

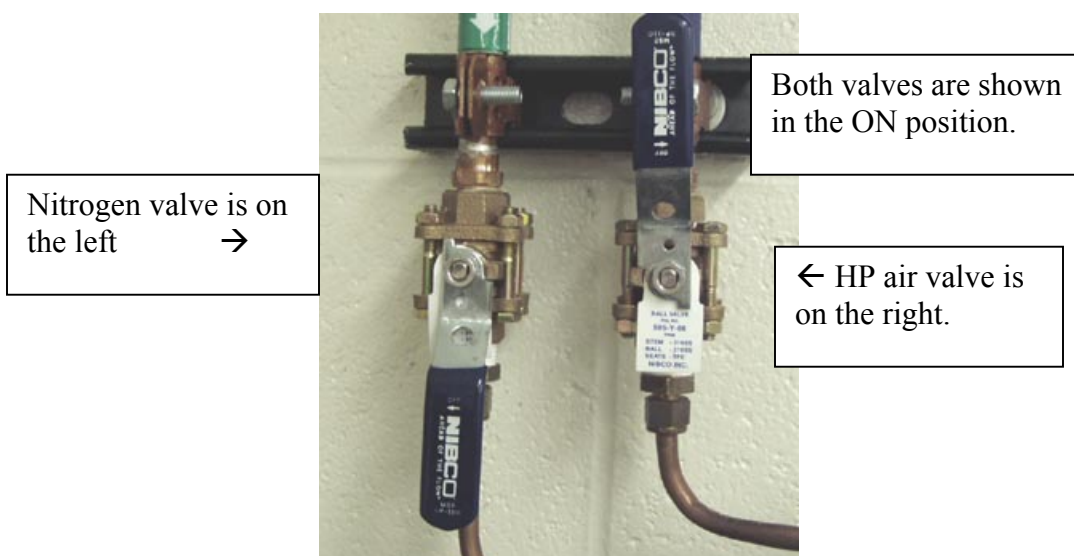
TGA-DSC User's Booklet

I. How to use the TGA

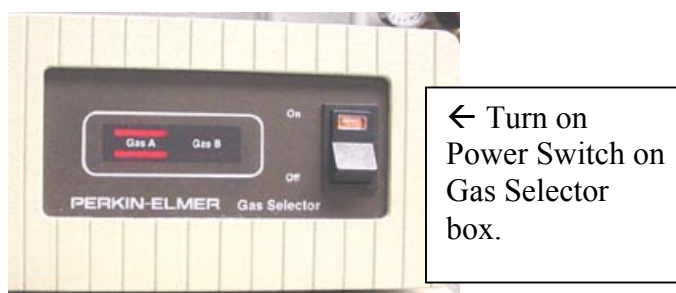
Please Note:

The order in which items are powered up is important. If they are not done in the proper order, the data system will not be able to communicate with the instrument. Please follow these steps in the exact order when powering up the system.

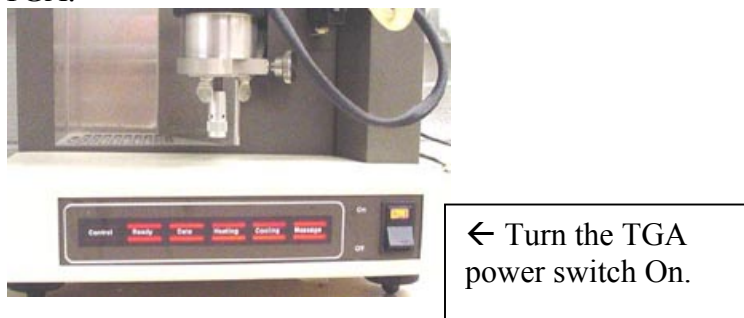
- 1) Turn on both the nitrogen gas and the high-pressure air, using the two blue-handled valves on the wall. The valves are ON when the blue handle is parallel to the pipe. The valves are OFF when they are perpendicular to the pipe.



- 2) Turn on the power to the Gas Selector Box, which is located on top of the TGA.



- 3) Turn on the power to the TGA.

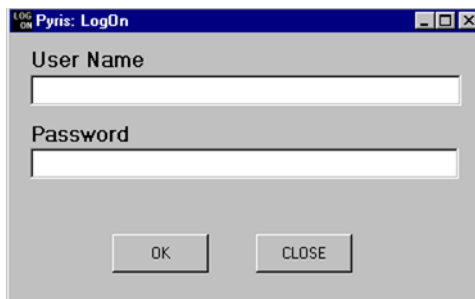


- 4) Turn on the Power to the TAC (Thermal Analysis Controller) for the TGA.

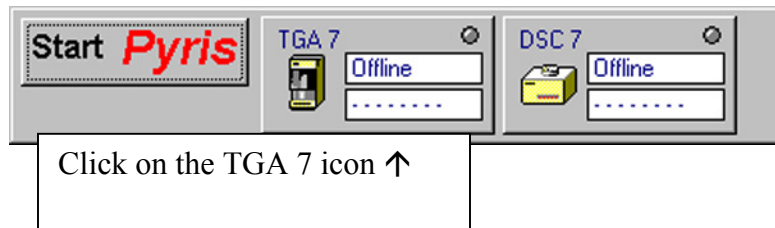


← Turn on the Power switch for the TGA TAC.

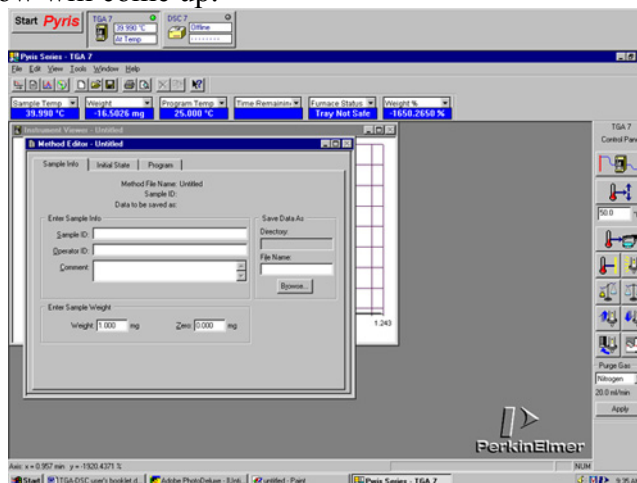
- 5) Log into Windows
- 6) After Windows comes up, double-click the LOG ON icon. The LOG ON window will come up. Type in your username & password.



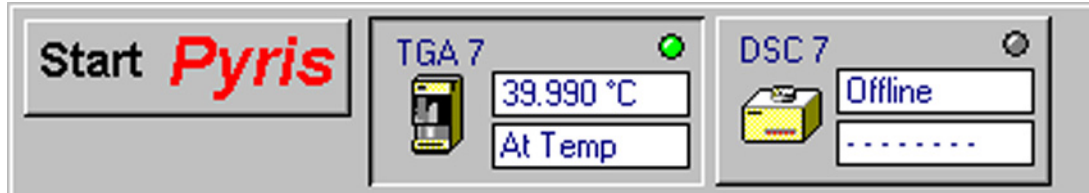
- 7) After you login, the Pyris software will load. You will see a menu bar at the top of the screen, which will indicate that the TGA 7 is Offline. Click on the TGA 7 icon to bring it online:



