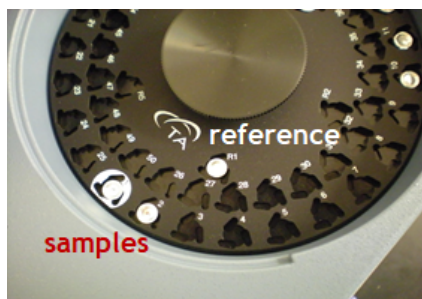


# Differential Scanning Calorimeter

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## Sample Preparation

1. Using the Sartorius Microbalance, weigh and record the mass of your empty aluminum Tzero pan and lid. Then tare the pan and weigh 1-10 mg of sample into it, recording the sample mass to 4 significant figures. Eliminate static electricity on your pan/sample, as the balance is highly sensitive.
2. Seal the sample pan
  - a) Place the pan in the blue die, and place a lid on the pan
  - b) Move the die into the press:
3. Seal the pan using the Tzero Press. *Caution: the press exerts an enormous amount of pressure on the aluminum pan, never insert your fingers in the press!*
4. Place your sample pan in one of the autosampler positions, labeled 1-50.
5. Place an empty pan (with sealed lid) into one of the positions labeled R1-R4 to be used as the reference mass. *Be sure that the reference pan/lid is the same type as your sample pan.*



## Turning on the DSC

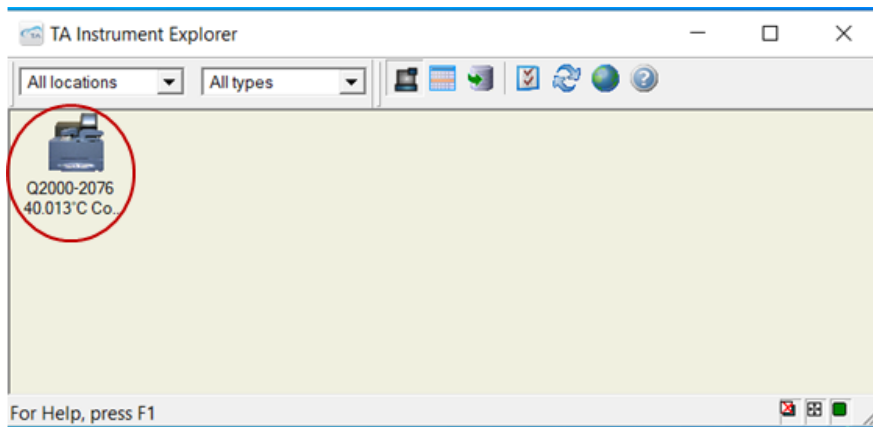
1. Check that the house nitrogen line (to the left of DSC, behind Tosoh) is ON (handle parallel to piping):



2. Open the TA software by clicking on “TA Instrument Explorer” icon.



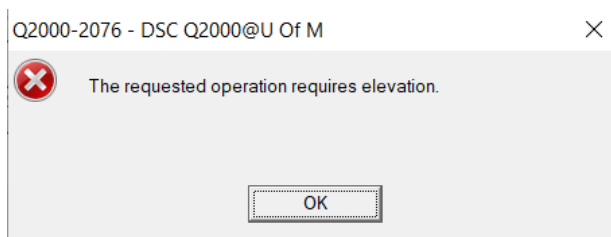
3. Double click the Q2000 icon to connect to the instrument. A two-toned beep and a yellow icon indicates that the instrument has successfully connected.



Successful connection:



4. The following window may pop up, click “OK”



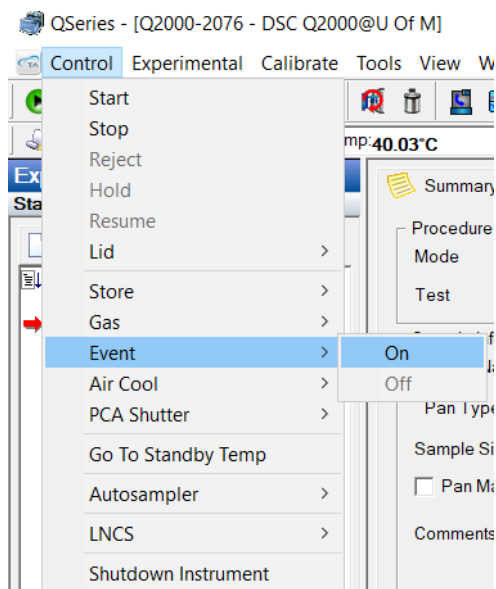
5. Check that the chiller is powered ON. ***Caution: Never turn the chiller on without nitrogen flow! If left on without N<sub>2</sub>, ice/condensation will build up in the cell and damage it!*** There are two ways to turn on the chiller:

a. Through the software - This method is preferred because it will ensure that the chiller gets automatically turned off in the event of a power outage or instrument failure.

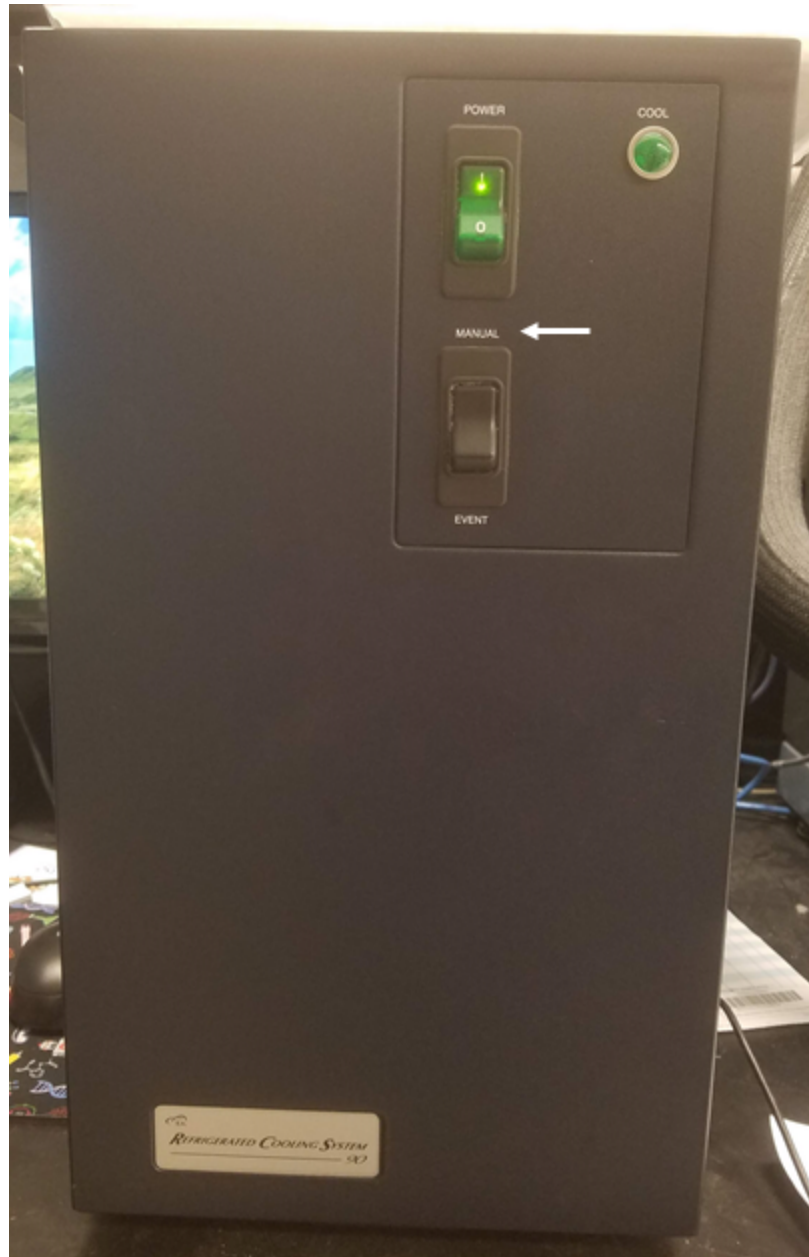
i. Check that the switch on the front of the chiller is flipped to “Event”

[photo]

ii. In the TA software, click “Control.” From the dropdown menu, select “Event,” and click “On.” Once clicked, “on” will be greyed out and “off” will be black.



