAM Assistant also contains powerful packages for data analysis, data export, creation of reports, etc. For this reason, results data has to be transferred from the TAM III system to an external computer loaded with TAM Assistant.

Therefore, the external computer must be able to communicate with the TAM III system in order to download data. This communication is normally made through a local area network (LAN).

An external computer can also be connected directly to the TAM III system by use of an external network card and a cross-over cable. It should be noted that the communication status of the TAM III system in a local area network is comparable to that of a printer, *i.e.*, it will not negatively affect any other components on the network or beyond.

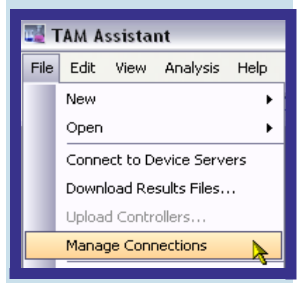
Communication between client computers on the network and the servers on the TAM III device is made with dynamic IP addresses generated with DHPC.

Connecting TAM Assistant to a TAM III System

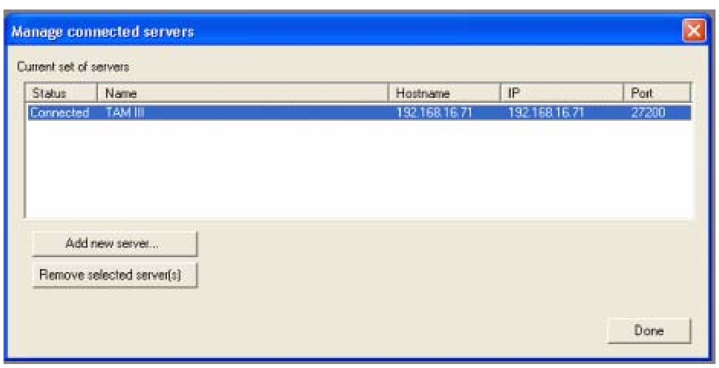
The following is only relevant for the external version of TAM Assistant.

To add (or remove) TAM III systems to TAM Assistant, follow these directions:

1. Select the **File** menu and choose **Manage Connections.**

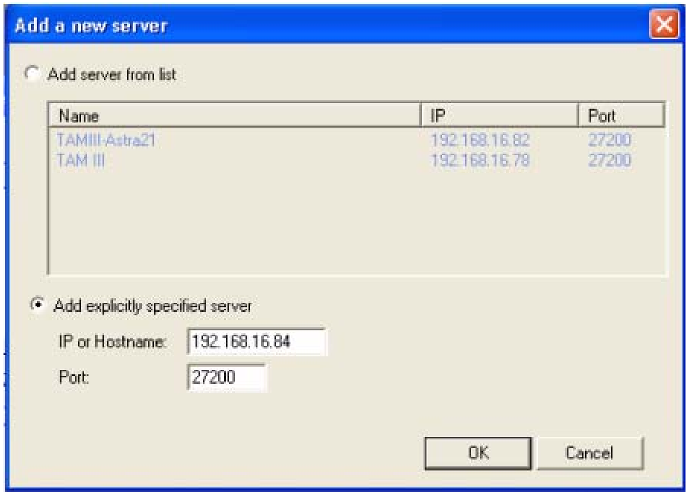


2. Use the dialog shown in the figure to the right to add or remove a connected instrument system along with its devices.



3. **Removing a system:**
   Select the name of the system that is to be removed and click **Remove selected server(s)**. The header corresponding to the selected TAM III system disappears from the Devices group. The removed instrument system can be added again at any time.

4. **Adding a system**: Click **Add new server...** The dialog shown in the figure to the right is displayed. TAM Assistant seeks out TAM III systems that are connected to the Local Area Network and lists the identified instruments. Select the desired system from the list displayed. If the system that is to be added is not included in the list, click the radio button labeled **Add explicitly specified server** and enter the IP number. When the information has been entered, click OK.



5. Click **Done** on the **Manage connected servers** dialog to return to the basic user interface. Within a few seconds the added devices can be seen under the Device group.

Chapter 2:

Calibrating the TAM III Calorimeter

Background

Calibration helps to assure that the calorimetric signal, as delivered by the software, corresponds to the heat flow generated by an arbitrary process. The calibration process closely mimics any measured process. This is achieved by electrical heaters mounted in the sample holder in close proximity to the sample ampoule.

NOTE: For detailed information on running a TAM III experiment, refer to Chapter 3.

In the TAM III System, the calibration procedures are included in the TAM Assistant software.

The primary signal, as generated by the heat flow sensors (thermopiles), is an electromotive force directly proportional to a temperature gradient over the sensor. The gradient is a direct consequence of the heat flow generated by an arbitrary non-inert sample in the calorimeter. The magnitude of the electromotive force is directly proportional to the size of the heat flow.

The signals, as generated on the sample and reference side of the calorimeter, enter an amplifier where they are differentiated and amplified approximately 100 times. An RC filter reduces the noise generated by the amplifier. The bandwidth of the filter is 30 seconds, which is approximately 10 times lower than the time constant of a calorimeter.

After the amplification step the filtered analog signal is converted to a digital signal by a 24-bit A/D converter. The dynamic range of the calorimetric signal is -20 to 20 mV. This results in a resolution of 7 nW bit^{-1} (the sensitivity of the heat flow modules are approximately 3 W V^{-1}).

The digital signal is received by a software filter function with one data point per 1/10 second. The window size of the filter function is one second, meaning that filtered data is collected with one data point per second.

The next step in data processing is the conversion of the signal from the A/D converter digits to heat flow values. The first step is the multiplication with a temperature-dependent calibration function, determined at four different temperatures. This procedure is done before you receive your instrument. A certified service technician should make subsequent temperature-dependent calibrations. The main reason for the temperature dependence is that the “Seebeck coefficient of the heat flow” sensors change slightly with changing temperature.

Relative deviation from the temperature-dependent calibration constant (which is, unity, by definition) is called the *Gain factor*. This is a factor that you will determine through the TAM Assistant software. Procedures for the determination of the gain factor are described in detail in this chapter.

The Gain Factor

The calculated calibration constant is called a *Gain factor*, as described in the previous section. This factor is independent of temperature to within TA Instruments specifications. The gain constant is always calculated relative to a temperature-dependent calibration factor. After a temperature-dependent calibration has been performed, the gain factor in TAM Assistant is automatically set to one. A subsequent gain calibration reveals any deviation of the recorded signal relative to the expected signal. The result is expressed the fractional deviation from 1. For example, a gain factor of 0.992 indicates a deviation of -0.8% from the signal obtained by the temperature-dependent factor alone.

Calibration with TAM Assistant

Calibration Procedures

Electrical calibration with the TAM Assistant can be made directly from the device interface under the **Devices** group, or while collecting data with the **Generic** experiment type.

The procedures are very similar, and the calibration experiment consists of a 10-minute baseline recording followed by a heat flow release with the internal heaters of one hour. After the power has been switched off, the calorimeter is given one hour to return to a steady baseline. A final five-minute baseline is recorded.

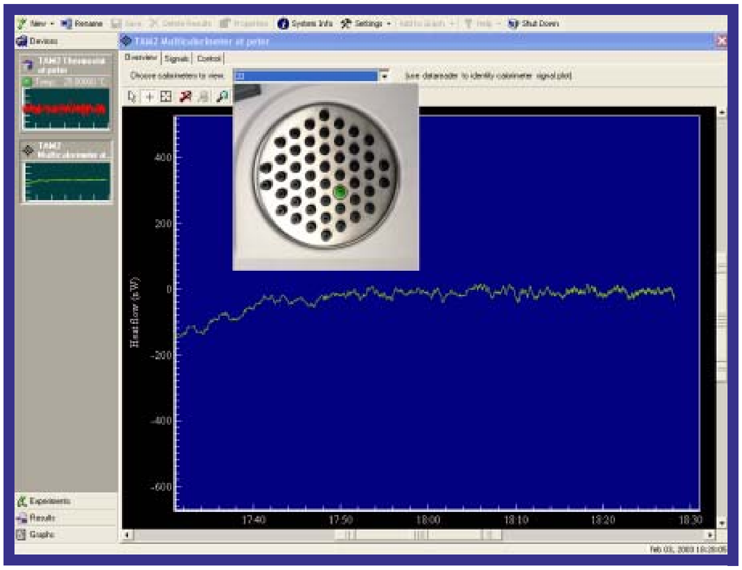
Calibration Directly from the Device Control

Follow these steps to calibrate from the Device Control:

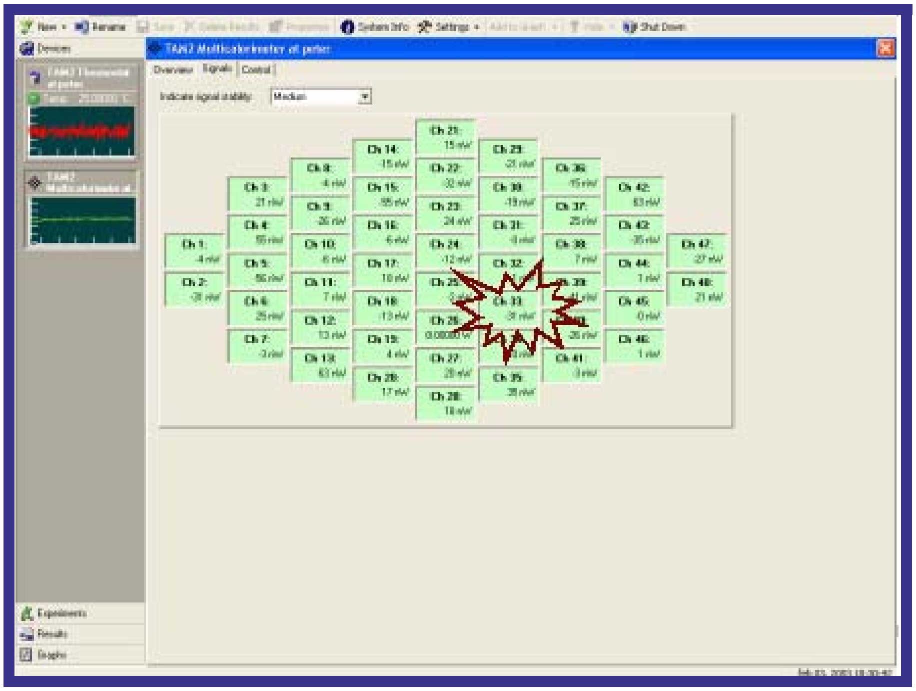
1. Click on the calorimeter miniview button in the **Devices** group mode.

2. Choose the channels for calibration from the multicalorimeter representation under the drop-down. One or more channels can be chosen for simultaneous calibration.

   In the example to the right, Channel 33 from the TAM 48 was chosen.



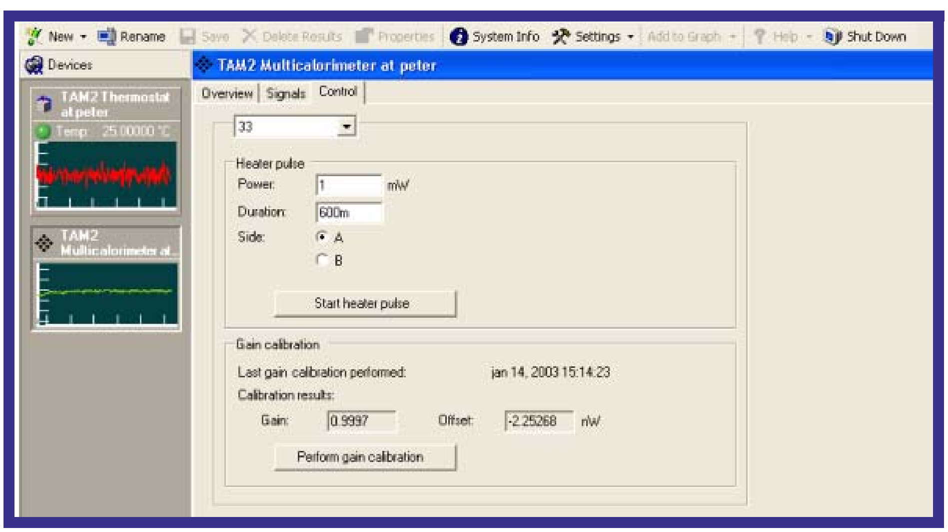
3. Press the **Signals** tab to show the current signals from the calorimeters as shown in the figure below.



4. Select the desired **Signal stability** condition from the drop-down list (moderate is the default).

   When the square representing the channel that is to be calibrated has the color green, the signal is stable in accordance with the chosen stability conditions.

5. Click the **Control** tab to access the information shown in the figure below.



6. Start the calibration by clicking the button labeled **Perform gain calibration**. The time taken for a calibration is approximately one hour for the 3206 calorimeters. A heat pulse of 3 mW is released by the inbuilt calibration heaters during a time period of 30 seconds giving a total heat of 90 mJ. The **Abort gain**

NOTE: Erroneous calibration values may result if the stability criteria are not met when starting the calibration.

NOTE: It is possible to abort the calibration at any time and the calibration values (i.e. gain and offset values) will not change from the previous calibration.

calibration text on the click button will change back to **Perform gain calibration** when the calibration is ready.

The new calibration constants will replace the old values when ready. It is not possible to change these values manually.

The calculated calibration values will remain until the next calibration is performed.

Calculation of the Calibration Constants

TAM Assistant automatically calculates the calibration parameters when the calibration recording is ready. The two parameters calculated are the Gain factor, described above, and an Offset value describing the deviation of the steady state signal from the instrument zero level, *i.e.*, no thermal activity in the calorimeters.

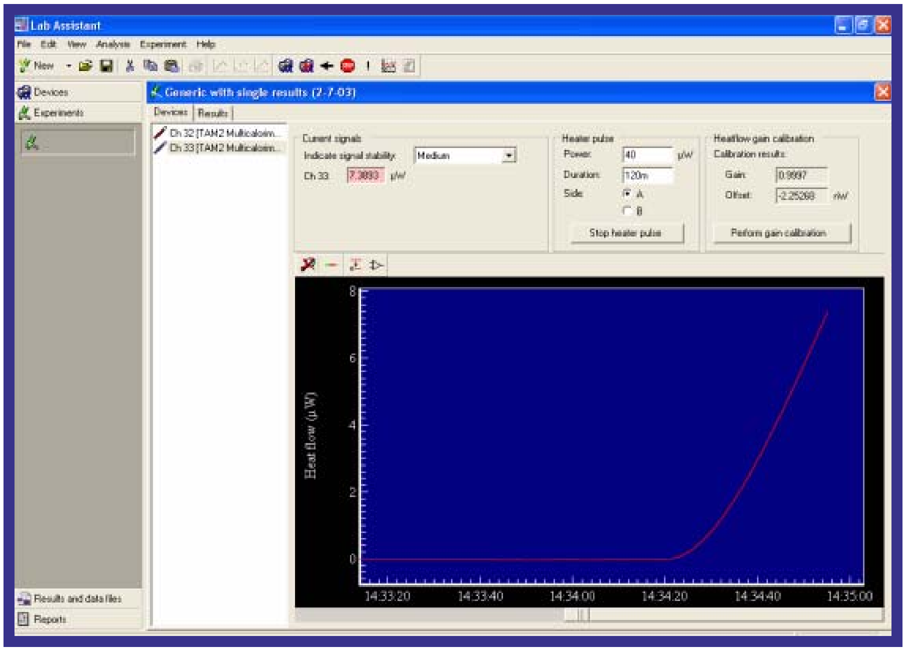
Calibration in a Generic Experiment

To save the data for the calibration in a results file, make sure that you perform the calibration while collecting data to a results file. The Generic experiment type allows you to do this. This section describes one of several different ways to perform a gain calibration.

1. Start a Generic experiment as described in Chapter 3, " Running Generic Experiments." Include all the calorimeters that are to be calibrated in the experiment.

2. Make sure that the signal is stable, according to the stability criterion, before starting the calibration. The signal is stable when the background color in the current signal text box is green.

3. Start the calibration on several calorimeters simultaneously by marking more than one in the device field to the left of the experiment view, see figure below.



Simply press down the Ctrl or Shift key, while clicking on the calorimeters to be calibrated.

4. When the stability criterion is met press the **Perform gain calibration** button in order to start the calibration.

   The text on the **Perform gain calibration** button changes to **Abort gain calibration** once the electrical calibration has been started.

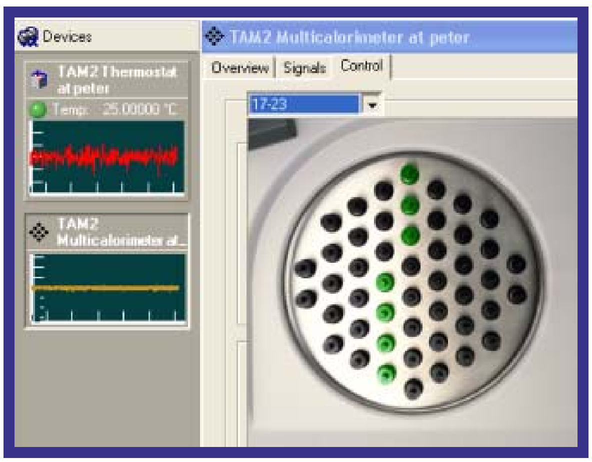
   When the calibration recording is ready, the software automatically calculates a new calibration constant and signal offset that automatically replaces the old values. The change of the calibration values is not unique for the Generic experiment. The change is global, meaning that the calibration values under the device control are also changed. These values will remain until the next calibration is performed.

   When the text on the calibration button has changed back to **Perform gain calibration,** the calibration is finished.

5. End the generic experiment.

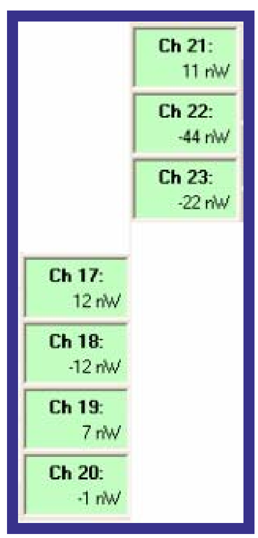
Manual Calibration

You can also use an alternative method to perform calibration with more flexibility regarding the heat flow. In this approach the calibration heaters deliver a thermal power of choice.



Follow these steps to manually calibrate:

1. Access the **Control** tab for the multicalorimeter device under the **Devices** group.

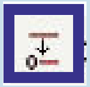


2. Mark the calorimeters to calibrate. Calorimeters 17-23 have been marked in the figure to the right.

3. Wait until the stability criterion has been met. When the images representing each chosen calorimeters have turned green, as shown in the figure to the right, the stability is reached.

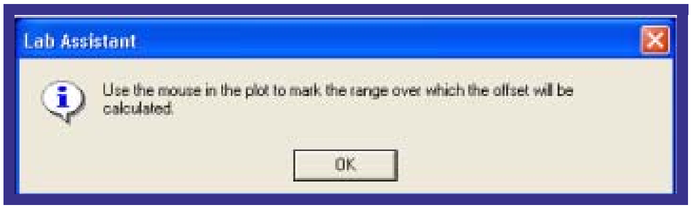
4. Press the **Overview** tab to access the plot window.

   The first step in the calibration procedure is to determine the zero offset of each calorimeter. This is the signal deviation from zero when there is no thermal activity in the calorimeters.



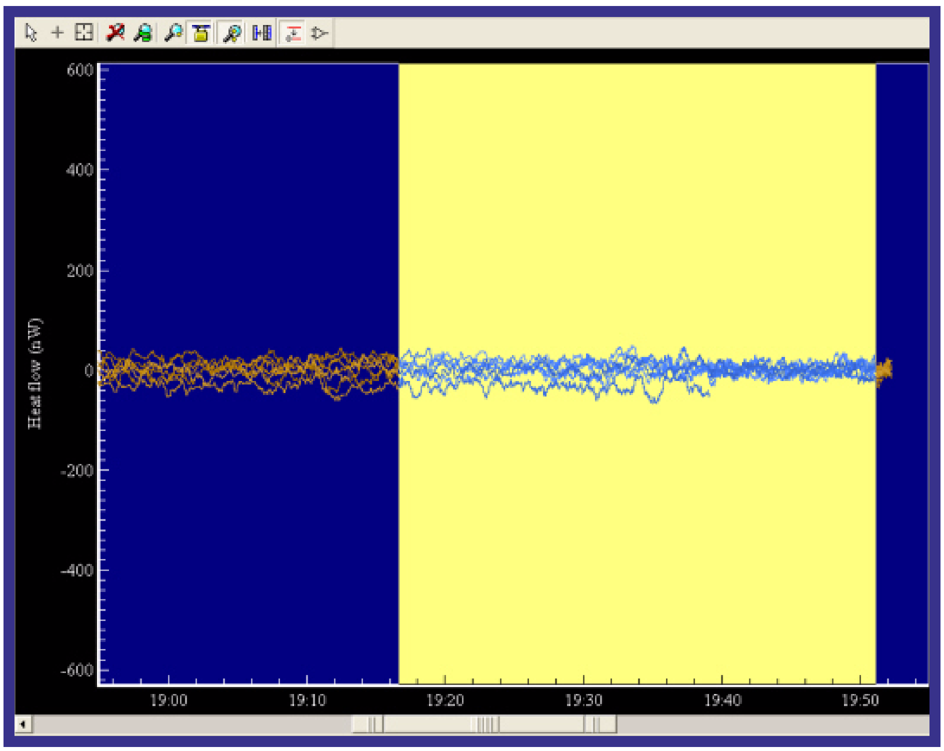
Set Offset Button

5. Press the **Set Offset** button in the toolbar of the plot window. The dialog shown in the figure to the right appears.



6. Press **OK**.

7. Mark an interval of the stable baseline by clicking and dragging in the plot. A 20- to 30-minute interval on the stable graph (or graphs) is sufficient for the software to calculate an offset that is representative for the real case. See the figure below for an example.



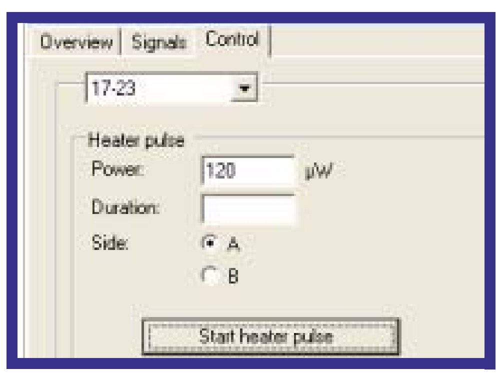
After the section has been marked, the offset from the zero level is calculated and the curve is shifted to zero.

8. Turn on the calibration heaters as follows:

   a. Select the **Control** tab.

   b. Make sure that the channels to be calibrated are chosen.

   c. Enter a thermal power of choice in the text box labeled **Power**.



d. Leave the **Duration** text box empty. The heaters will run indefinitely and must be turned off manually when the calibration is finished.

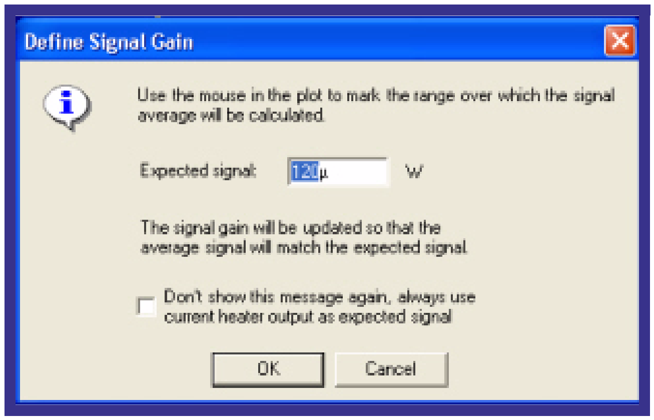
e. Choose **Side A** (default) and press the **Start heater pulse** button. It is now seen in the plot (Overview tab) that the signals are increasing towards a steady state value close to the entered power value.

f. Wait approximately 1.5 hours to reach a true steady state.

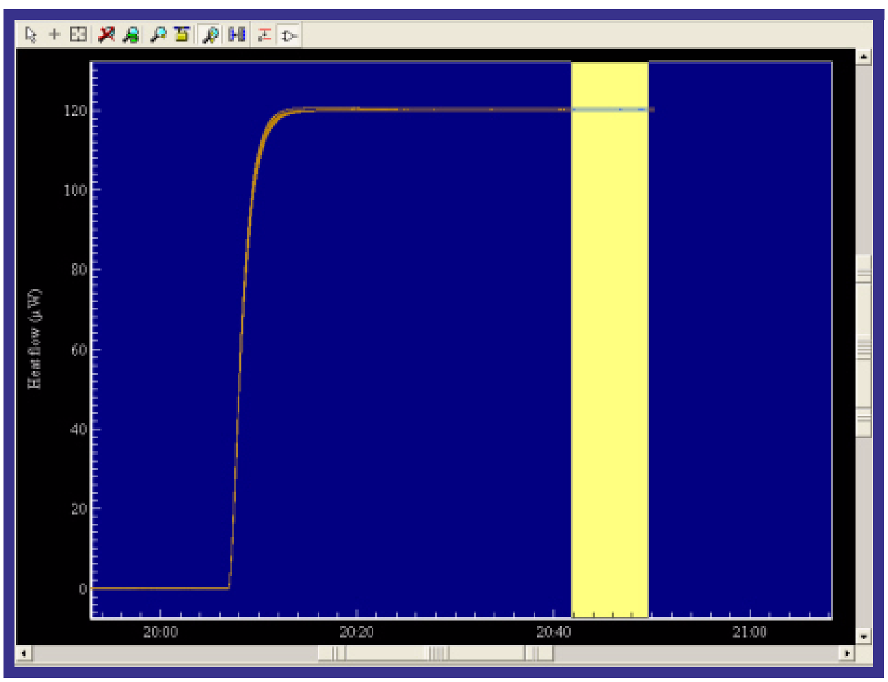
9. When the steady state has been reached, and there are 10 to 20 minutes with a constant signal, press the **Define signal gain** button in the plot toolbar.

Define Signal Gain Button

The dialog shown in the figure to the right appears, unless the check box labeled **Don't show this message again, always ...** has been marked previously. The text box labeled **Expected signal** has already been filled in with the current power value of the heaters.

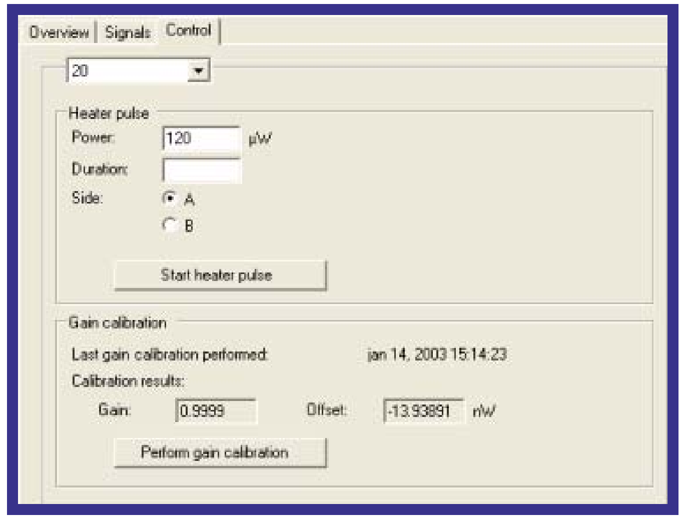


10. Press the **OK** button and mark a region of 10-20 minutes of the graph were the signal is at steady state. See the image below for an example.



After the section has been marked, the offset from the true power value is calculated, and the curves are shifted to the true power value.

11. Press the button currently labeled **Stop heater pulse** under the Control tab to turn of the heaters. If only one of the calibrated calorimeters is chosen at a time, the Calibration results can be seen in the **Gain calibration** frame.



NOTE: The date for the **Last gain calibration** doesn't change when performing a manual calibration. The date from the last automatic gain calibration remains.

In the procedure described for the manual calibration, the heat flow data has not been stored in a results file. In order to collect data from a manual calibration, a generic experiment should be started before the manual calibration starts. See the section in this chapter called “Calibration in a Generic Experiment.”

Dynamic Calibration

The heat flow produced by a sample or disturbance will be measured across the heat detector. However, due to the heat capacity and heat exchange coefficient the heat flow monitored by the heat detector may be different from the rate of heat production from the sample. For slow processes this effect is insignificant, but for rapid processes this effect must be considered. From knowledge of the time constant of the calorimeter it is possible to apply a correction procedure termed the Tian correction (below).

$$P_C = P_R + (\tau_1 + \tau_2)\frac{dP_R}{dt} + \tau_1\tau_2\frac{d^2P_R}{dt^2}$$

In this equation, P_R is the raw data signal monitored by the heat detector and P_C is corrected data, which closely represents the true rate of heat production by the sample. The time constant (τ) of the calorimeter can be calculated by using the calibration heater to apply known calibration powers in two consecutive steps. The response on the heat flow will exhibit exponential curvature. An error function, representing the sum of the squares of the difference between the estimated P_c and the actual calibration power, is calculated and a minimization routine is utilized to reduce the error function and calculate the values of τ_1 and τ_2. TAM Assistant uses two time constants, rather than one, to get a better precision in the correction (*cf*. Taylor expansion). In this case the fitting parameters has no relevant physical interpretation

Performing a Dynamic Calibration

For fast reaction, *i.e.*, in titration experiments, it is necessary to take into account the thermal inertia of the calorimeter, ampoule and sample. This is done by running the experiment in a dynamically corrected mode.

The Nanocalorimeter and the 20-mL Microcalorimeter can be run in a dynamically corrected mode, and to do this a dynamic calibration should be performed.

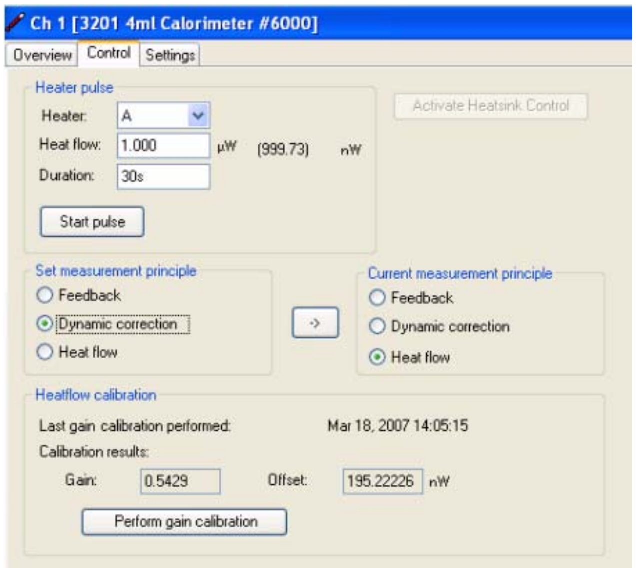
To perform a dynamic calibration, load the calorimeter with the ampoule (*i.e.*, titration ampoule) in measuring position and with the sample loaded. The reason for this is that the thermal inertia, τ, depends on the heat capacity and heat transfer from the sample to the surroundings.

1. Lower the sample and reference ampoules into the calorimeter.

2. Wait for a stable baseline.

3. Click on the calorimeter device and on the **Control** tab.

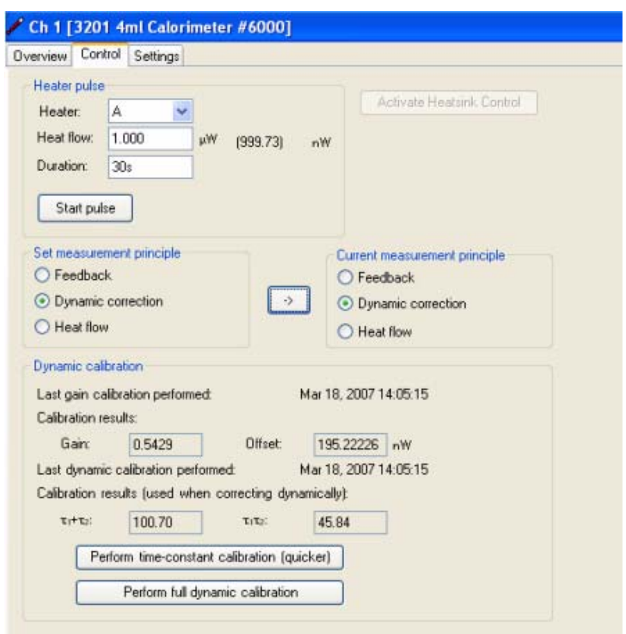
4. Click the **Dynamic correction** radio button in the **Set Measuring Principle** box. See the figure to the right.



5. Press the arrow to execute the command. The following dialog is displayed.

6. There are two options on the dynamic calibration:

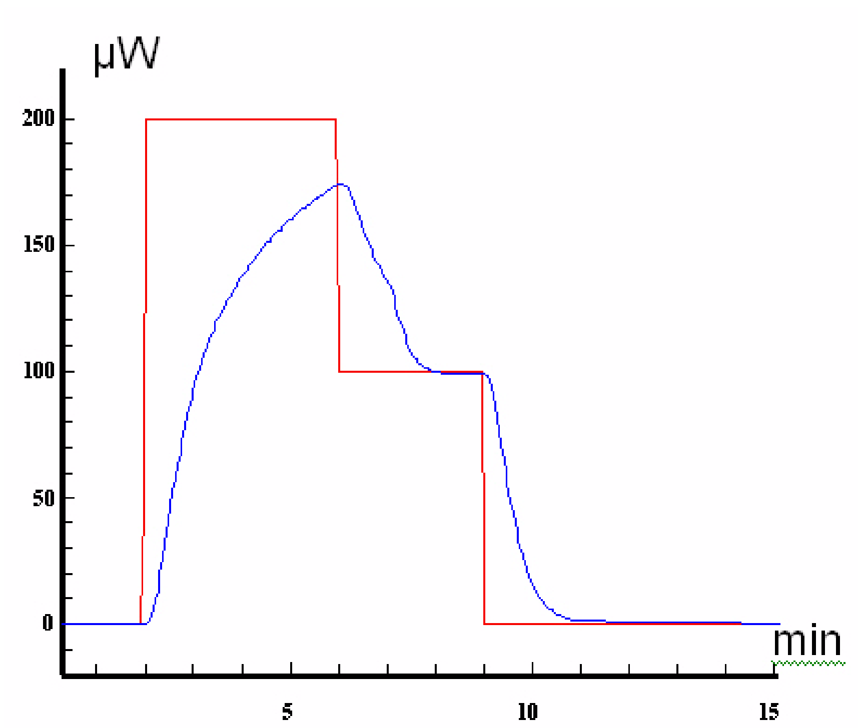
   a. **Perform time-constant calibration (quicker)**: With this option only the time constants of the system is calculated. This option can be used if a full dynamic calibration is previously performed with the same experimental setup.



This would be done if the sample volume is changed, solvent is changed or the stirring rate is significantly changed.

b. **Perform full dynamic calibration**:
With this option time constants as well as Gain and Offset is calculated. This option should when a new ampoule type is used.