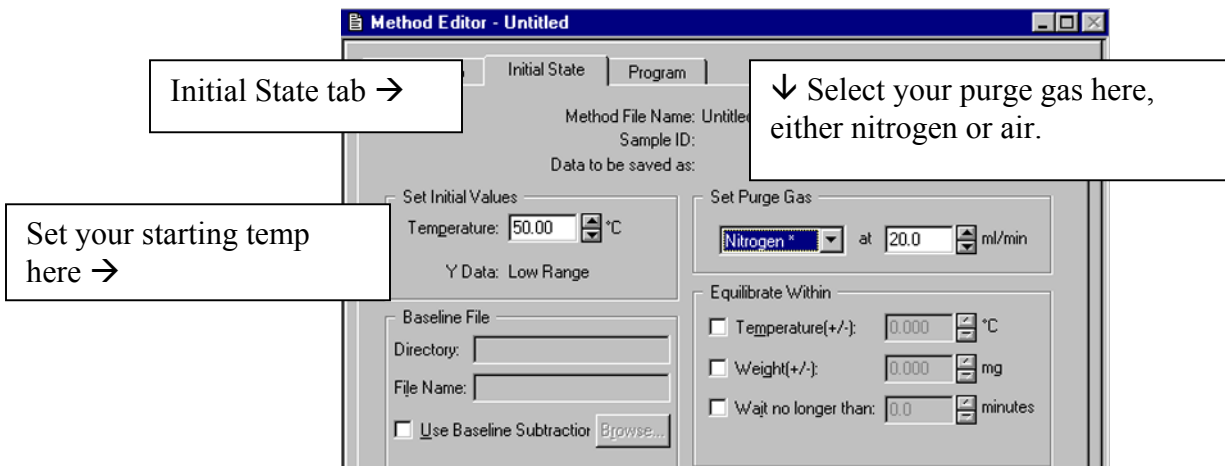
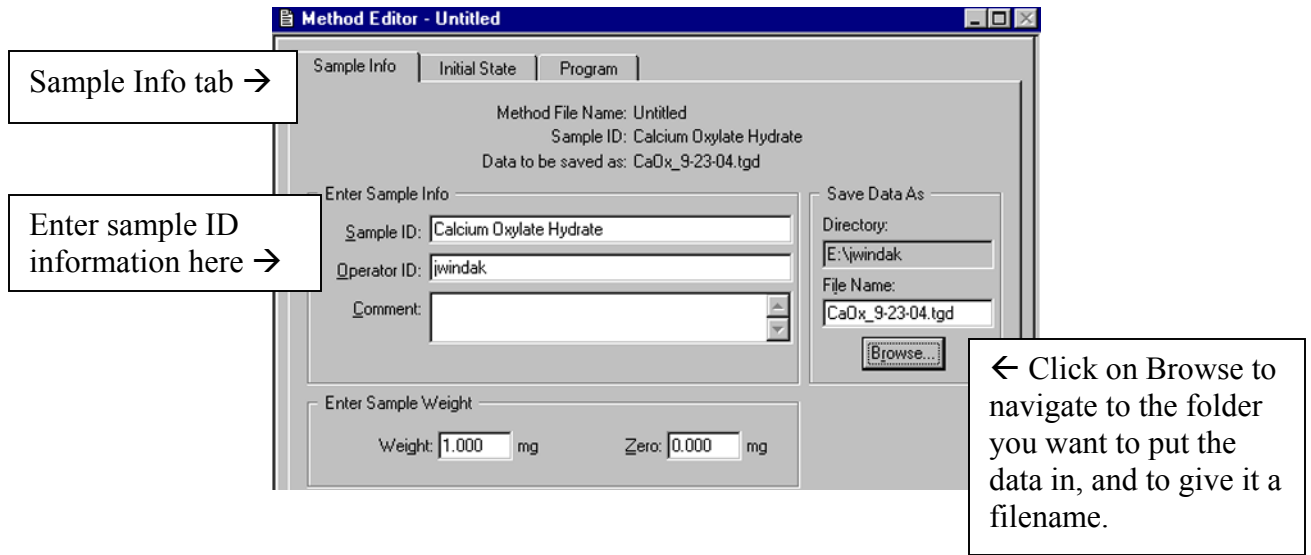
- 8) The TGA 7 window will come up.

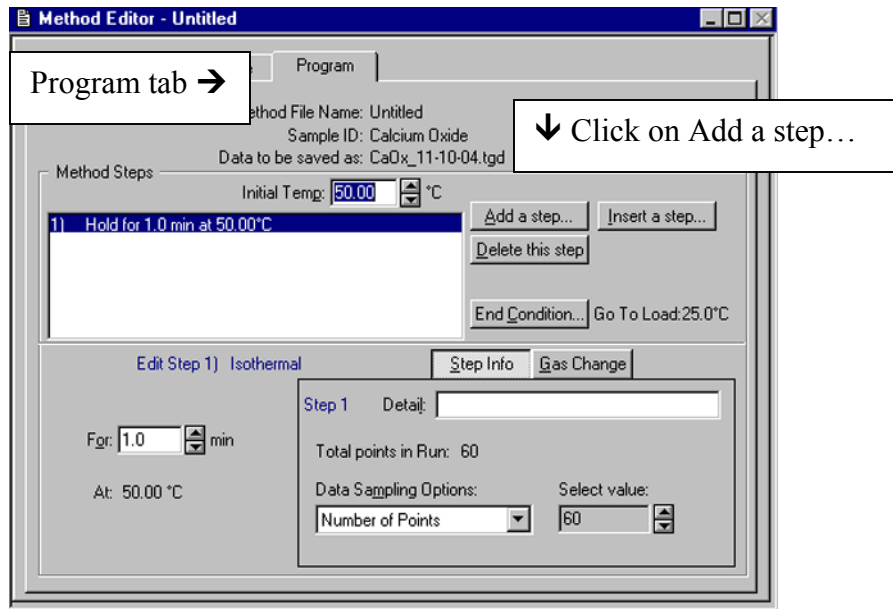


- 9) You should see temperature status information for the TGA 7 if everything is working properly:

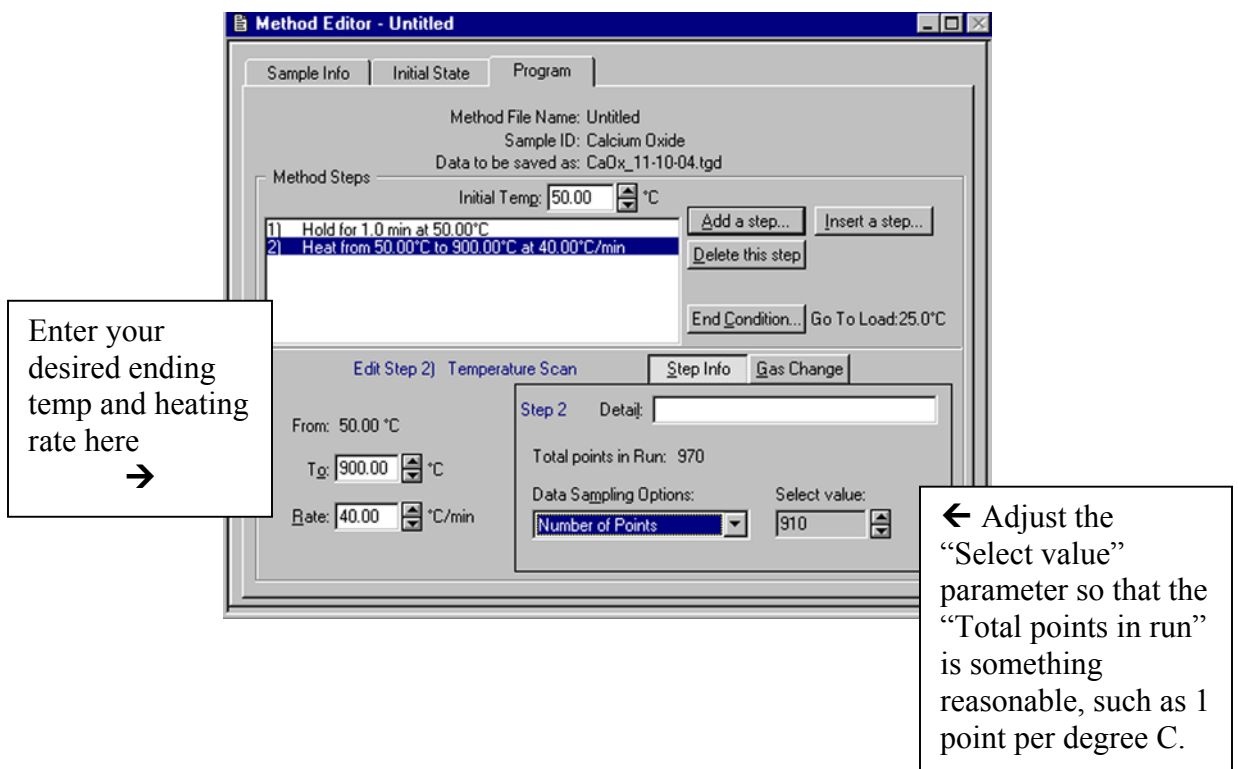
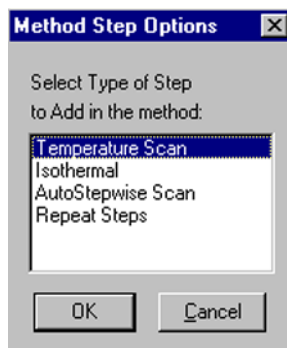


- 10) Enter the sample information into the method editor:



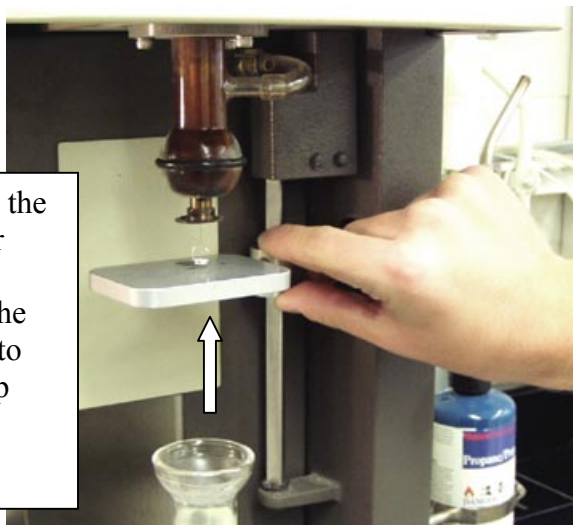


After you click on “Add a step...” the following box appears to let you choose what type of step to add. Usually, this will be a Temperature Scan. Then click on OK.