- b. Manually - This method is not preferred because the chiller may stay on even if the DSC shuts down or the N<sub>2</sub> is turned off, damaging the cell. *Note: As of April 2021, the chiller is turned on in manual mode because the chiller was turning off unexpectedly during sample runs. The TA technician suggested leaving it on manual to solve the issue. As of December 2021, manual mode caused ice build-up in the instrument, chiller returned to event mode.*
  - i. Flip the switch on the front of the chiller to “Manual.” The “Event” option in the software will now appear as if the chiller is turned off (“off” = greyed out, “on” = black)



6. Wait until the Flange Temperature reaches about  $-80\text{ }^{\circ}\text{C}$  before beginning a run. You will see this temperature in the real-time signal monitor window at the upper right of the software:

Signal	Value	^
Method Time	0.00 min	
Segment Time	0.00 min	
Remaining Run Time	0 min	
Temperature	40.03 °C	
Heat Flow	0.106 mW	
Heat Capacity	0.000 mJ/°C	
Set Point Temp	40.00 °C	
Heater Power	42.642 W	
Flange Temperature	-82.19 °C	∨

# Running a Sample

## Entering Sample Information

1. In the experiment tab of the TA Instrument Explorer software, you may create a new sequence by clicking “**new sequence**” on the left.
2. Alternatively, you may add your sample to an existing sequence by clicking “**append run**” or “**insert run**,” depending on where you want the new run in the sequence.

The screenshot shows the TA Instrument Explorer software interface. The 'Experiment' tab is selected, and the 'Standard Sequence' window is open. The 'Standard Sequence' window displays the following information:

- Procedure Summary: Mode: Standard, Test: Custom - \\Chem-2ua42428h5\ta\QProcedures\DSC
- Sample Information: Sample Name: (empty), Pan Type: Tzero Aluminum Hermetic, Sample Size: 0.000 mg, Pan No. 1, Ref. 2, Pan Mass: 0.000 mg (Sample), 0.000 mg (Reference), Comments: (empty)
- Data File Name: \\Chem-2ua42428h5\ta\Data\DSC\2021\EAM\EAM\_5-1-121P

The 'Experiment' tab is highlighted in red, and the 'Run 1' entry in the sequence list is highlighted in blue. A graph on the right shows Heat Flow (mW) vs Temperature (°C) with a plot area from 0.80 to 2.00 on the x-axis and 0.90 to 2.00 on the y-axis.

3. “Run 1” will be added to the sample sequence. Click the text “Run 1” so that it turns blue to edit the sample information.

This close-up screenshot shows the 'Experiment' tab and the 'Standard Sequence' window. The 'Run 1' entry in the sequence list is highlighted in blue, indicating it is selected for editing.



4. In the center panel, select the “Summary” tab to enter your sample information.

The screenshot displays the QSeries software interface. The central panel is on the 'Summary' tab, showing the following fields:

- Procedure Summary:** Mode: Standard; Test: Custom - \\Chem-2ua42428h5\ta\QProcedures\DSC
- Sample Information:** Sample Name: (empty); Pan Type: Tzero Aluminum Hermetic; Sample Size: 0.000 mg; Pan No.: 1; Ref.: 2; Pan Mass: 0.000 mg (Sample) / 0.000 mg (Reference); Comments: (empty); Data File Name: \\Chem-2ua42428h5\ta\Data\DSC\2021\EAM\EAM\_5-1-121P; Network Drive: (empty)

The right sidebar contains a 'Signal' table and a 'Running Segment Description' table:

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	40.03 °C
Heat Flow	0.104 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	40.00 °C
Heater Power	42.698 W
Flange Temperature	-82.15 °C

#	Running Segment Description
1	Equilibrate at 0.00 °C
2	Isothermal for 5.00 min
3	Mark end of cycle 0
4	Ramp 10.000 °C/min to 350.00 °C
5	Isothermal for 5.00 min
6	Mark end of cycle 1
7	Ramp 10.000 °C/min to 0.00 °C
8	Isothermal for 5.00 min
9	Mark end of cycle 2

The bottom right graph shows Heat Flow (mW) on the y-axis (0.90 to 2.00) and Temperature (°C) on the x-axis (0.80 to 2.00). The status bar at the bottom indicates 'Standby' and 'Standard'.

5. Under “Procedure Summary” select “standard” for mode and leave “Custom...” for test.

The screenshot shows the QSeries software interface. The 'Procedure Summary' section has 'Mode' set to 'Standard' and 'Test' set to 'Custom - \\Chem-2ua42428h5\ta\QProcedures\DSC'. The 'Signal' section shows a list of parameters and values, including Method Time (0.00 min), Segment Time (0.00 min), Remaining Run Time (0 min), Temperature (40.03 °C), Heat Flow (0.104 mW), Heat Capacity (0.000 mJ/°C), Set Point Temp (40.00 °C), Heater Power (42.698 W), and Flange Temperature (-82.15 °C). The 'Running Segment Description' table is as follows:

#	Running Segment Description
1	Equilibrate at 0.00 °C
2	Isothermal for 5.00 min
3	Mark end of cycle 0
4	Ramp 10.000 °C/min to 350.00 °C
5	Isothermal for 5.00 min
6	Mark end of cycle 1
7	Ramp 10.000 °C/min to 0.00 °C
8	Isothermal for 5.00 min
9	Mark end of cycle 2

The 'Sample Information' section shows 'Sample Name' as an empty field, 'Pan Type' as 'Tzero Aluminum Hermetic', 'Sample Size' as 0.000 mg, 'Pan No.' as 1, and 'Ref.' as 2. The 'Data File Name' is set to '\\Chem-2ua42428h5\ta\Data\DSC\2021\EAM\EAM\_5-1-121\P'. The status bar at the bottom indicates 'Stand by' and 'Seg 0 in Run 1'.

6. Under “Sample Information,” enter a name for your sample. **NOTE:** This is **NOT** the name that your data will be saved under! It only identifies your sample in the sample sequence.

The close-up screenshot shows the 'Sample Information' dialog box. The 'Sample Name' field contains the text 'DSC\_test-run', which is highlighted by a red arrow. Other fields include 'Pan Type' (Tzero Aluminum Hermetic), 'Sample Size' (0.000 mg), 'Pan No.' (1), 'Ref.' (2), 'Pan Mass' (0.000 mg (Sample) and 0.000 mg (Reference)), 'Comments' (empty), 'Data File Name' (\\Chem-2ua42428h5\ta\Data\DSC\2021\EAM\EAM\_5-1-121\P), and 'Network Drive' (unchecked).