7. Press the button of the preferred calibration option. [The button will change to **Abort Calibration**while the calibration is in progress.] The calibration will be automatic and the signal will look like the blue curve below.



Red curve - Power to calibration heater
Blue curve - Measured heat flow

8. When the calibration is finished the button will change back to **Perform Calibration**. The values for the time constants as well as gain and offset is now displayed.

Chapter 3:

Running Experiments

Experimental Practices

This chapter deals with some practical aspects of experimental design and methods used to run texperiments. Balancing between the reference and sample sides of a calorimeter, especially the 3206 minicalorimeters, is an important step that must be performed before running experiments. The noise level of the calorimetric signal and thus the quality of the measurements may be affected if this is not performed.

Sample Ampoules and Reference Inserts

Each minicalorimeter is equipped with a reference ampoule holder similar in design to the ampoule holder on the sample side of the instrument. Each reference ampoule holder contains an inert Aluminum reference body, which is intended to balance the sample side relative to the reference side, with respect to the heat capacity. An important purpose of this design is to reduce the short-term noise and also the offset signal during a temperature scan. The long-term stability of the calorimetric signal is not affected by an unbalanced system.

The Reference Inserts of the 3206 Minicalorimeter

Each 3206 Minicalorimeter is equipped with a fixed reference insert. There are a number of reference inserts with different mass to create heat capacity balance for a number of different applications. The reference inserts consists of an aluminum plug screwed into the reference side of the calorimeter.

The Sample Ampoules

Selecting a reference insert depends on the type of sample ampoules that are to be used with the minicalorimeters, as well as the type and amount of sample.

The table below shows the heat capacities of the ampoules (empty) that are used for static measurements.

Sample Ampoules

Product No.	$C_{p,a}$ / J K^{-1}	Description
2509-51	2.34	5-mL Heat Seal Glass Ampoule
95.53.1015	4.32	3-mL Disposable Glass Ampoule
24.20.0400	5.84	4-mL Disposable Glass Ampoule
2502-40	11.9	4-mL Stainless Steel Ampoule Circlip Cap
2277-301	11.5	4-mL Stainless Steel Ampoule Threaded Cap
3320	10.8	4-mL Stainless Steel Ampoule Threaded Cap

Balancing the Minicalorimeters

NOTE: The heat capacities given include all details that constitute the ampoules, *i.e.*, caps, gaskets, eyelets, etc.

An ideal twin heat conduction calorimeter should not have any noise produced by the calorimeters other than that produced by the electronics. This is because the differential nature of the measurements should cancel out all thermal fluctuations from the environment. However, in our practical world it is not possible to create a perfectly balanced system.

The lowest heat capacity on the sample side is achieved with an empty ampoule holder. The percentage difference in heat capacity, for example, becomes -48.2%

for reference insert 1, without any ampoule in the holder. The minus sign implies that the mass should be increased on the side of the sample ampoule holder in order to improve the balance and thus the noise level.

For a given ampoule and reference insert the heat capacity balance can be improved by optimizing the amount of substance in the sample ampoule. The percentage difference in heat capacity, between the sample and reference sides of a minicalorimeter, Δ, can be calculated by the following formula:

$$\Delta = 100 \cdot \frac{C_{p,ri} - 0.24 - C_{p,a} - C_{p,c} \cdot m}{C_{p,ri} + 7.18}$$

Where:

$C_{p,ri}$ = Heat capacity of reference insert

$C_{p,a}$ = Heat capacity of ampoule

$C_{p,c}$ = Heat capacity of the compound used in the measurement

7.18 = Heat capacity of the ampoule holder on the reference side, reference insert excluded

0.24 = The diff[illegible]olders on the sample and referenc[illegible]

m = The ma[illegible] be optimized so that the absolute value of Δ approaches zero.

The maximum deviation specification for the signal is ±20%.

Values of Δ larger than 20% may be accepted in many cases as the quality of the measurements are dependent on the signal to noise ratio rather than the absolute value of the noise level. A large sample size may be preferred instead of a close balance if this results in a larger signal-to-noise ratio. It is important to note that the specifications regarding the short term noise applies only when the heat capacity difference is within the specified limits.

Common Heat Capacities

In the table a few compounds are listed with the heat capacity at ambient temperature.

Heat Capacity at Ambient Temperature

Compound	Cp /J K^{-1} g^{-1}
Liquids	

Compound	Cp / J K^{-1} g^{-1}
Water	4.18
Ethanol	2.43
Propanol	2.40
Benzen	1.73
Toluene	1.71
Pentane	2.33
Heptane	2.24
DMSO	1.93
Solids	
Inorganic	
NaCl	0.86
Quartz (SiO2)	0.76
Organic	
Lactose	1.22
Urea	1.55
Glucose	1.24
Salicylic acid	1.16
Common gun powders	1.28

An Example

For this example the following are the experimental paramters:

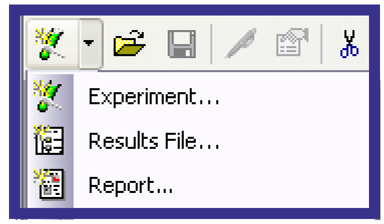
- Sample is a water solution
- Minicalorimeter is equipped with reference insert 1.
- Ampoule is the 3-mL glass vial
- To get a perfect balance ($\Delta = 0$), a mass of 0.61 g of solution is required as calculated by the equation on the previous page.

This would give the lowest absolute value of the noise level. However, it might not give the best signal to noise ratio. If more sample mass can be used (in this case 3 g), the quality of the measurements might improve. It is recommended that you take into consideration the positive and negative factors that are unique for each experimental setup when planning the experiment.

Running Experiments

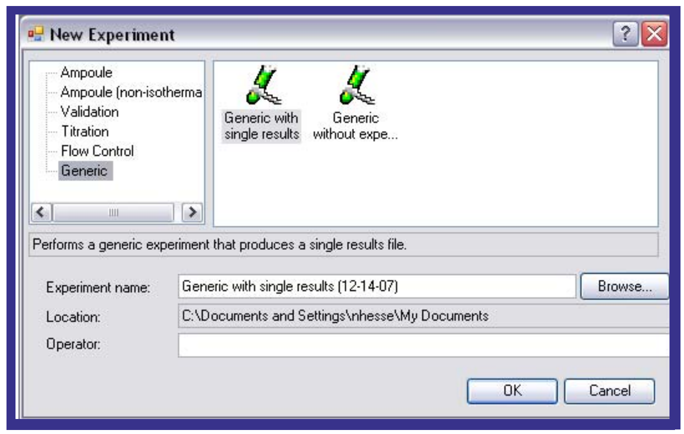
The remainder of this chapter describes how to run experiments with TAM Assistant, using the experimental wizards. Initiating, running, and finalizing experiments with TAM Assistant are described in detail. The images used throughout this chapter are from TAM Assistant Embedded. Although the user interface of the TAM Assistant is somewhat different than that of TAM Assistant Embedded, the procedures of designing and running experiments are identical in the two versions.

Starting a New Experiment



When starting a new experiment, independent of what type, select **Experiment** from the **New** menu. See the figure to the right

1. Choose the type of experiment from those shown in the **New Experiment** window (see the figure to the right).



   There are six basic types of experiments included in TAM Assistant: **Ampoule** and **Non-isothermal ampoule** experiments, **Validation**, **Titration** , **Flow control**, and **Generic** experiments.

   Generic experiments are the simplest type. In Generic experiment, data is collected without any division into pause, baseline and main sections. Actions such as a temperature jump or scan can be implemented in a generic experiment, but it has to be made manually under the device group. See the next section for more details on Generic experiments.

2. Enter **Experiment name.** The experiment name is the filename where the result and additional information is stored. A default name, stating the type of experiment and date will automatically appear as the experiment name. You can alter this name, if desired.

3. Enter the **Operator**'s name or initials.

4. Click **OK**.

Running Generic Experiments

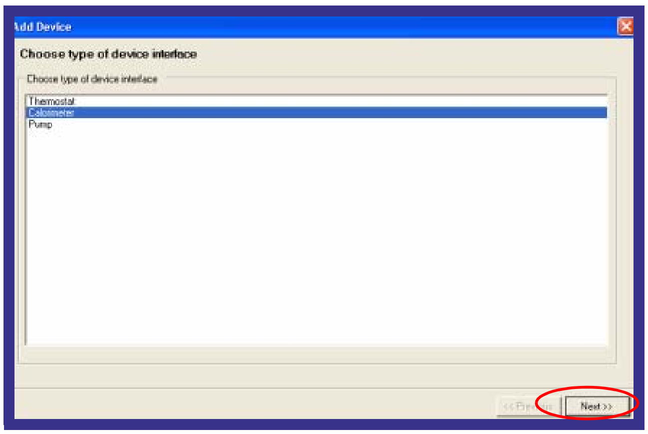
The *Generic experiment* is the simplest type of experiment run by the TAM Assistant. It may be considered as pure data collection and the collected data is not divided into baseline, pause, or main sections. This means that there are no possibilities to perform baseline corrections on saved data or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

Actions such as a temperature jumps or scanning can be performed in a generic experiment, but the necessary control actions has to be made manually from the thermostat device in the Devices group.

Setting Up a Generic Experiment

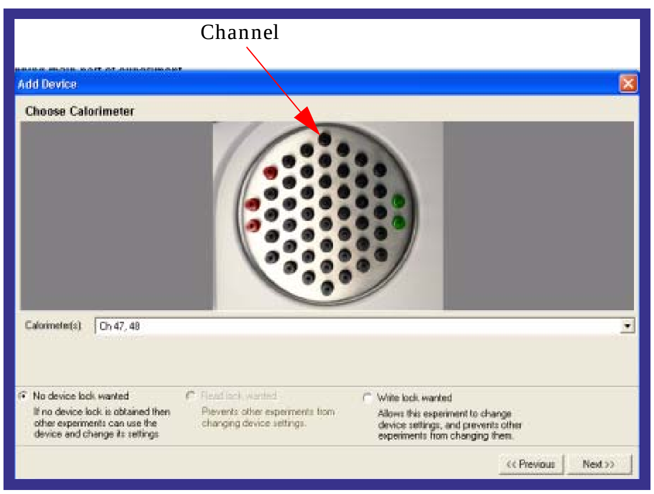
1. Choose the Generic type of experiment from those shown in the **New Experiment** dialog. The dialog shown in the figure to the right is displayed.

2. Select the device(s) to be included in the experiment. Press **Next**.



The following view appears, see the figure below.

3. Click on the calorimetric channels that you want to use in the experiment. To choose several channels, press and hold the Ctrl-key, while clicking the respective channel(s). (You can also click on one channel and drag the cursor over the remaining channels that you wish to select).



4. Choose from a set of security options at the bottom of the dialog above:

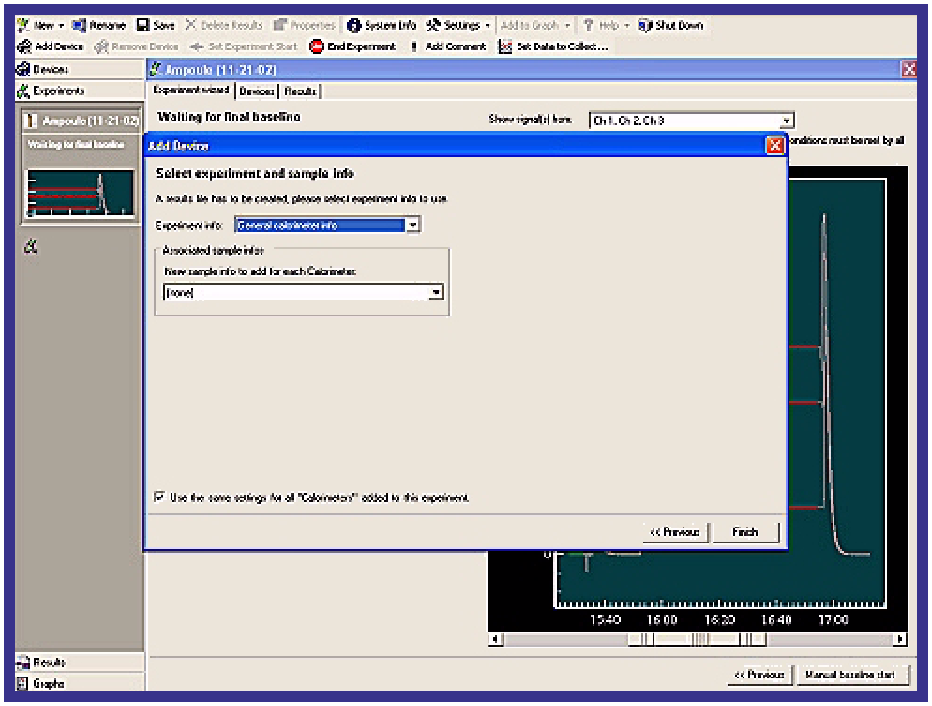
   - If you select the option **Write lock wanted**, the minicalorimeters will not be available for different kinds of control actions from other experiments, *i.e.*, others cannot write to the devices.

NOTE: If channels are occupied by another experiment (see the figure to the left), they will be displayed as red.

The color on the top of the channel turns green as it is chosen.

- If you choose the option **Read lock wanted,** it is not possible for any other experiment (and, therefore, other users) to collect data to another experiment.
- If you select **No device lock wanted**, full access to the chosen devices are possible from other experiments and users.

5. Click **Next**. The dialog shown in the figure below is displayed.



NOTE: The choices made on experimental information cannot be changed after the experiment.

NOTE: Experimental information can be entered while the experiment is running or after the experiment has been saved in an experimental file.

6. Select the type of experimental information to include in the results file using the two drop-down menus are provided.

   If **None** is chosen in both drop down menus, no sample information can be included in the experimental file.

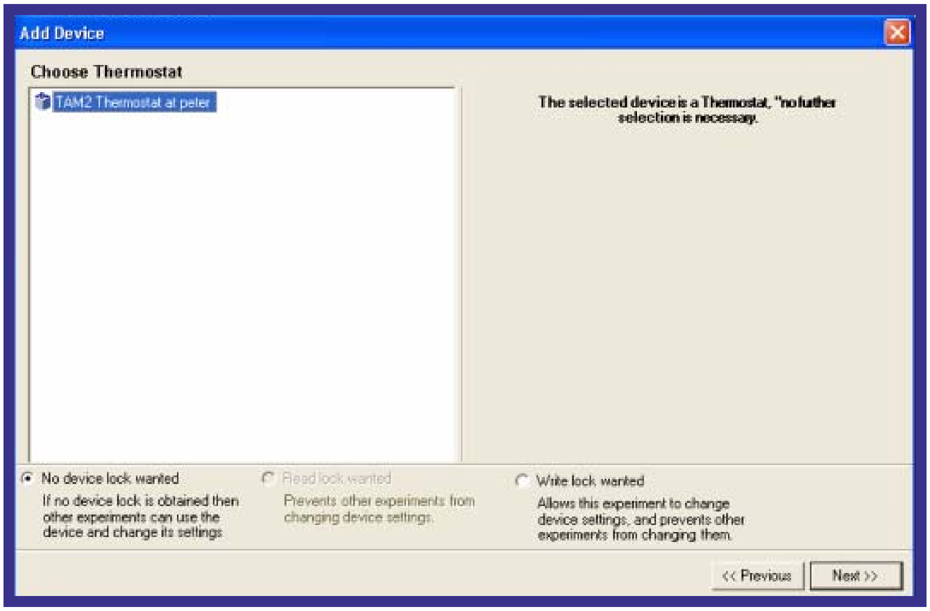
7. Click **Finish** to start the data collection. A miniview button (see the figure to the right) appears in the Experiment group. This can be used to check the progress of the experiment at any time.

Adding Additional Devices

At this stage you can add more devices than were selected before the data collection was initiated. For instance, the thermostat device or additional minicalorimeters can be added.

To add a new device follow these steps:

8. Click the **Add Device** button (shown to the right). The dialog shown on the next page appears.



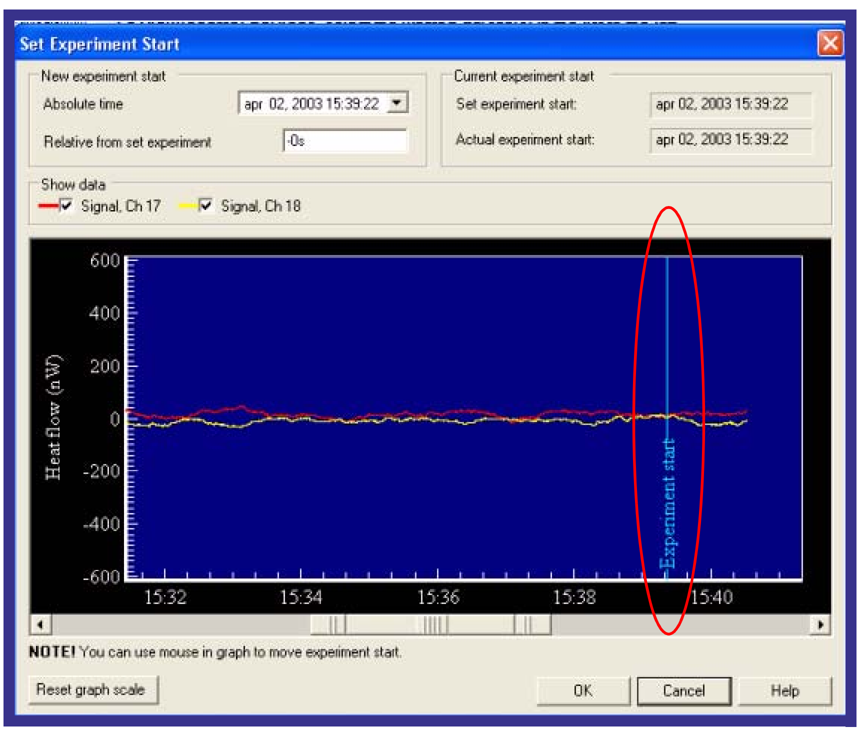
9. Select the device to be added, and press **Next**. A dialog is displayed to allow you to decide on experimental information, see the previous page for details.

10. Click **Finish** in order to start the data collection from the newly added device.

Setting the Experiment Start

After the collection of data has been initiated, you can set the time to begin data collection.

11. Click **Set Experiment Start**. The following dialog is displayed.

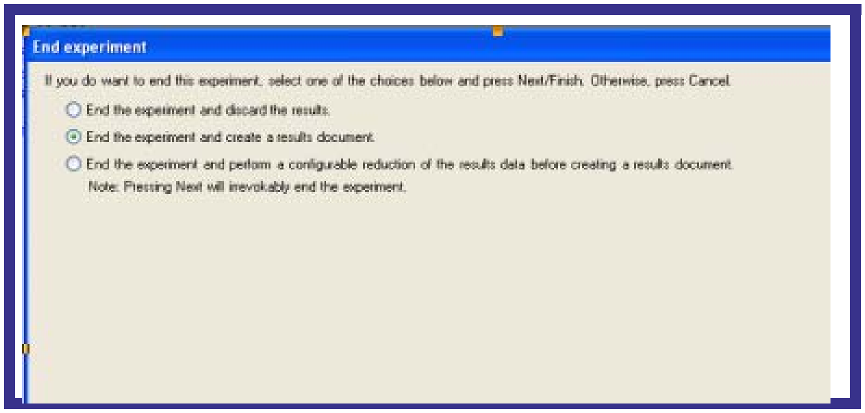


12. Move the cursor to the time marker labeled **Experiment start.** Drag the time marker to the start time of choice. This can be done back or forward in time.

Finalizing a Generic Experiment

You will need to manually finalize data collection in a Generic experiment in order to save the data in a results file.

13. Press the **End Experiment** button in order to finalize the data collection. A dialog is displayed to let you decide whether to end the experiment and, if so, discard the results or reduce the experimental data. How data reduction is performed is described in the Ampoule experiment section.



Ampoule Experiments

An *Ampoule experiment* is a static form of calorimetric experiment. In other words, there is no control of auxiliary devices such as the stirring motor, the mass flow controllers for perfusion, or the pumps for injection. The ampoules containing the sample are either metallic (stainless steel or hastelloy) or disposable glass vials.

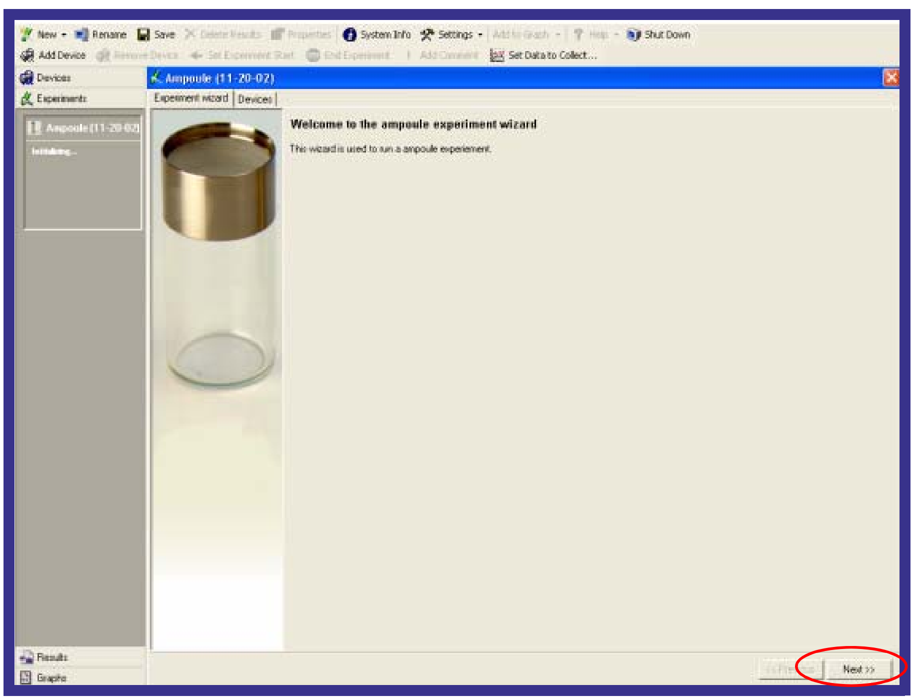
You can use the *Ampoule experiment wizard* to guide you through a number of steps where instructions on operational details are given. You will need to enter information about the experiment throughout the wizard.

Ampoule experiments can be run in isothermal or non-isothermal mode. An experimental wizard exists for each of them. In this section the ampoule wizard is described in detail. Additional features belonging to the non-isothermal ampoule wizard are also described, *e.g.*, how to define a temperature program etc.

The Ampoule Wizard

Follow these steps to run the Ampoule wizard:

1. Choose **Ampoule experiment** and click **OK**. The first dialog is displayed, together with an icon under the Experiment Manager. The icon shows the experimental name and the current status of the experiment.



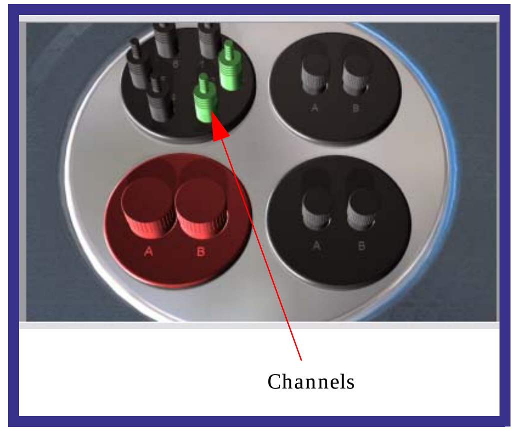
2. Click **Next** to continue.

NOTE: If channels are occupied by another experiment (see the figure to the right), they will be displayed as red.

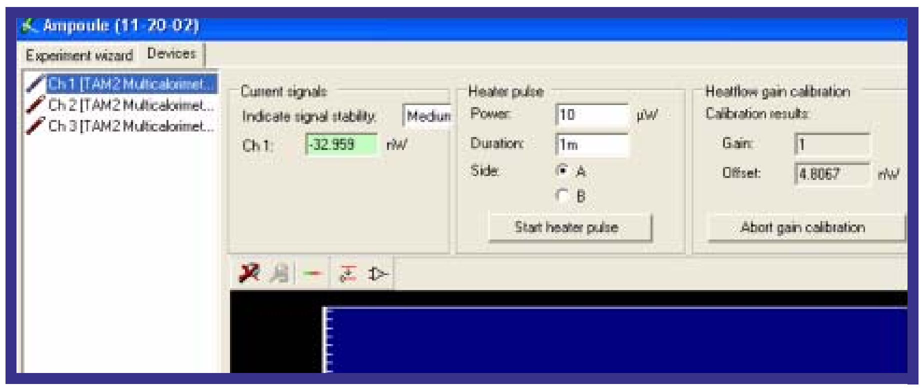
The color on the top of the channel turns green as it is chosen.

3. Choose the calorimeter channels to use in the experiment by clicking on the channel in the figure. To choose several channels, press and hold the Ctrl-key, while clicking the respective channel(s). (You can also click on one channel and drag the cursor over the remaining channels that you wish to select).

4. Click **Next**.



At this stage, the chosen channels are listed under the tab labeled **Devices** (see below). By marking a calorimeter device on the left, a view appears that lets you control the actions of the device and/or examine the status (*e.g.*, signal evolution). A plot displays the signal from the chosen device.



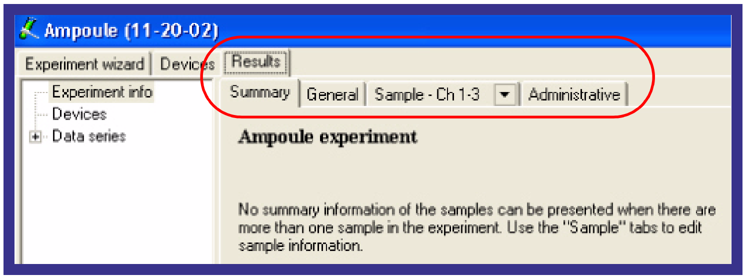
It is possible, at this stage, to add additional devices such as an additional minicalorimeter.

5. Click on the **Add Device** button at the top left of the main program window. See the figure to the left. This opens a window allowing you to choose additional device(s) to add.

6. Return to the Experimental wizard and click **Next**.

Ampoule Wizard Results Tab

At this point in the Ampoule wizard a new tab is displayed labeled **Results**. See the figure to the right.



7. Enter experiment information under the **Results** tab (sample size, concentration etc.).

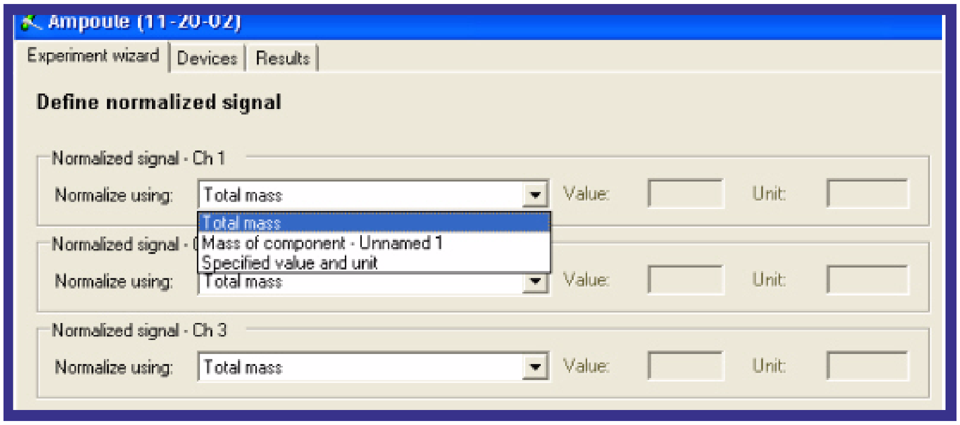
   a. Select the **Sample** tab to enter experimental information such as the name of substance, lot/batch number).

   b. Select the **Administrative** tab to enter information such as the project name, affiliation etc.

   c. Choose the **General** tab to view the Experiment Start, Stop and Temperature field, which will be entered automatically during and after the experiment. Any text can be entered in the field labeled Additional information.

8. Return to the Experiment wizard and click **Next**.

Normalization Criteria for the Signal



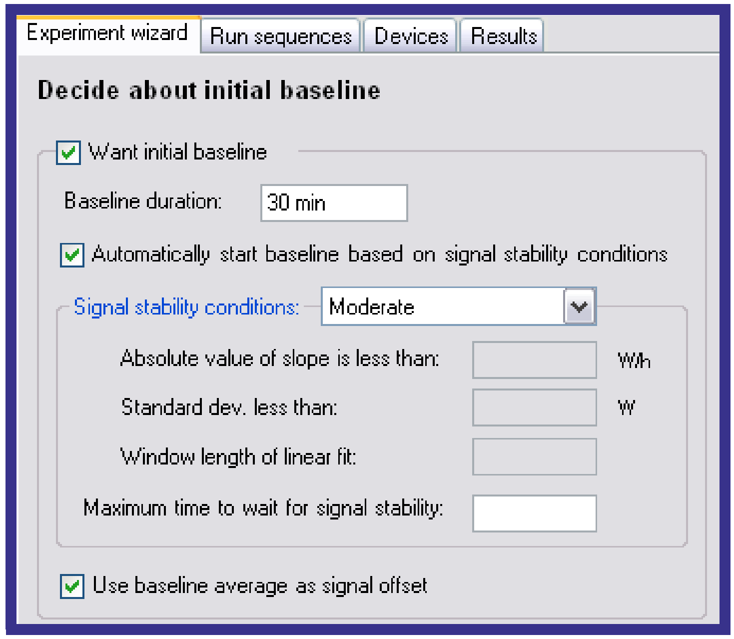
This window in the Experiment wizard lets you enter information on how the signal should be normalized.

9. Determine how to normalize the signal. Choose the desired options from the available drop-down lists. The information on sample size of the component(s) is optional.

10. Select the **Experimental wizard** tab then click **Next**.

Decide about Initial Baseline

The next window (shown in the figure below) lets you decide about initial baseline for the experiment. You can enter the time for recording the baseline. The default value, as given by the software, is 30 minutes.

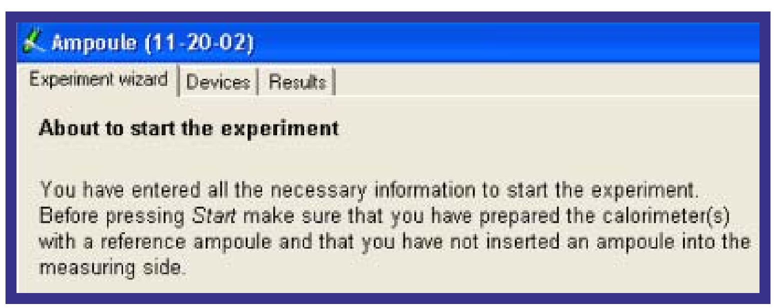


The baseline can be started manually, when desired, or automatically, when the signal is stable according to a specified baseline criterion. The default value for the stability criterion as entered by the software is **moderate** signal stability condition. These values will vary depending on calorimeter type.

11. Select the duration and stability conditions for the initial baseline. If the checkbox **Use baseline average as signal offset** is marked, TAM Assistant automatically uses the calculated mean value of the initial baseline to correct the signal in the main part of the experiment.

12. Click **Next** to continue.

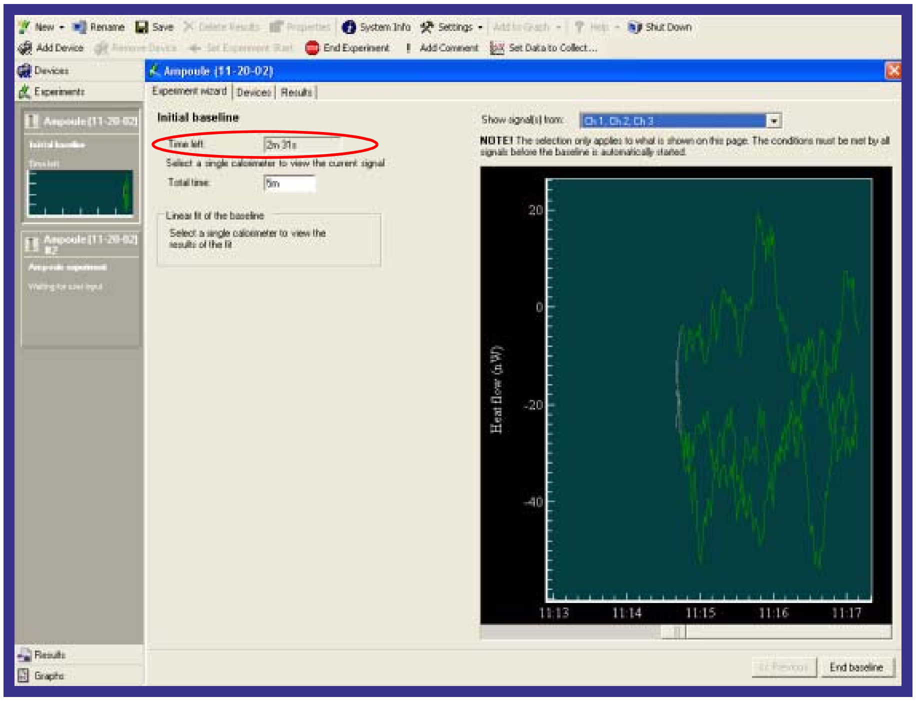
Usually the baselines are run without an ampoule in the measuring position in order to be sure that no offset due to any thermally activity is recorded.



When you are sure that the measuring position of the minicalorimeter is empty, you may start the experiment.

13. Click **Start**. The following window appears. The green curve indicates the baseline and the grey part of the curve indicate the pause section prior to the baseline. The baseline section starts as soon as the chosen **Signal stability condition** is met.

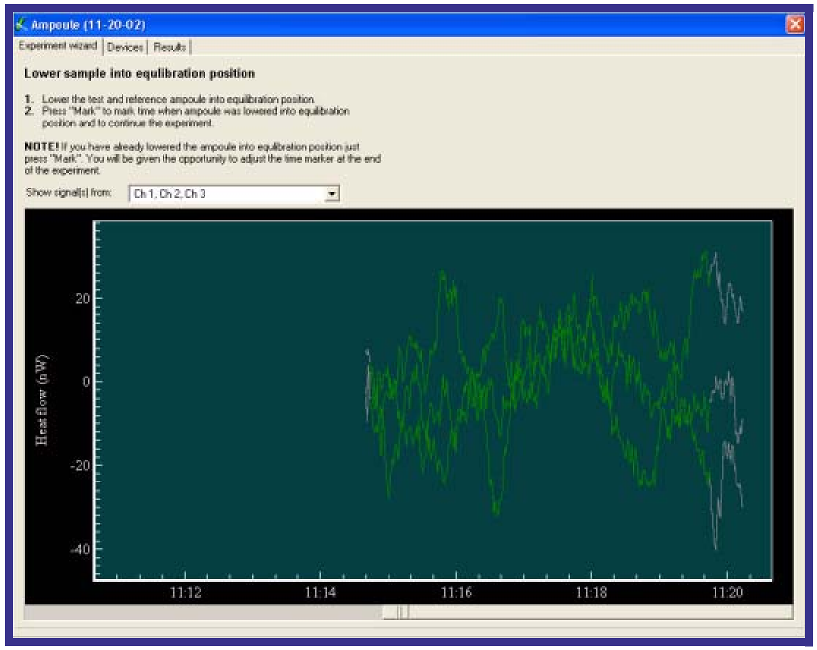
14. *Running initial baseline*: The program counts down the time specified for the baseline duration, and the remaining time is specified in the text box labeled **Time left**. When the baseline recording is finalized, you are instructed to introduce the sample ampoule into the equilibration position. Proceed to the next section.