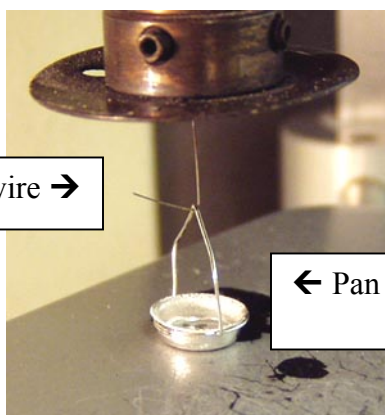


- 11) Carefully hang the empty pan and stirrup onto the hang-down wire, by raising the pan and stirrup using the tray. Please be careful not to raise it too high, or you might crush the platinum stirrup.

Carefully raise the tray using your thumb and forefinger, to the height needed to push the stirrup onto the hang-down wire.

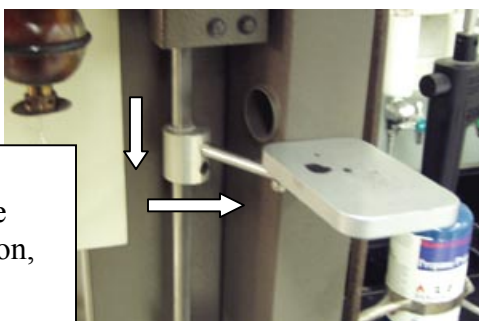


Hangdown wire →



← Pan & Stirrup

Push the tray down, and then all of the way to the side, in the "safe" position, when done. →



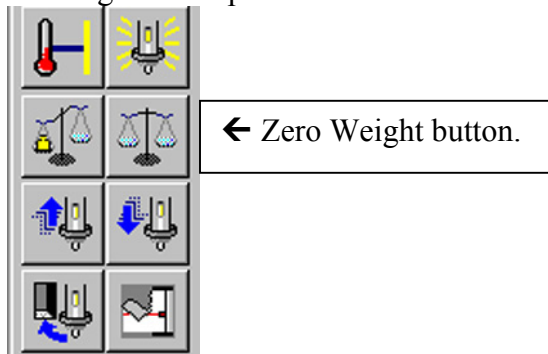
Please note:
The furnace will not raise unless the tray is all the way in the safe position.

- 12) Raise the furnace by clicking on the raise furnace button.

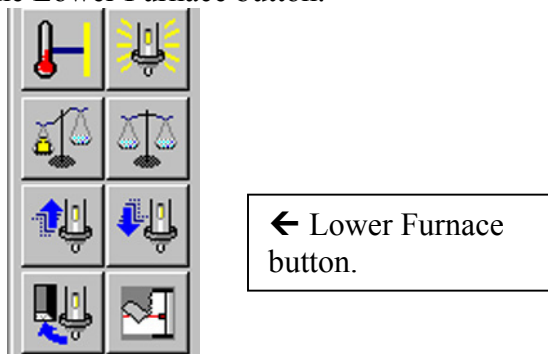
Raise furnace button →



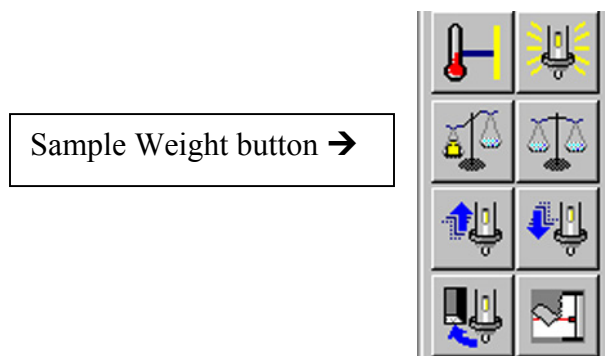
- 13) After the furnace has raised, and after the stirrup & pan have settled down and stopped swinging, click on the Zero Weight button to subtract out the tare weight of the empty pan. After this has been done, the indicated weight of the pan should be close to zero.



- 14) Lower the furnace by clicking on the Lower Furnace button.

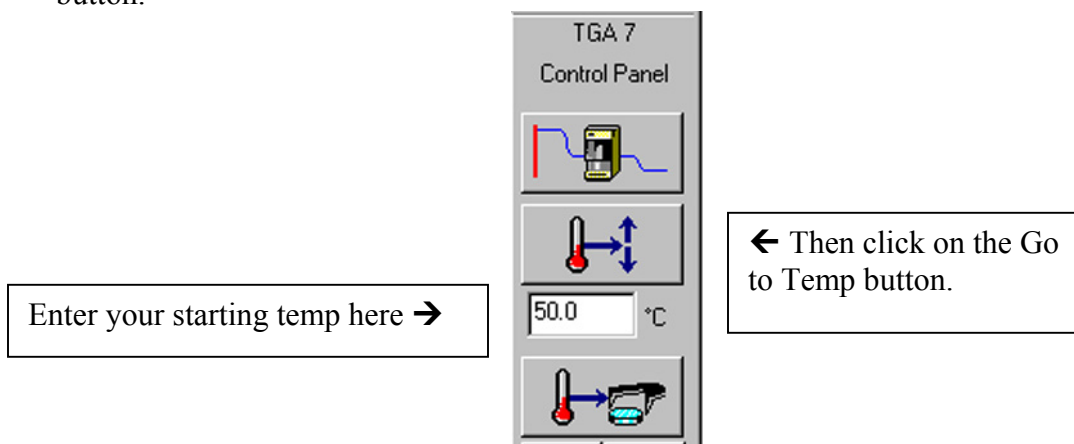


- 15) Remove the stirrup and pan by using the tray to raise them up so that they can be slipped off of the hang-down wire. Please be careful not to raise them too high or you will crush the stirrup.
- 16) Remove the pan from the stirrup using tweezers. Fill the pan with sample. Then put the sample pan back into the stirrup with tweezers. Re-hang the stirrup and pan onto the hang-down wire, using the tray.
- 17) Raise the furnace by clicking on the Raise Furnace button.
- 18) Record the starting weight of the sample by clicking on the Sample Weight button. This will automatically enter the weight into the program, and set the % Weight to 100%.

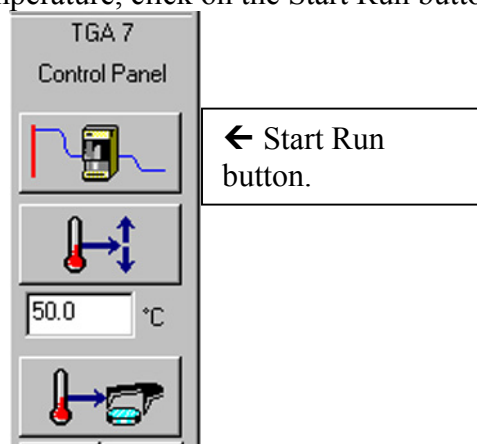


19) **Lower the safety shield in front of the furnace. Important!**

20) Go to your starting temperature, by entering that temperature and clicking on the Go to Temp button.



21) After the TGA is at the starting temperature, click on the Start Run button.



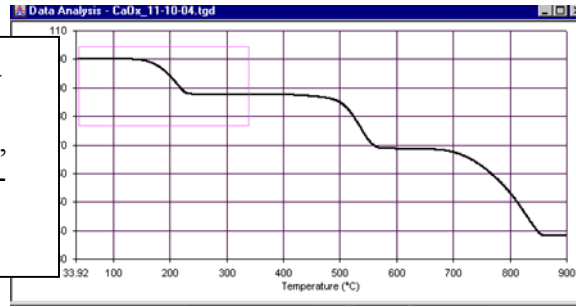
22) After the run has started, click on the Instrument Viewer to monitor the progress of the run. Note that the default setting for the graph is to have "Time" displayed on the X-axis. If you want to change this to Temperature, click on the Display menu, and click on Rescale X, and you will see an option to change the X-axis to Temperature.