7. Choose the appropriate pan type from the drop-down menu. Choose “Tzero Aluminum Hermetic” for standard and low-mass hermetic pans.

Sample Information

Sample Name: DSC\_test-run

Pan Type: Tzero Aluminum Hermetic

Sample Size: [dropdown menu]

Pan Mass

Comments: [text area]

Data File Name: \\Chem-2ua42428h5\ta\Data\DSC\2021\EAM\EAM\_5-1-121\P

Network Drive

8. Enter the appropriate sample mass, sample pan mass (includes pan and lid), reference mass (includes pan and lid), sample pan number, and reference pan number (from auto sampler).

Sample Information

Sample Name: DSC\_test-run

Pan Type: Tzero Aluminum Hermetic

Sample Size: 3.456 mg

Pan Mass: 53.458 mg (Sample)

Pan No.: 1

Ref.: 1

53.604 mg (Reference)

Comments: [text area]

Data File Name: \\Chem-2ua42428h5\ta\Data\DSC\2021\EAM\EAM\_5-1-121\P

Network Drive

9. Click on the “browse” icon, which will open a pop-up window for you to choose a file location and name for your data. **NOTE:** This is the name that your data will save under!

Sample Information

Sample Name: DSC\_test-run

Pan Type: Tzero Aluminum Hermetic

Sample Size: 3.456 mg

Pan Mass: 53.458 mg (Sample)

Pan No.: 1

Ref.: 1

53.604 mg (Reference)

Comments: [text area]

Data File Name: \\Chem-2ua42428h5\ta\Data\DSC\2021\EAM\EAM\_5-1-121\P

Network Drive

10. Click “Apply” at the bottom of the center panel to save your sample information.

## Setting Method Parameters

1. In the center panel, select the “Procedure” tab to enter method parameters.

The screenshot shows the QSeries software interface. The 'Procedure' tab is selected in the center panel. The 'Method' section displays a list of segments:

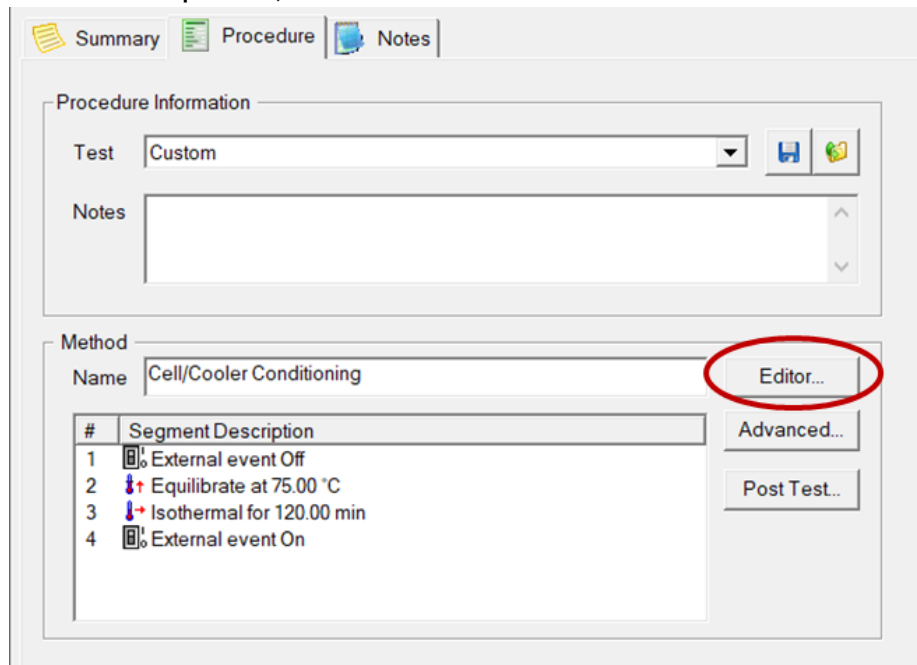
#	Segment Description
1	External event Off
2	Equilibrate at 75.00 °C
3	Isothermal for 120.00 min
4	External event On

The graph on the right shows Heat Flow (mW) on the y-axis (ranging from 0.90 to 2.00) and Temperature (°C) on the x-axis (ranging from 0.80 to 2.00). The status bar at the bottom indicates 'Stand by' and 'Seg 0 in Run 1'.

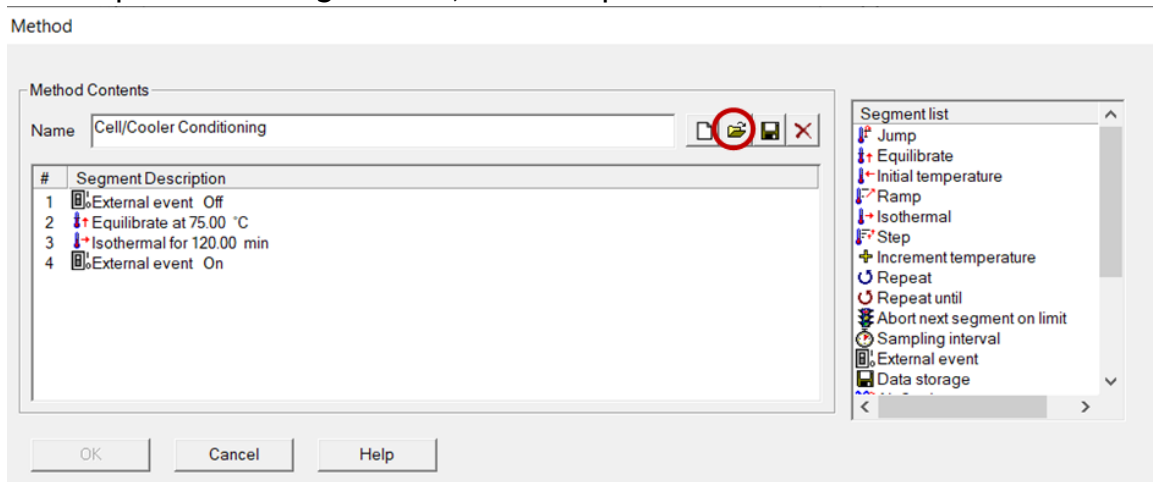
2. Under “Procedure,” select a pre-programmed method or “Custom.” Custom is often more useful because you can choose your desired parameters. You may open a previously saved method by clicking “open.”

The close-up screenshot shows the 'Procedure Information' panel. The 'Test' dropdown menu is open, displaying a list of methods. A red circle highlights the 'open' icon (a folder with a green arrow) next to the 'Custom' option. A red text label 'open pre-saved method' points to this icon.