Thermal Equilibration of the Sample

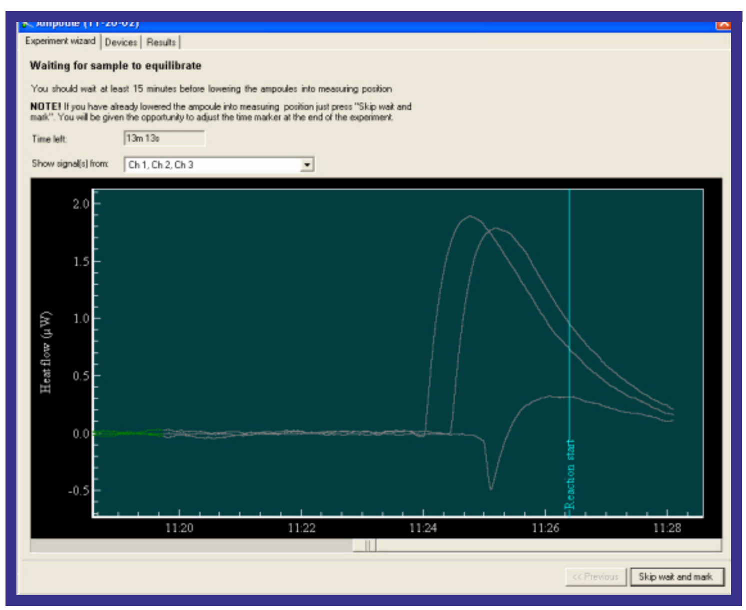
The sample has to be thermally equilibrated to the calorimetric temperature before the actual measurement can be started. Follow the instructions given.

15. Carefully and slowly lower the sample into the equilibration position of the calorimeter.



When lowering the sample into the equilibration position a disturbance of the signal is seen. This disturbance originates from temperature differences between the sample and the calorimeter.

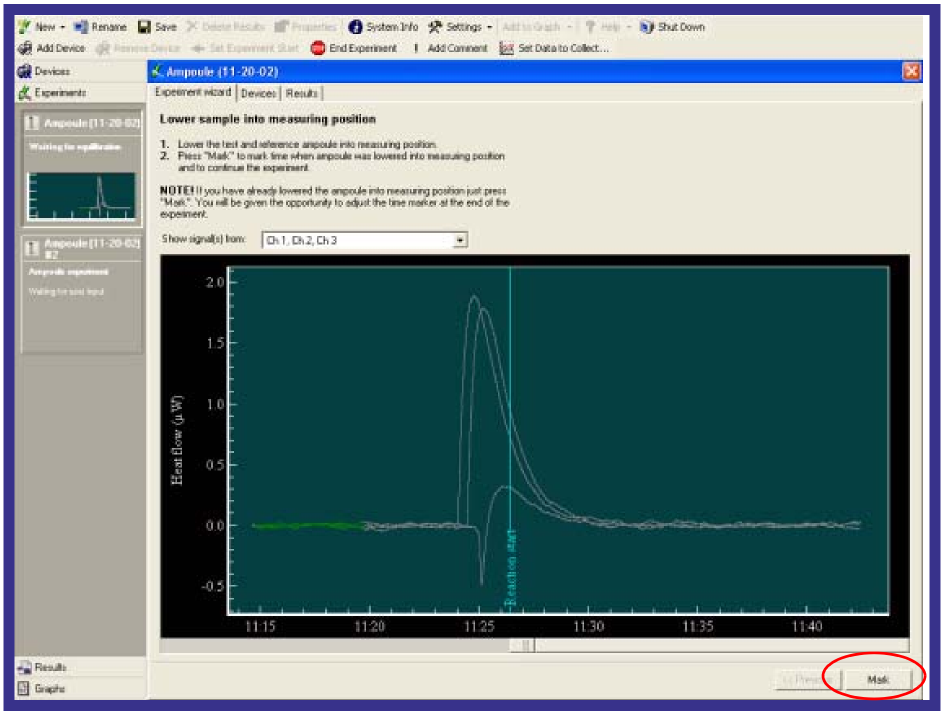
16. Click the **Mark** button when the ampoule is in position. The software starts to count down the time starting from 15 minutes. This is the normal time required for the sample to equilibrate, and reach the same temperature as the bath. The following dialog is displayed.



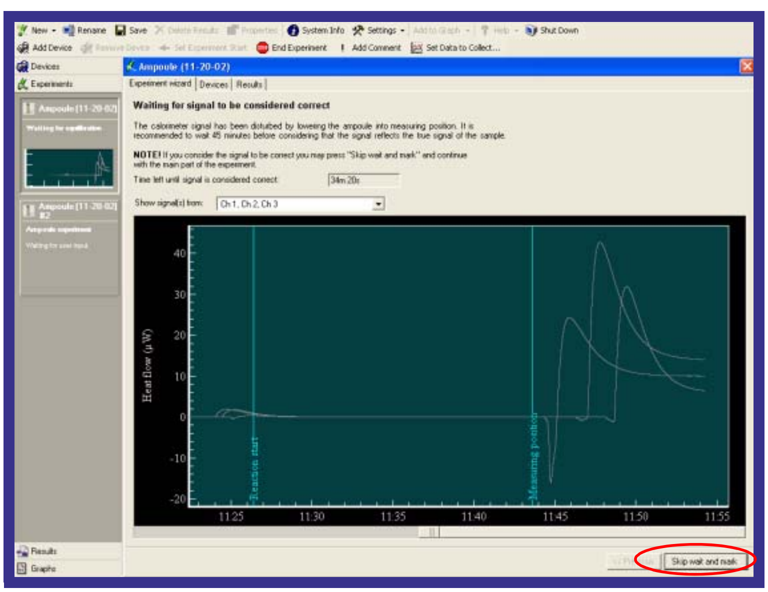
17. Wait for equilibration to be reached.

Insertion into the Calorimetric Measuring Position

After 15 minutes of equilibration you are prompted to lower the sample into measuring position as seen in the figure below.



18. Click **Mark,** and lower the sample ampoule slowly into the measuring position. This introduces an initial disturbance of the signal, mainly caused by the friction generated between the sample holder and the walls of the ampoule, as the ampoule is introduced into the ampoule holder of the calorimeter.



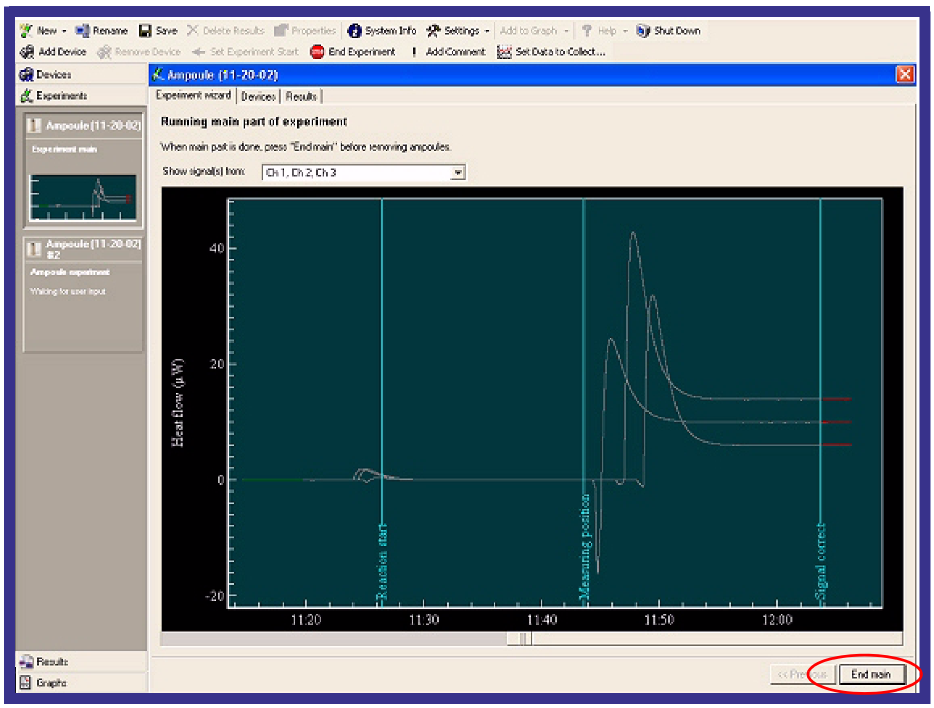
The software waits until the signal is considered correct, that is free from the disturbances generated because of the introduction of the sample ampoule. The default time is 45 minutes.

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

Managing the Main Part of the Experiment

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

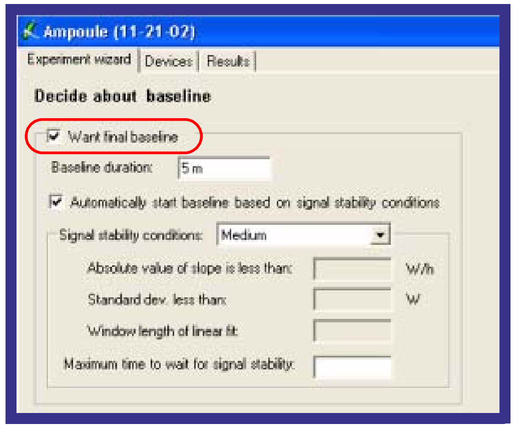
At this point the signal is considered correct from the instrument's point of view. Unwanted effects in the sample such as redistribution of solvent in a solid sample, or different types of sorption phenomena, etc., which might occur when the sample is exposed to sudden temperature changes, is not included.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



19. Press the **End main** button when the main part of the experiment is considered to be ready. A new dialog is displayed so that you may decide about the final baseline.

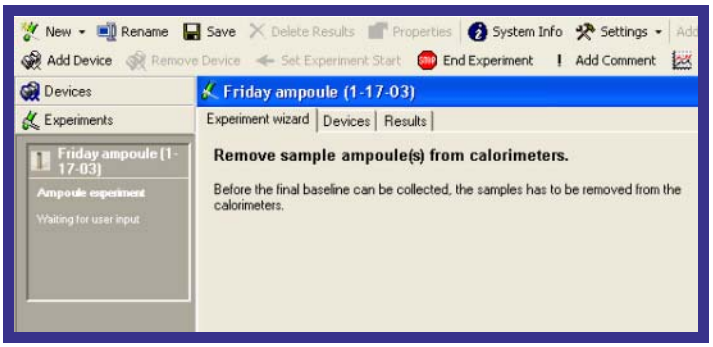
Decide about Final Baseline

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



20. Select the duration and stability conditions for the final baseline- and click **Next**.

You will be prompted to remove the sample ampoule (or ampoules) from the calorimeter. See the figure below.

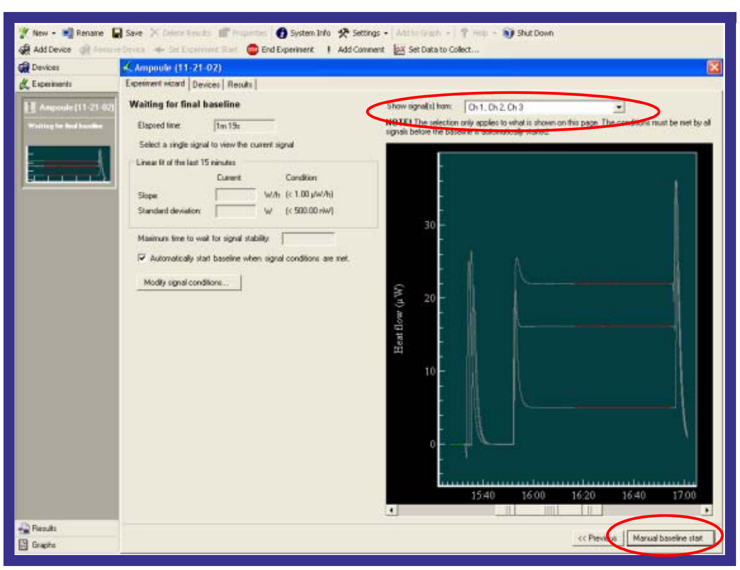


21. Remove the ampoules. After removal, place the ampoule lifter back in position then insert the lifters into the lowered position.

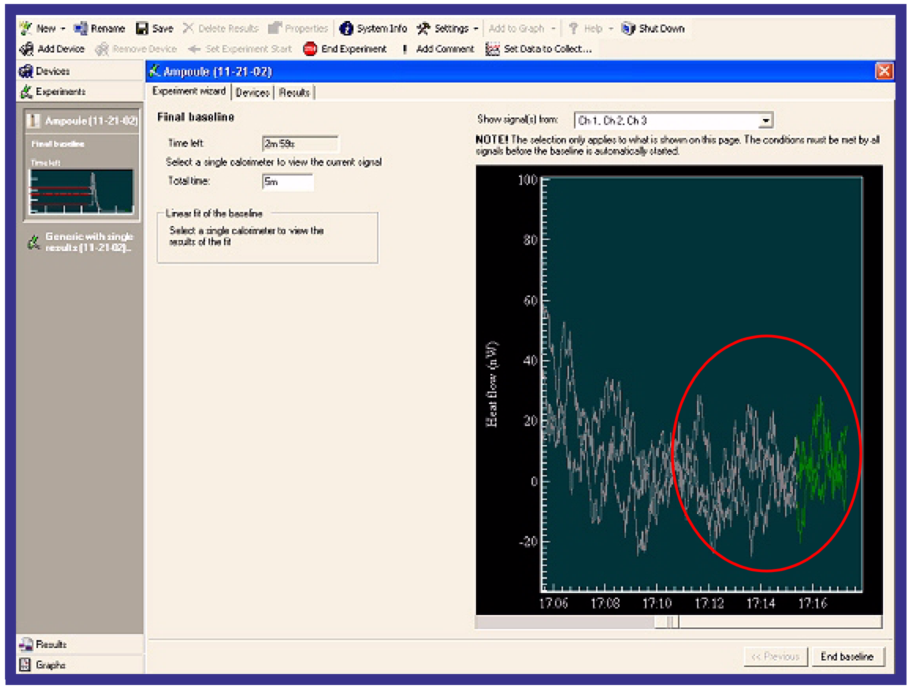
22. Click **Next** when the ampoules has been removed. The software now waits until the chosen stability criterion is met before starting the final baseline.

You can follow the current slope and standard deviation for any channel in the experiment by choosing the channel in the drop-down menu above the plot window. See the figure to the right.

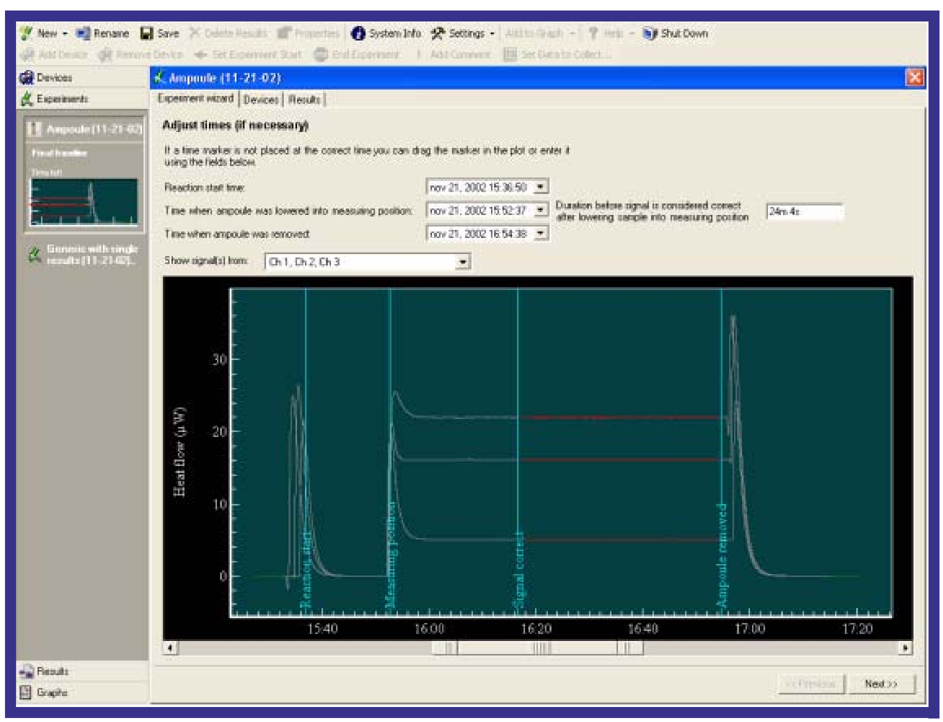
To manually start the baseline before the stability criterion has been reached, press the **Manual baseline start** button.



When the signal is stable in accordance with the set stability criteria, the final baseline section will start. The plots change color from gray to green when the baseline starts.



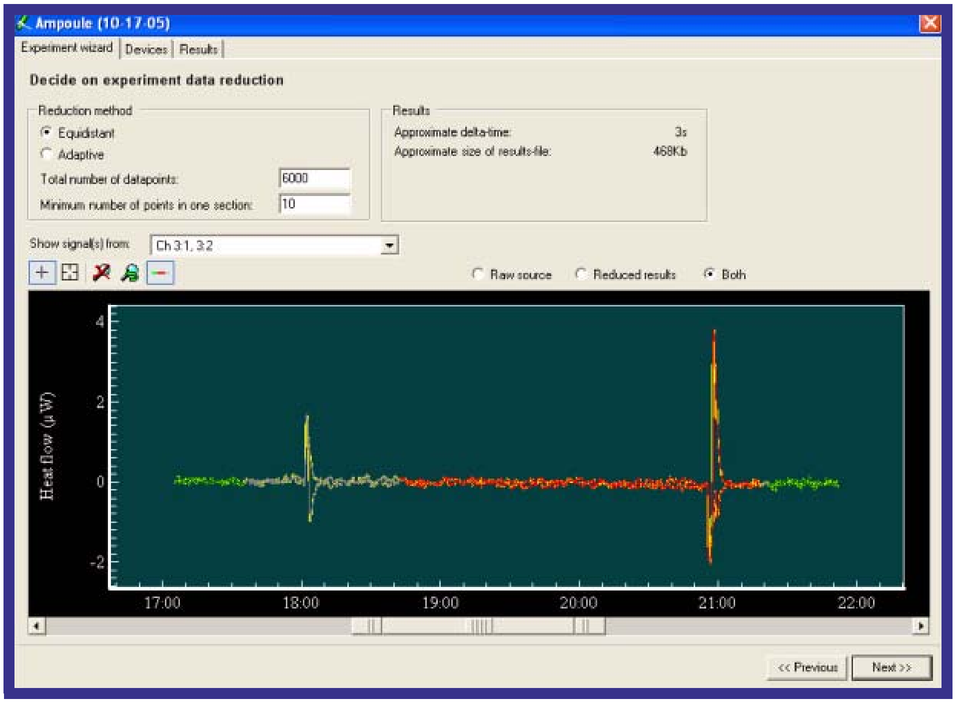
23. When the final baseline recording is finished, a new window in the wizard automatically appears to let you adjust the time settings (see the figure below). This may be done if the previous time settings are considered to be incorrect, *e.g.*, the duration before the signal was considered correct.



24. Click and drag a section change time marker to the correct time. Then adjust the time settings, using the drop-down list, if necessary. Click **Next**.

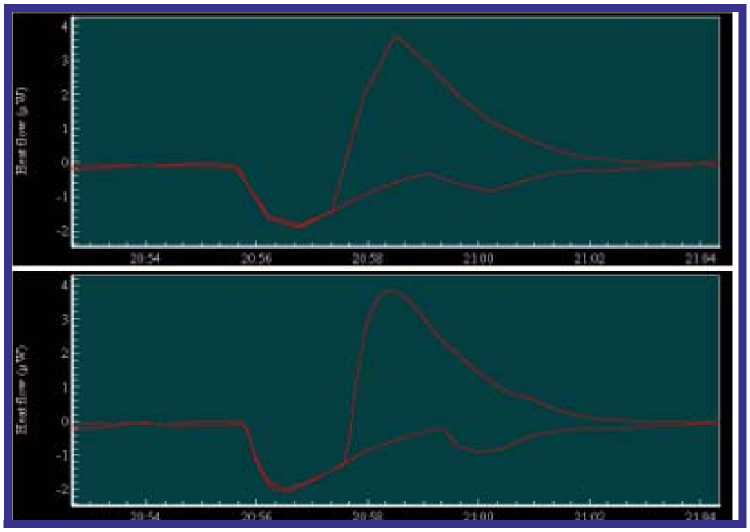
Decide on Experimental Data Reduction

In all experiments, data points are collected approximately every second. When running either the Ampoule or Non-isothermal Ampoule wizard you have the opportunity to choose how to save the data after the experiment. The following form appears when the experiment has been finalized in which one can decide 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 previewed 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 parts 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 previous page 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.

25. Click **Next**. A new form appears to let you finish the calorimetric experiment.

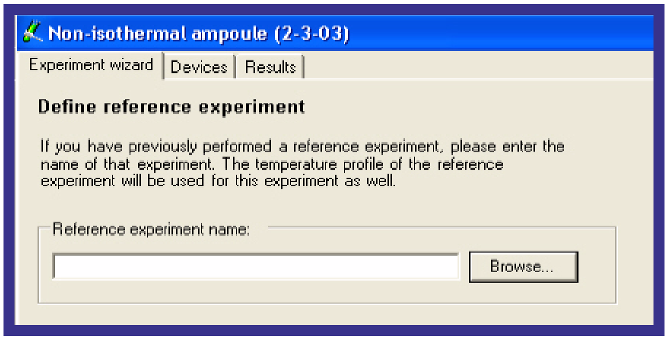
26. Mark the check box if the results file should not appear.

27. Click **Finish**.

The results have now been saved as a results file. To view the results file, click the mini-view button representing the file under the **Results** group.

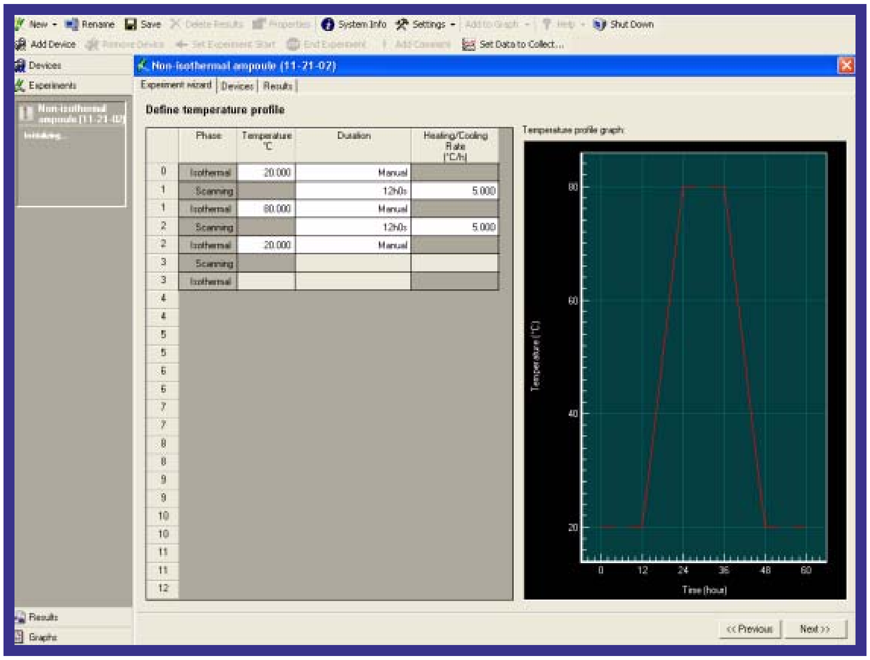
Non-Isothermal Measurements

Most features of the Non-Isothermal Ampoule Wizard are similar to the Ampoule wizard described on the previous pages. In the non-isothermal ampoule wizard a window for the definition of a temperature program is included. The temperature program can be continuous, *i.e.*, scanning or stepwise temperature jumps.



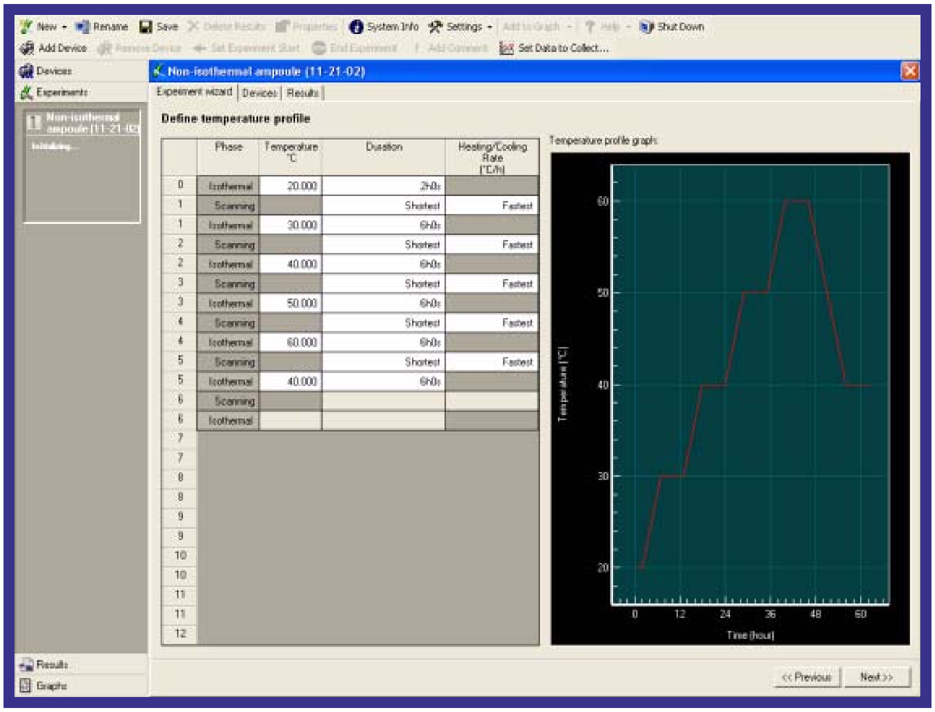
Setting Up a Temperature Profile

The following is an example of how a continuous wizard is defined.



In the example shown on the previous page, the settings for a scanning experiment are displayed. The initial temperature is set to 20 °C, and normally a stable signal is achieved before the scanning initiates. It is seen that the final temperature of 80 °C should be reached after 30 hours, leading to a scanning rate of 2 °C/h. The experiment is finalized with a down scan to 20 °C. The scanning periods are initiated manually as indicated in the example.

The temperature profiles can also be defined as stepwise as shown below.



In this example a stepwise temperature profile is defined. The initial temperature is 20°C, with duration of 2 hours, followed by a number of 6 hour steps with 10 °C intervals up to 60 °C. The experiment is finalized by decreasing the temperature to 40 °C. The duration of each step is chosen as the shortest corresponding to the fastest temperature change rate. The fastest achievable temperature change varies in the interval 2-10 K h^{-1}, depending on the absolute temperature, and whether the change is cooling or heating.

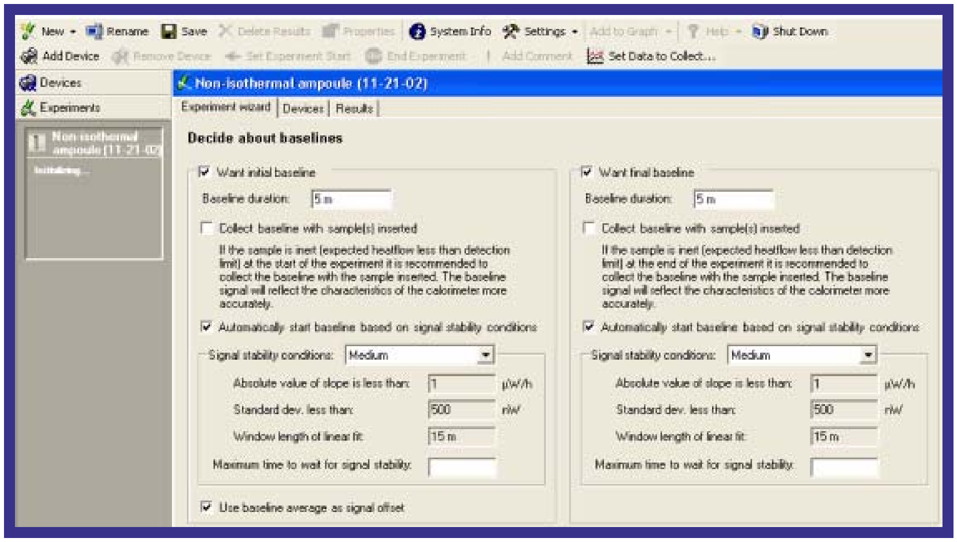
1. Set up the desired temperature profile.

2. Click **Next**. A new window is displayed to allow you to define the initial and final baseline.

NOTE: The maximum time to wait for stability conditions can be entered. The means that the temperature change will take place, if the conditions have *not* been met within the specified time period.

Defining a Baseline

You can collect a baseline with the sample ampoule inserted by marking the check boxes as shown in the figure below. When measuring phase transitions, *e.g.*, melting of lipids etc., this is the preferred experimental design. In cases where a baseline corresponding to zero activity is required, such as when measuring compatibility, stability, etc., a baseline should be recorded with the ampoule holders empty. In cases where the sample has zero activity at the start (and/or end) temperature, the sample can be inserted in the calorimeter when recording the baseline.

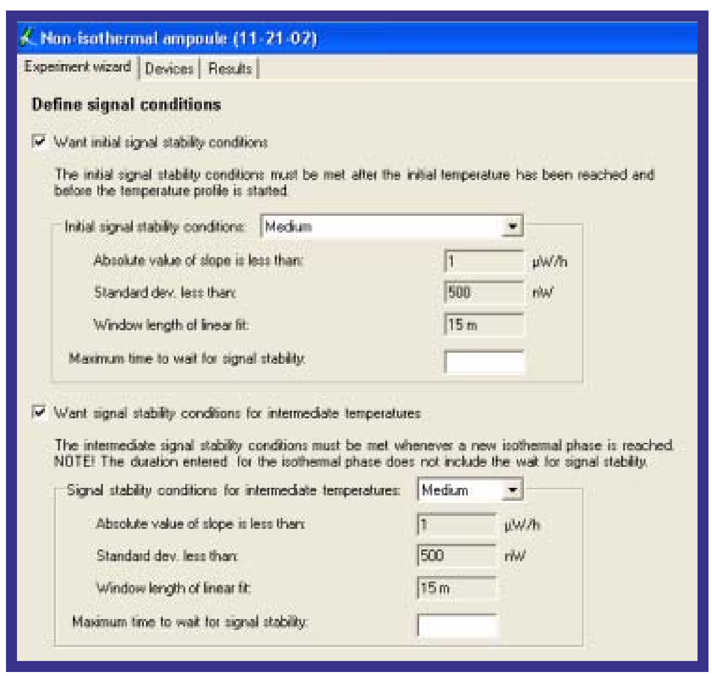


3. Enter the baseline criteria and click the **Next** button.

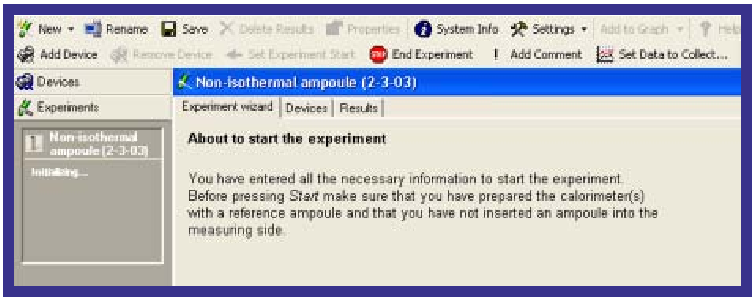
The next window (shown in the figure below) lets you define the **Initial** signal stability conditions to be met after the initial temperature has been reached, and before the temperature program starts.

You will also need to define the **Intermediate** signal conditions. This is the stability condition that should be met at each isothermal period between temperature jumps or scans, other than the Initial.

4. Click the **Next** button when all necessary information has been entered.



Starting the Non-Isothermal Experiment



At this point it is possible to review the experimental information given by clicking **Previous**.

When all setting are considered to be correct click **Start** to begin the non-isothermal experiment.

RH Perfusion Experiments

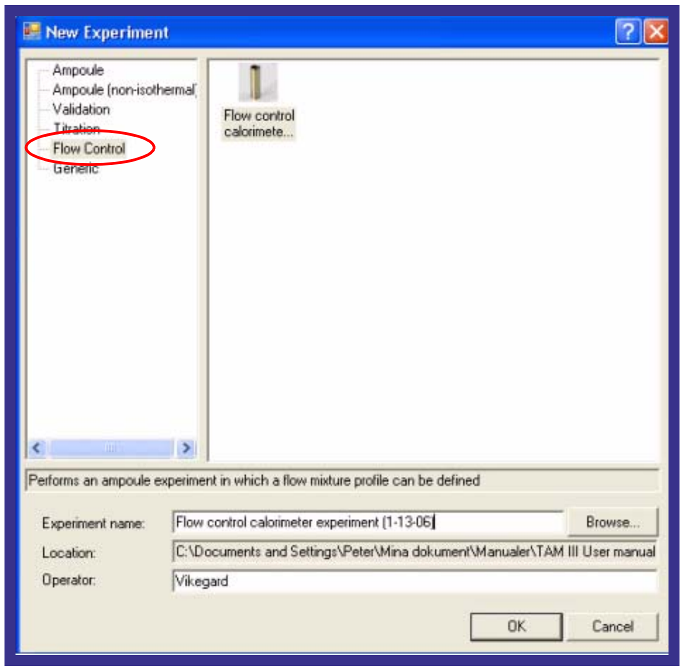
The RH perfusion ampoule can be used to measure interactions between a vapor and a solid material, *e.g.*, a powder. An inert carrier gas, usually nitrogen, with a set vapor pressure, is passed over the sample and any interaction, in terms of heat, is measured as a function of time.

The vapor pressure is set by use of two computer controlled mass flow controllers and can be changed stepwise or continuously in a ramp.