23) When the run is done, the furnace will automatically lower, and go to the cool down position, where air is blown over the furnace to cool it.

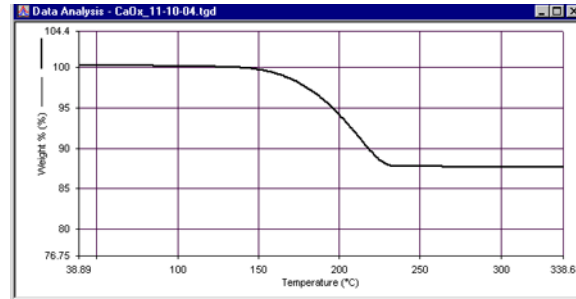
II. TGA Data Analysis

1) If you want to zoom into a smaller region on the graph, click and drag a box around the region, and then double-click inside of the box.

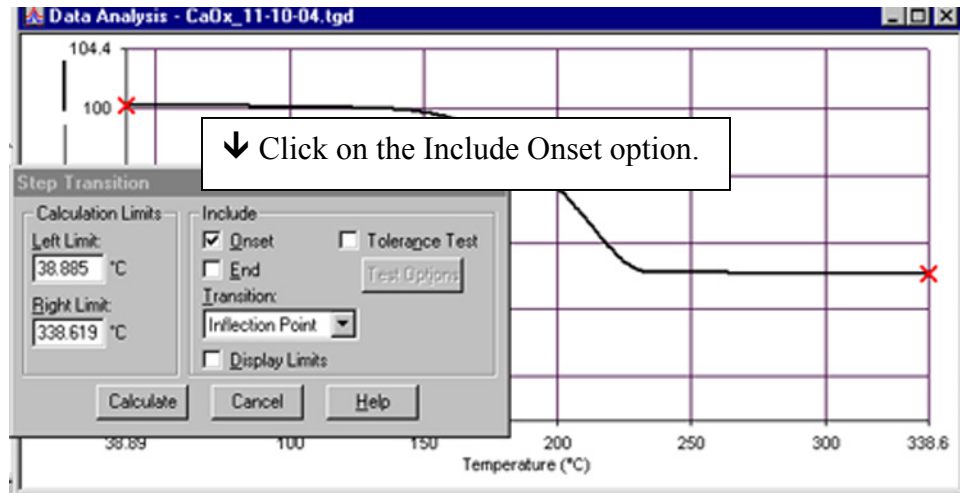
Click and drag a box around the region you want, and then double-click inside the box.



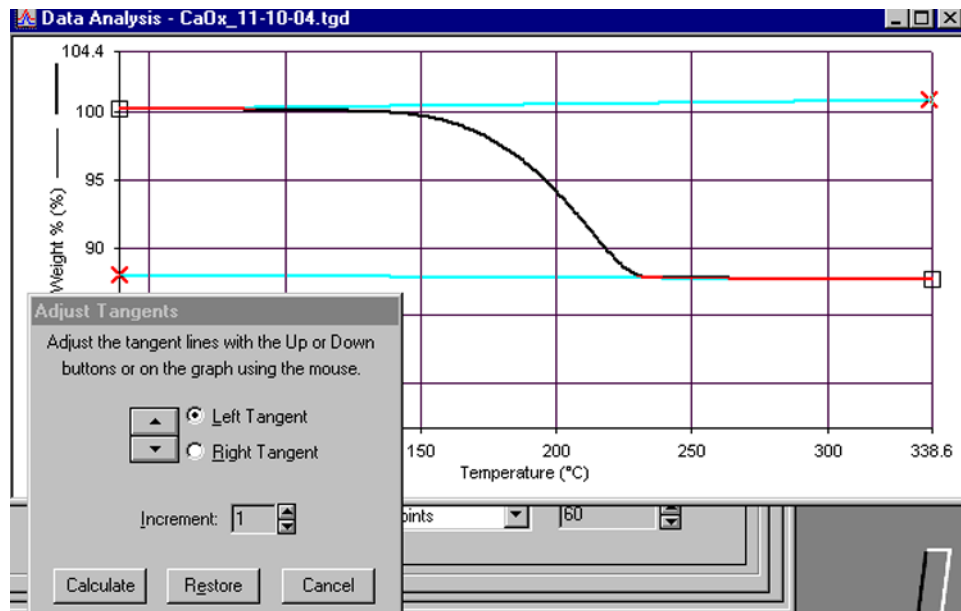
The graph will then zoom into that area.



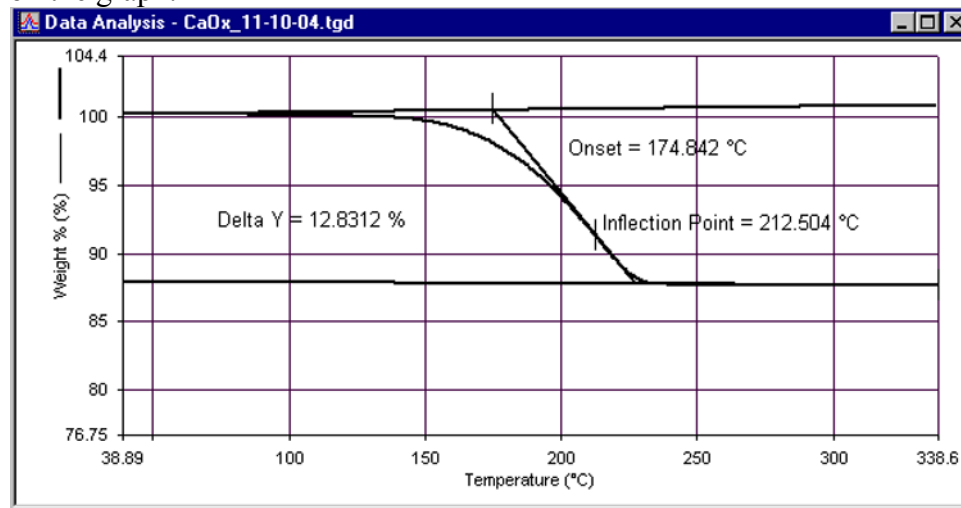
- 2) If you want to calculate both the onset temperature, and the change in weight percent, then click on the Calc menu, and click on Step. The following Step Transition box will appear. Click on the Include Onset option. Then click on Calculate.



- 3) After you click on Calculate, two tangent lines will appear. You can adjust these up or down in order to make them tangent to the baseline on the curve. Then click on Calculate.



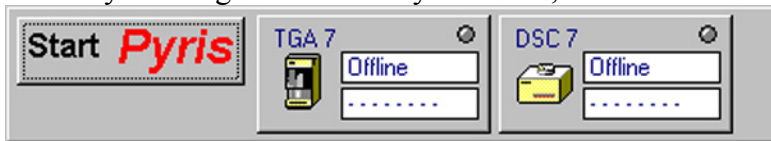
- 4) After you click on Calculate, the onset temperature, the inflection point, and the DeltaY will be displayed on the graph. You can click and drag these labels to move them wherever you want on the graph.



- 5) You must click on File and Save Data, in order to save the calculated results with the raw data file.
- 6) To convert the graph to ASCII format, click on the Edit menu, and click on Copy. Then go to a text program, such as Word, and click on Edit and Paste.

III. How to shut down the TGA when you are finished.

- 1) Close the software by clicking on the Start Pyris button, and then clicking on “Close All”.



- 2) After the software closes, turn off the power to the TAC box (Thermal Analysis Controller) for the TGA.



← Turn off the power for the TGA TAC.

- 3) Turn off the power for the TGA.



← Turn off the power to the TGA.

- 4) Turn off the power to the Gas Selector box on top of the TGA.



← Turn off the Gas Selector power.

- 5) Turn off the high-pressure nitrogen and air. The gases are off when the blue handles are perpendicular to the pipe.

The valves are both shown in the OFF position.



IV. How to use the DSC

Please Note:

The order in which items are powered up is important. If they are not done in the proper order, the data system will not be able to communicate with the instrument. Please follow these steps in the exact order when powering up the system.

- 1) Turn on the nitrogen gas, using the blue handled valve on the wall. The valve on the left is for nitrogen. If you are only using the DSC, then only the nitrogen gas need be turned on. If you are using the TGA also, then both of the gases need to be on.