3. To edit a custom sequence, click “Editor.”

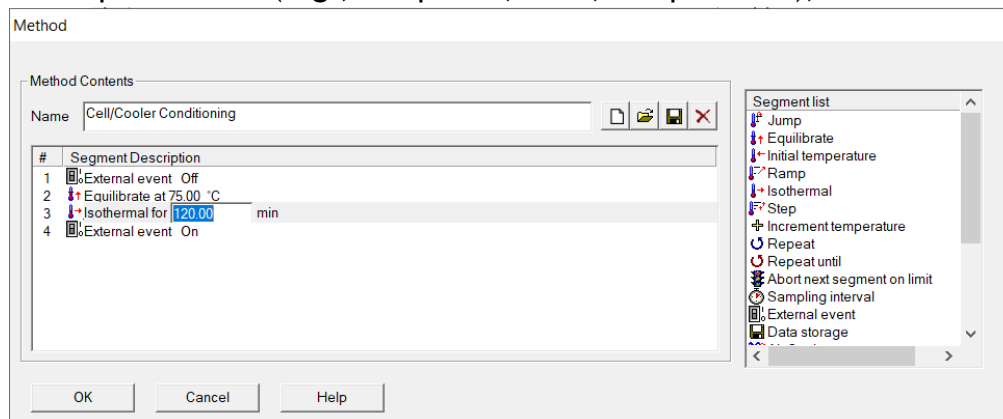


4. In the resulting pop-up, you can edit your method. There are several actions to edit the method:
- To open an existing method, select “open method file.”

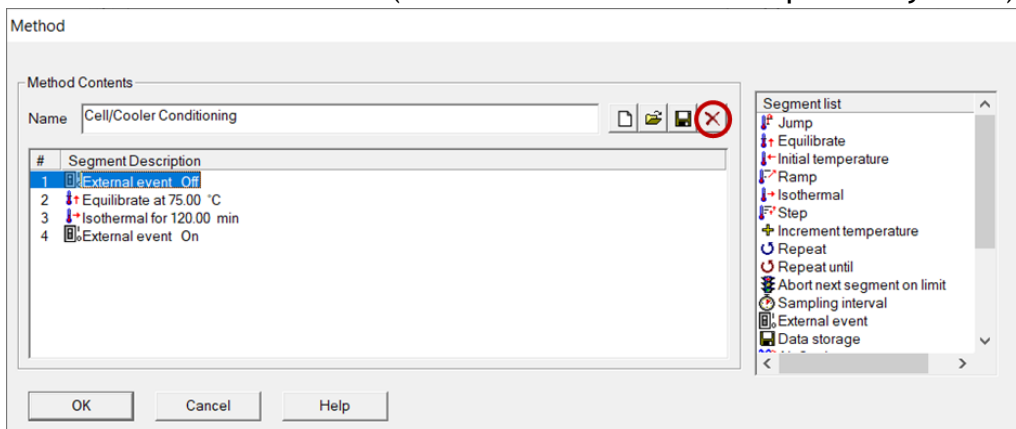


- To add a segment to the end of the existing method, double click on a segment from the list at the right.
- To add a segment anywhere in the method, click and drag a segment from the list at the right anywhere in the method.
- To move an existing segment to a different location in the method, simply click and drag to the desired position.

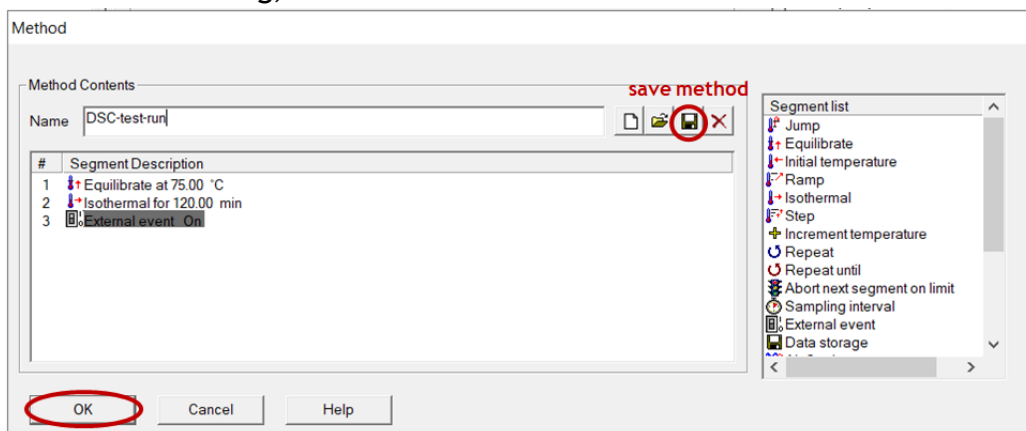
- e. To edit parameters in a given segment, double click on the segment, enter the desired parameters (e.g., ramp rate, time, temperature), then hit “enter.”



- f. To remove an existing segment from the method, single click on the segment and click the “delete” icon (or hit “delete” on the computer keyboard).



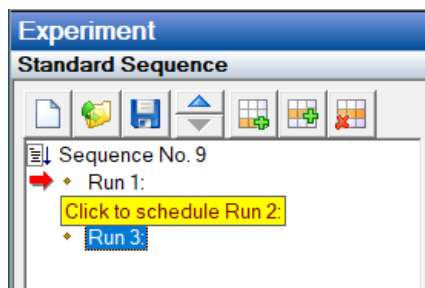
5. To save your custom method for future use, name it and click “save.” To use the method without saving, click “OK.”



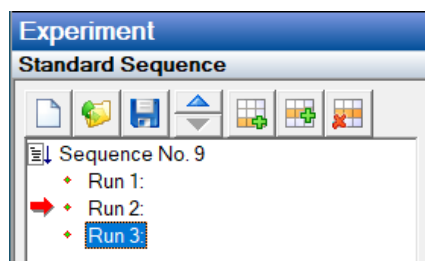
11. Click “Apply” at the bottom of the center panel to save your method parameters.

## Starting the Sample Run Sequence

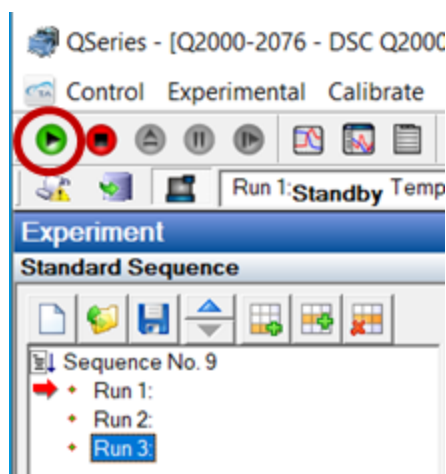
1. The software will begin analysis at whichever sample is marked with the red arrow. Check that the red arrow is at the sample that you wish to analyze. To move the red arrow, hover your mouse over the sample you wish to begin with; a yellow arrow and banner will appear.



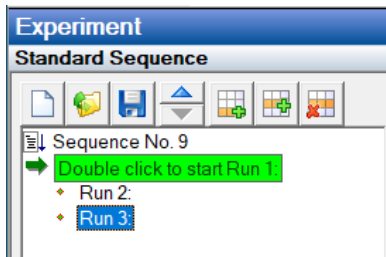
2. Single left click to place the red arrow at the new sample.