Initiating an RH Perfusion Experiment

Follow the instructions below to initiate an RH perfusion experiment:

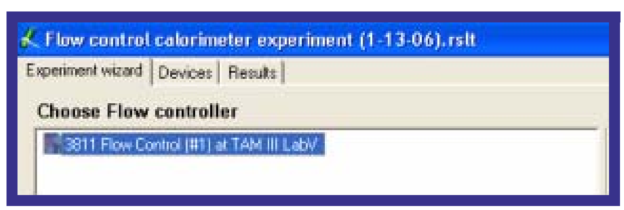
1. Choose **New Experiment** in the TAM Assistant menu. The following dialog is displayed, which allows you to chose from a number of different experiments. Choose the group named **Flow Control** to initiate the Wizard.



2. Click **OK** and click **Next** when the next view appears.

3. Choose the calorimeter to be used in the experiment by clicking on the picture that appears on the screen.

4. Click **Next** and the next view lets you choose the flow controller to be used in the experiment.

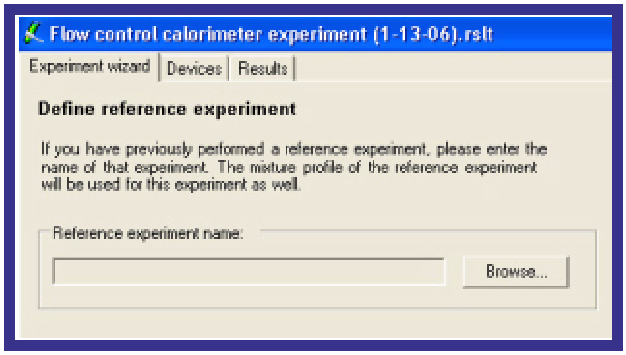


5. Mark the controller, even if there is only one connected.

6. Click **Next**.

At this point you can choose a blank measurement as a *reference measurement*, that will be subtracted from the main experiment. When the result from a blank reference experiment is to be subtracted, it is important that the main part of the experiment is run in exactly the same way for the blank reference to be a good representative.

NOTE: When choosing a previously run reference measurement, make sure that the subsequent measurement is run in an identical way.



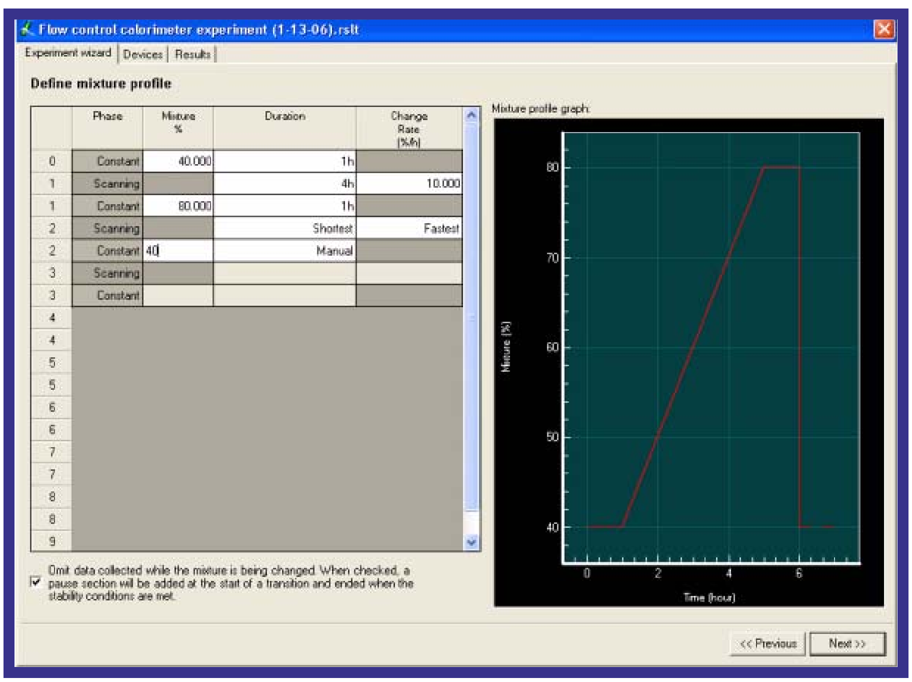
If this is not relevant, or if no reference experiment exists, leave the text frame empty.

7. Click **Next**.

Setting Up the RH Ramp

You can program an RH program as continuous or stepwise. Below is an example of how a continuous ramp is defined starting from 40 %RH to 80 %RH. The change rate is 10 %RH units per hour.

The ramp is followed by a 1-hour delay, which in turn is followed by a fast (step wise) return to the initial value.



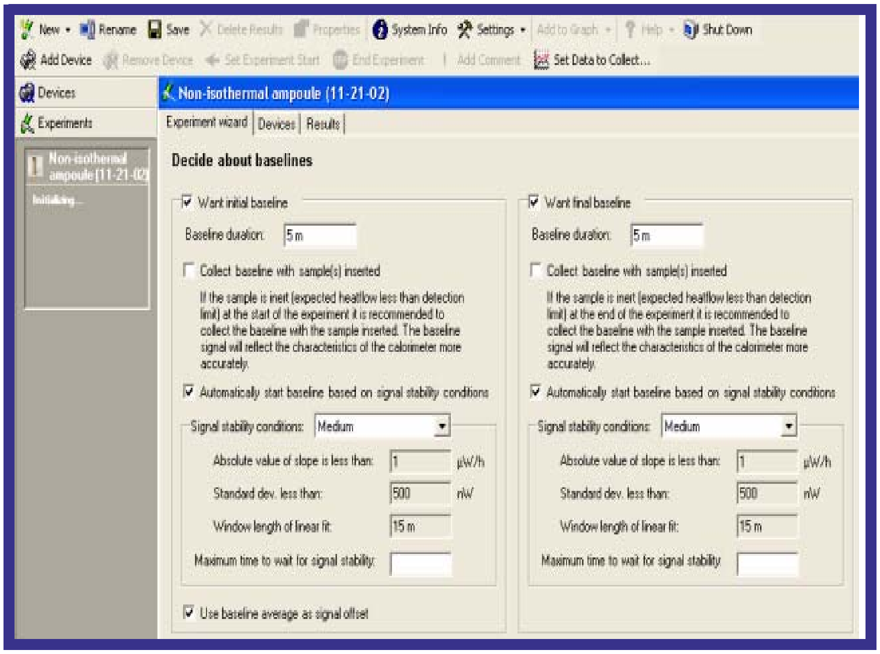
1. Define the desired RH profile.

2. If a ramp is defined, the signal of interest could be when the RH is being changed. In this case uncheck the box, **Omit data collection,** under the RH profile table.

3. Click **Next** when finished. A new window is displayed to allow you to define the initial and final baseline. See next page.

Baseline Definitions

You can collect a baseline, with the sample ampoule inserted, by marking the check boxes on the dialog displayed in the figure below. When measuring phase transitions, *e.g.*, a solvate formation at a critical vapor activity, this is the preferred experimental design. In cases, when the sample has zero activity at the initial (and/or final) vapor activity, the sample can be inserted in the calorimeter when recording the baseline.



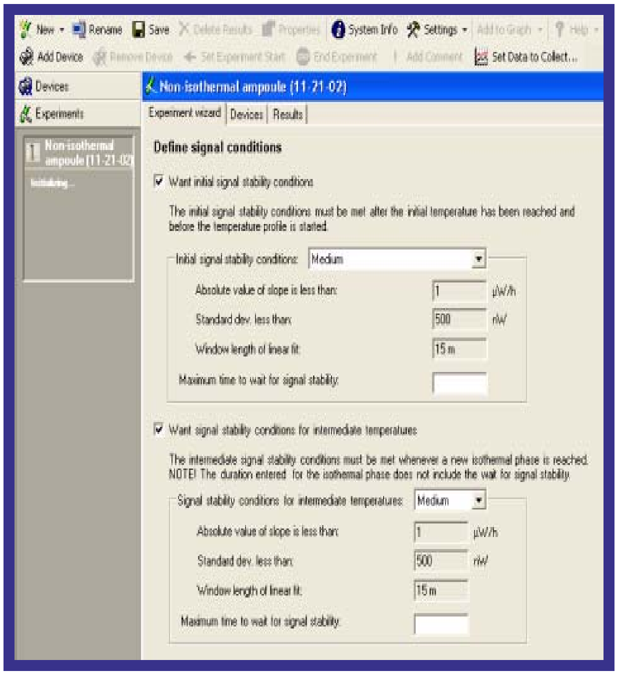
1. Enter the baseline criteria.

2 Click the **Next** button. The next window, shown in the figure below, lets you define the **Initial signal stability conditions**, which is to be met after the initial vapor activity has been reached and before the program starts.

NOTE: You can enter a maximum time to wait for stability conditions. This means that the temperature change will take place, if the conditions have not been met within the specified time period.

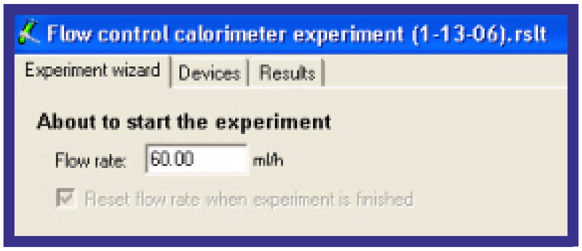
The **Intermediate signal conditions** should also be defined. This is the stability condition that should be met at each period of constant vapor activity between step wise changes or scans others than the initial.

3. Click the **Next** button when all necessary information has been entered.



Start the RH Control Experiment

1. Click **Previous** to review and change the experimental information entered.



2. Set the **Flow rate** between 60 to 180 mL/h.

3. When all of the settings are considered correct, click **Start** to begin the RH-perfusion experiment.

Titration Experiments

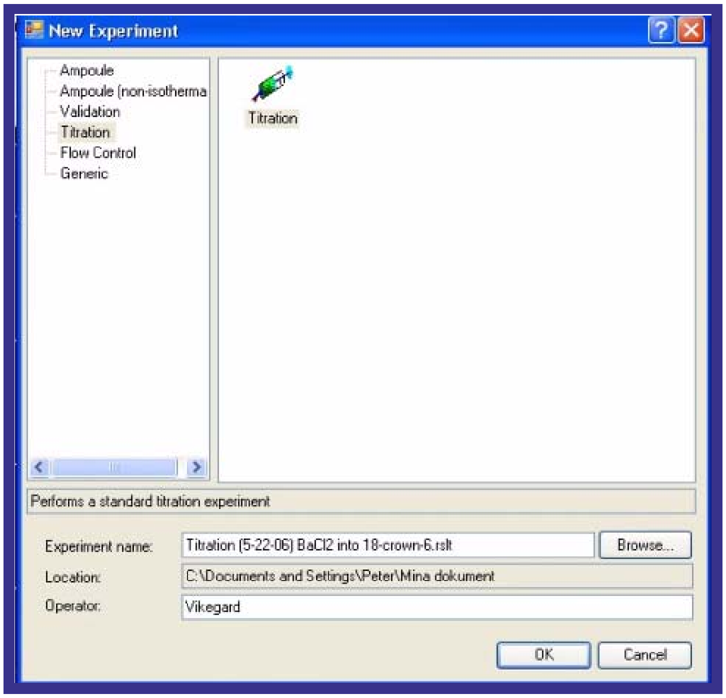
Titration experiments generally consist of initiating a physical, chemical or biological process by injecting a reactant liquid with a syringe into another liquid contained inside the calorimeter so as to initiate a chemical or physical process *in situ*.

This technique can be used to study slow chemical reactions (reaction time > 1 h or higher) or various physico-chemical interactions in solution. A widely used application is the investigation of complexation phenomena for determination of binding affinities and enthalpy changes, *e.g.*, ligand binding between in biological or chemical systems.

The Titration Experiment Wizard

Follow the instructions in this section to use the Titration Wizard:

1. Choose **New/Experiment...** from the menu on the top left of the main view. The **New Experiment** dialog is displayed as seen in the figure below.



2. Choose the **Titration** wizard in the branch structure.

3. Choose Titration or any saved predefined experiment controller.

4. Enter the desired **Experiment name** (*i.e.*, file name) and **Location**. (It is only possible to choose location if the experiment is started from the external computer.)

5. Enter an **Operator** name to continue.

6. Click **OK**. When the "welcome" view appears, click **Next** to continue to the next view.

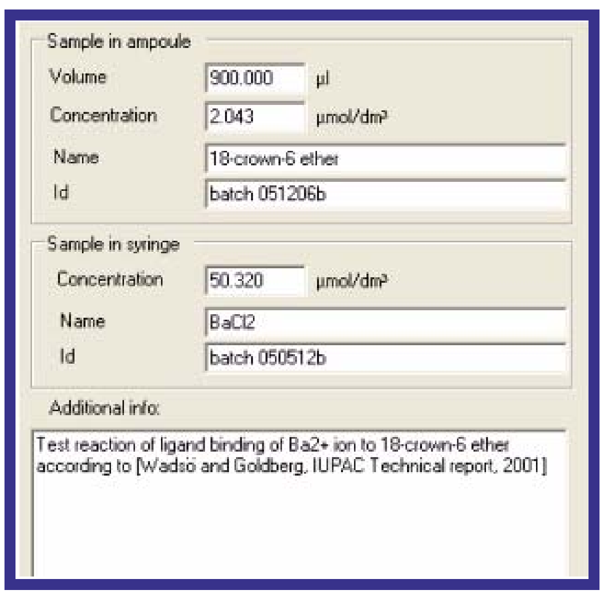
7. Choose the calorimeter to be used in the experiment. In the Titration wizard only one calorimeter can be chosen due to the fact that only one calorimeter can be associated per one injection pump. Calorimetric channels already occupied by ongoing experiments are shown in red color.

Click on the calorimetric channel to be used in the experiment. When chosen, the calorimeter will be shown in green color.

8. Click **Next**. You will be prompted to choose an injection pump (named the Lund Syringe Pump) as shown in the figure to the right. If several pumps are connected to the TAM III instrument, chose the one to be used in the experiment.

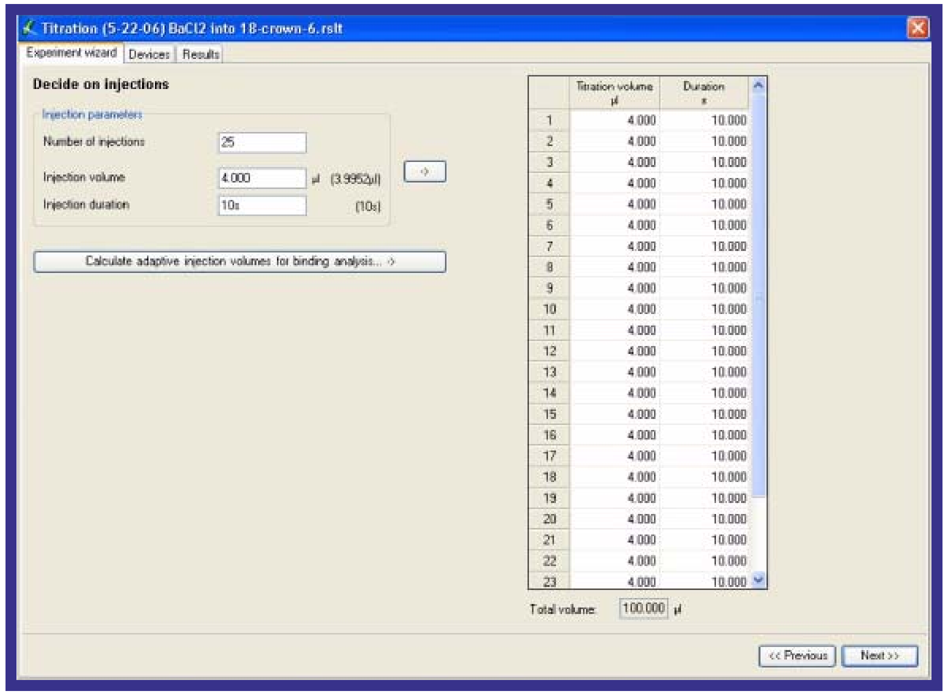
9. Click **Next** after selecting a pump. The following dialog is displayed.

10. Enter information about the sample. This can also be done at a later stage, either during the experiment, or after. If the sample information will be added after the experiment has already been started, click the **Results** tab to access the sample information sheet.



11. Click **Next** to continue. The dialog shown in the figure on the next page is displayed.

NOTE: Before deciding on the number of injections and volume when determination of binding affinities is to be made, you may wish to conduct a ligand binding simulation. See “Ligand Binding” in Chapter 6 for details on how to use the binding simulation feature in TAM Assistant.



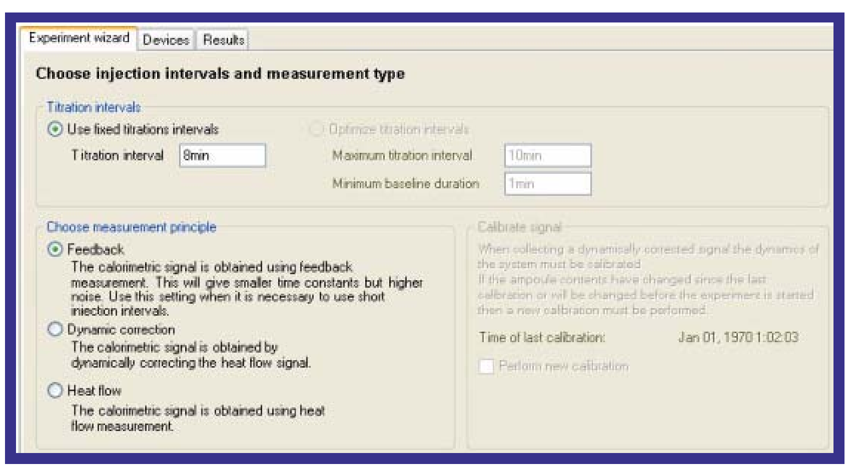
12. Enter the **Number of injections**, **Injection volume**, and **Injection duration** and click the arrow to move the information to the table.

13. *Binding Analysis feature*: In cases when the binding constant is known, or can be estimated within a certain error range (*e.g.*, within ½ order of magnitude), a feature called **Calculate adaptive injection volumes for binding analysis ...** can be used to optimize the precision in the determination of the binding constant.

    If you click the button, you will be prompted to make a binding simulation, see the NOTE above left. When TAM Assistant has all the necessary input data, it makes an optimization calculation of the number of injections in the region where the measured heat changes rapidly with the injected amount of reactant.

    This feature can be used to increase the precision in determinations of binding constants in a number of successive repetitive measurements.

14. When finished, click **Next**. The dialog shown in the figure below is displayed.



15. Determine the *injection interval, i.e.*, the time between injections, using the guidelines described here.

    The time required between injections is naturally related to the response time of the calorimeter, as well as the reaction time. Most complexation reactions are very fast compared to the calorimetric response time. In such cases, approximately 7-8 minutes between injections should be chosen to ensure that the signal has time to return to baseline and record a small baseline section. This is the case only when the instrument is run in **Feedback** or **Dynamic correction** mode, selected on the **Settings** tab (see Chapter 3). If the experiment is run with the instrument in **Heat flow** mode, at least 20 minutes is required between injections.

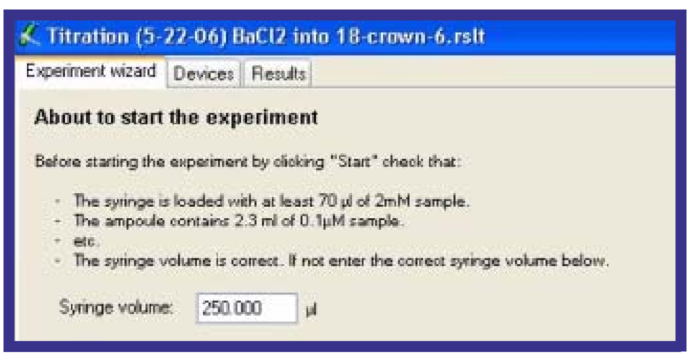
    In the case of slow reactions, initiated by the injection of a reactant, the time between injections must be judged in accordance with the reaction rate.

    The measurement principle, marked in the frame **Choose measurement principle,** is the same as the previously set current measurement principle. (**Feedback** was chosen in the example shown in the figure on the previous page.) However, any measuring principle can be chosen at this stage.

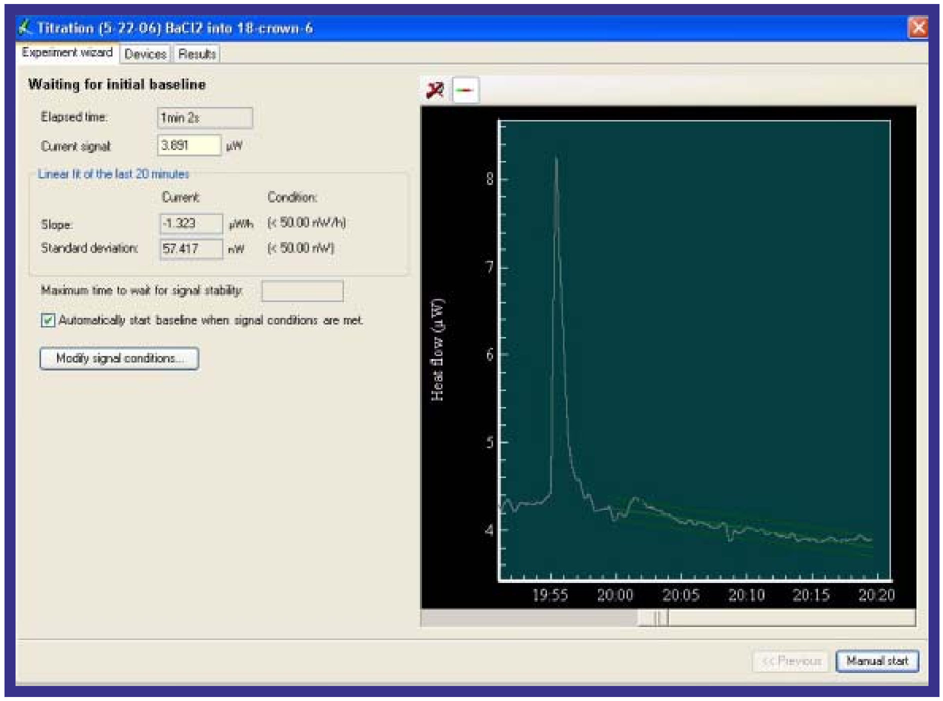
    If the measurement is to be run in **Dynamic correction** mode, the dynamics of the system must be calibrated with the ampoule and initial sample volume in the measuring position. This can be made from the **Devices** control before the Titration wizard is started, or it can be chosen to be included in the experiment. Just mark the box **Perform new calibration,** if a dynamic calibration with the current sample, or equivalent, has not already been made.

16. Select your choices and enter the desired values. Click **Next** to proceed. Review the information displayed and go back through the wizard, if something needs to be changed.

17. Cick **Start** to initiate the experiment according to the values defined in the wizard.



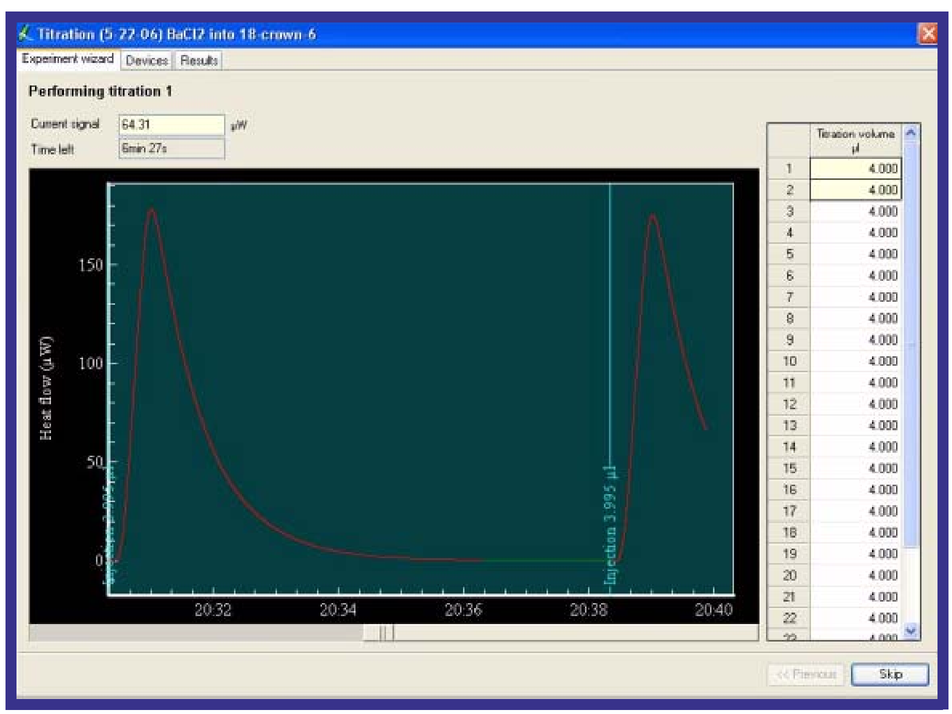
If the signal is not stable with respect to the chosen stability conditions, the dialog shown in the figure on the following page appears.



In the example above, the signal is considered stable whenever a linear fit over the last 20 minutes has a slope less than 50 nW/h, and the residuals have a standard deviation below 50 nW. These conditions correspond to the predetermined "Moderate." When they are met the software starts the initial baseline.

The initial baseline runs for a period of 5 minutes.

After the initial baseline has been completed the first injection starts automatically.



In the figure on the previous page, the value of the current signal and the time left is shown in the upper left corner. A table with the injection volumes is shown to the right. You can alter the injection volumes of individual injections, if they have not already been made as marked in the table.

When all the injections have been made, and the measurement has reached its end, click **Finish** to create the results file. The information that previously could be accessed from the Results tab in the experiment view, is now stored in the results file.

The results can now be analyzed with the ligand binding module in the analysis menu, see Chapter 4 for details.

Run Sequences

NOTE: Examples given in this section are based on an Ampoule Experiment wizard, If you use the other wizards, some screens and options will be different.

The run sequences option is used to program different sequential conditioned events within an experiment. This allows for higher flexibility and more automated and complex experimental setups.

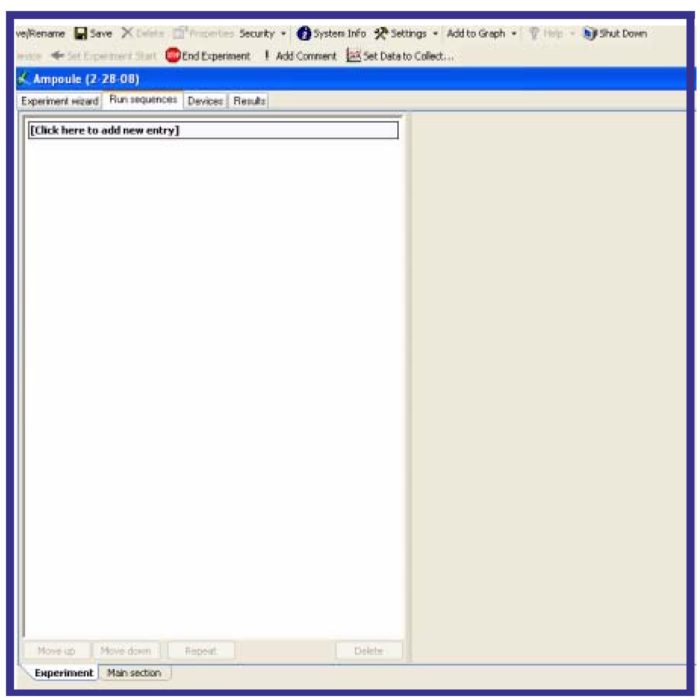
When starting a new experiment the wizard (shown in the figure to the right) will contain four tabs, of which one is called **Run sequences**. How to use the other parts of the Experimental wizards were described previously in this chapter.

To use the run sequences function, follow these steps:

1. Click on the **Run sequences** tab. The window will appear as shown in the figure below. To the left there is a square [Click here to add new entry] and on the bottom of the screen there are two tabs, **Experiment** and **Main section**.

   If the entries are programmed using the Experiment tab, the programmed sequences can be executed in any parts of the experiments, *i.e.*, also baseline and pause sections.

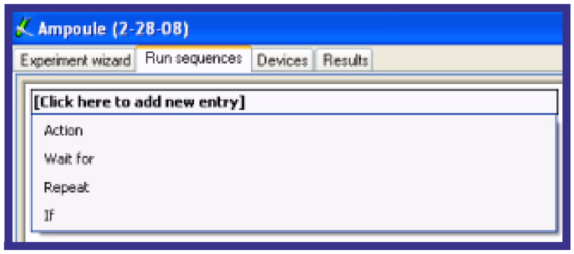
   If entries are programmed in the Main section tab, the events will only occur in the main section of the experiment.



These two can be programmed independently of each other. The Main section tab is only available in the Ampoule experiment wizard.

2. Click on **[Click here to add new entry]** and four options are shown. See the figure to the right.

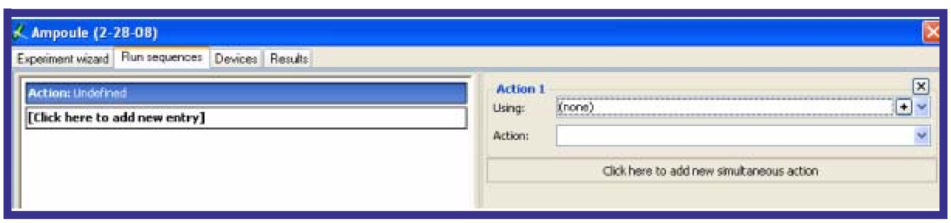
3. Select one of the four options described briefly in the list below:



- **Action** - Some kind of function should occur. It could be a calibration, an injection or change in relative humidity.
- **Wait for** - The next action will not occur until the defined condition(s) has been met. For example, you can set the next action to occur after a certain time period has elapsed or a stability condition has been met.
- **Repeat** - This repeats the last action or sequence of actions.
- **If** - An action will only occur if a certain condition is met.

Using the "Action" Option

If an **Action** is chosen, a new text box will be displayed to the right as shown in the figure below. This box will have several options. The drop down menu will look a little different depending on if devices have been added to experiment or not.



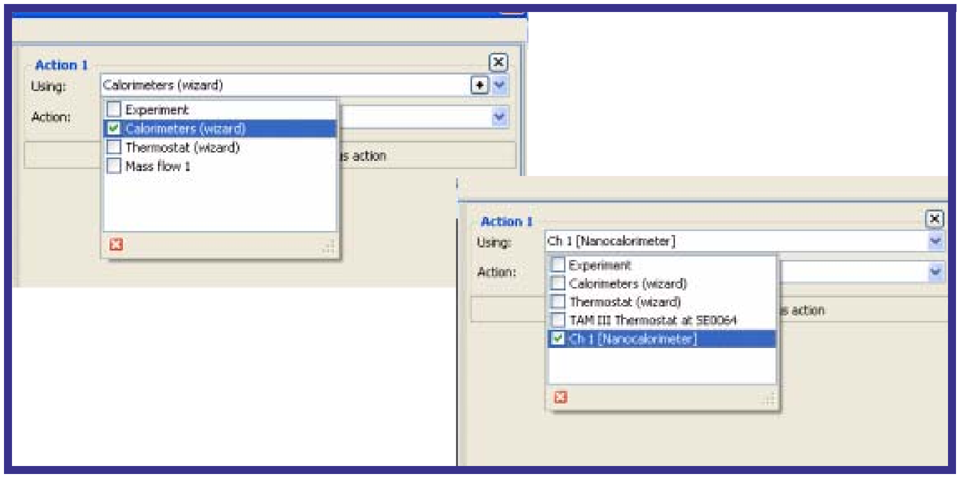
1. Add a device by clicking the **[+]**. The window shown to the right will appear.

2. Choose a **Device type**. Different device types can be chosen, e.g., Thermostat, Calorimeter, Accessory, Pump, Stirrer, Flow controller or Thermometer.

3. Name the device in the **Device description** field. For the other options refer to the section on how to run a Generic experiment.

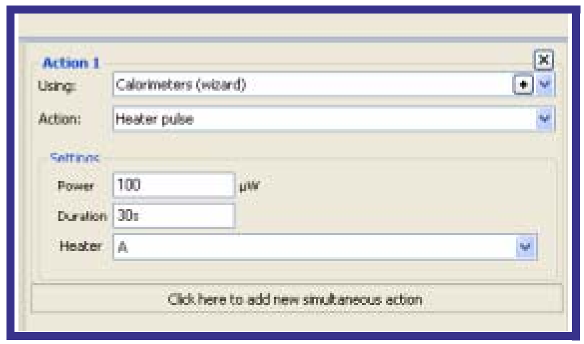
4. Click **OK**.

5. Click the arrow to open the drop-down menu and choose one to use. See the figures on the next page.



In the first case no calorimeters have yet been chosen in the wizard, but when this is done the action will be on the chosen calorimeter(s).

In the second example, the calorimeter in Channel 1 has already been chosen using the experimental wizard.



6. Choose an **Action** from the drop-down list depending on the type of device that is used. For a Calorimeter the following choices are available: Heater pulse, Dynamic calibration, Gain calibration.

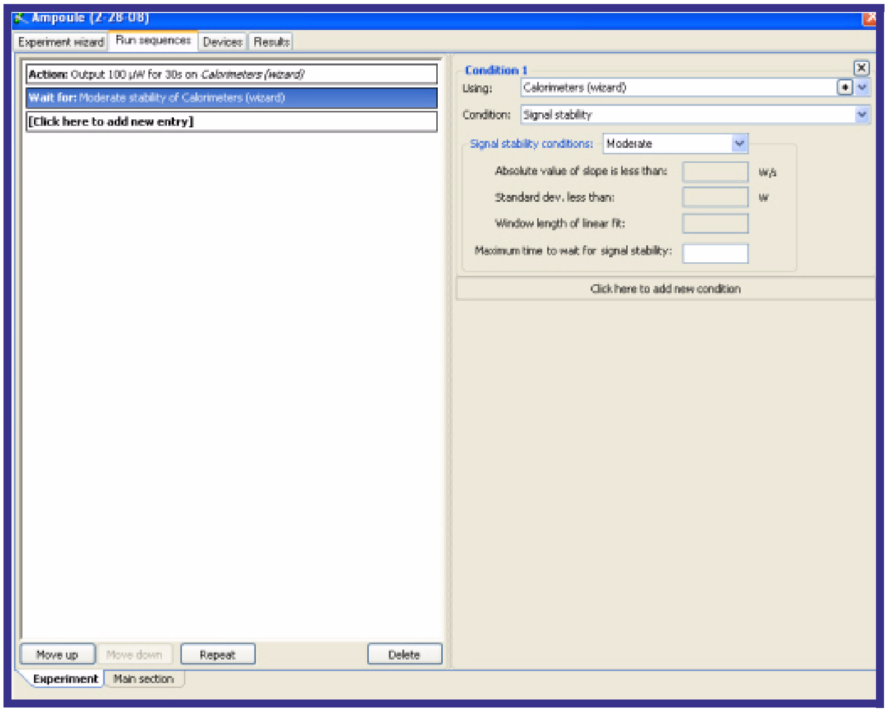
   If heater pulse is chosen, options of power, duration, and heater will be displayed.