The valve on the left is for nitrogen, which is shown in the ON position.



- 2) Turn on the power switch on the front of the DSC.



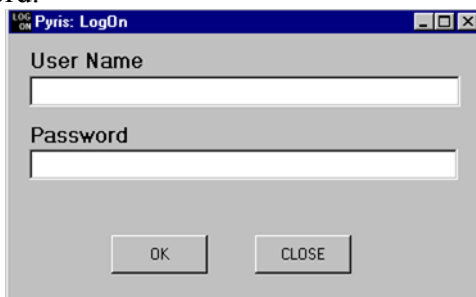
← Turn ON the power switch for the DSC.

- 3) Turn on the power for the DSC TAC (Thermal Analysis Controller).

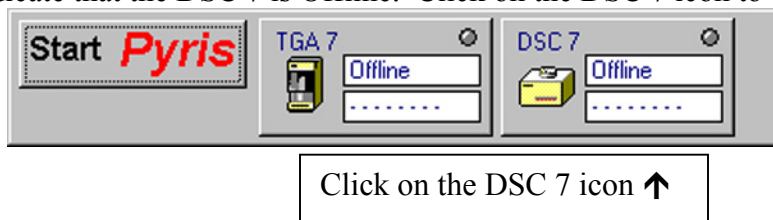


← Turn ON the power switch for the DSC TAC.

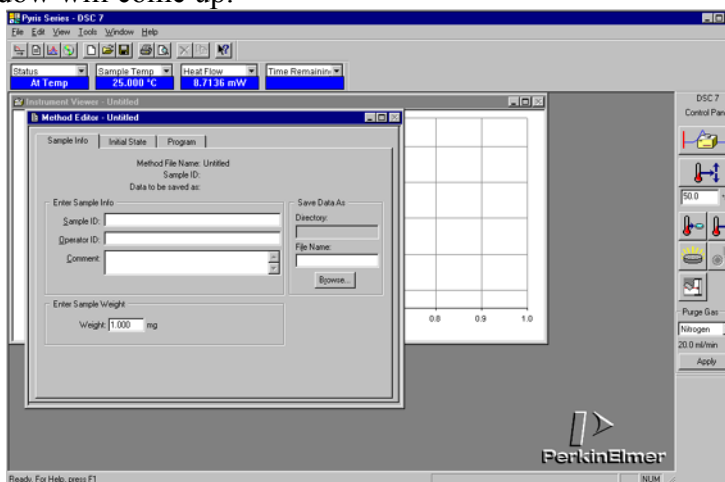
- 4) Log into Windows.
- 5) After Windows comes up, double-click the LOG ON icon. The LOG ON window will come up. Type in your username & password.



- 5) After you login, the Pyris software will load. You will see a menu bar at the top of the screen, which will indicate that the DSC 7 is Offline. Click on the DSC 7 icon to bring it online:



- 6) The DSC 7 window will come up.



- 7) You should see temperature status information for the TGA 7 if everything is working properly:



- 7) Fill the DSC with ice water, with the metal funnel in place.

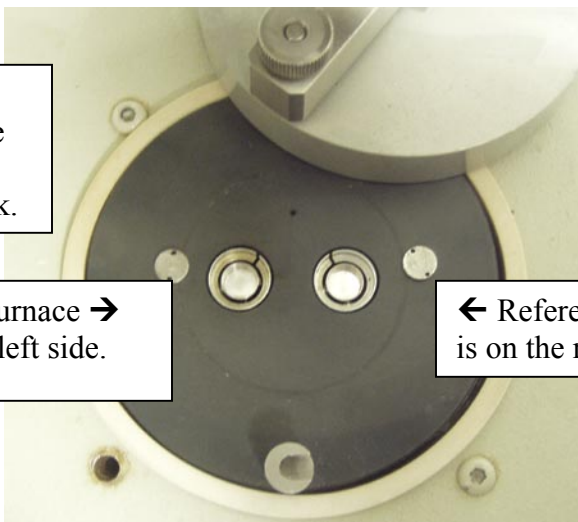


8) First, we need to run a blank spectrum to verify that the DSC was found in good condition. After you run your blank spectrum, print it out, and put your name on it and place it in the tray by the computer monitor. Before you run the blank, check to make sure that both the sample and reference furnaces are empty.

Make sure that both furnaces are empty before running the blank.

Sample furnace → is on the left side.

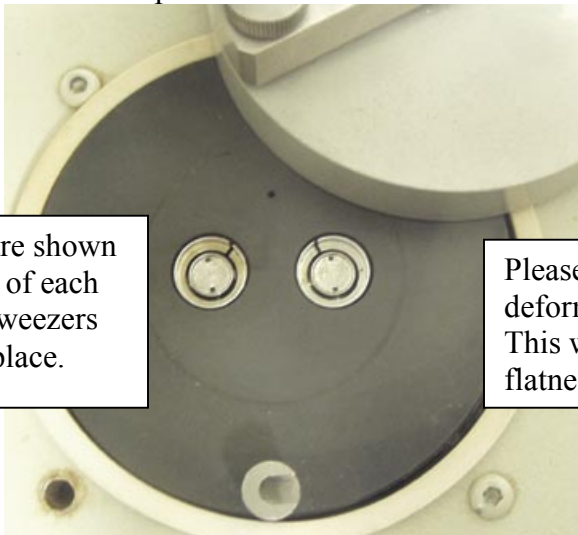
← Reference furnace is on the right side.



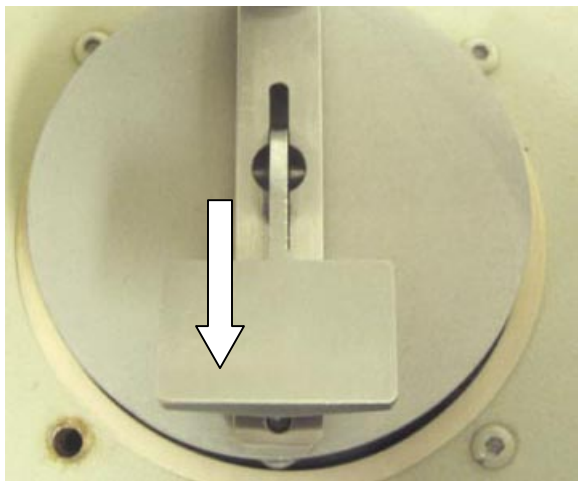
9) After you have checked that both furnaces are empty, put their platinum lids on, close the swing-arm cover, and put the dome on top.

Platinum lids are shown in place on top of each furnace. Use tweezers to set them in place.

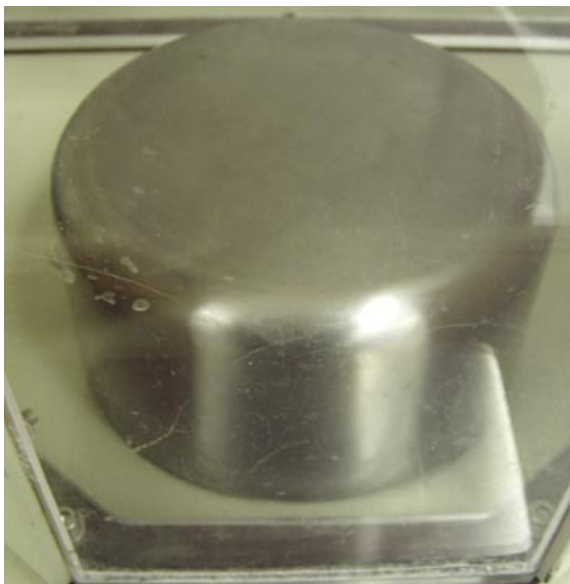
Please do not bend or deform the platinum lids. This will affect the flatness of the baseline.



Swing arm cover is shown in place over the furnaces. Push down on the lever to lock it in place.



Dome cover is shown in place over the furnace assembly.



10) Set up your data acquisition using the method editor.