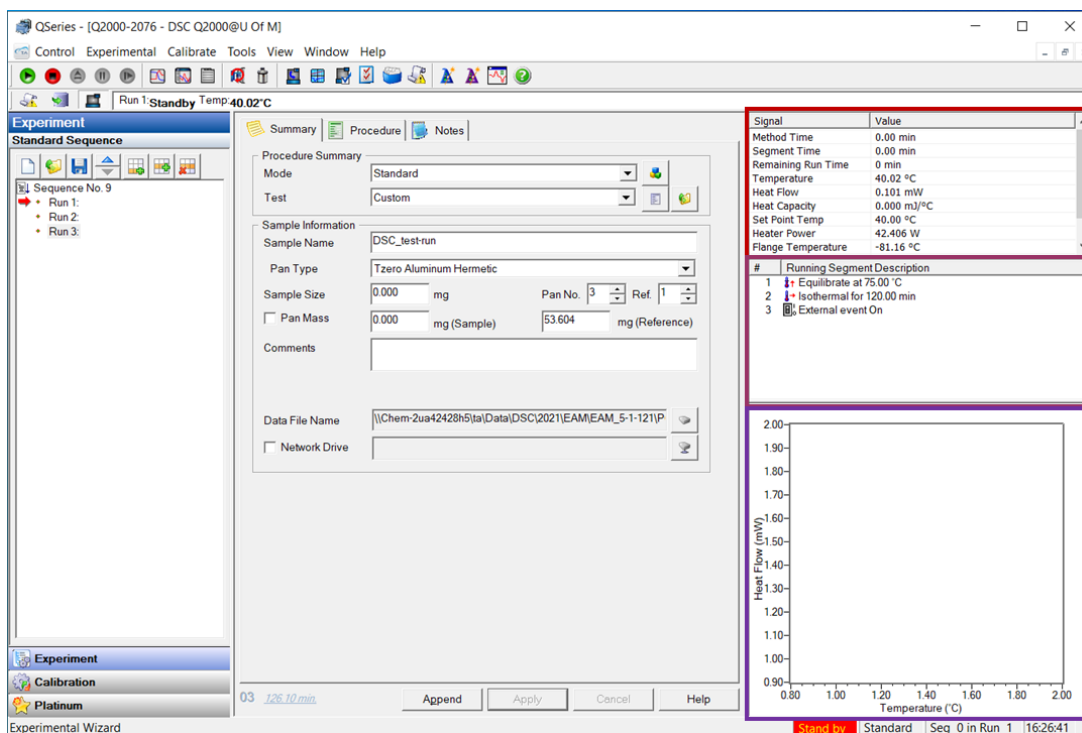
3. There are two ways to start running the sample sequence:
  - a. Click the “play” icon at the upper right to begin the sample sequence.



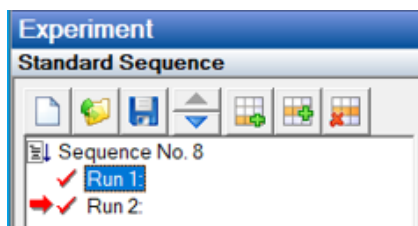
- b. Hover the mouse over the red arrow until a green banner pops up. Double left click to begin the sample sequence.



4. A running sample can be monitored by the right hand panel. The topmost window gives the **real-time instrument conditions**. The middle panel indicates the **current method segment** being run, and the bottom panel displays the **real-time thermogram** for the current sample.



5. After a given sample is finished running, a red check mark will appear in the sample sequence panel at the left.





## Processing the Data

### Opening and Viewing Thermograms

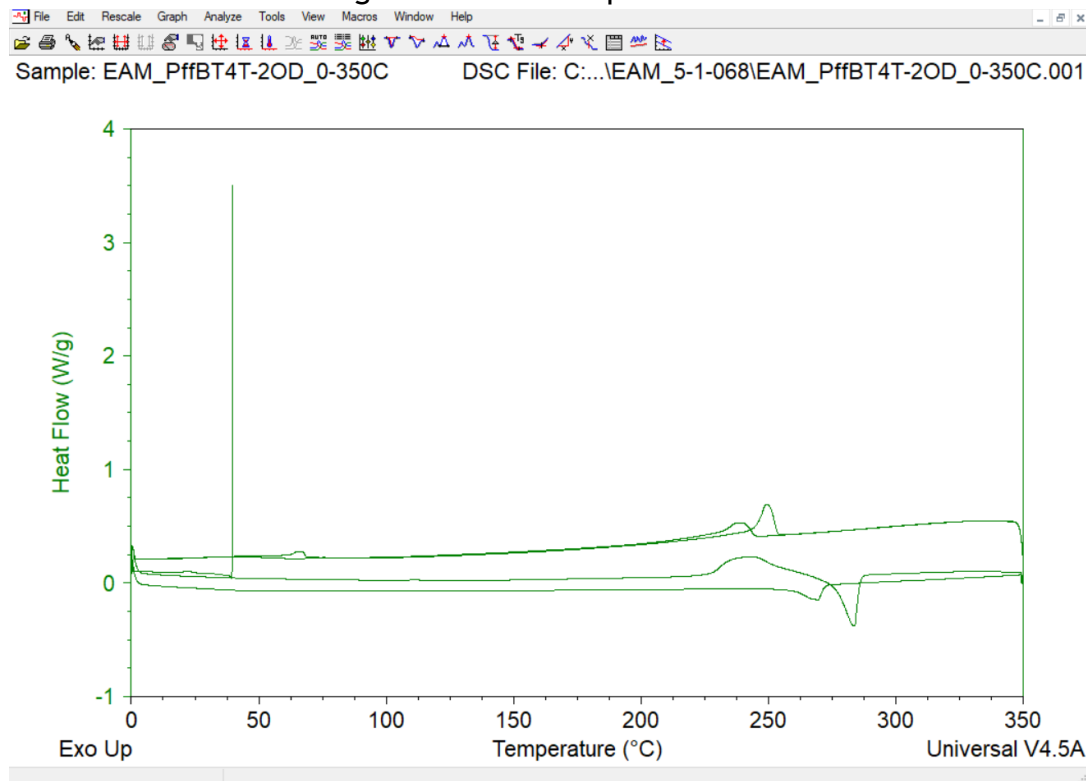
1. On the desktop, open the TA Universal Analysis software by clicking the icon.



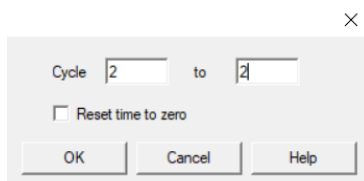
2. To open a data file go to File → Open. Raw data files are “File 00X” file types and have the extension .00X. Select the desired file, then click OK.
3. A window will pop up. You may choose to plot the exotherm (i.e., which direction the exothermic [crystallization] peaks point in the thermogram) up or down. Make sure to label the chosen direction in your final worked-up plot. Click OK.

A screenshot of the 'Open' dialog box in TA Universal Analysis software. The window has a title bar with a close button (X). It contains several sections: 'File' with fields for Name (C:\...\DSC\2020\EAM\EAM\_5-1-048\EAM\_PfBT4T-20D.002) and Date (12-Aug-2020 10:38), and buttons for 'Signals...', 'Units...', 'Instr Params...', and 'Data Limits...'; 'Module type' set to 'DSC Standard Cell RC'; 'Parameters' section with fields for Sample (EAM\_PfBT4T-20D), Size (4.4970 mg), Operator (DEF), Method (EAM\_OPV\_materials), and Comment; an 'Exotherm' dropdown menu set to 'Up' (highlighted with a red box); and a 'Cell constant' field set to 1.0317. A checked 'Restore analysis' checkbox is at the bottom. At the very bottom are buttons for '<< Previous', 'OK', 'Same As', 'Cancel', and 'Help'.