7. Now it is possible to add a simultaneous action in the same or in a new device by selecting **Click here to add new simultaneous action**.

8. Program additional events if desired by selecting **[Click here to add new entry]** to the left. See the figure on the next page.

Using the “Wait For” Option

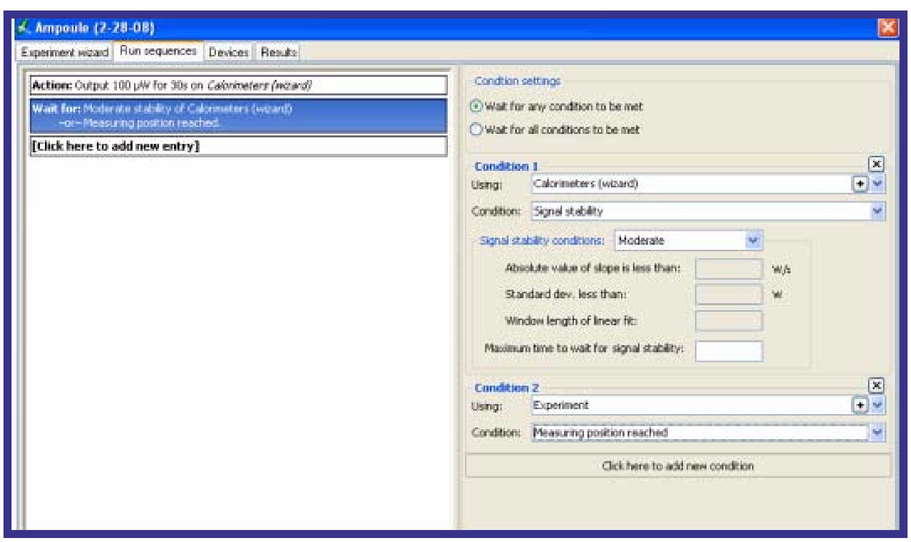
In the example below **Wait for** is chosen and a stability condition for the calorimeter is defined.



In the left list the events are shown as an overview. To the right the events are defined.

The following functions can be performed from this window:

- To change the order of any event, use the **Move up** and **Move down** buttons at the bottom.
- To repeat an event, press the **Repeat** button.
- To remove an event, mark that event and press the **Delete** button.
- To add more conditions, select **Click here to add new condition**.

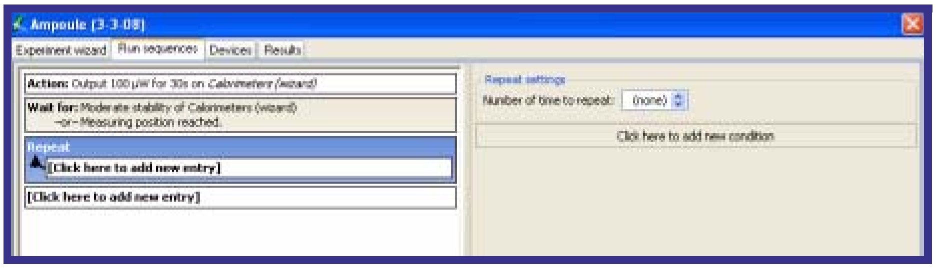


- To choose more than one condition, check the desired conditions in the Conditions settings box.

The conditions can also be set by using the Experimental events, such as Equilibration position reached, Measuring position reached, Baseline started or Baseline ended.

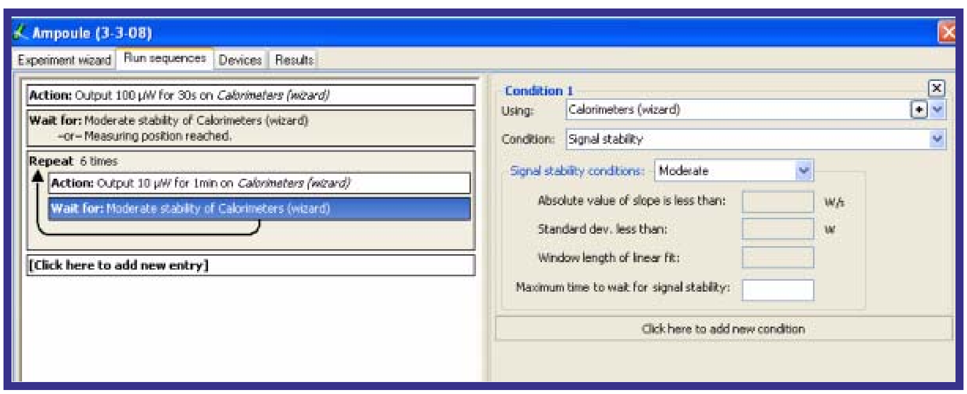
Using the “Repeat” Option

The **Repeat** function can be used when adding a new entry if, for example, several sequential heat pulses in a calorimeter should be performed.



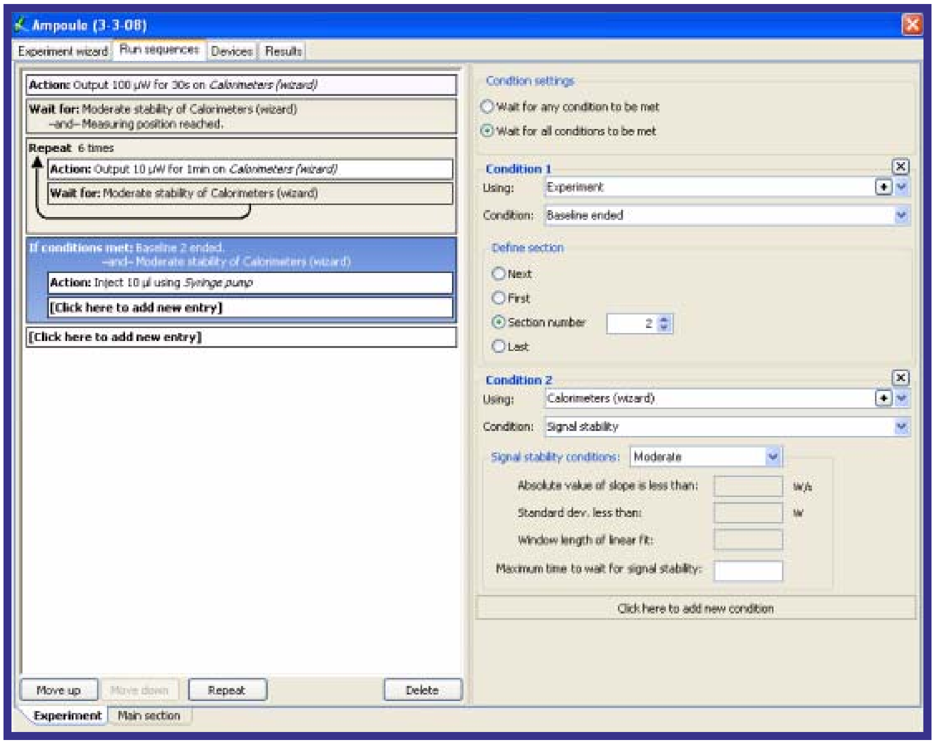
In the right-hand box, the **Number of times to** perform the action can be set.

In the example below, calibration pulses are performed six times with a stability condition between each one.



Using the “If” Option

The **If** entry can be used to choose whether or not an action should take place.



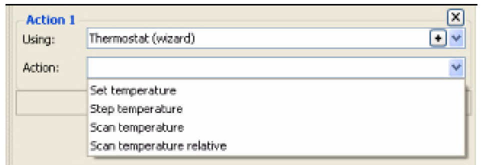
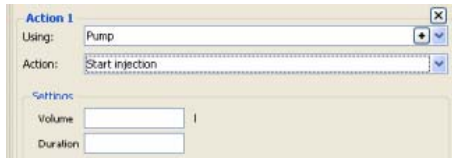
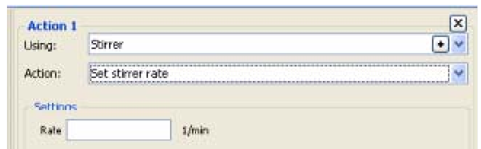
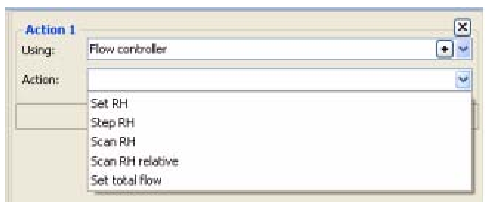
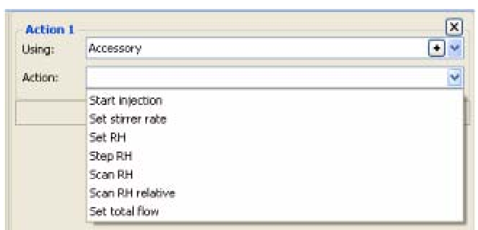
In the above example an injection should be performed if a baseline section is ended and the calorimeter is stable.

Option Overview

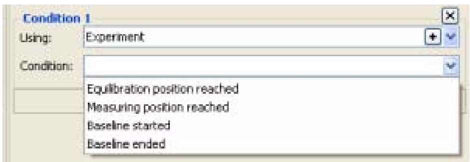
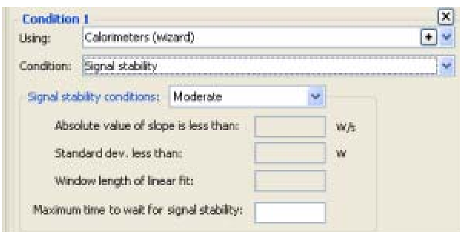
The following tables provide an overview of the different options and their functions.

Actions

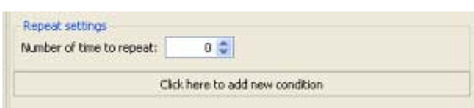
Actions	Description
	**Add comment**: Used To add a comment that is saved in the experimental file. This could be a comment about the action in the experiment.
	**Heater Pulse, Dynamic Calibration**, and **Gain Calibration**: Used to program calibrations and heater pulses. You can chose a specific calorimeter or any calorimeter connected to the experiment.

	**Set Temperature, Step Temperature, Scan Temperature**, and **Scan Temperature Relative**: Used to program a change in temperature.
	**Start Injection:** Used to program injections, volume and speed of injection of a syringe pump.
	**Set Stirrer Rate**: Used to set the stirrer rate and start or stop the stirrer via the pump control module.
	**Set RH, Step RH, Scan RH, Scan RH Relative**, and **Set Total Flow**: Used to set or change the relative humidity via the flow control module.
	**Start Injections, Set Stirrer Rate, Set RH, Step RH, Scan RH, Scan RH Relative**, and **Set Total Flow**: Used to control an Accessory such as the pump, stirrer, or the relative humidity during the experiment.

Wait For / If Conditions

Wait For/If Conditions	Description
	**Elapsed Time Over**: If **(none)** is selected it is possible to set a time delay.
	**Equilibration Position Reached, Measuring Position Reached, Baseline Started**, and **Baseline Ended**: Uses the selected experimental events as conditions to wait for to continue the **Experiment**.
	**Signal Stability**: Uses the calorimeter stability as a condition to continue the experiment.

Repeat

Repeat	Description
	**Number of times to repeat**: Used to set the number of times an action should be performed.

Chapter 4:

Data Handling & Analysis

Data Handling

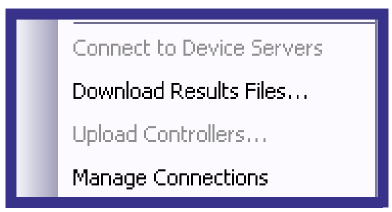
This section describes how experimental results files are downloaded from TAM III to an external computer connected to the network. It also describes in detail how data from a results file is exported to a format that can be imported by software like Excel, etc.

Downloading Results Files

Results files that have been saved on the instrument system should be moved to an external computer at regular intervals in order to avoid overloading the memory resources.

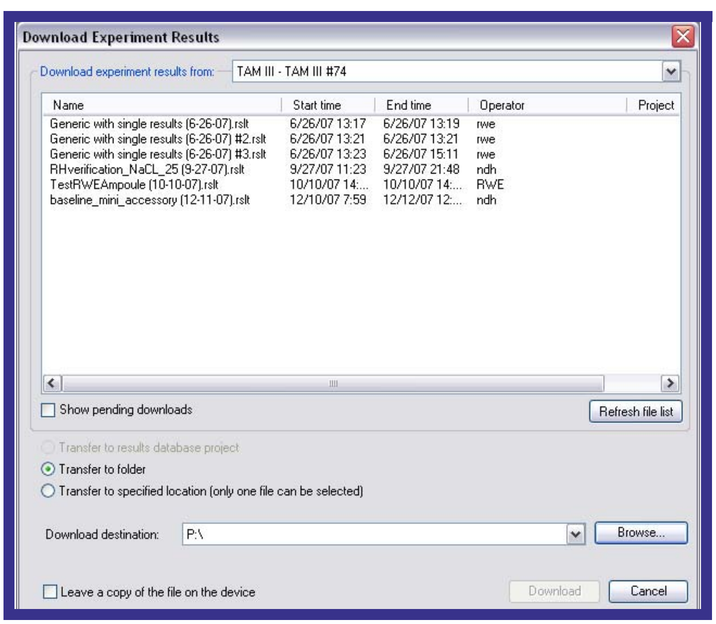
Follow these instructions to download the results files:

1. Choose **Download Results Files...** under the File menu. A dialog opens with the results files that currently exist on the instrument system listed.



2. Choose the desired results file by clicking on the target directory for the downloaded file.

3. Select the method to use:

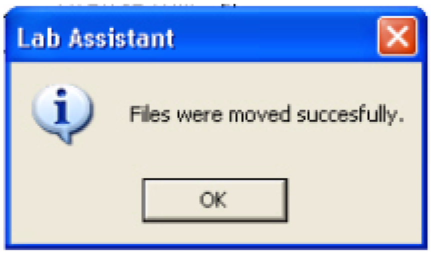


- Use the **Transfer to Folder** function to move the multiple results files to the chosen folder.

- Use the **Transfer to specified location** function to move one results file to the chosen location.

- If the **Leave a copy of the file on the device** option is left blank, the results file will irreversibly be moved from the instrument system.

- We recommend that file transfer be performed regularly to unload the system and release computer memory.

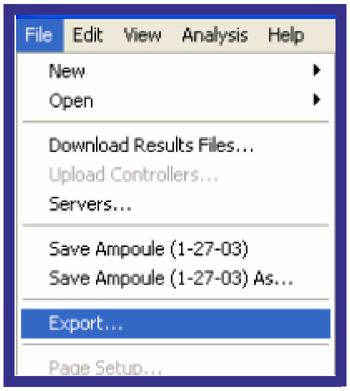


4. Select the results file, target location, and the **Move** or **Copy** option then click the **Download** push button to complete the procedure. If the operation was performed successfully the following information appears.

5. Click **OK**.

6. Choose another file to download or press the **Cancel** button to exit the download dialog.

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:

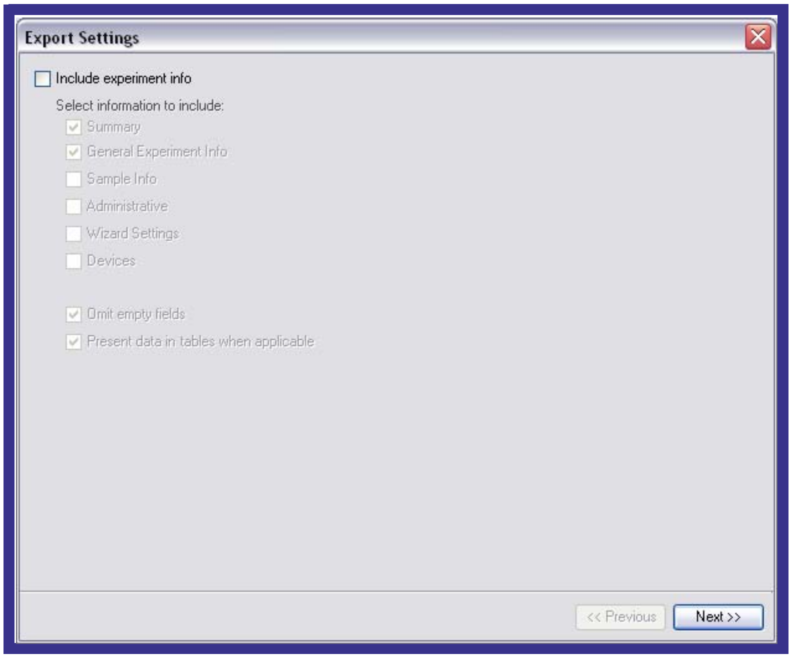
1. Choose **Open/Results File...** under the **File** menu.

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 window shown in the figure below is displayed, which allows you to choose the content of the exported data.



5. Check the desired options then click the **Next** button to proceed. You will be offered the opportunity to include experiment information, various data series, raw data, and/or statistics.

   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.

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

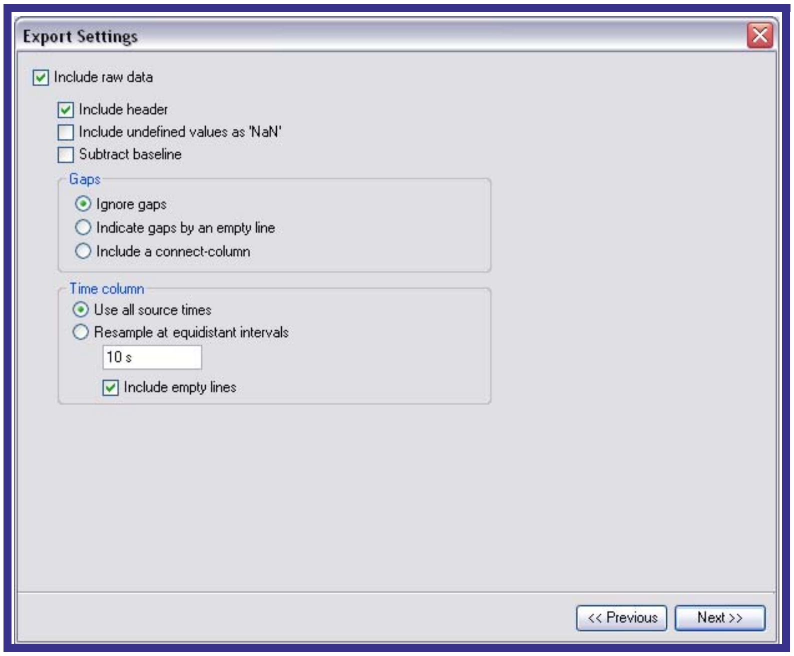
7. Click **Next** again.

8. Choose the desired raw data options shown in the figure below. The following guidelines should be considered:

NOTE: Scroll down to select the information for all channels.

- 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 Experiment

If you are going to be running a long experiment, it can be useful to export and present the data collected during the initial part of the experiment. This is possible by following the instructions given in the previous section.

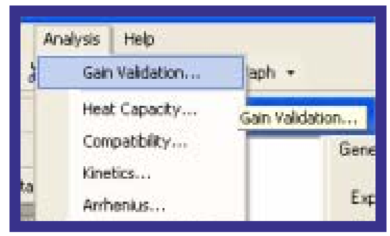
Results Analysis

The results analysis in TAM Assistant is performed by use of analysis modules, with each module dedicated to a certain application.

All analysis modules of TAM Assistant are accessed from the **Analysis** menu. In order to perform an analysis, the data files have to be downloaded from the TAM III system and analyzed by the external TAM Assistant.

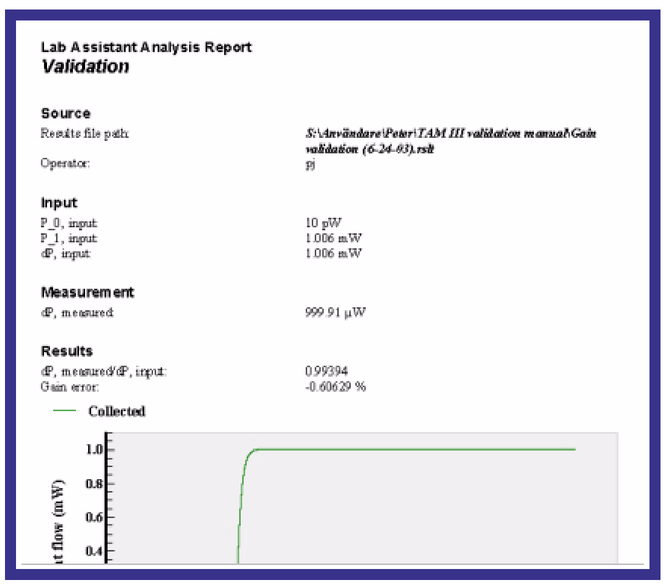
Gain Validation

The Gain validation analysis is only applicable to results files that have been generated by the Gain Validation wizard.



For evaluation of the data and creation of a report, select **Gain Validation...** under the **Analysis** menu. Open and view the results file when making the analysis.

A report is created automatically containing a graph of the calorimetric signal and validation parameters as calculated by TAM Assistant. Click on the icon shown in the figure to the right to open the report. See the example below.



Lab Assistant Analysis Report
Validation

Source

Results file path:	*S:\Användare\Peter\TAM III validation manual\Gain validation (6-24-03).rslt*
Operator:	pj

Input

P_0, input:	10 pW
P_1, input:	1.006 mW
dP, input:	1.006 mW

Measurement

dP, measured:	999.91 µW

Results

dP, measured/dP, input:	0.99394
Gain error:	-0.60629 %

The report contains the values of the input parameters as well the results.

- **Measurement**: Provides the calculated mean value of the steady state signal, corrected for the baseline offset.
- **Results**: Provides the difference between the gain factors as obtained by a standard calibration, and the results given by the Validation procedure.

- The Gain error is calculated according to the equation below.

Where P is the measured steady state signal and P_{in} is the heat flow released by the calibration heater when exposed to a known electrical current.

$$GainError = ((P - P_{in}) / P)$$

The Gain error is an estimation of the accuracy of the calorimeter and, as such, it should be within the accuracy given by the specifications for the validated calorimeter type.

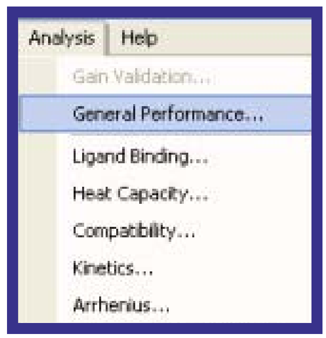
General Performance

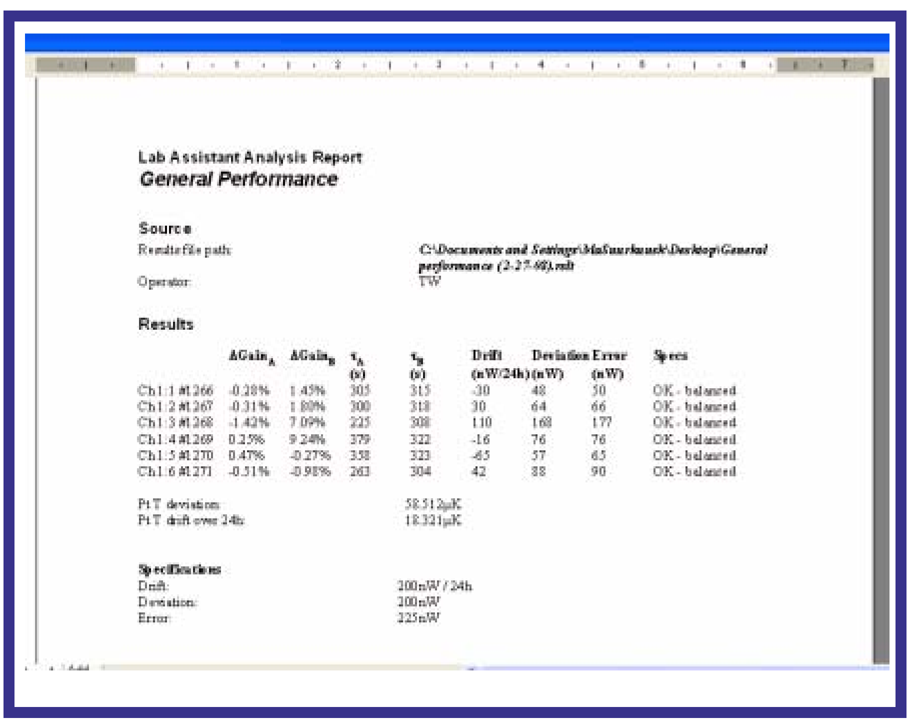
To run the General Performance analysis, follow these steps:

1. Open the GPT results file and select the file in the Results file manager.

2. Start the analysis by choosing **General Performance ...** from the **Analysis** menu. The analysis report is then automatically generated.

The General performance analysis is only possible with results files generated by the experimental wizard called **General performance**.





Lab Assistant Analysis Report

General Performance

Source

Results file path: *C:\Documents and Settings\MaSuurkuusk\Desktop\General performance (2-27-08).rslt*

Operator: TW

Results

	ΔGain$_A$	ΔGain$_B$	τ_A (s)	τ_B (s)	Drift (nW/24h)	Deviation (nW)	Error (nW)	Specs
Ch1:1 #1266	-0.28%	1.45%	305	315	-30	48	50	OK - balanced
Ch1:2 #1267	-0.31%	1.80%	300	318	30	64	66	OK - balanced
Ch1:3 #1268	-1.42%	7.09%	225	308	110	168	177	OK - balanced
Ch1:4 #1269	0.25%	9.24%	379	322	-16	76	76	OK - balanced
Ch1:5 #1270	0.47%	-0.27%	358	323	-65	57	65	OK - balanced
Ch1:6 #1271	-0.51%	-0.98%	263	304	42	88	90	OK - balanced

PtT deviation: 58.512µK

PtT drift over 24h: 18.321µK

Specifications

Drift: 200nW / 24h

Deviation: 200nW

Error: 225nW

The example in the figure above shows the result from a general performance test on two sets of TAM III 3208 Multicalorimeters (a total of 12 3206 Minicalorimeters). The calculated parameters in the results table must have values consistent with TA Instruments' specifications. The parameters reflect the performance of a calorimeter and thermostat.

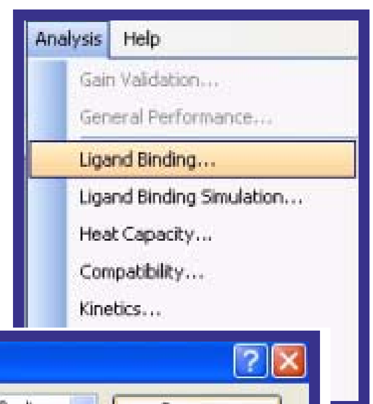
The **DGainA** is the relative difference between the calibration heat supplied and the measured heat. The **DGainB** is the relative difference between the gain factors obtained for the Sample side (A) relative, to that of the reference side (B) of a calorimeter.

Tau A and **Tau B** are the time constants obtained for the A and B sides respectively. For a calorimeter well balanced with respect to the heat capacity, the difference between the two time constants should be small. In the example given on the previous page, the General performance test was run with the insert on the reference side, but with the sample side empty. This is clearly reflected in the lower time constant for the A side.

The parameters **Drift**, **Deviation** and **Error** reflect the long term stability of a calorimeter. The drift is simply the slope of a fitted line over 24 hours, and the deviation is twice the standard deviation over the residuals with respect to the fitted line. The error is the quadratic mean value of the drift and the deviation.

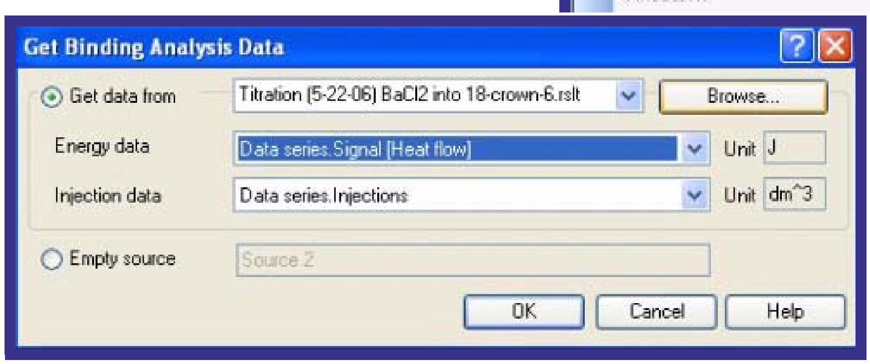
Ligand Binding

The Ligand Binding analysis module is used to analyze the results from complexation (or binding) reactions obtained by isothermal titration experiments (ITC). The analysis consists of a numerical non-linear regression procedure where the fitting parameters generally are the binding (or affinity) constant and the apparent enthalpy change for the process.



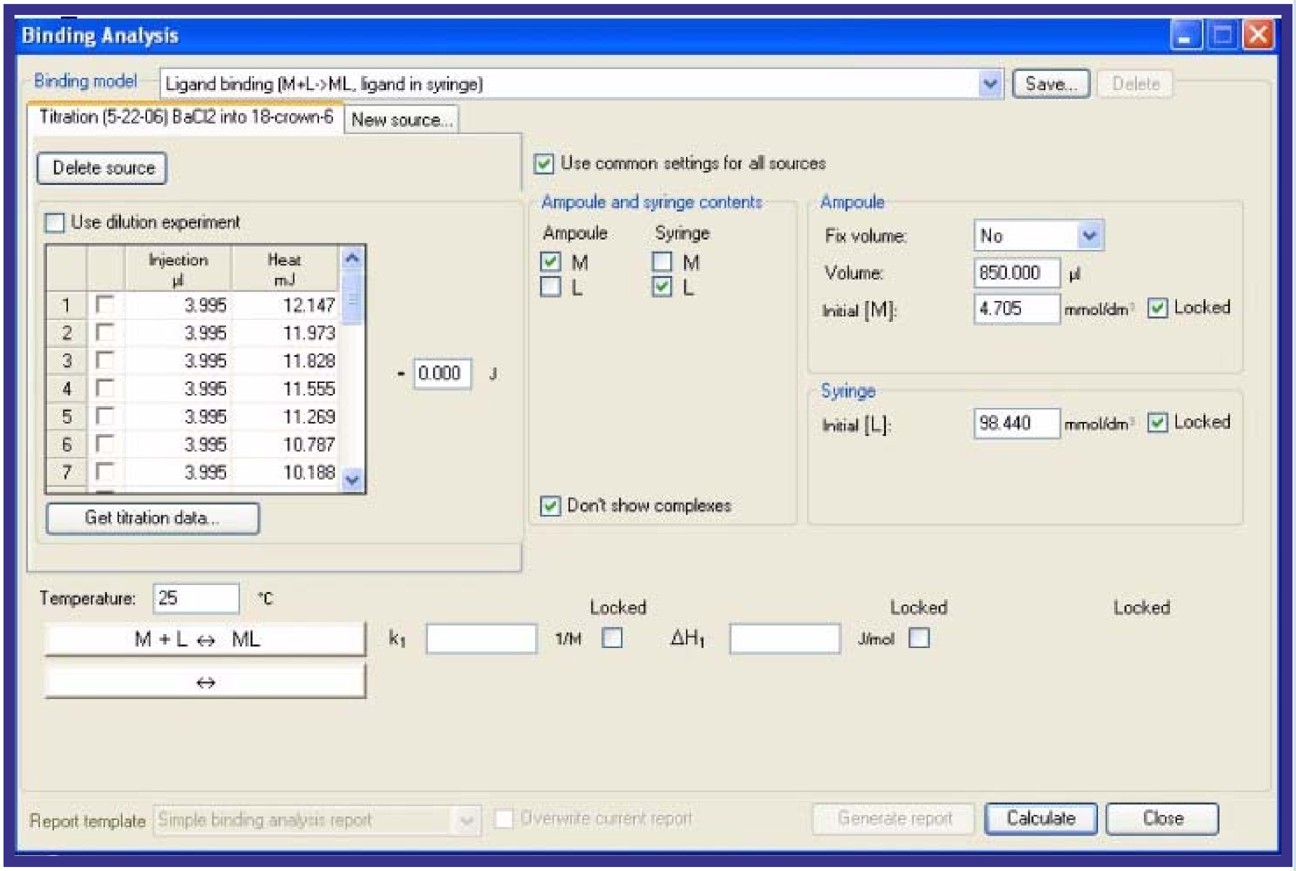
Follow these steps:

1. Choose **Ligand Binding...** from the **Analysis** menu to start the program. The dialog shown in the figure below is displayed.



2. Use the Browse button to find the file to be analyzed and click **OK**.

   The Binding Analysis form (shown on the next page) appears with the tabulated injection volumes and the integrated titration calorimetric data.



3. Add new sources by clicking **Get titration data...** then browse for the results file to be included.

4. Select additional input for the analysis: reaction volume, initial concentration in the ampoule and in the syringe.

   If this data has been included in the results file, it is imported automatically into the **Binding Analysis** dialog. Otherwise, you will need to add it manually.

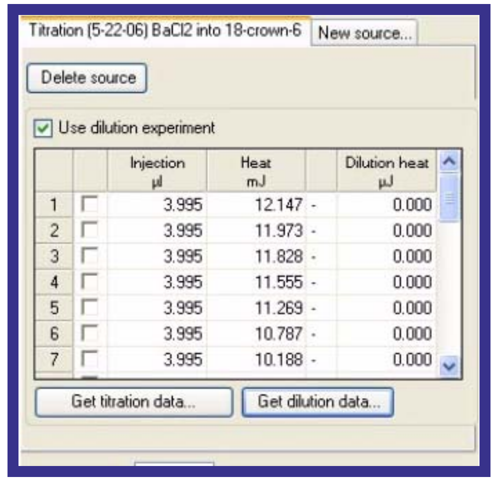
   Another input is the binding model, which can be changed with the buttons below the data table. The subscript **M** represents "Macromolecule," or host molecule, and **L** represents "Ligand."

   The *ligand* is considered to be the substance that is injected. This is the common terminology used in biophysics or biochemistry where the host molecule might be a protein and the ligand is a smaller molecule, for instance a drug compound. In metallo-organic chemistry for instance, the host molecule is considered to be a metal ion and the ligand(s) are larger organic molecules.

   The default model chosen is the most common model used, namely the 1:1 model. See the next section, "Choosing a Binding Model," for details on how to choose models.

   In many cases, correction for dilution effects of the injected substance should be made. The heat of dilution can be determined in a separate measurement and the obtained heat subtracted from that of the main result.

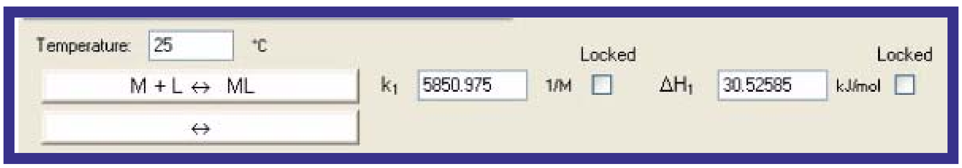
5. Check the box **Use dilution experiment** to subtract dilution heat. See the figure to the right.