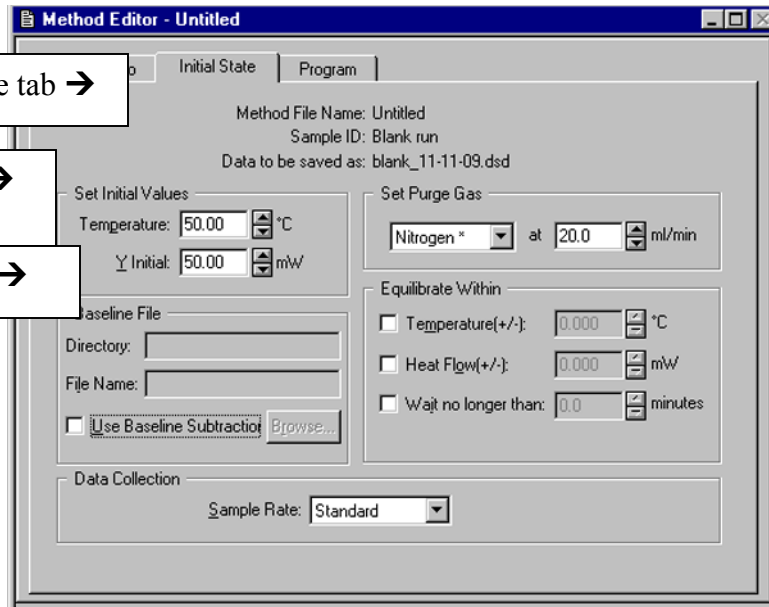
Sample Info tab →

Enter sample ID information here →

For the blank run, you do not need to enter anything for the sample weight. For an actual sample, you must enter its weight if you want to measure ΔH .

The screenshot shows the 'Method Editor - Untitled' window. It has three tabs: 'Sample Info', 'Initial State', and 'Program'. The 'Sample Info' tab is active. The window displays the following information: 'Method File Name: Untitled', 'Sample ID: Blank run', and 'Data to be saved as: blank_11-11-09.dsd'. Below this, there are three input fields: 'Sample ID' (containing 'Blank run'), 'Operator ID' (containing 'jwindak'), and 'Comment' (empty). To the right, there is a 'Save Data As' section with 'Directory' (E:\jwindak), 'File Name' (blank_11-11-09.dsd), and a 'Browse...' button. At the bottom, there is an 'Enter Sample Weight' section with 'Weight' (1.000) and 'mg'.

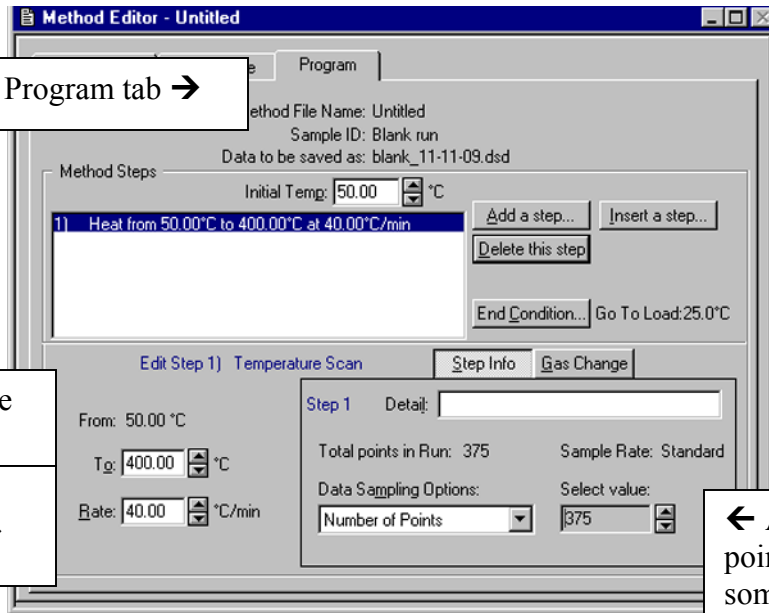
← Click on Browse to navigate to the folder you want to put the data in, and to give it a filename.



Initial State tab →

Set initial temp here →
50 C for the blank run

Set Y baseline here →



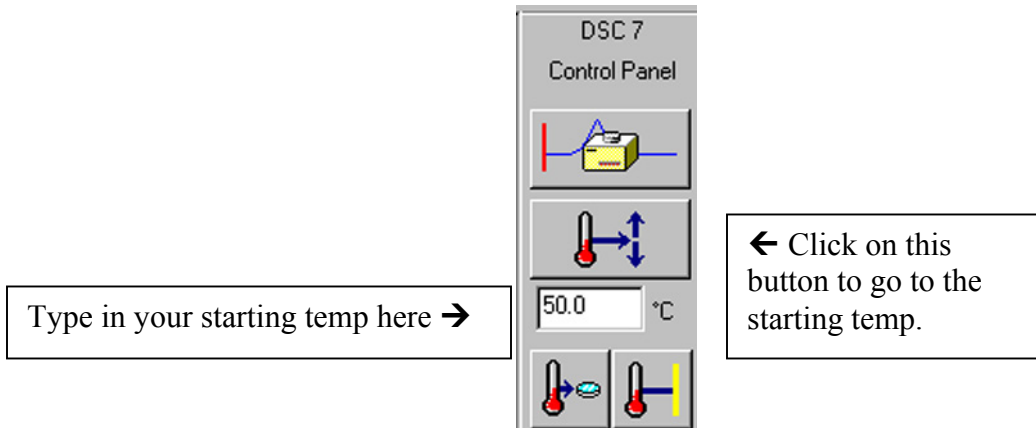
Program tab →

For the blank run, set the
final temp to 400 C →

For the blank run, set the
heating rate to 40 C /min →

← Adjust the total
points in the run to
something reasonable,
such as 1 point per
degree C.

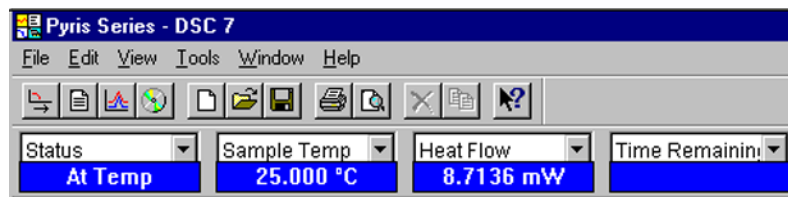
11) Set the DSC to the starting temperature in your run.



Type in your starting temp here →

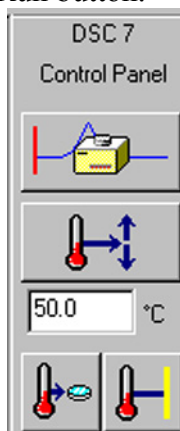
← Click on this
button to go to the
starting temp.

- 12) After the DSC status indicates that is “At Temp”, look at the Heat Flow reading, and wait until the Heat Flow is stable to 0.001 mW.



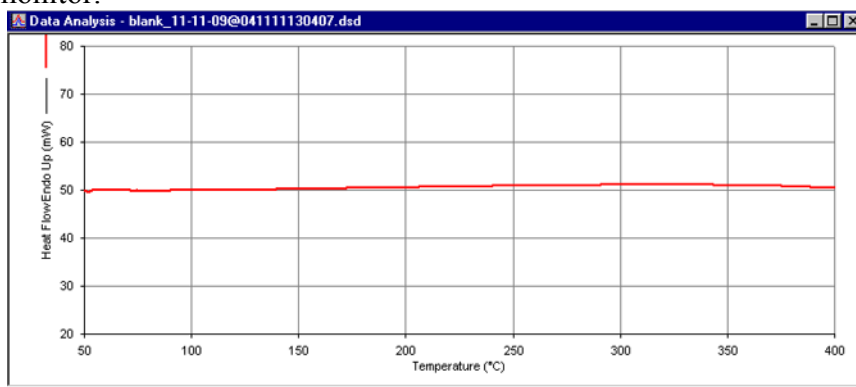
The Heat Flow should stabilize to within 0.001 mW before you start the run. ↑

- 13) Start the run by clicking on the Start Run button.



← Click on the Start Run button.

- 14) After the run has started, click on the Instrument Viewer to monitor the progress of the run. Note that the default setting for the graph is to have “Time” displayed on the X-axis. If you want to change this to Temperature, click on the Display menu, and click on Rescale X, and you will see an option to change the X-axis to Temperature.
- 14) Please print out your blank spectrum, and put your name on it, and place it in the tray next to the computer monitor.



- 15) To run an actual sample, you must first encapsulate your sample in a special aluminum sample pan. For instructions on how to do this, please see the Appendix on sample preparation. Place your sample pan into the middle of the sample furnace, and then put an empty aluminum pan into the reference furnace.