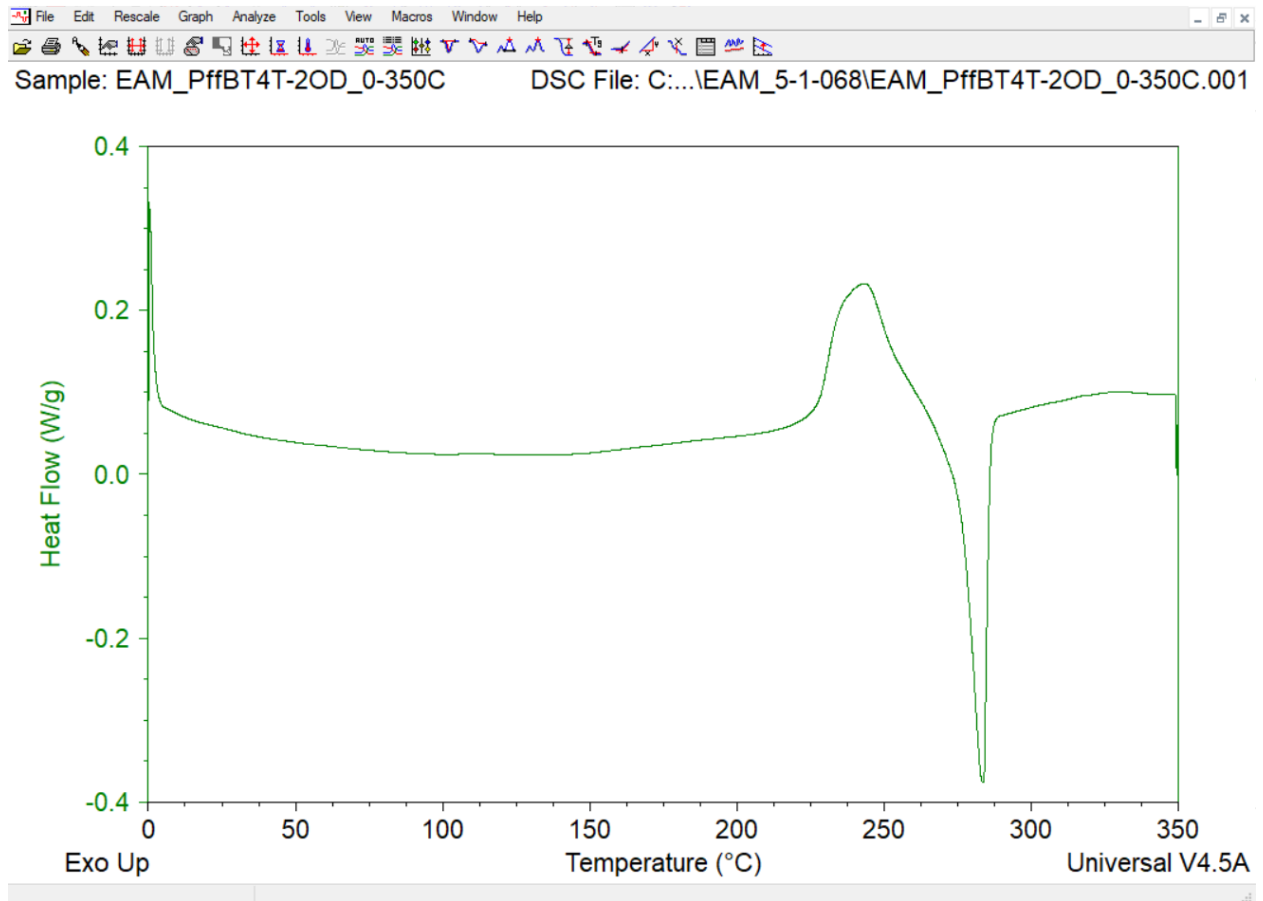
4. The file will look something like this when opened:



5. To isolate a particular cycle (e.g., first heat, second cool), select “Graph” → “Data Limits” → “Cycle.” Input the number for the cycles you wish to view and click “OK.” Note that the cycles will be numbered according to the cycle markers you chose in your method (see pg 11). For example, in the plot above, cycle 2 is the first heat. Selecting “cycle 2 to 2,”



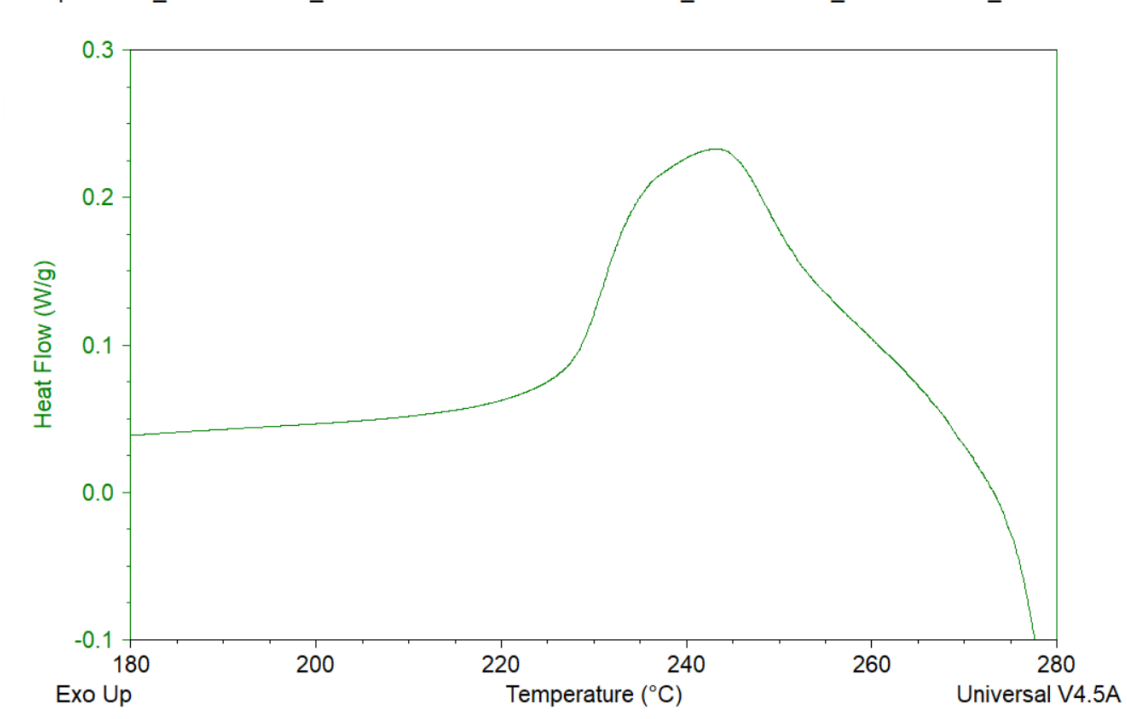
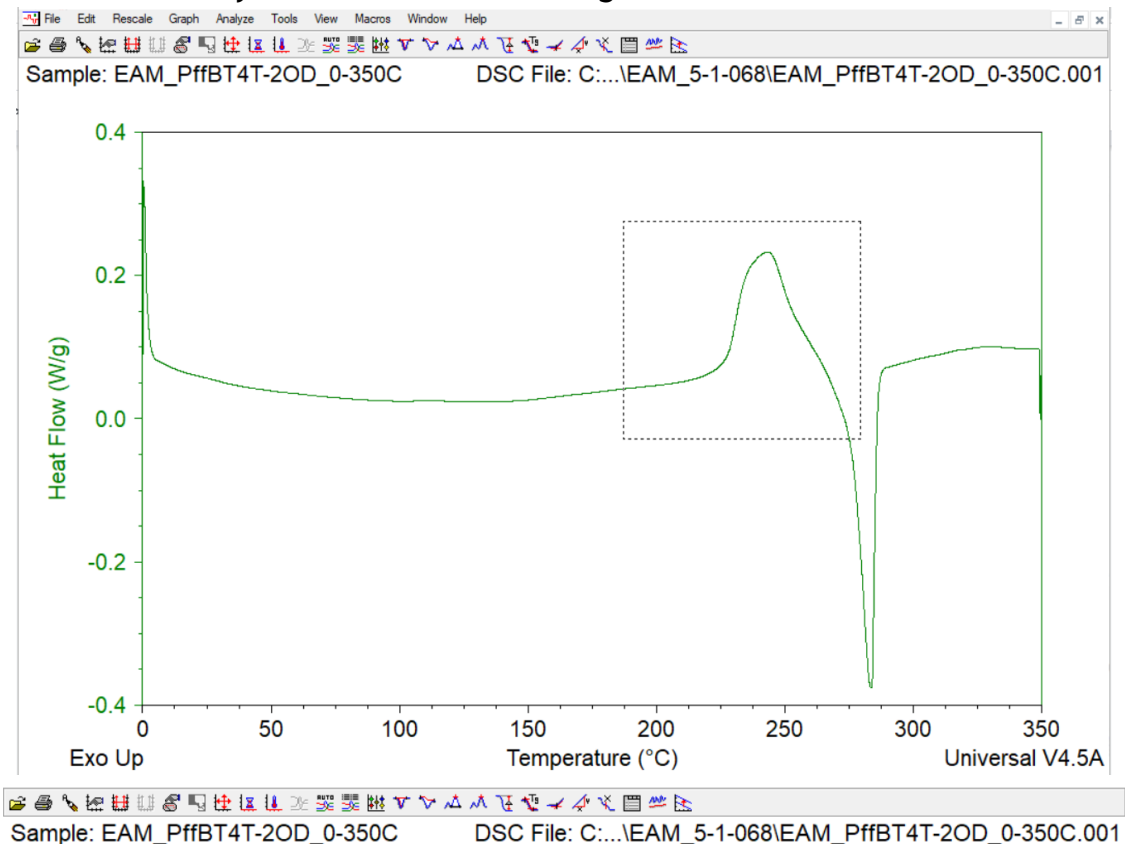
now shows:



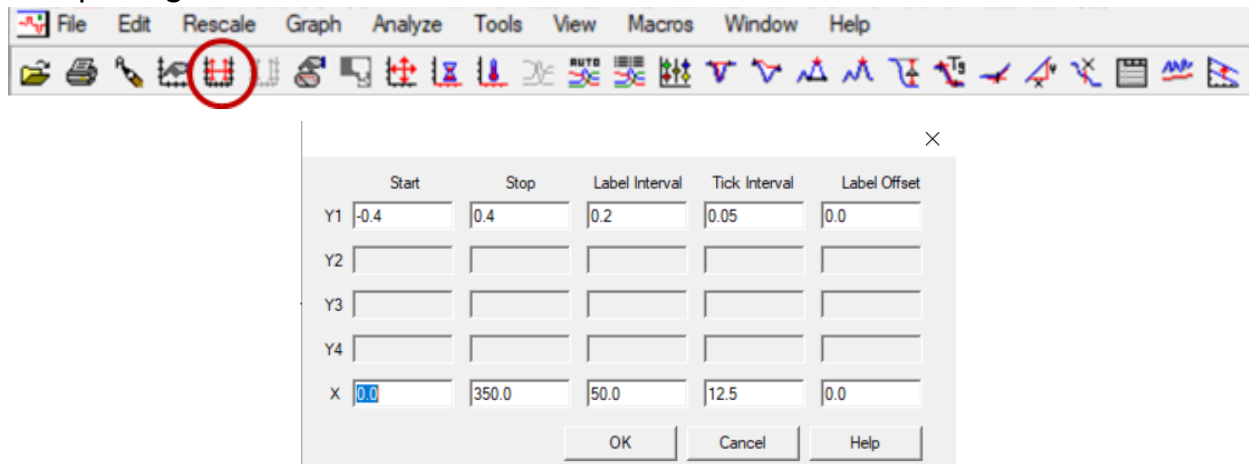
6. To view the entire trace at any time, click the “Data Limits Full Range” icon from the top toolbar.



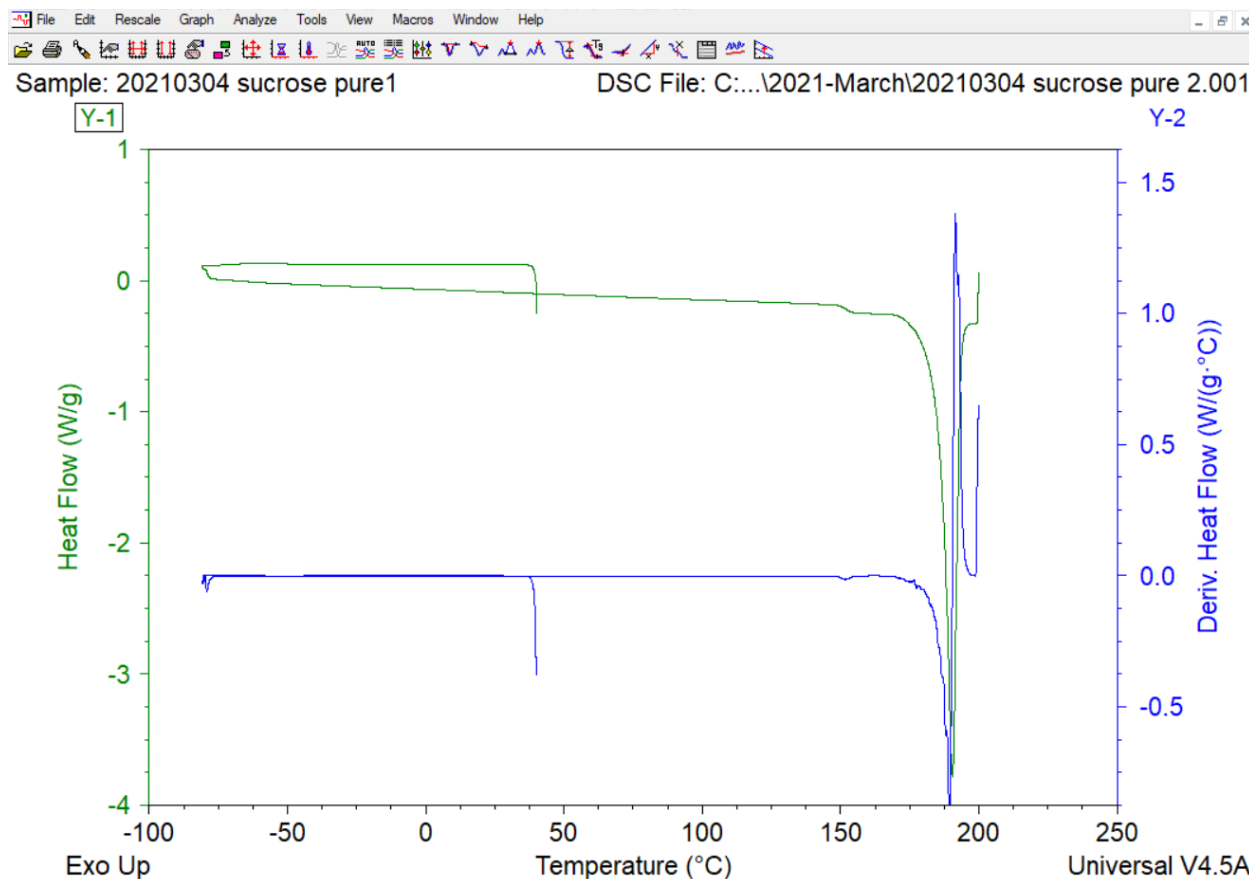
- 7. To zoom in on any part of the trace, click and drag a rectangle around the area of interest. Click anywhere within the rectangle to zoom. Hit "Ctrl+z" to zoom out.