6. Click the **Get dilution data...** button to import the dilution heat in a separate column of the table.

   If the dilution heat doesn't change significantly with the concentration interval used in the experiment, a single heat of dilution can be used instead. Enter this constant dilution heat in the text box to the right side of the table.

7. When all the settings have been made, click **Calculate.** The software will calculate the affinity constant and the apparent enthalpy change for the binding reaction.



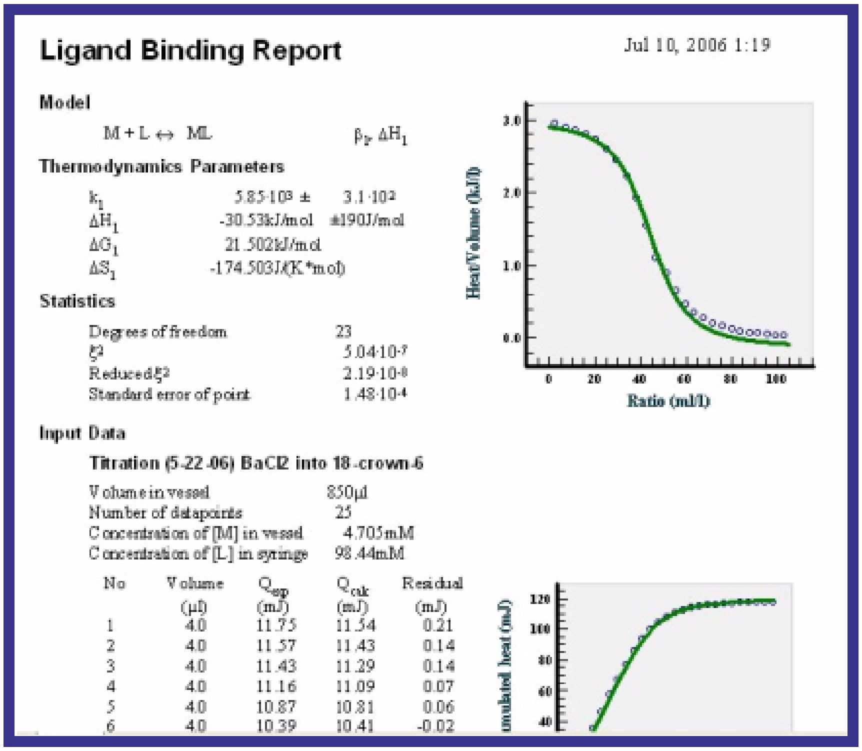
   You can "lock" or unlock" certain parameters in the form. The binding constant and enthalpy changes are commonly used fitting parameters and are, therefore, un-locked by default.

   However, if you have a good estimation of the binding constant, by other means of determination, you can choose to use only the enthalpy change as the fitting parameter.

   Alternatively, one of the initial concentrations might not be well known (*e.g.*, a concentration of an unstable protein). In this case you can unlock the protein concentration and this will be used as a fitting parameter. Hopefully you will have a good physical argument for doing this as adding fitting parameters to experimental data inevitably will give a better fit. Hence, adding a fitting parameter must be substantiated by solid scientific arguments.

8. Click **Generate report** when the calculation has been made, and a ligand-binding report (see the figure on the next page) is made with the estimated fitting parameters, together with the calculated free energy and entropy changes for the reaction.

   The plots in the report are heat per amount of injected volume versus the ratio of the injected volume to the initial volume. The lower graph shows the accumulative heat versus ratio of the injected volume to the initial volume. To refine the analysis it is possible to return to the Ligand binding form by choosing **Refine Analysis** under Report in the top menu.



Ligand Binding Report Jul 10, 2006 1:19

Model

M + L ↔ ML β_1, ΔH_1

Thermodynamics Parameters

k_1	$5.85 \cdot 10^3$ ±	$3.1 \cdot 10^2$
ΔH_1	-30.53kJ/mol	±190J/mol
ΔG_1	21.502kJ/mol	
ΔS_1	-174.503J/(K*mol)	

Statistics

Degrees of freedom	23
ξ^2	$5.04 \cdot 10^{-7}$
Reduced ξ^2	$2.19 \cdot 10^{-8}$
Standard error of point	$1.48 \cdot 10^{-4}$

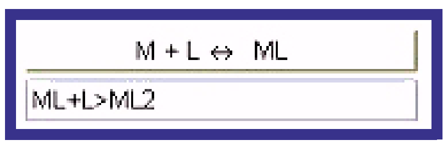
Input Data

Titration (5-22-06) BaCl2 into 18-crown-6

Volume in vessel	850µl
Number of datapoints	25
Concentration of [M] in vessel	4.705mM
Concentration of [L] in syringe	98.44mM

No	Volume (µl)	Q_{exp} (mJ)	Q_{calc} (mJ)	Residual (mJ)
1	4.0	11.75	11.54	0.21
2	4.0	11.57	11.43	0.14
3	4.0	11.43	11.29	0.14
4	4.0	11.16	11.09	0.07
5	4.0	10.87	10.81	0.06
6	4.0	10.39	10.41	-0.02

Choosing a Binding Model



Use the model buttons to choose different types of models. Click a button to choose the right the model as in the table. Single equilibrium steps with one affinity with different stoichiometry can be entered or, alternatively, two or several equilibrium steps as seen in the figure to the right.

The reaction is added as for example: ML+L>ML2 and entered by clicking anywhere outside the button area.

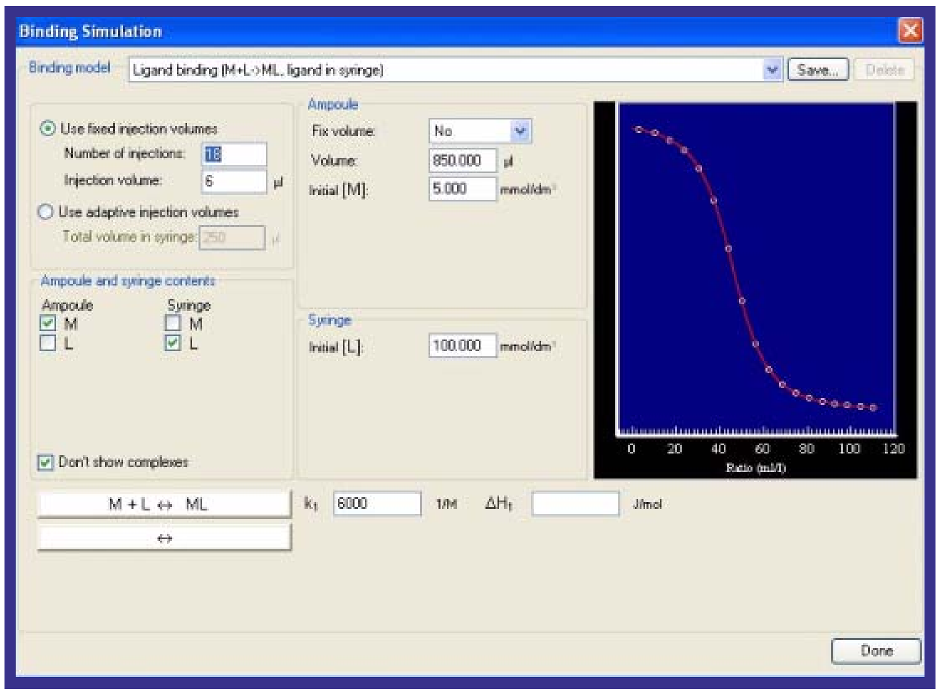
Ligand Binding Simulation

The Ligand Binding Simulation module is very useful when determining concentrations for the experiment. This simulation provides input to the use of adaptive injection volumes as determined in the experimental setup wizard.

Follow these steps:

1. Select **Ligand Binding Simulation...** from the **Analysis** menu. The following simulation view appears, in which the binding constant and concentrations is entered.

2. Choose the binding model as described in the previous section "Choosing a



Binding Model."

3. Set the ampoule concentration in relation to the magnitude of the binding constant. This is important to get a reasonably good estimation of the binding constant. For example, an acceptable estimation of the binding constant for a 1:1 stoichiometric binding reaction can be obtained, if the concentration is chosen in the following interval:

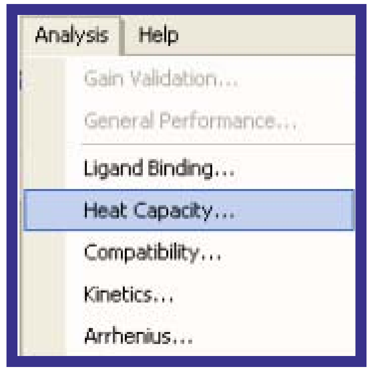
   10 < (concentration*binding constant) < 1000.

   This ensures that a sigmoidal shape of the binding curve as seen in the figure above can be obtained.

Heat Capacity Analysis

The heat capacity analysis function is used to analyze experimental data generated with the non-isothermal experimental wizard. The experiments can be run either in scanning mode or in step isothermal mode for heat capacity determinations.

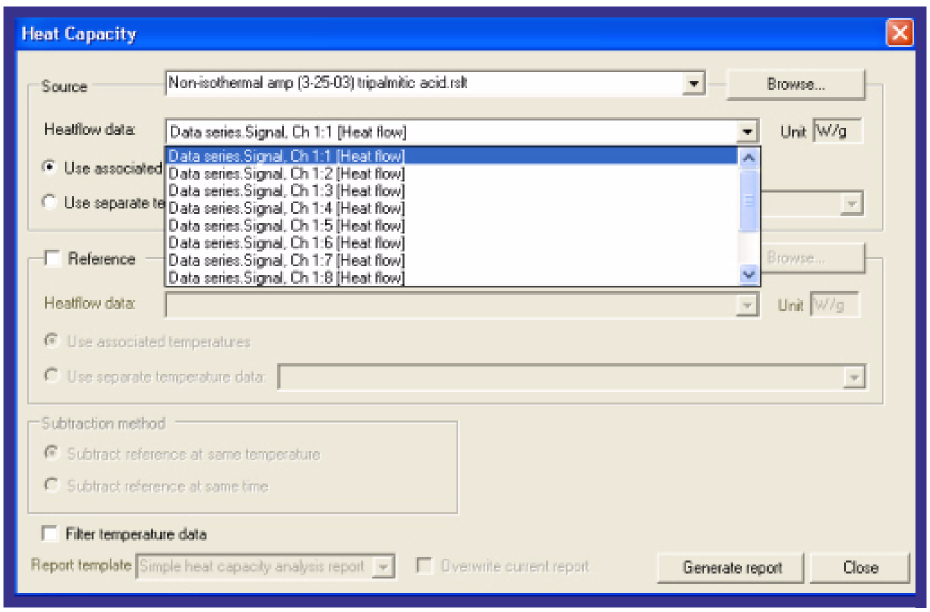
Follow these steps:



1. Select **Heat Capacity...** from the **Analysis** menu. The heat capacity form is displayed. See the figure below.

2. Click Browse to find the source file with the non-isothermal heat flow data.

3. For files containing data from several channels, choose which signals to include in the analysis from the drop down list labeled



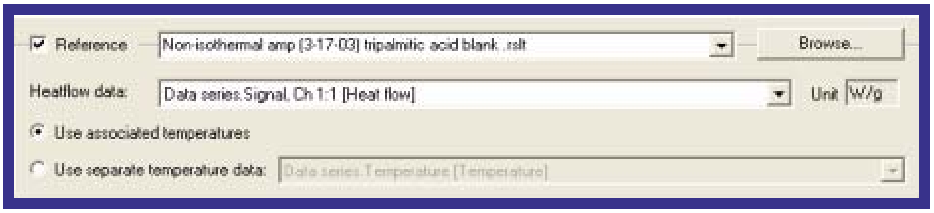
Heat flow data.

With each calorimetric heat flow signal there is a calculated associated temperature. This temperature is calculated from the measured temperature of the thermostat liquid and the thermal inertia of the calorimeter heat sinks to get a temperature that is close to the real temperature of the sample.

4. Choose the temperature to use in the analysis by clicking either **Use associated temperature** or **Use separate temperature data**. The separate temperature data is generally referred to the measured bath temperature.

   If a blank experiment has been run in a separate experiment, you can subtract the blank signal from the main. The best result is obtained if the blank has been run on the same calorimeter using the same temperature profile.

5. Check the **Reference** box and choose the results file and the corresponding



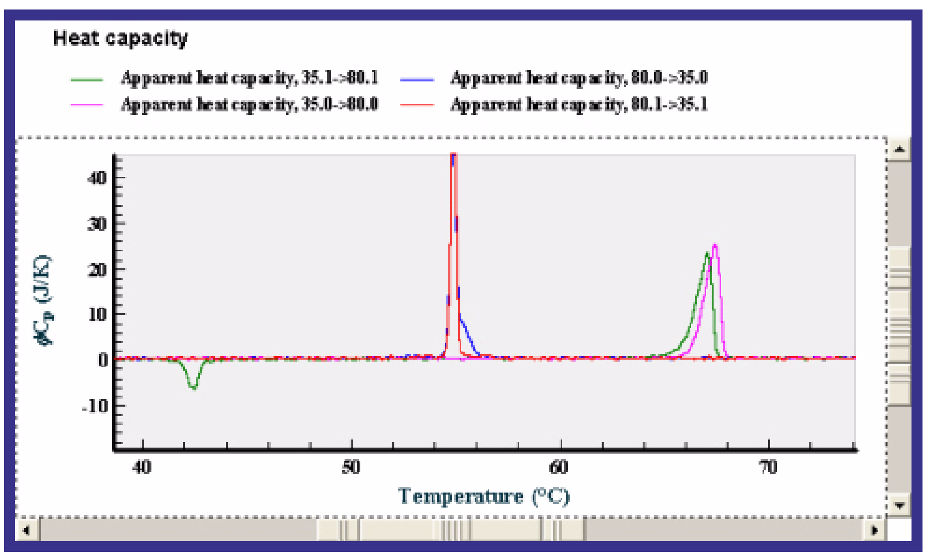
signal (calorimeter).

6. Choose the same temperature data for the reference and the sample data to obtain the best results.

7. Decide if you wish to subtract the heat flow data at the same time, or at the same temperature. For scanning experiments, you normally subtract at the same temperature to make sure that temperature dependent events are subtracted correctly.

   When calculating the heat capacity, the heat flow vector is divided by the chosen temperature vector. This is a mathematical operation that increases the noise level in the resulting heat capacity vector quite substantially as compared to the original heat flow and temperature data. For this reason a filter function has been added.

8. Click the **Filter** box before creating the report.



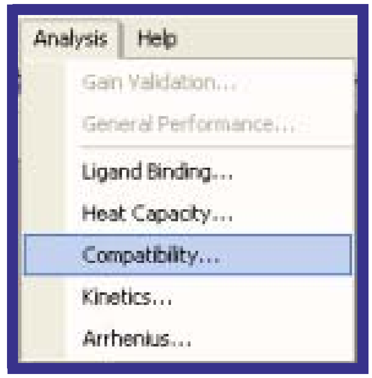
9. Click **Generate report** after all settings have been selected.

A plot of apparent heat capacity versus temperature is displayed, along with the sample information and source of the data.

The example shows an irreversible polymorphic phase transition and subsequent melting of Tripalmitic acid. Two pairs of up and down scans in the temperature interval 35 to 80 °C are shown and distinguished by the different colors.

Compatibility

The compatibility program is used to analyze heat flow data of mixtures. The sections below show the procedures for making compatibility analysis with TAM Assistant.

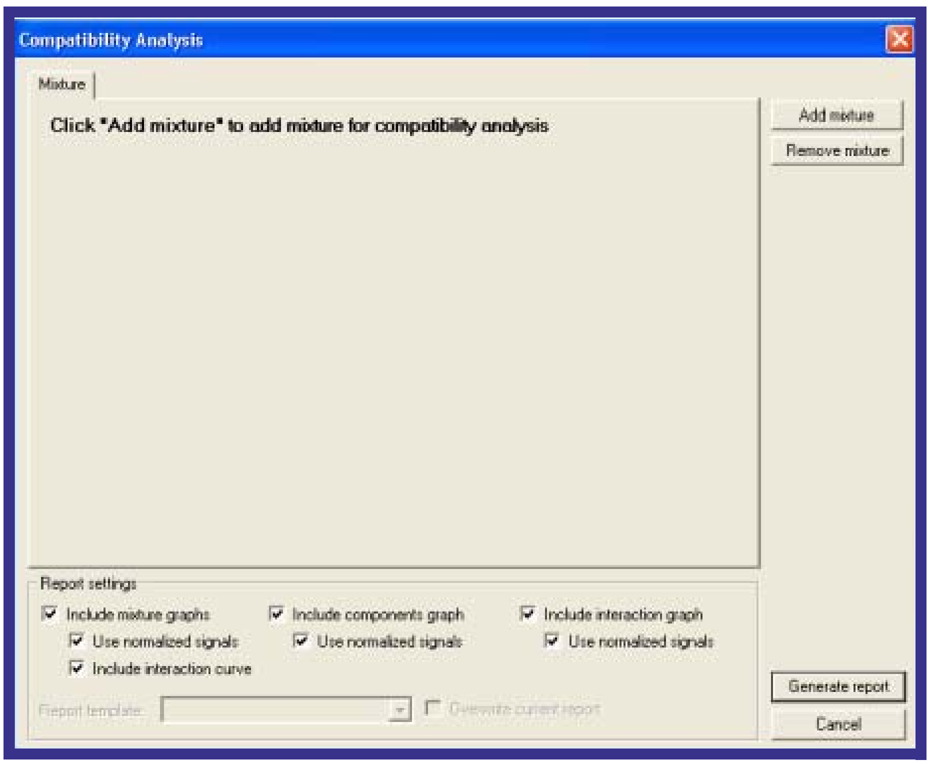


Loading Compatibility Data

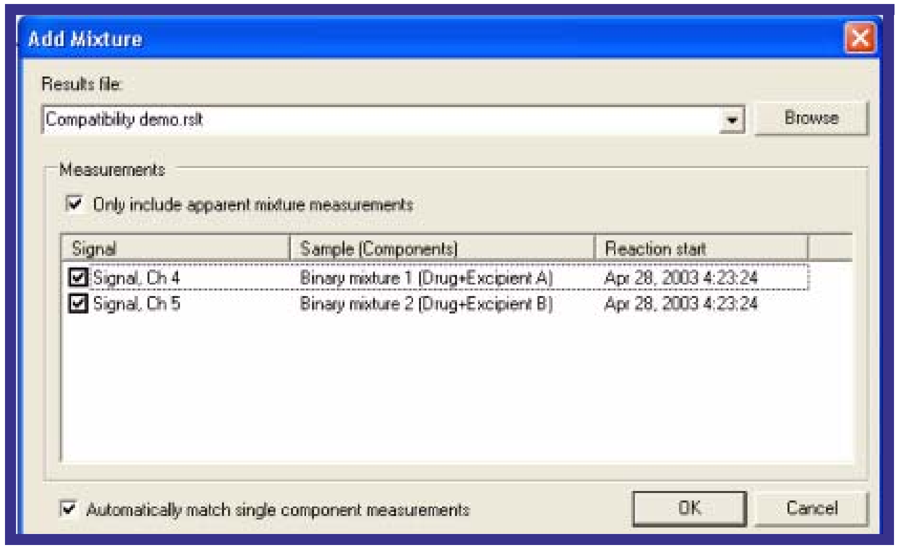
Follow these steps:

1. Select **Compatibility...** from the **Analysis** menu to start the compatibility program in TAM Assistant. The Compatibility Analysis dialog is displayed, see below.

2. Click the push button **Add mixture** to find the results file and load compatibility data.



The following dialog is displayed (see the next page) to let you choose which binary mixtures to be used in the calculation. If all component information has been entered in the results file, the program recognizes the signals (calorimeters) that represent the mixtures.

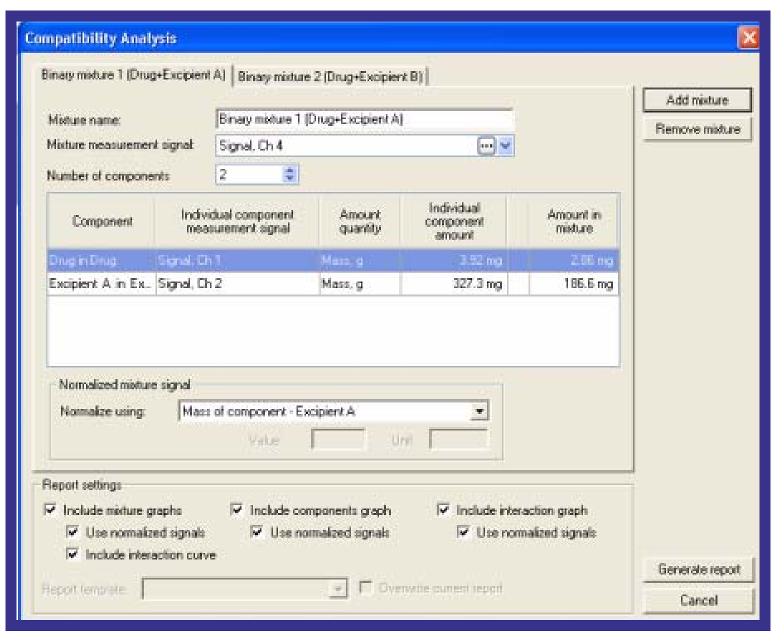


In the example given here, the name of the results file is "Compatibility demo.rslt." All measurements (components and mixtures) are contained in a single results file. In this example, a total of 5 measurements were made: 1) a drug component, 2) excipient A, 3) excipient B, 4) mixture of drug and excipient A and 5) mixture of drug and excipient B.

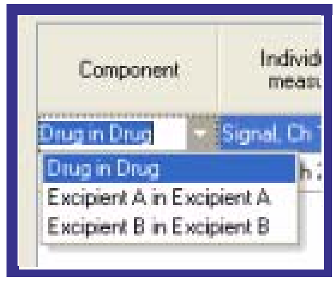
3. Choose the mixture signals to include in the calculation.

4. Click **OK** to import the data to the compatibility analysis.

The following dialog is displayed with the mixtures included in the experiment file and the individual components. See figure below. The software identifies the single component measurements with the mixture measurement. This is done automatically if you check, **Automatically match single component measurements**.



If **Automatically match single component measurements** is not checked, you need to match the single component measurements to the mixture measurements manually by choosing the correct data in the **Components** column, shown in the figure to the right.

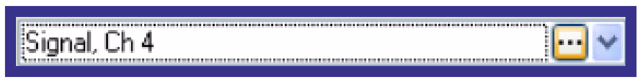


5. Use the tabs on top of the **Compatibility Analysis** dialog to choose the mixture that will have information displayed.

   In the component table, information about the individual components, from the single measurements and the mixture measurements, is shown.

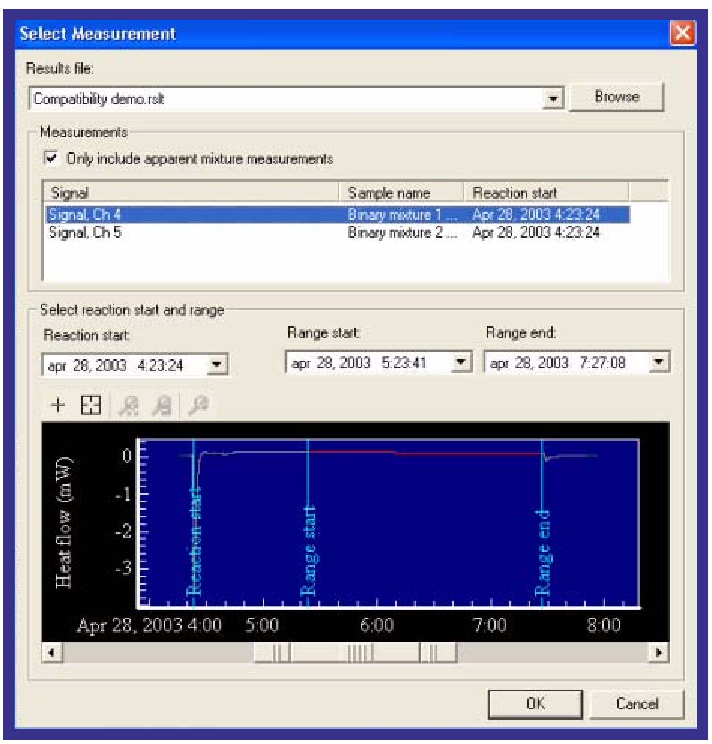
   The range over which the analysis shall be made can be determined from the heat flow plot for the different mixtures and components.

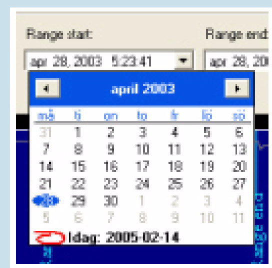
6. Click the three-dotted button on the right side of the text box named **Mixture measurement signal** to display a graph.



   The default range is the measurement period from the point at which the signal is considered correct after introducing the ampoule, to the time when the main experimental section was finalized and the sample was removed from the calorimeter.

7. Change the range for the individual signals, if desired, by moving the range markers back and forth along the time axis, or by using the drop- down menus above the graph.



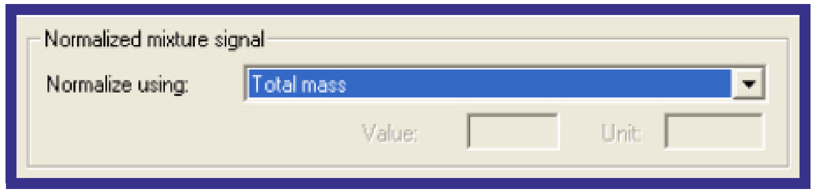


8. Repeat the procedure for all the signals related to the current mixture, before proceeding to the next mixture.

9. Press **OK** when the settings for all mixtures have been completed.

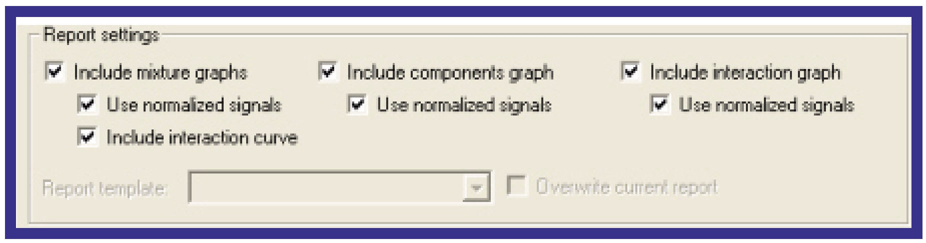
10. Normalize the mixture signal, taking into consideration the following points.

    - The signal is generally normalized with respect to the size of the sample (mass, molar mass or volume).



    - You can determine if the signal is to be divided by total sample size, size of an individual component in the mixture or any chosen factor and unit.

11. Determine what to include in the Compatibility report. There is a predefined template that can be used to assist you.

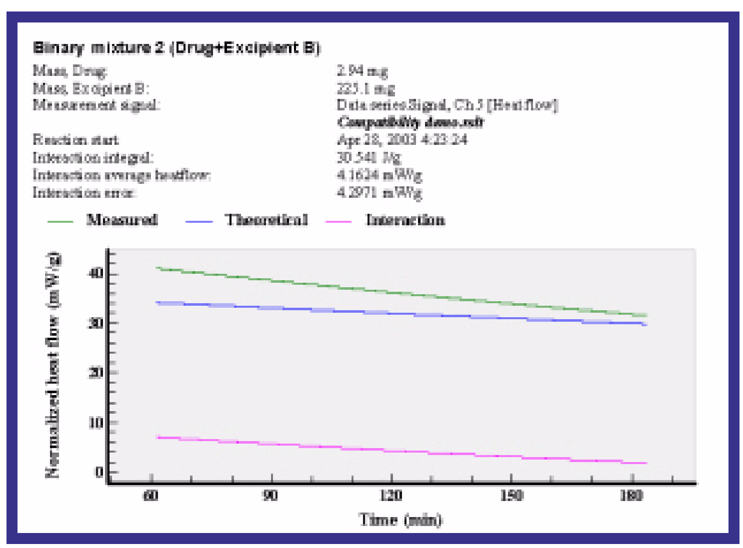


    This is done in the **Report settings** frame (shown in the figure above) where a number of boxes can be checked.

    - The box labeled **Include mixture graph** refers to one graph for each mixture. Each mixture graph contains three plots: *Measured*, *Theoretical*, and *Interaction*.

    - If the boxes **Include components graph** and/or **Include interaction graph** are checked, separate graphs will be created displaying the results from the respective individual components and the individual interaction plots.

12. Press the **Generate report** button to create the report when the settings are satisfactory. The figure to the right is an example showing the measured signal from one of the mixtures,



the theoretical curve and the interaction curve. The three calculated parameters from the data are: 1) interaction integral, 2) interaction average heat flow and 3) interaction error, *i.e.*, the standard deviation for the interaction (high slope, high interaction error).

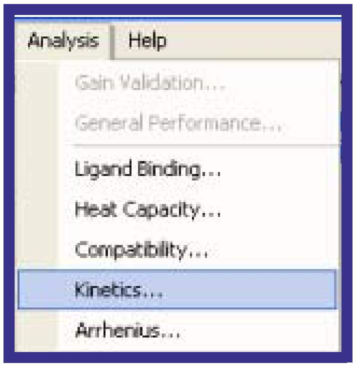
The different pages in the report can be accessed either with the triangular arrows or the page tabs at the lower left side of the graph window.

Kinetic Analysis

Since the heat production rate associated with chemical, physical and biological processes is related to the conversion rate of such processes, heat flow data can, in some cases, be analyzed with various kinetic models. In TAM Assistant you can import signals from results files into the kinetic analysis program for analysis with a variety of different models.

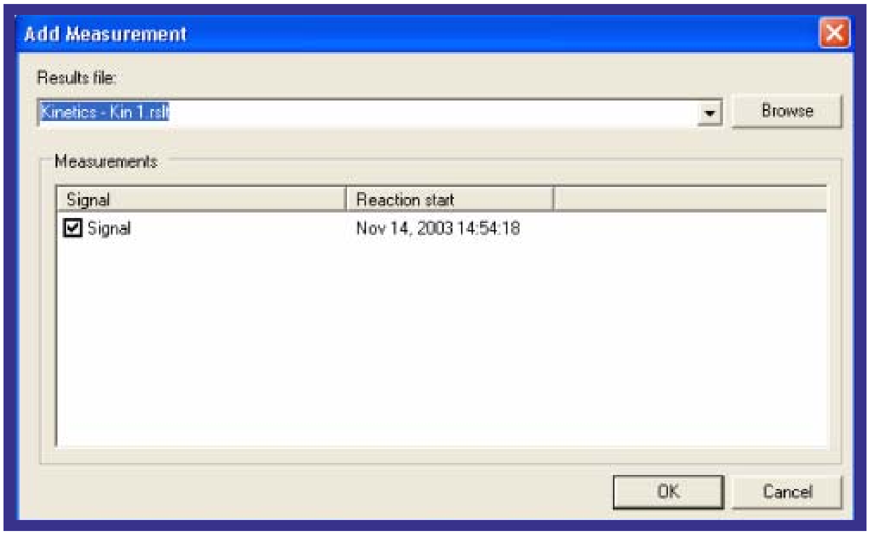
NOTE: You can try different models without changing the entered settings.

Follow these steps:



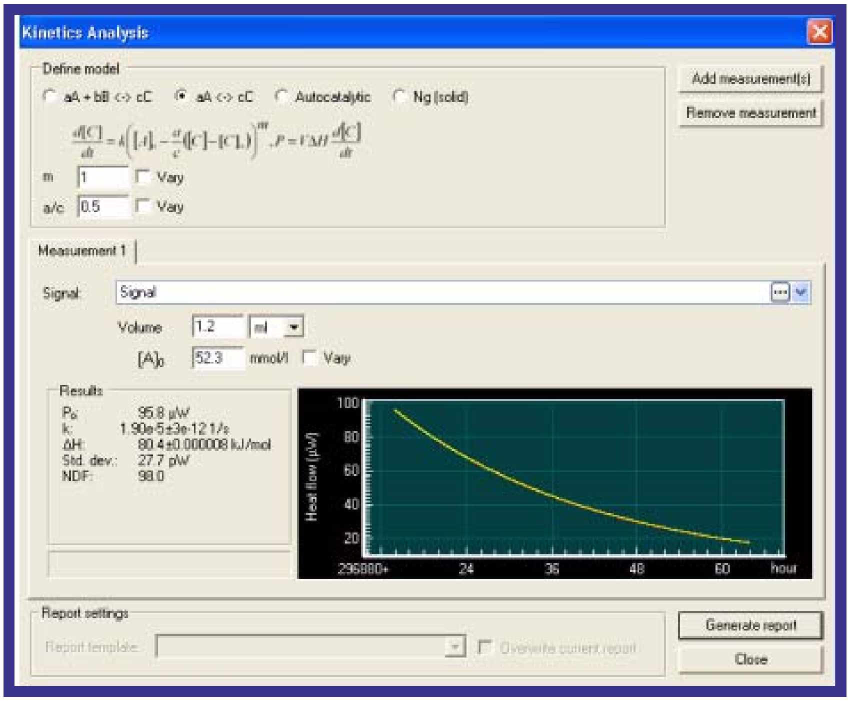
1. Select **Kinetics...** from the **Analysis** menu. When the Kinetic Analysis dialog is displayed, click the **Add measurement** button to find the results file to use for data extraction. See the figure below.

2. Browse the file system to find the results file, and mark which calorimeter signals should be included in the analysis (there is only one signal in the example).



3. Click **OK** when the choice has been made. The data is now loaded into the Kinetic analysis form and is seen in the graph window.

4. Enter details about the reaction, such as stoichiometric information (a/c and/or b/c), volume and concentration or amount. When all model input data has been entered the fitted constants are shown in the Results frame to the left of the graph. See the figure on the next page.



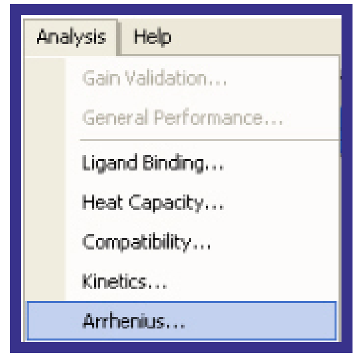
5. Adjust the time interval over which the fitting is to be made, if desired, by using the three dotted button on the **Signal** drop-down.

6. Click the **Generate report** button when all of the settings have been selected to create the report.

Arrhenius Analysis

Since heat production rates are generally related to the rate of chemical, physical or biological processes, temperature analysis by the Arrhenius model can be made on calorimetric data collected for a process at different temperatures.