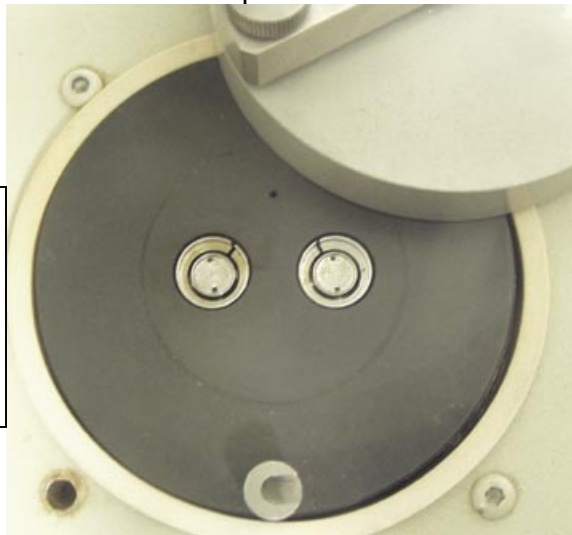
Sample should be centered in the sample furnace, which is on the left side →



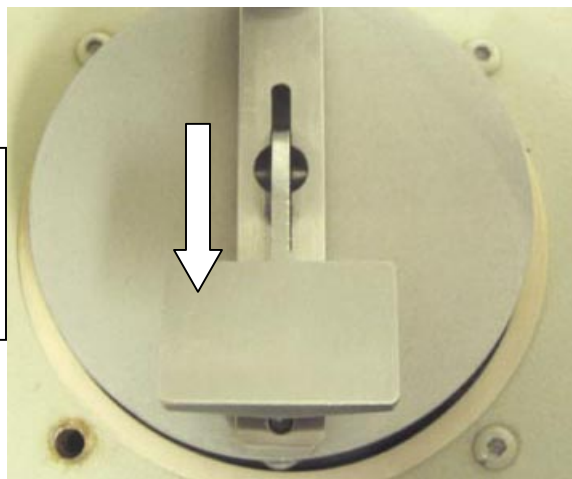
← An empty aluminum pan should be centered in the reference furnace, which is on the right side.

- 16) Carefully put the platinum lids on top of each furnace, then secure the swing-arm cover over the furnaces, and then put the dome lid on top.

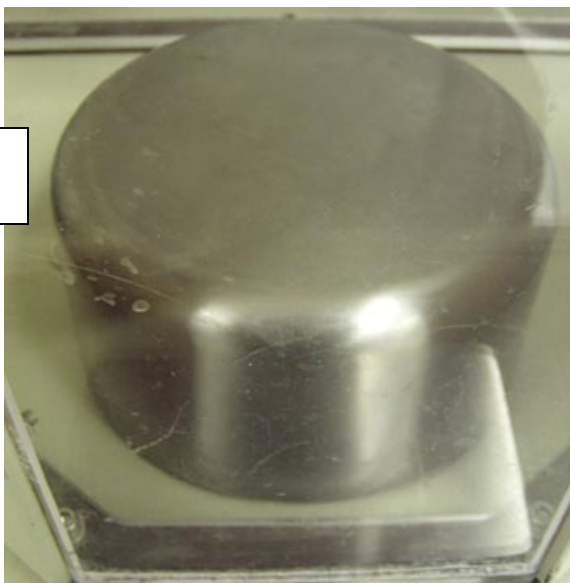
Platinum lids are shown in place. Use tweezers to gently put them in place. Please do not bend or deform the lids.



Swing-arm cover is shown in place. Push gently down on the lever to lock it in place.



Dome cover is shown in place.



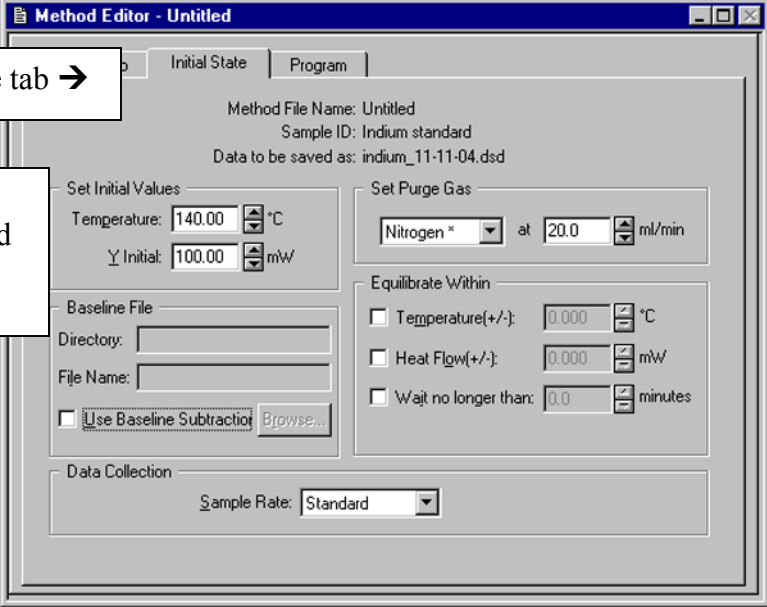
17) Set up your data acquisition using the method editor.

The screenshot shows the 'Method Editor - Untitled' window. It has three tabs: 'Sample Info', 'Initial State', and 'Program'. The 'Sample Info' tab is active. The window contains the following fields and sections:

- Method File Name: Untitled
- Sample ID: Indium standard
- Data to be saved as: indium_11-11-04.dsd
- Enter Sample Info section:
 - Sample ID: Indium standard
 - Operator ID: jwindak
 - Comment: (empty)
- Save Data As section:
 - Directory: E:\jwindak
 - File Name: indium_11-11-04.dsd
 - Browse... button
- Enter Sample Weight section:
 - Weight: 8.022 mg

Four callout boxes provide instructions:

- 'Sample Info tab →' points to the 'Sample Info' tab.
- 'Enter sample ID information here. →' points to the 'Sample ID' field.
- 'Enter an accurate sample weight here, if you want to measure ΔH .' points to the 'Weight' field.
- '← Click on Browse to navigate to the folder you want to put the data in, and to give it a filename.' points to the 'Browse...' button.



Initial State tab →

Set your starting temperature here, and the level for the Y baseline in mW.

Method File Name: Untitled
Sample ID: Indium standard
Data to be saved as: indium_11-11-04.dsd

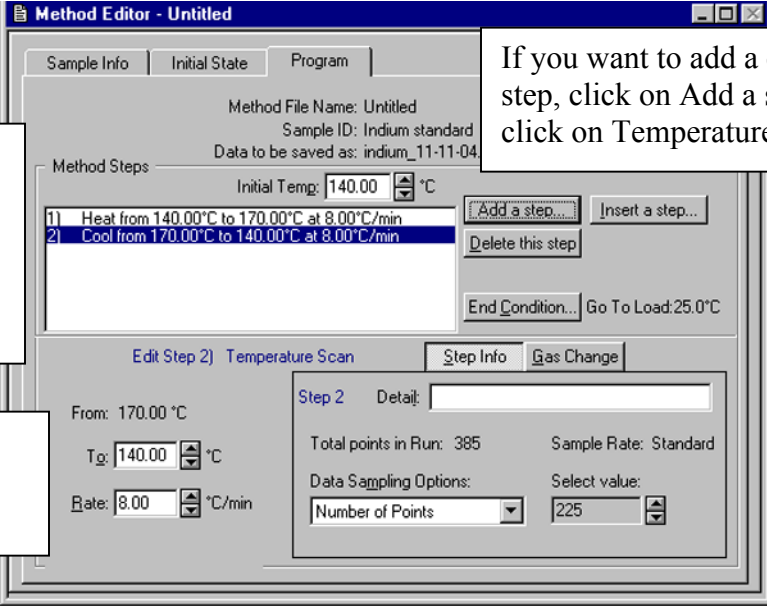
Set Initial Values
Temperature: 140.00 °C
Y Initial: 100.00 mW

Set Purge Gas
Nitrogen * at 20.0 ml/min

Equilibrate Within
 Temperature(+/-): 0.000 °C
 Heat Flow(+/-): 0.000 mW
 Wait no longer than: 0.0 minutes

Baseline File
Directory:
File Name:
 Use Baseline Subtraction

Data Collection
Sample Rate: Standard



Method Steps

1 Heat from 140.00°C to 170.00°C at 8.00°C/min
2 Cool from 170.00°C to 140.00°C at 8.00°C/min

Buttons: Add a step..., Insert a step..., Delete this step, End Condition..., Go To Load: 25.0°C

Edit Step 2) Temperature Scan

From: 170.00 °C
To: 140.00 °C
Rate: 8.00 °C/min

Step 2 Detail

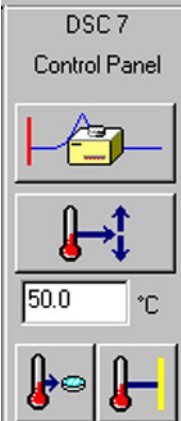
Total points in Run: 385
Sample Rate: Standard
Data Sampling Options: Number of Points: 225

If you want to add a cooling step, click on Add a step, and click on Temperature Scan.