Follow these steps:



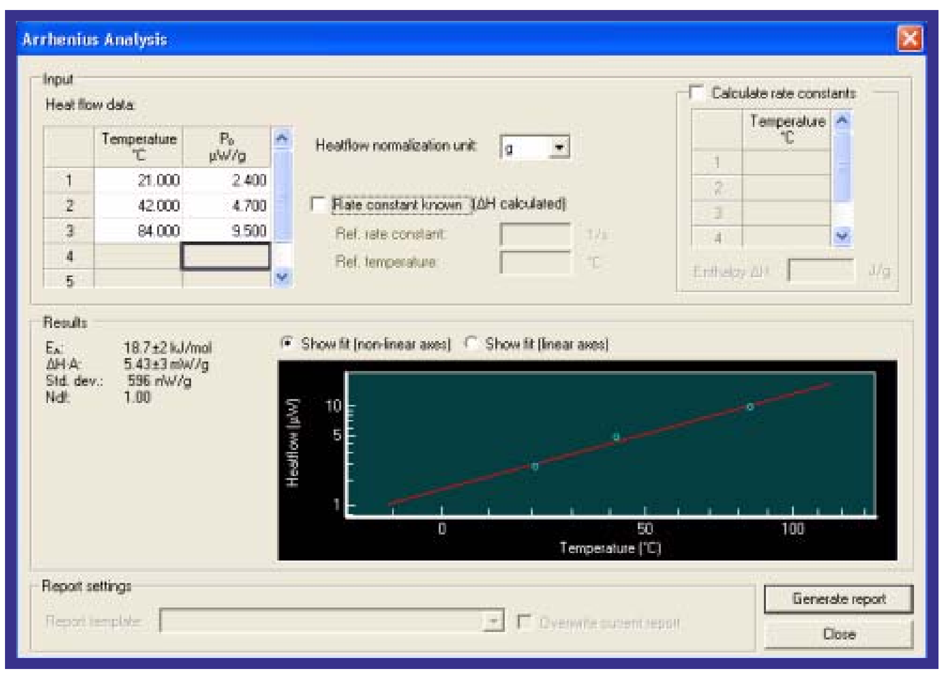
1. Select **Arrhenius...** from the **Analysis** menu, shown here.

2. Enter at least three representative experimental values for the heat production rate at a few different temperature values. You can choose heat flow values at time zero or a time later than zero, if the extent of the reaction is approximately the same for the different temperatures.

   If you choose heat flow values at a time later than zero, it is important that the heat flow values are chosen at a point where the **extent** of reaction is the same, *i.e.*, the integrated heat, up to the point where the heat flow data is taken, should be the same at the different temperatures.

3. Enter the temperatures in degrees Celsius. Heat production data is usually entered as normalized against sample size.

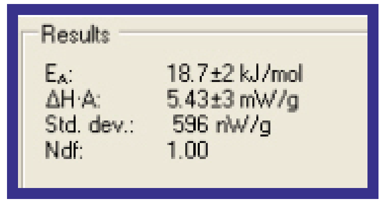
4. Choose the unit from the drop-down menu labeled **Heat flow normalization unit**.



In the plot, the scale on the ordinate is given as logarithmic in terms of heat flow. The unit on the abscissa gives the temperature in degrees Celsius. This scale is proportional to 1/T with the unit reciprocal Kelvin (K-1).

In the example on the previous page, three temperature values and three values for the heat production rate were entered.

The plot is made automatically when three values in both columns have been entered, and the result of the linear regression is shown in the **Results** frame.



If the analysis is made with the heat flow data only, the activation energy is gained from the slope and the term ΔH*A from the ordinate intercept.

If the enthalpy change for the reaction, ΔH, is known, and can be assumed to be independent of temperature, the pre exponential factor, A can be calculated. This enables the rate constants for the reaction to be calculated at different temperatures by use of the conventional Arrhenius equation. Naturally, the rate constant is only relevant if the mechanism of the reaction is known, *e.g.*, first order kinetics.

5. Press **Generate report** to generate the Arrhenius report.

Chapter 5:

TAM Assistant Security

Introduction

The TAM Assistant Security system provides user and permissions management for devices and experiments controlled by TAM Assistant. It also provides audit trails for keeping track of all changes to devices and all experiments.

The following functionality is provided:

- **User and group management**
  The security system includes a built-in user database that can be used to define the users that have access to the system, as well as define groups of users for easier permissions management. Additionally, the security system can be integrated with Active Directory, allowing the standard Windows login to be used.

- **User login**
  When accessing an instrument that is connected to the security system, the user needs to login to the system. It is the responsibility of the security system to authenticate the user. The authentication can be performed either against the built-in user database or, if Active Directory integration is enabled, using the standard Windows login. If Active Directory integration is enabled, it is possible to automatically login as soon as the TAM Assistant software is started, without providing any additional credentials.

- **Permissions management**
  Permissions can be assigned to all parts of the security system. Securable objects (*i.e.*, devices, instruments or the security system itself) are arranged in a hierarchical structure. Permissions can be assigned for the whole system, an instrument or even down to a specific calorimetric channel.

- **Audit trails**
  Another part of the security system is the audit trails. The audit trails are used to keep track of all events that happen on the system. The events can for instance be: setting new temperature on a thermostat, performing a calibration, starting an experiment, or any system problem. Each entry in the audit trail contains information like time, user, severity, and a description. The entries can be filtered based on different criteria and exported to a file or sent to a printer.

- **CFR Part 11 support**
  The above functionality provides the basis for implementing a system that is compliant with the CFR Part 11 regulations.

Setting up TAM Assistant Security

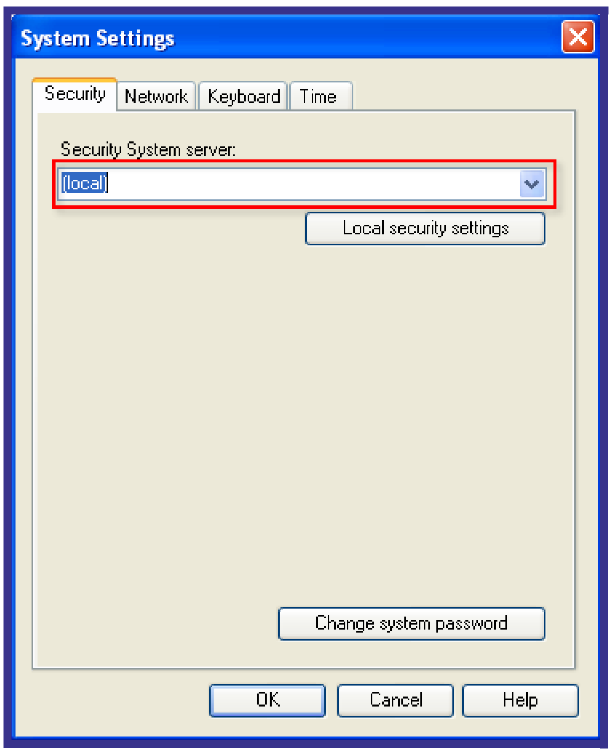
To use the security system, TAM Assistant Security has to be available. There are two options for setting up the security system. Either the security system embedded on the TAM III instrument can be used, or it can be installed on a standalone computer.

Using TAM Assistant Security on a TAM III Instrument

If the instrument setup consists of a single TAM III instrument, the easiest way to set up TAM Assistant Security is to use the embedded security system on the TAM III instrument. The embedded security system is identical to the security system that can be installed on an external computer, with the limitation that Active Directory integration is not available.

To enable the embedded security system, perform the following steps:

1. Select **System** settings from the **Settings** menu on the TAM III.



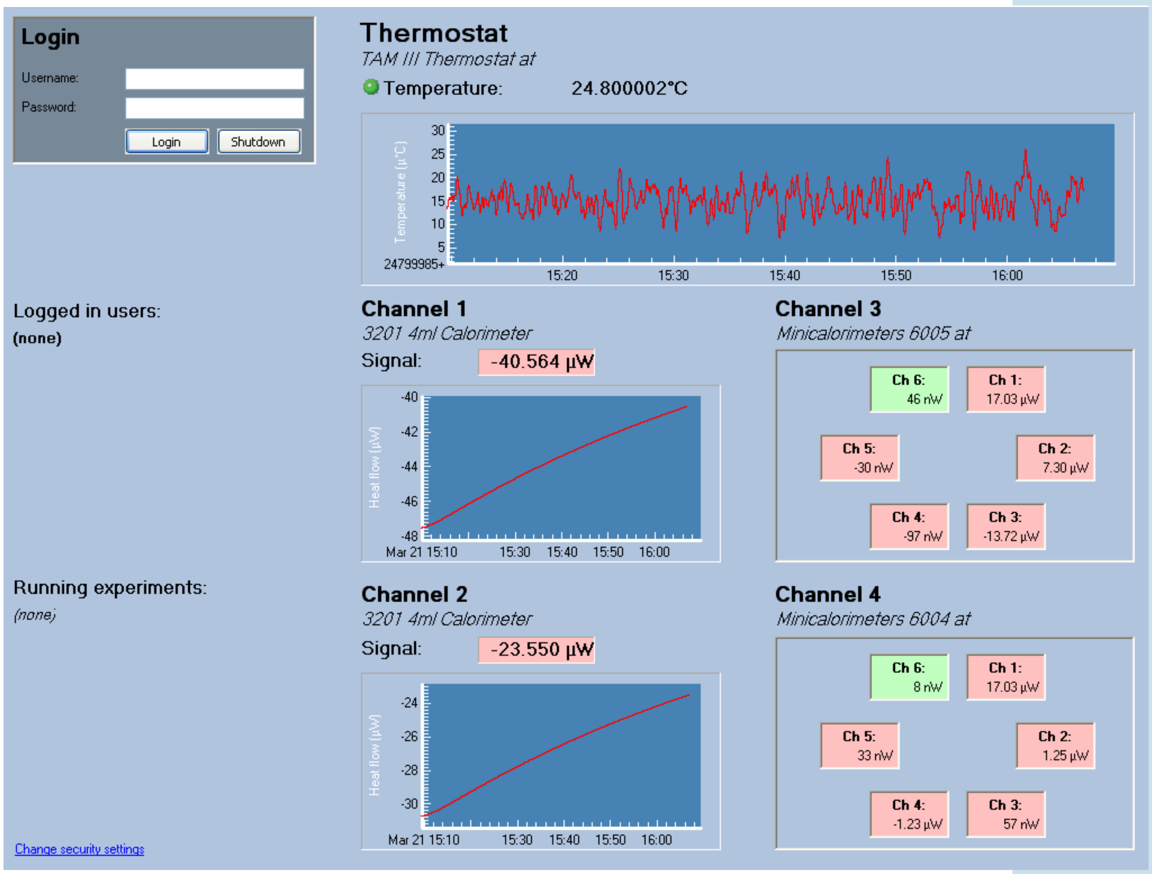
2. Under the Security page, select “(local)” as the **Security system**.

3. Click **OK**. A prompt for the system password will appear. The system password is used to protect the TAM III system from unauthorized changes. However, the default password is empty, so unless a system password has been previously set, no password needs to be entered.

4. The first time the embedded security server is used it needs to be initialized. The security server is initialized by adding an

Administrator account to the user database, as well as the default **Administrators** and **Users** groups. Members of the Administrators group are assigned full permissions to the whole security system. To be able to initialize the user database, the password for the **Administrator** needs to be defined.

5. After entering the **Administrator** password, the system will restart. On the next startup, the system overview and login screen will appear.



The overview screen provides information about the thermostat and all calorimeters installed in the TAM III, as well as information about currently logged in users and running experiments.

6. To login to the TAM III system, enter “Administrator” as the user name and specify the password defined when initializing the embedded security system.

For information on how to setup additional users, see the sections “Settings up Users and Groups” on page 129 and Assigning Permissions on page 137.

NOTE: If a previous version of TAM Assistant or TAM Assistant has been installed before, it is highly recommended that it is uninstalled before installing the new version.

Installing TAM Assistant Security on a Standalone Computer

If more than one TAM III instrument should be connected to the security system, or if Active Directory integration is wanted, it is recommended that the TAM Assistant Security system is installed on a standalone computer. The security system can be installed on the same computer as the TAM Assistant software, but if possible it is recommended that it is installed on a server computer which is always running.

To install the TAM Assistant Security software, perform the following steps:

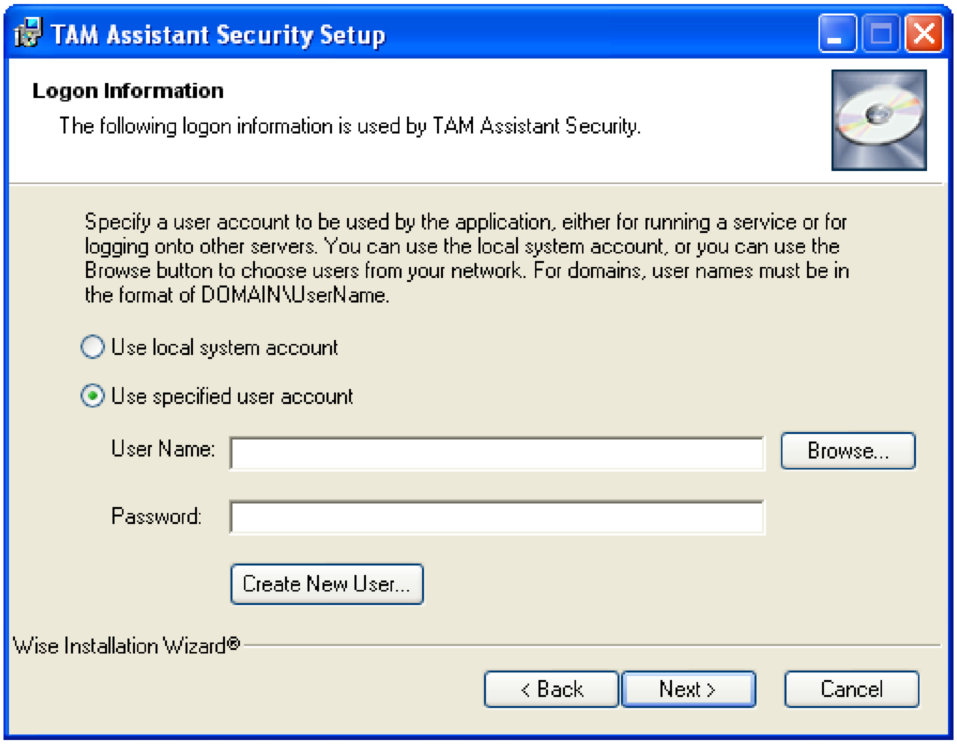
1. Insert the TAM Assistant CD into the computer. The installation start screen should appear automatically.

2. Click **Install TAM Assistant Security.**

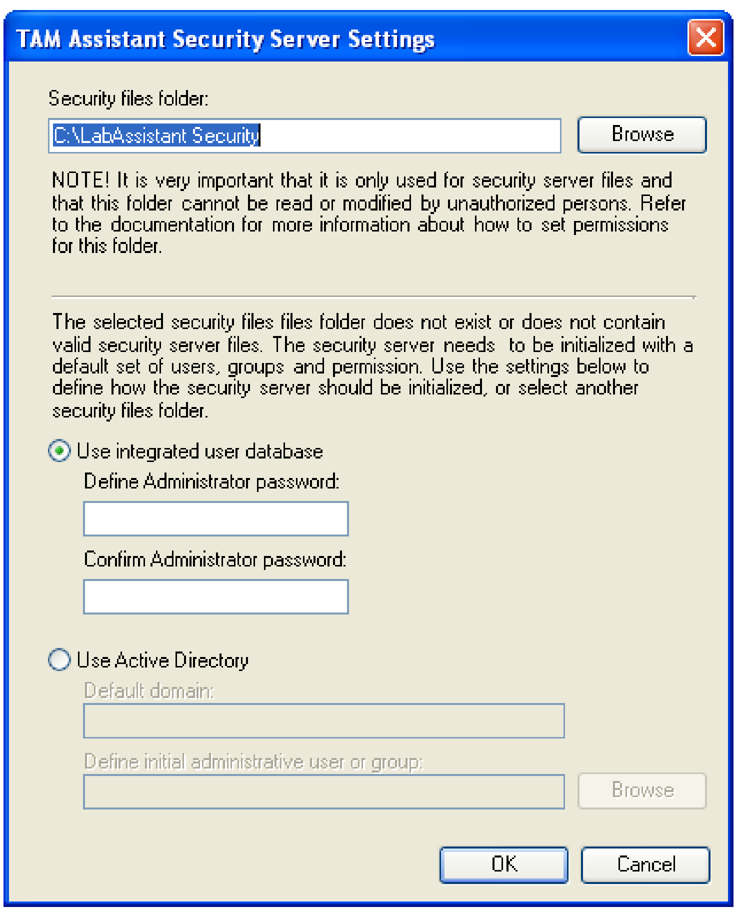
   If the installation start screen didn't appear in step 1, select **Run** from the Windows **Start** menu, enter "D:\ServerSetup.exe" (where D is the driver letter for the CD-drive) and click the **OK** button.

3. The TAM Assistant Security installation wizard is started. Follow the instructions provided by the wizard until the Logon Information page is reached.

4. On the Logon Information page, the account to use for the TAM Assistant Security service should be selected. It is possible to select either the built-in local system account, or to select or create a normal user account.



5. If TAM Assistant Security has not been previously installed, the security settings window will appear at the end of the installation.



Here the folder where the security files should be stored is specified. The folder should either be a folder containing TAM Assistant security files from a previous installation, or it should be an empty folder. For security reasons, the selected folder should not be used for any other data. For more information, see “Security Considerations and Backup of Security Data” on page 151. If the specified folder does not contain any security files, a first time initialization must be performed.

The security system can be initialized to use either the built-in user database, or to enable Active Directory integration.

- **Use built-in user database**
  This option enables the built-in user database. The database will be initialized with a single **Administrator** account, an **Administrators** group and a **Users** group. The Administrators group will be assigned full administrative rights to all objects in the security system.

  To initialize the Administrator account a password must be specified using the **Define Administrator password** and **Confirm Administrator password** fields.

- **Use Active Directory integration**
  This option enables the integration with standard Windows user accounts and groups using Active Directory. All users and groups that are defined in Active Directory are available to the security system.

- **Default domain**
  Defines the domain that should be used when looking up users and accounts. If the standard domain should be used (i.e., the domain on which the security server is installed), this field can be left empty.

- **Define initial administrative user or group**
  Defines the user account or a group that should initially be assigned full administrative rights to the security system.

6. Finish the installation.

7. The installation is finished.

- Use the TAM Assistant Security Manager to set up users and permissions. For more information, see “TAM Assistant Security Manager” on page 129.

- Connect the TAM Assistant software and TAM III instruments to the security system. For more information, see “Connecting a TAM III Instrument to a Standalone Security System” and “Connecting External Computers Running TAM Assistant to the Security System” in this chapter.

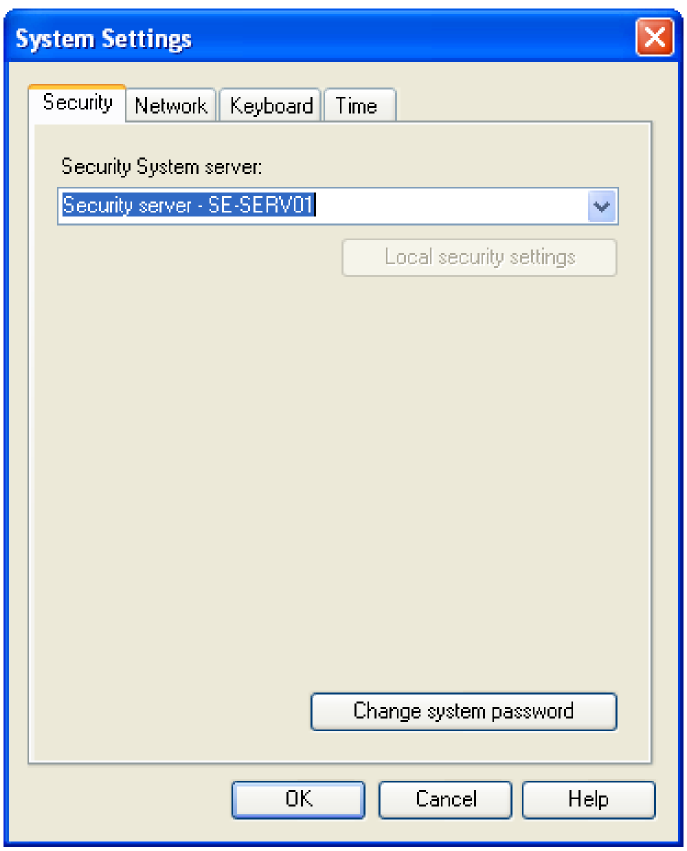
Connecting a TAM III Instrument to a Standalone Security System

To enable the security system on a TAM III instrument, the security server to use must be defined. If the embedded security server should be used, follow the instructions under the "Using TAM Assistant Security on a TAM III Instrument" section on page 120.

To enable the embedded security system, perform the following steps:

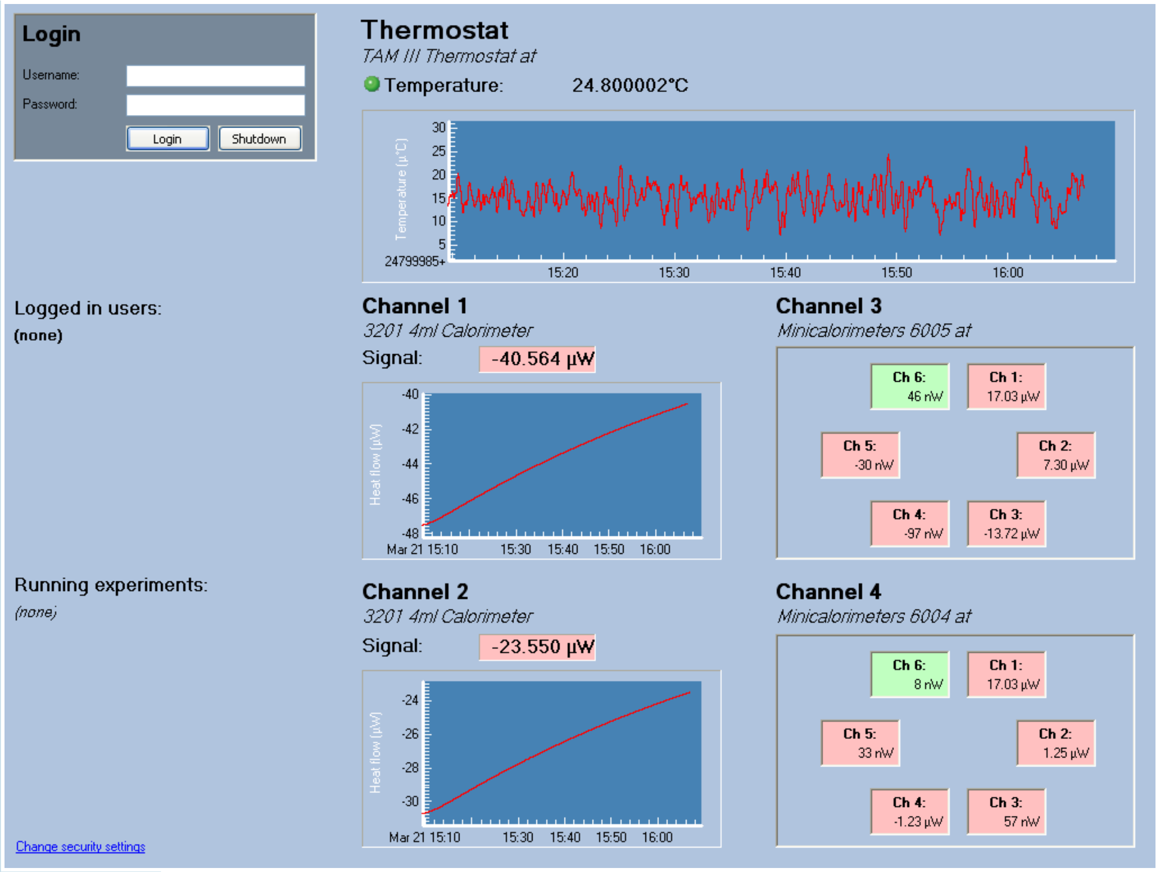
1. Select **System** settings from the **Settings** menu on the TAM III.

2. Under the Security page, select the wanted **Security system**.



3. Click **OK**. A prompt for the system password will appear. The system password is used to protect the TAM III system from unauthorized changes. However, the default password is empty, so unless a system password has been previously set, no password needs to be entered.

4. After entering the system password, the system will restart. On the next startup, the system overview and login screen will appear.



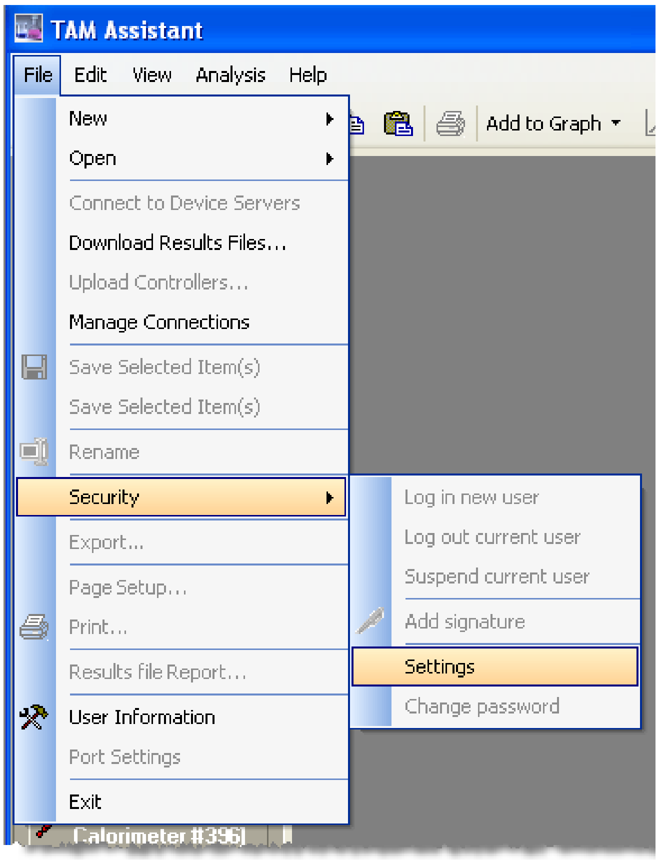
The overview screen provides information about the thermostat and all calorimeters installed in the TAM III, as well as information about currently logged in users and running experiments.

5. Use the **Login** area to enter the user name and password to login to the TAM III system.

Connecting External Computers Running TAM Assistant to the Security System

To be able to use the TAM Assistant software to connect to an instrument connected to the TAM Assistant Security system, the TAM Assistant software must be connected to the same security server. To select security system server for TAM Assistant, perform the following steps:

1. Select **Settings** from the **File-Security** menu in TAM Assistant.

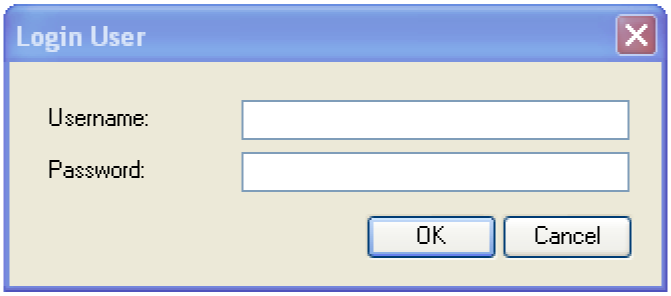


2. In the **Security Settings** window, select the wanted **Security system**.

   For information about the other options, see “Security Settings” on page 148.

3. Click OK. The selected security server will be used the next time TAM Assistant is started.

4. Exit TAM Assistant and restart it. If the **Automatically login at startup** option is enabled, a login screen will appear.



   If the **Automatically login at startup** option is not enabled, it is necessary to use the **Log in new user** command under the **File/Security** menu.

5. Use the **Login User** screen to enter the user name and password to login to the TAM Assistant Security system and gain access to secured instruments.

TAM Assistant Security Manager

The TAM Assistant Security Manager is used to make define security settings, work with users and groups, and assign permissions. It is installed together with the TAM Assistant Security system. It can also be installed by doing a custom install of TAM Assistant. For more information, see the TAM Assistant installation instructions.

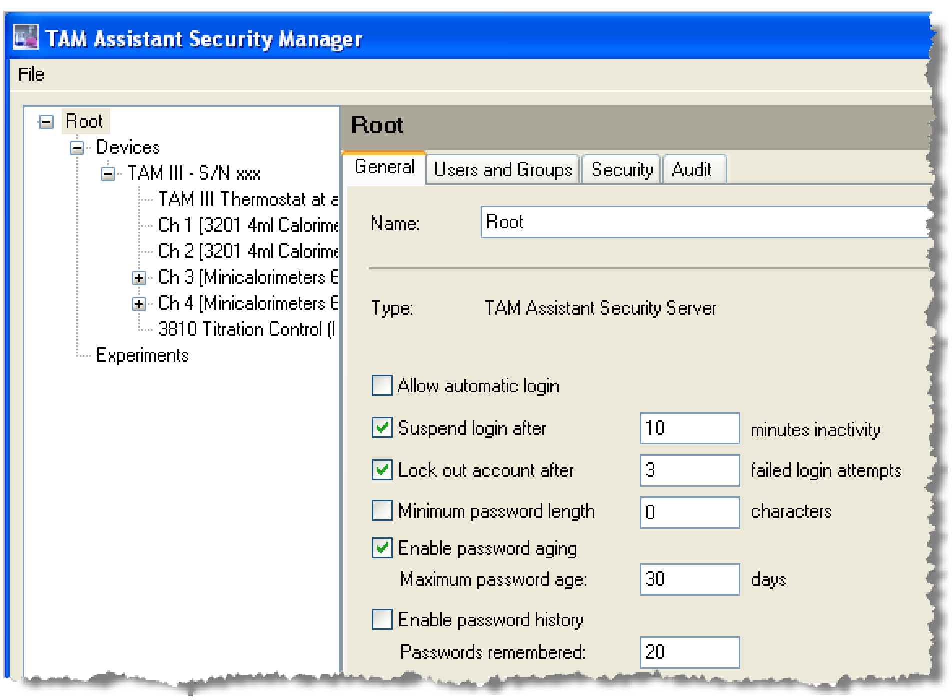
The TAM Assistant Security Manager can be started from the Windows Start menu or it can be accessed from a TAM III instrument, using the **Security Manager** command under the **Security** menu.

Connecting to a Security Server

If TAM Assistant Security Manager is started on a standalone computer, the security server to connect to must be specified. To define the security server, the **Connect** command under **File** menu should be used. This will bring up a window that provides the possibility to select one of the available security servers. The selection window will present all available security system servers. If the security managed is running on the computer where the TAM Assistant Security Server is running, one addition option will also be present: **<Direct>.** This option will connect the security manager directly to security server files, bypassing the login to the security system. Only users which have access to the security files can use this option. For more information, see "Security Considerations and Backup of Security Data" on page 151.

The Security Manager Window

The security manager window contains two panes. The left pane presents all available security objects (*e.g.*, devices and the security server itself) in a tree hierarchy. This tree can be used to select the object that should be viewed or edited.



Depending on the selected object in the tree, the right pane shows the available security properties for the object. The properties are divided into several pages. All items include the Security and Audit pages. The top-level item (which represents the security system itself) includes additional pages: **General** and **Users and Groups**.

Apply

This button is used to apply the changes made to the security settings or user permissions.

Cancel

This button is used to cancel any changes made to the security settings or user permissions.

Security Server Settings

The global settings for the security system can be accessed by selecting the top-level item in the navigation tree, and then selecting the **General** page. This page provides access to the settings:

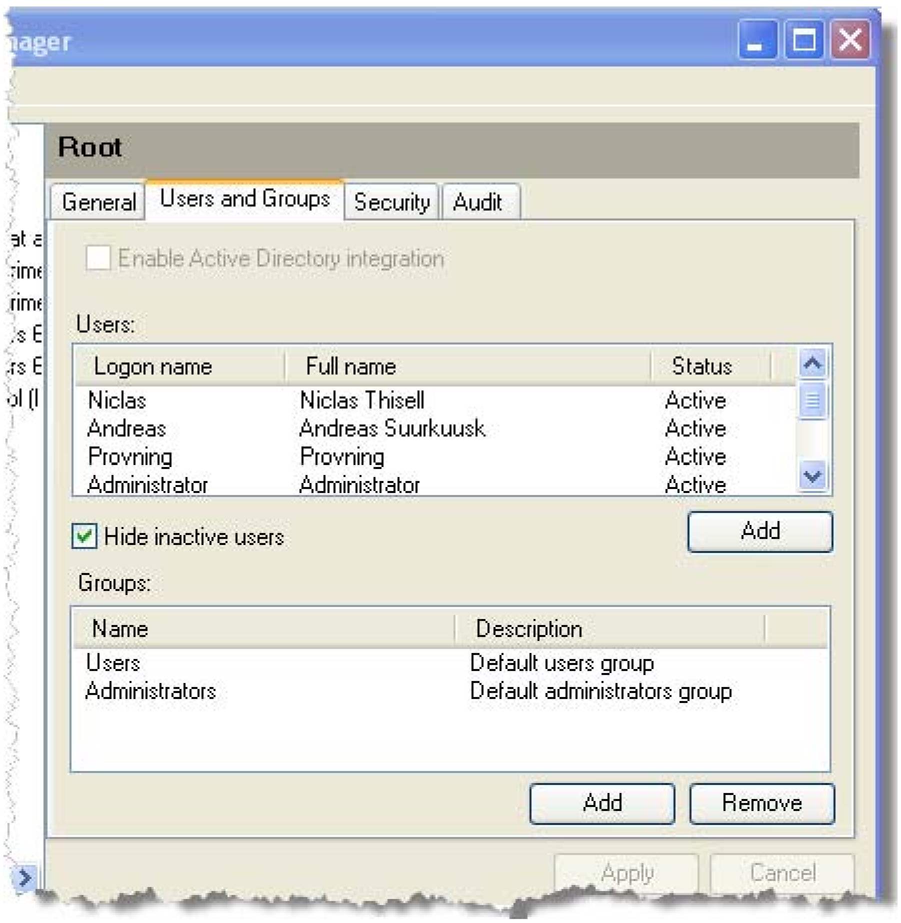
- **Name**: This field is used to define the name of the security system. The default name is "Root" and it can be changed to any suitable name.
- **Allow automatic login**: This field is used to indicate whether TAM Assistant is allowed to login automatically using the current Windows credentials. Automatic login is only possible if Active Directory integration is enabled.
- **Suspend login after ... minutes inactivity**: This setting is used by the TAM Assistant Software and the TAM III instrument. It defines the number of minutes of inactivity is allowed before suspending the current login. After the login has been suspended, the user name and password needs to be reentered to continue to use the TAM III instrument or TAM Assistant software.
- **Lock out account after ... failed login attempts**: This field defines the number of failed logins that are allowed before locking out a user account. For information about how to re-enable a locked out account, see "Setting up Users and Groups" on page 131.
- **Minimum password length**: Defines the minimum allowed length of a user password. This setting is only used if the integrated user database is active.
- **Enable password aging**: This field is used in conjunction with the **Maximum password age** field to define the maximum age of a password. If this option is enabled and a user logs in with a password that is older than the specified age, the user will be prompted to enter a new password. This setting is only used if the integrated user database is active.

- **Enable password history**: This field is used in conjunction with the **Passwords remembered** field to define the password history. If this option is enabled, then the **Passwords remembered** field is used to define the number of passwords that are stored for each user. When assigning a new password, it is not allowed to use a password that exists in the password history. This setting is only used if the integrated user database is active.

Users and Groups

In order for a user to login to the system, information about the user must be added to the system. The TAM Assistant Security system keeps track of all users, either by using a built-in user database or by using existing user accounts in Active Directory.

To simplify permissions management, it is also possible to combine users into user groups. The user groups are either handled by the built-in user database, or by Active Directory, if Active Directory integration is enabled.



The **Users and Groups** page provides access to the users and groups that have been added to the security system. This page can be used to add or edit users, add or edit groups and assign members of groups.

To change the settings on this page, the logged in user must have the **"Change security settings"** permission on the root object. For more information, see "Permissions and Roles" on page 140.
The settings on the next pages are available on the **Users and Groups** page.

Enable Active Directory Integration

This option indicates whether Active Directory integration is enabled. It can only be changed if the security manager is connected directly to the security data.

Default Domain

This field is only available if Active Directory integration has been enabled. It can be used to define the domain that should be used when looking up users and accounts. If the standard domain should be used (*i.e.*, the domain on which the security server is installed), this field can be left empty.

Use All Users and Groups from Active Directory

If this option is enabled, it is possible to log in to the system using any available Active Directory user account. If the user has not previously logged in to the system, it will be automatically added to the list of users.

Only Use Selected Users and Groups from Active Directory

If this option is enabled, only users whose account has been explicitly added to the user list can login to the system. For more information on adding users, see “ Editing Users” on page 133.

Users

This list contains all users that are registered with the security system. New users can be added using the **Add** button below to list and user information can be viewed and edited by double-clicking a user entry. For more information, see “Adding New Users” on page 175.

Hide Inactive Users

If this option is enabled, then only active users will be listed in the **Users** list.

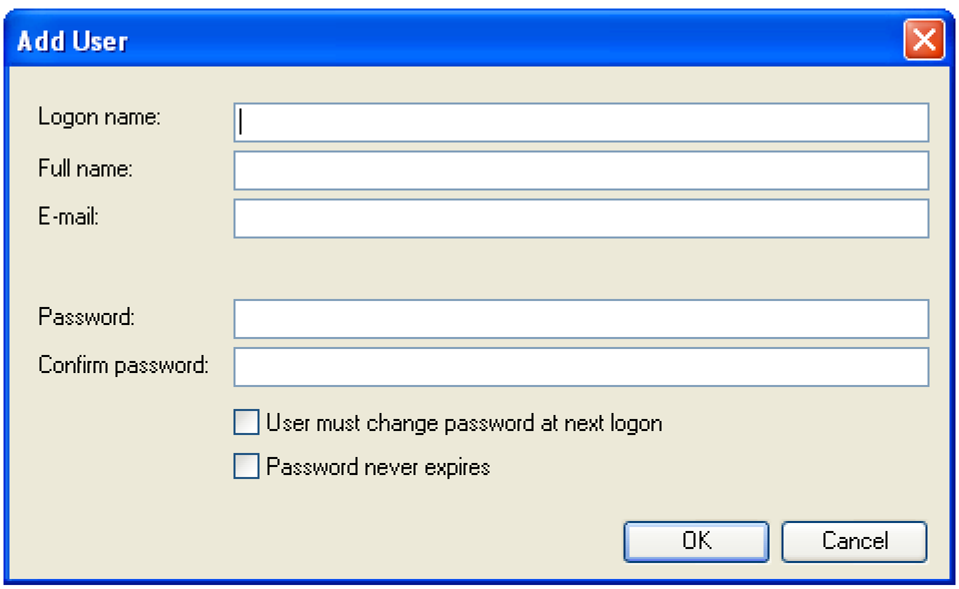
Groups

The Groups list contains all groups that are registered with the security system. A group can be used to assign permissions to a group of users (and groups), thus simplifying permissions management. If Active Directory integration is enabled, the group list is not available, however, all groups in Active Directory are available when assigning permissions. For more information about groups, see “Working with User Groups” on page 137.

Adding New Users

New users can be added to the security system by using the **Add** button under the **Users** list. To add new users, the logged in user must have the **“Modify users”** permission on the root object. For more information, see “Permissions and Roles” on page 140.

If Active Directory integration is enabled, a user selection screen will appear, allowing the selection of an Active Directory user to add. For more information on selecting the user, see “User and Group Selection” on page 138.
If the built-in user database is enabled, the **Add User** screen will appear.



This screen is used to define the most important settings for the new user.

Logon Name

This is a mandatory field where the logon name of the user should be entered. The logon name is used to identify the user when logging in to the system.

Full Name

Use this field to enter the full name of the user. The full name is used when presenting the name of the user, for instance in audit trails entries.

E-mail

This field can be used to enter the e-mail address of the user.

Password/Confirm Password

The password fields are used to define the initial password for the user.

User Must Change Password at the Next Logon

This option indicates that a new password should be requested the next the user logs in to the system. It is recommended that this option is enabled when the password has been reset.

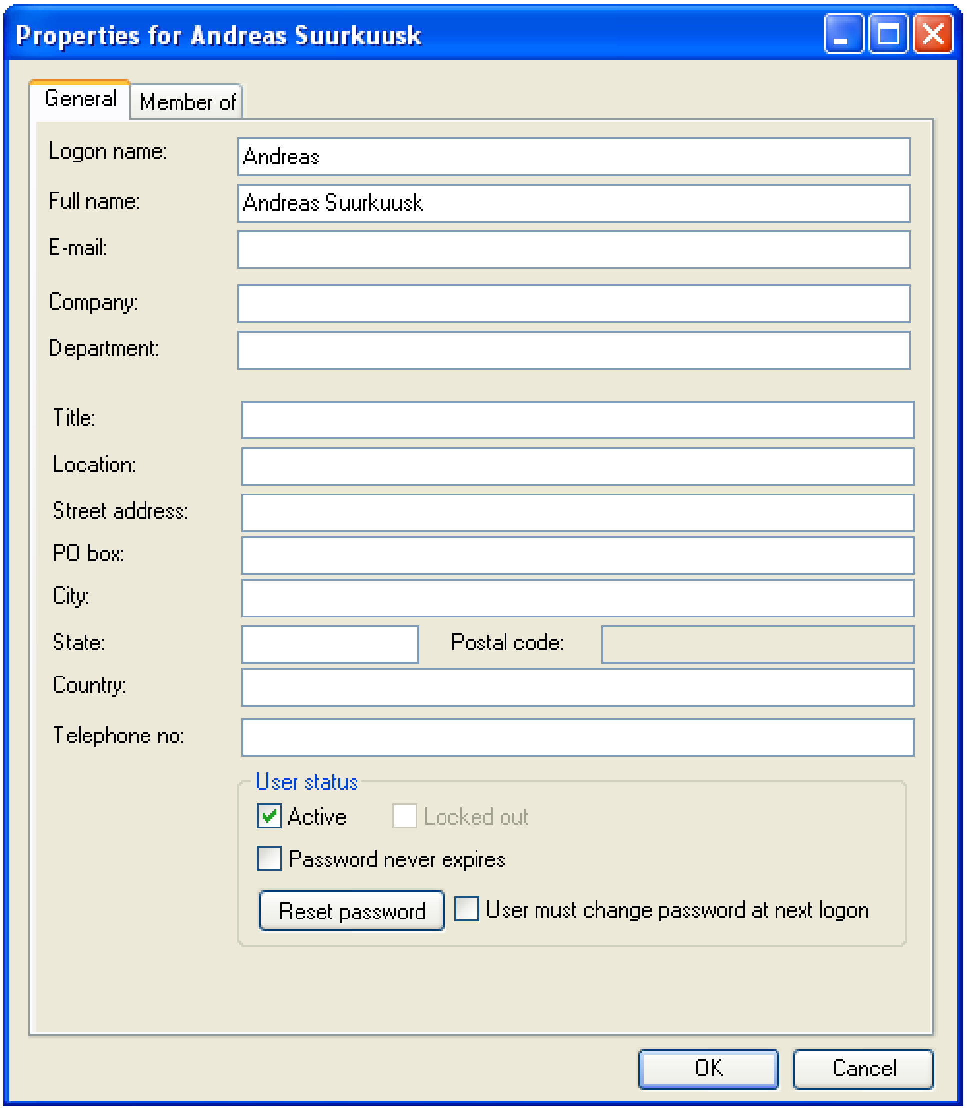
Password Never Expires

This option indicates that the password of the user never expires, even if password aging is enabled.

Editing Users

The settings for a user can be viewed and modified by double-clicking the user in the **Users** list. This will bring up a window that provides access to all user properties. If Active Directory integration is enabled, the properties are extracted from Active Directory and cannot be modified, except the **Active** and **Locked out** options. To change the settings of an Active Directory user, the standard Active Directory interface should be used.

The user properties window consists of two pages. The **General** page provides access to the standard user properties and the **Member of** page shows the groups that the user is a member of.



Logon Name

This is a mandatory field where the logon name of the user should be entered. The logon name is used to identify the user when logging in to the system.

Full Name

Use this field to enter the full name of the user. The full name is used when presenting the name of the user, for instance in audit trails entries.

E-mail/Company/Department/Title/Location/Street address/PO box/City/State/Postal code/Country/Telephone Number

These fields can be used to enter optional contact information for the user.

Active

This field indicates whether the user is active. A user account cannot be removed from the security system, but by clearing the **Active** option, the user will be prevented from logging in to the system.

Locked out

This option indicates that the user has been locked out. It is only available when the user has been locked out by making too many failed login attempts. Clearing this option will allow the user to login to the system again.

Password never expires

This option indicates that the password of the user never expires, even if password aging is enabled.

Reset Password

This button can be used to reset the password for the user. Clicking this button brings up a window prompting for a new password (and password confirmation). To reset the password, the logged in user must have the “Reset passwords” permission on the root object. For more information, see “Permissions and Roles” on page 140.

User Must Change Password at Next Logon

This option indicates that a new password should be requested the next the user logs in to the system. It is recommended that this option is enabled when the password has been reset.

Member of

The **Member of** page is only available when the built-in user database is enabled. It contains a list of all groups that the user is a member of. To add the user as a member of a new group, click the **Add** button. To remove the user as a member of a group, select the group and click **Remove**. For more information, see the next section.

Working with User Groups

A user group can be used to assign permissions to a group of users (and groups), thus simplifying permissions management. Permissions assigned to a user group are automatically assigned to all members of the group.

If Active Directory integration is enabled, the security groups in Active Directory are available when assigning permissions, and group management is performed using the standard Active Directory administration tools.

If the built-in user database is used, the available groups are managed using the **Groups** list on the **Users and Groups** page.

Creating a New User Group

When the built-in user database is initialized, two user groups are automatically added; the **Administrators** group and the **Users** group. The Administrators group initially contains the Administrator user account. Additional user groups can be created to provide more specialized groups. For instance, all users that are allowed to access a specific instrument might be added into a new group. To create a new user group, perform the following steps:

1. Click the **Add** button under the **Users** list.
2. In the screen that appears, enter a name for the group, and optionally a description.
3. Add members to the group, as described in the next section.

Adding Members to a Group

There are two ways of adding members to a group. Either by using the **User properties** screen, or the **Group properties** screen. To add a user to one or more groups using the User Properties screen, perform the following steps:

1. Double-click the user in the User list. This will bring up the User properties screen.
2. Select the **Member of** page.
3. Click the **Add** button. This will bring up the “Select Groups” screen.
4. Select the group or groups that the user should be a member of. For more information, see “User and Group Selection” on page 138.
5. Click OK to accept the group selection.
6. Click OK again to close the User properties screen and save the changes.

To add members to a group using the Group properties screen, perform the following steps:

1. Double-click the group in the Groups list. This will bring up the Group properties screen.

2. Click the **Add** button. This will bring up the “Add Users and Groups” screen.

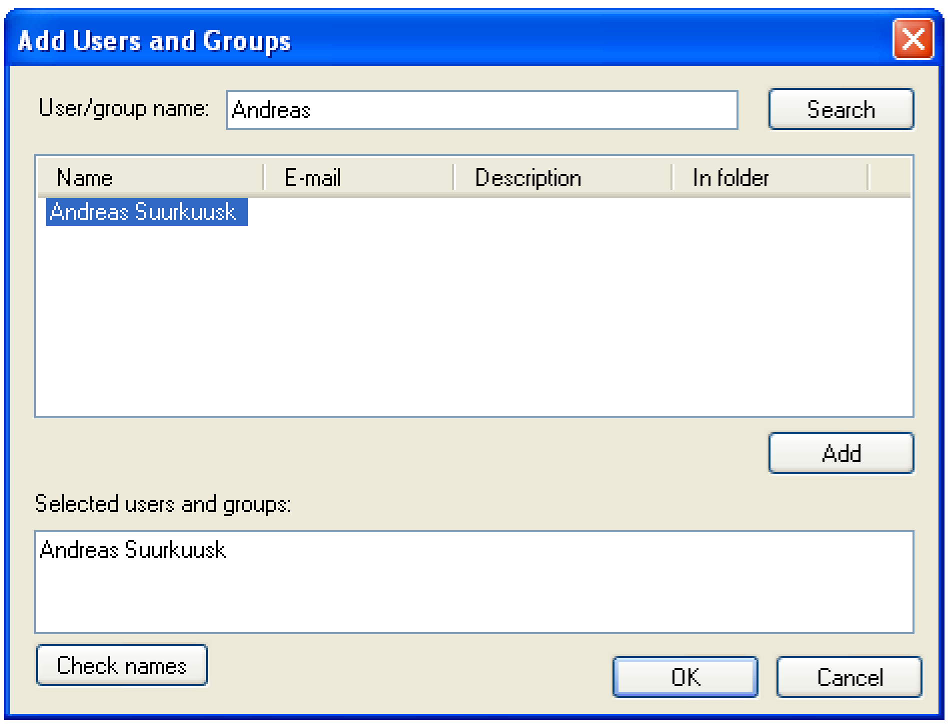
3. Select the users (or groups) that should be added to the group. For more information, see “User and Group Selection” on page 138.

4. Click OK to accept the group selection.

5. Click OK again to close the Group properties screen and save the changes.

User and Group Selection

Users or groups need to be selected when assigning permissions, working with group memberships, or filtering audit trails. This is done by using the users and groups selection screen.



The top part of this screen is used to search for users and groups. The bottom part is used to specify the selected users/groups.

Searching for Users and Groups

The **User/group name** field is used to define the user or group to select. Clicking the **Search** button will search for users and groups matching the text in the field and the matching items will be presented in the list below. The list can be used to select the wanted users or groups by either clicking the **Add** button or by double-clicking the name in the list. This will add the user or group to the **Selected users and groups** field.

Selected Users and Groups

This field contains a list of names (delimited using ';') of the users or groups to select. This field can either be filled in by typing the name of the user or group, or by using the search feature in the top of the screen.

Check Names

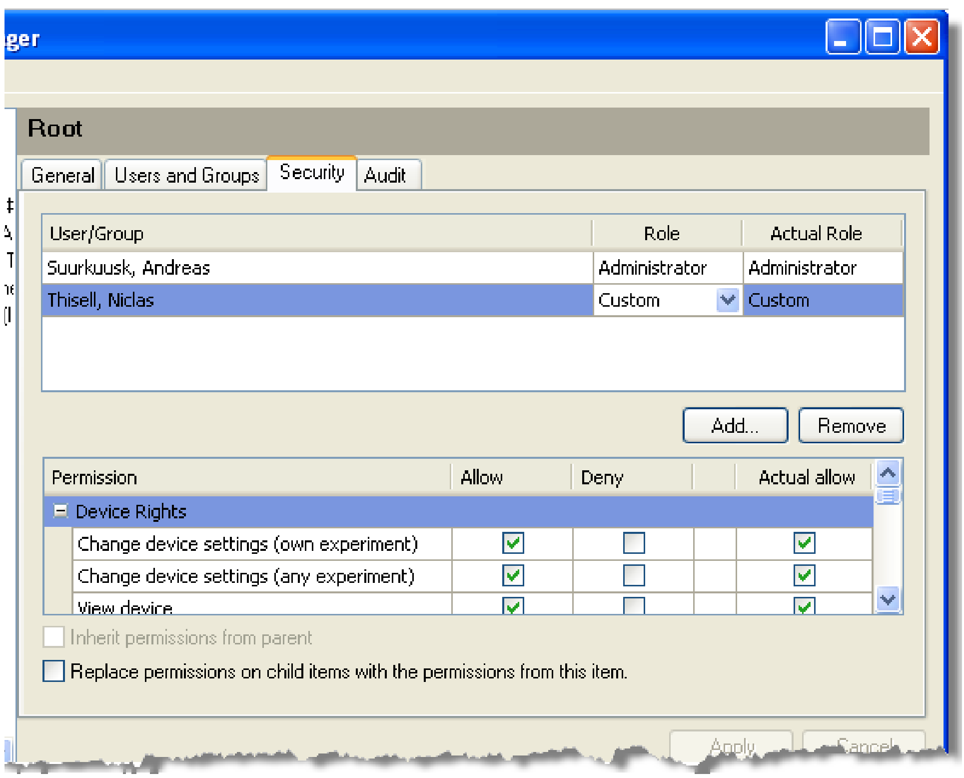
This button can be used to validate the names in the **Selected users and groups** field. If a name cannot be found among the available users and groups, or if multiple users or groups match a name, a prompt will appear.

Permissions and Roles

Permissions and roles determine the actions that are allowed by a specific user or group on the selected object.

For each selected item a set of permissions is available. The actual permissions available depend on the type of the item. For each permission it is possible to define whether the selected user or group is allowed to perform the related action (**Allow**), or if the user or group is explicitly denied to perform the action (**Deny**). Permissions assigned to one item are normally inherited by child items (unless prevented by clearing the **Inherit permissions from parent** option). For example, if a user has permission to change device settings on an instrument, then, by default, the user has permission to change device settings on all devices installed in the instrument.

It is also possible to define permissions by assigning roles to the user or group. A role is essentially a set of predefined permissions. When a role is assigned to a user or group, all associated permissions will be granted to the user or group.



The **Security** page is used to define the permissions and roles. It is accessed by selecting the top-level item in the navigation tree and contains two lists: the **User/Group** list, which lists all users and groups that have any permission setting for the selected item, and the **Permission** list, which lists the permission settings for the user or group that is selected in the **User/Group** list.

Users and Groups List

The User/Group list includes all users and groups that have any permission setting for the selected item. It has three columns:

- **User/Group:** This is a read-only column that shows the name of the user or group
- **Role:** This column shows the role that is explicitly assigned to the user or group for the selected item. The role of the user and group can be modified by using the drop-down list in this column and selecting one of the predefined roles. If no permissions have been assigned to the user or group, the text **None** will be presented in this column. If custom permissions have been set using the **Permissions** list below, the text **Custom** will be presented.
- **Actual role:** This is a read-only column that presents the actual role of the user or group. The actual role is derived from the permissions assigned to the selected item, and all permissions derived from parent items.

To add a new user or group to the list, use the **Add** button. This will allow a user or group to be selected using the User and group selection screen. When a new user or group is added to the list, it has no defined permissions. Use the **Role** drop-down list to assign a role to the user or group, or use the **Permissions** list to grant or revoke specific permissions.

Permissions List

The permissions list includes all available permissions for the selected item. It can be used to view or define the specific permissions for a user or group. The list contains the following columns:

- **Permission:** This column presents the name of the permissions
- **Allow:** This column indicates whether the selected user or group has been explicitly granted the permission for the selected object.
- **Deny:** This column indicates whether the permission has been explicitly revoked for the selected user or group.
- **Actual Allow:** This is a read-only column that indicates whether the permission is actually allowed for the selected user or group. This is derived from the permissions assigned to the selected object, and all permissions derived from parent objects.

Inherit Permissions from Parent

This option indicates whether permissions set on parent objects should be inherited by the selected object.

Replace Permissions on Child Items with the Permissions from This Item

This option indicates that the permissions settings for this item should be assigned to all child items, when the changes are applied. This will remove any specific permission settings on the child items.

Available Roles

Currently only two roles are defined:

Administrator

This role includes all permissions available. It is recommended that this role is assigned to the Administrators group at the root item. It can also be assigned to other groups and users at lower level items. It can for instance be assigned at instrument level to the user responsible for the instrument.

User

This role includes the permissions needed to work with the instruments and run experiments. It does not include permissions that allow the user to change advanced settings on devices, or to modify security settings.

Available Permissions

In this section is a list of the default permissions that are available:

Device Rights

- **Change device settings (own experiment)**
  Allows the user to make changes to a device, as long as it is not in use by an experiment run by another user.
- **Change device settings (any experiment)**
  Allows the user to make changes to a device, even if it is in use by an experiment run by another user.
- **View device**
  Allows the user the view the device, e.g., its signal histories and any settings.
- **Change device settings**
  Allows the user to make changes to a device, as long as it is not in use by any experiment.
- **Change advanced settings**
  Allows the user to make changes to the advanced settings of a device.

Experiment Rights

- **View any experiment:** Allows the user to view any running experiment, even if it is started by another user.
- **Create experiment:** Allows the user to start new experiments
- **Change any experiments:** Allows the user to make changes and control any running experiment, even if it is started by another user.

Generic Rights

- **View permissions:** Allows the user to view the security permissions of the selected item.
- **Reset password:** Allows the user to reset the password of other users. This permission is only available on the top-level item (the security system itself).
- **Change permissions:** Allows the user to make changes to the security permissions of the selected item.
- **Modify users:** Allows the user to modify the user database, e.g., adding, editing or removing users and groups. This permission is only available on the top-level item (the security system itself).
- **Change security settings:** Allows the user to make changes to the security settings of the system. This permission is only available on the top-level item (the security system itself).
- **Connect:** Allows the user to connect to the Device and Experiment server (*e.g.*, a TAM III instrument)

Audit Trail

The audit trail is used to keep track of all events that happen on the system. The events can for instance be: setting new temperature on a thermostat, performing a calibration, starting an experiment, or any system problem. Each entry in the audit trail contains information like time, user, severity, and a description. An audit trail is automatically collected by the security system. As soon as an instrument is connected to the system, audit entries are collected from the instrument as well. The audit trail can be examined using the Audit page, which is available for all securable items.

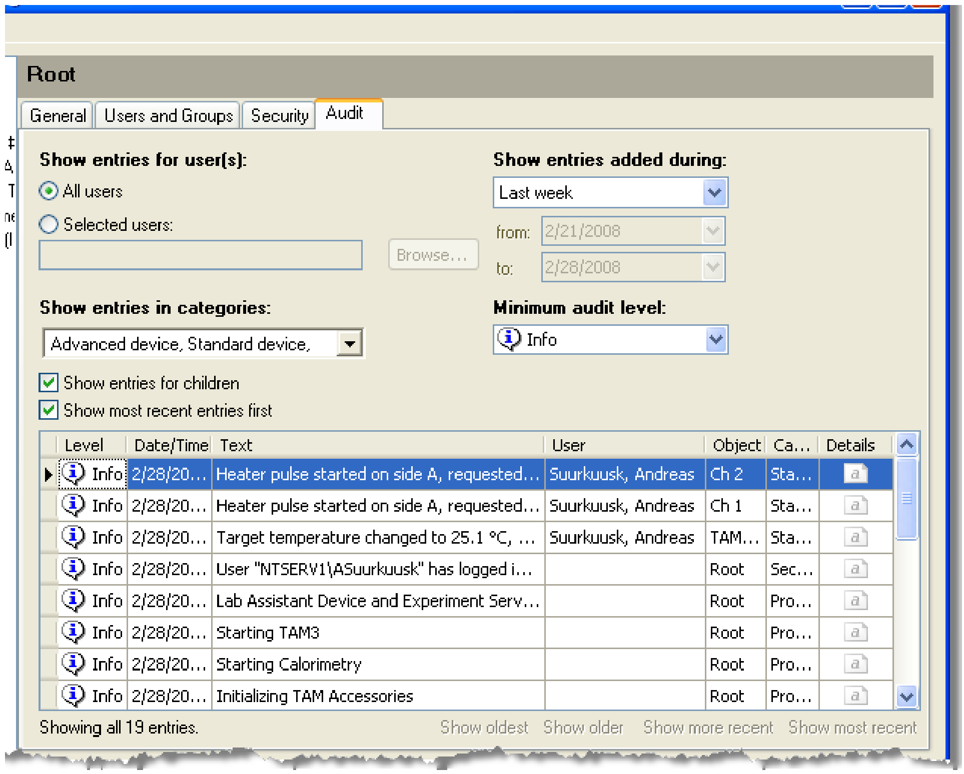
The Audit page consists of two parts. The top part is used for setting up a filter for the audit entries that should be presented. The bottom part contains a list of all the filtered audit entries.

Audit Entries Filter

The following fields are used to define the audit trail filter.

Show Entries for Users(s)

This option is used to filter audit entries based on the user that caused the event that was logged.



- **All users:** The audit trail will include entries logged by any user.
- **Selected users:** The audit trail will only include entries logged by the selected users. The list of users can be entered in the field below, or it can be defined using the **Browse** button.

Show Entries Added During

This area is used to specify the time period for the filter. Events that occurred during the specified time period will be included in the audit trail.

The time period can be specified using the following predefined intervals:

- **Any time:** No filtering of the time of the event is performed.
- **Last month:** Only events added during the last month are included.
- **Last week:** Only events added during the last week are included.
- **Last day:** Only events added during the last day are included.
- **Custom:** The time period is defined using the **from** and **to** fields below.

Show Entries in Categories

This drop-down list is used to specify the categories of the event entries that should be included. One ore more categories can be included by selecting them in the drop-down list.

Minimum Audit Level

This field is used to specify the minimum audit level of the event entries that should be included. The audit level indicates the severity of the event. The following levels are available:

- Info
- Warning
- Error
- Fatal

Show Entries for Children

This option indicates whether event entries that occurred in child items should also be included in the filtered audit trail.

Show Most Recent Entries First

If this option is enabled, the entries will be presented in reverse chronological order, *i.e.*, the most recent entry will be presented at the top.

Show Oldest, Show Older, Show More Recent, Show Most Recent

If too many entries match the specified filter, these fields are used to navigate through the entries.

Exporting Audit Trails

The filtered audit trail presented can be exported to a file by selecting **Export Filtered Audit Trail** from the File menu. (If the audit trail is view inside TAM Assistant, the **Export Filtered Audit Trail** command is found in the toolbar above the audit trail. For more information, see “Security in Property Windowsin Property Windows” on page 149.) This will bring up a standard save file window, providing the possibility to specify the file to export to and the file type.

The following export file types are available:

- **Text File**: If file type is selected, the audit trail will be exported as a plain human-readable text file.
- **Comma Separated Value File**: If file type is selected, the audit trail will be exported as a Comma Separated Value file (CSV).
- **Comma Separated Value File** (compatible): If this file type is selected, the audit trail will be exported as a Comma Separated Value file that is compatible with Microsoft Excel.
- **XML File**: If this file type is selected, the audit trail will be exported as an XML-file.

Printing Audit Trails

The filtered audit trail presented can be printed by selecting **Print Filtered Audit Trail** from the File menu. (If the audit trail is viewed inside TAM Assistant, the **Print Filtered Audit Trail** command is found in the toolbar above the audit trail. For more information, see “Security in Property Windows in Property Windows” on page 149.)

Commands in TAM Assistant Security Manager

The following commands are available from the **File** menu in TAM Assistant Security Manager:

Connect	Connects the TAM Assistant Security Manager to a security server. This command will bring a window that allows a security server to be selected.
Initialize Local Security Server	Initializes the local security server. This command is only available if a security server is installed on the computer running.
Export Filtered Audit Trail...	Exports the results of an audit trail search. This command is only available when viewing audit trails. For more information, see "Working with Audit Trails" on page 143.
Print Filtered Audit Trail...	Prints the results of an audit trail search. This command is only available when viewing audit trails. For more information, see "Working with Audit Trails" on page 143.
Exit	Exits TAM Assistant Security Manager

Security System Access in TAM Assistant

The TAM Assistant software and the TAM III instrument provides access to most functionality if the security system. It also provides functionality for specifying the security system to used, and other settings such as how login should be performed. Audit trail and permissions management can be accessed using the **Properties** for a securable object, such as a device or results file.

Security Settings

The Security Settings screen is available in TAM Assistant by selecting the **Settings** command under the **File/Security** menu.

It provides the possibility to change the following security settings.

Automatically Login at Startup

This option indicates whether TAM Assistant should automatically bring up the login window when the program is started. If this option is not selected, TAM Assistant will not login nor connect to any devices until the user explicitly logs in using the **Login** command under **the File/Security** menu.

Use Current Windows Credentials When Logging In

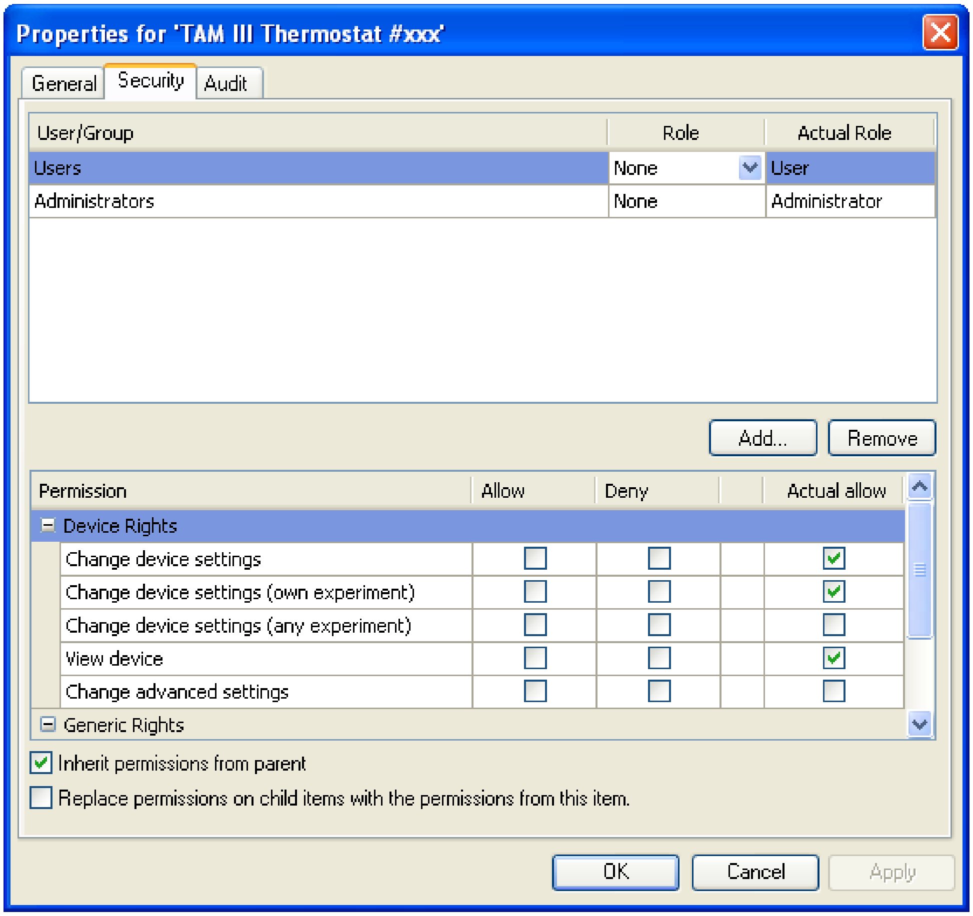
This option is used to decide whether the current Windows credentials should be used when logging in to the security system. If Active Directory integration is enabled and the security system settings allows automatic login, then it is possible for TAM Assistant to login without prompting for a username and password.

Automatically Connect to Devices at Login

Indicates whether TAM Assistant should automatically connect to all registered device servers when the user has logged in. If this option is not enabled, the **Connect to Device Servers** command under the **File** menu must be used to initiate the device connection manually.

Security in Property Windows

When a device or results file window is being shown, it is possible to use the **Properties** command (on the **View** menu) to view and edit the properties of the device or results file. If TAM Assistant is connected to a security server, the properties window will include two additional pages: **Security** and **Audit.** These pages are used to view and edit permissions for the object and to view the audit trail. They are equivalent to the **Security** and **Audit** pages in the TAM Assistant Security manager, but they only act on the selected device or results file.



Commands in TAM Assistant

The following commands are available from the **Security** submenu under the **File** menu in TAM Assistant:

Log in new user	Logs in a new user.
Log out current user	Logs out the currently logged on user. All open documents will be closed, but any running experiments will not be affected.
Suspend current user	Suspends the current user. The main windows will be closed and a login screen will appear, allowing another user to login. However, the original login is still active. If the same user logs in again, all open documents will be restored.
Change password	Changes the password of the currently logged in user.
Settings	Opens the security settings window. For more information, see “Security Settings” on page 148.

Commands on the TAM III Instrument (TAM Assistant Embedded)

On the TAM III instrument, the security related commands are found under the **Security** button in the top menu. The following commands are available:

Log out current user	Logs out the currently logged on user. All open documents will be closed, but any running experiments will not be affected. The system overview and login screen will be shown.
Suspend current user	Suspends the current user. The main windows will be closed and a login screen will appear, allowing another user to login. However, the original login is still active. If the same user logs in again, all open documents will be restored.
Change password	Changes the password of the currently logged in user.
Security Manager	Opens the Security Manager window. This window is equivalent to the TAM Assistant Security Manager main window. This allows security settings and permissions to be edited on the TAM III instrument.

Security Considerations and Backup of Security Data

NOTE: There is currently no way of backing up the security data files if the security system is running on the TAM III instrument. If the audit trails and user database is important, it is recommended that the security system is installed on a standalone computer which is properly backed up.

The security system server stores security data (user database and permissions) and audit trails in normal files. When installing the software, a folder where these files should be stored must be specified. In order to avoid tampering with the security files, this folder should only be used for the security files, and the access to the folder should be limited. The account that runs the security system service must have read/write access to the folder. To simplify file management and to allow direct access to the security system (as described below) it might be a good idea to allow the **LocalSystem** account and the Windows Administrators group to access the security files folder as well.

If a user (who should be an administrator) has access to the security files, it is possible to locally use TAM Assistant Security Manager, without first logging in to the security system. This provides the possibility to restore permissions and settings even if normal administrative access to the system is not possible, for instance if the administrator password has been lost, or administrative permissions have mistakenly been removed.

Backup

The security system service is designed to allow files to be backed up while the service is running. To prevent loss of audit trails and other security data, it is recommended that the folder containing the security files are backed up regularly, as part of the normal backup procedures.

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