By clicking on each step, you can highlight the step to change its temperatures & heating rate.

Enter your final temp and heating rate here.

18) Tell the DSC to go to your starting temperature.



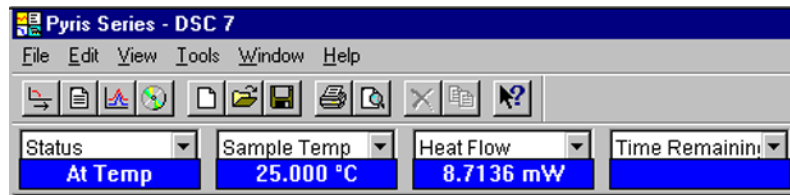
DSC 7 Control Panel

50.0 °C

← Click on the Go to Temp button.

Enter your starting temp here →

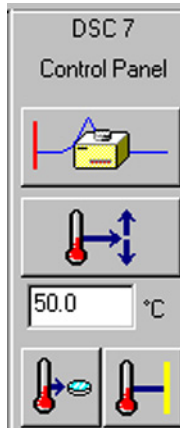
19) When it is "At Temp", monitor the Heat Flow. Wait until the Heat Flow is stable to 0.001 mW. Then click on the Start Run button.



Heat Flow should be stable to 0.001 mW ↑

20) Click on the Start Run button.

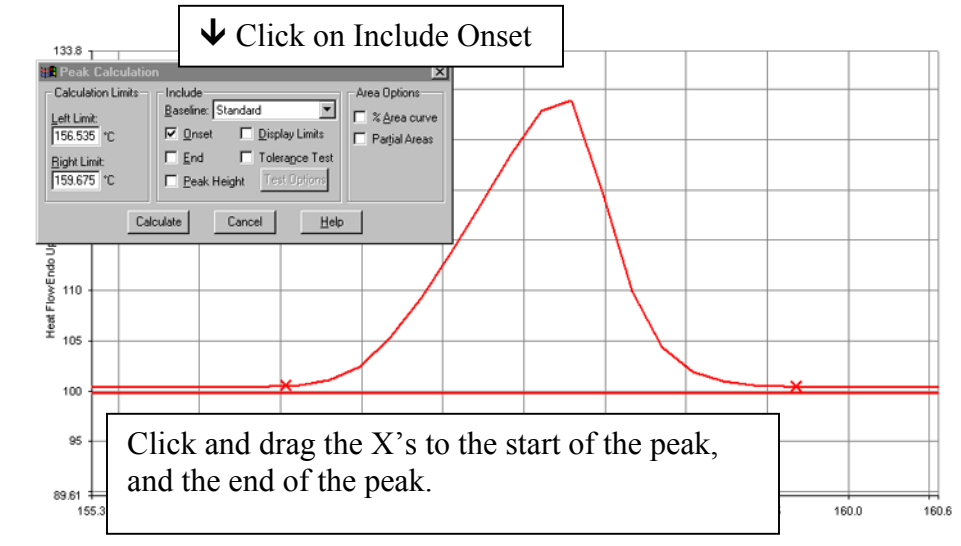
Start Run button →



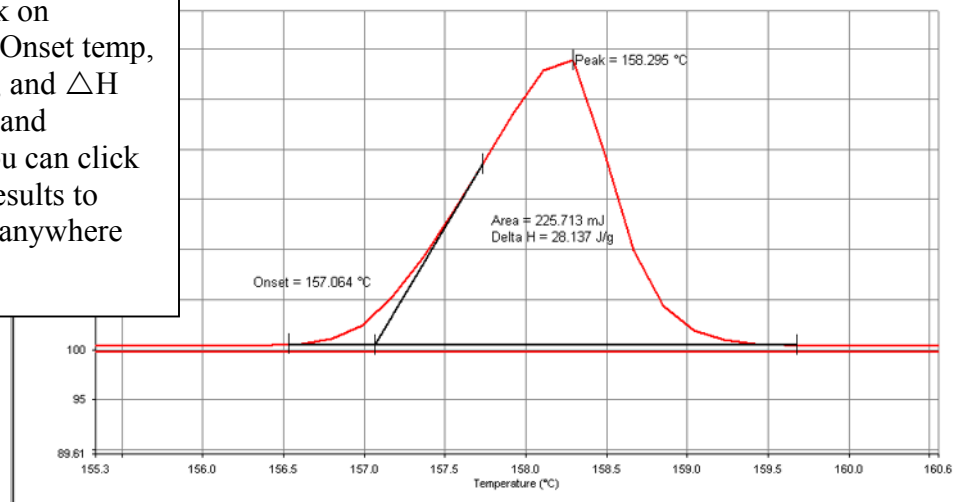
21) To monitor the progress of the run, you can click on the Instrument Viewer window.

V. DSC Data Analysis

- 1) To calculate the onset temperature, peak area, and enthalpy, click on the Calc menu, and click on Peak Area. Click on the option to include Onset. Position the X's at the start of the peak, and at the end of the peak. Then click on Calculate.



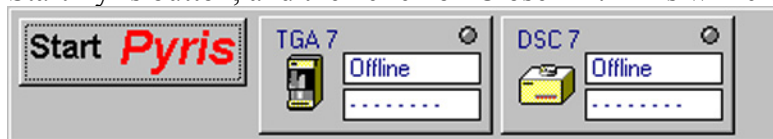
After you click on Calculate, the Onset temp, peak top, area, and ΔH are calculated and displayed. You can click and drag the results to position them anywhere on the graph.



- 2) You must then click on File, and Save Data, in order to save the calculated results with the raw data file.
- 3) To convert a spectrum to ASCII format, click on the Edit menu, and click on Copy. Then go to a text program such as Word, and click on Edit and Paste. The sample data points and the acquisition parameters will be displayed in text format, and can then be saved.

VI. How to shut down the DSC

- 1) First, run another blank spectrum to show that you have left the DSC in good condition. Please print out the blank graph, put your name on it, and put it in the tray near the computer monitor.
- 2) Click on the Start Pyris button, and then click on Close All. This will close the software.



- 3) Log out of Windows.
- 4) Turn off the DSC TAC (Thermal Analysis Controller).



← Turn off the power switch on the DSC TAC.

- 5) Turn off the power switch on the DSC.



← Turn off the power switch on the DSC.

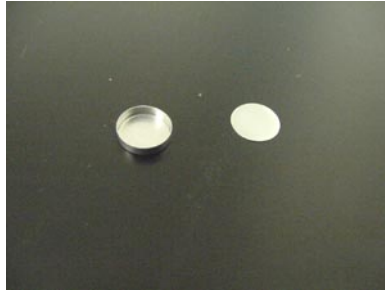
- 6) Turn off the nitrogen gas.



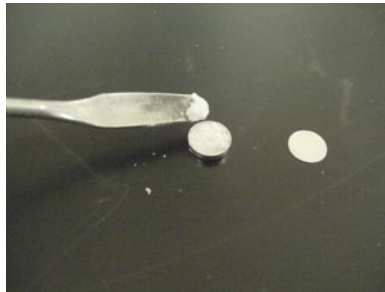
Appendix A

How to prepare samples for DSC

- 1) Tare an empty aluminum pan plus a lid on an analytical balance.



- 2) Put sample into the pan.



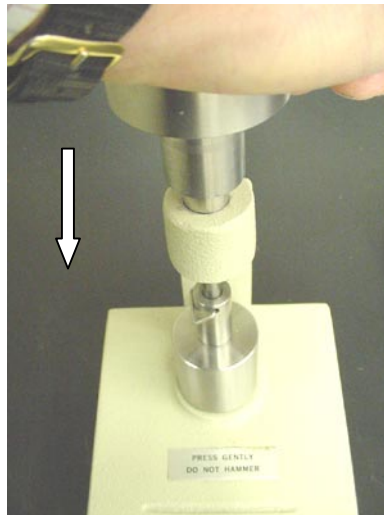
- 3) Put a lid on top of the pan.



- 4) Place the pan into the crimping tool, by lowering down the spring-loaded sample holder.



5) Push down on crimping tool to crimp the sample pan shut.



6) Remove crimped sample pan, and reweigh to get the weight of the sample.