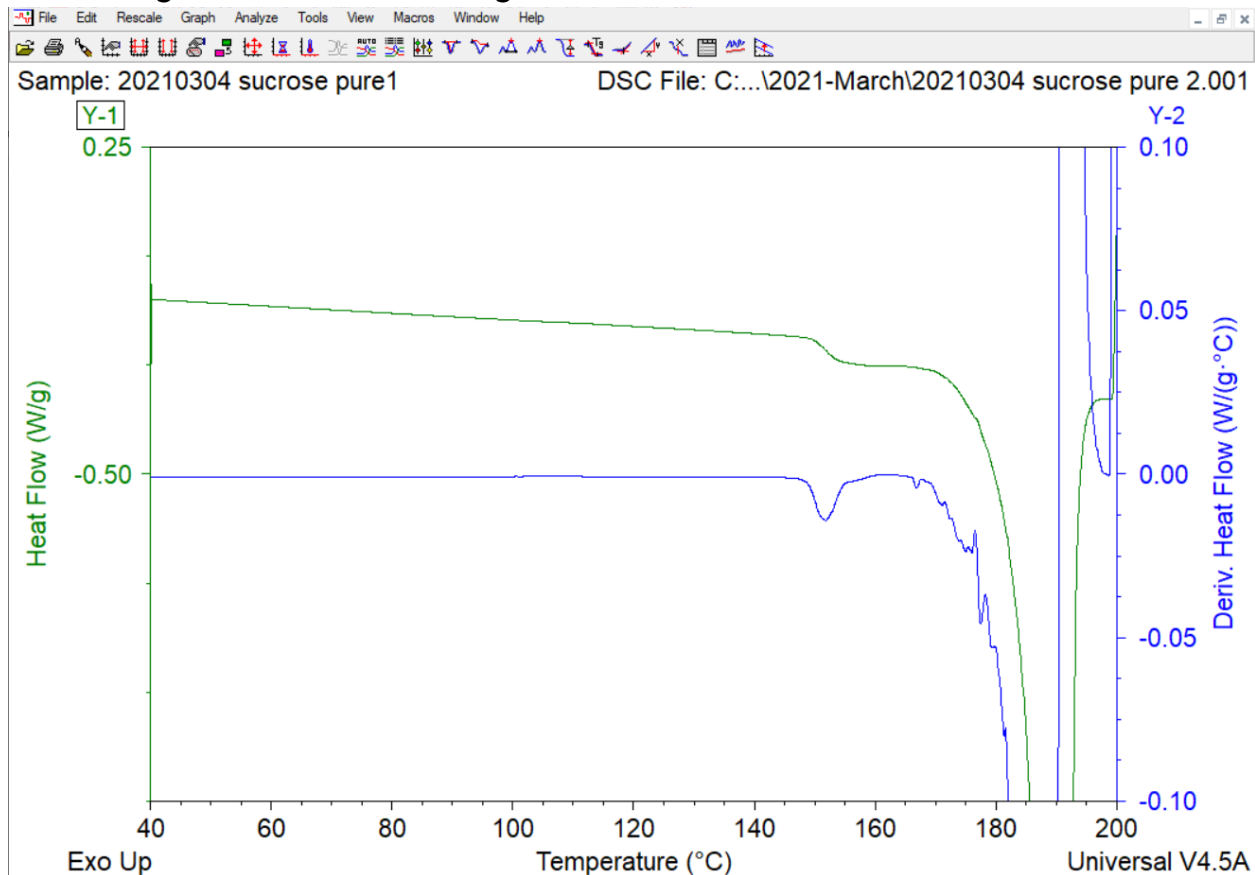
- To zoom back out, select the “Full Scale All” icon. Alternatively, you can manually choose axis limits for your thermogram by selecting “Rescale” → “Manual” and inputting the desired limits.



- To view other signals, like the first or second derivatives of your trace, select “Graph” → “Signals.” In the resulting pop-up window, select the signals that you want to appear. Multiple traces with multiple y-axes will now appear on the plot.



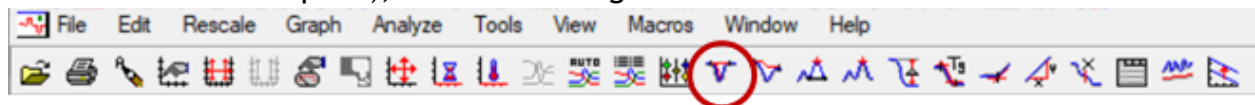
10. Optional: Click and drag on a given trace to adjust the offset relative to other traces. Manually adjust each y-axis by right-clicking on the axis numbering and entering desired numerical ranges.



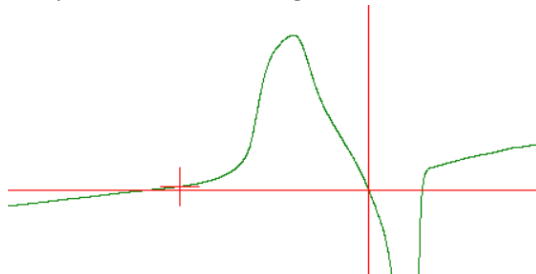
11. Click on a given trace to make it the active trace. The label at the top will now be boxed in (see Y-1, above). Integrate and label the desired peaks for that trace (see below).

## Labeling and Integrating Peaks

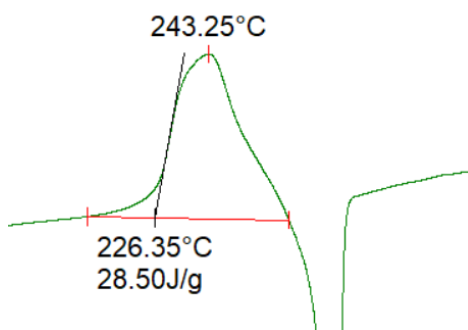
1. To integrate a peak with a symmetrical baseline (baseline is level or linear before/after the peak), click the “Integrate Peak Linear” icon.



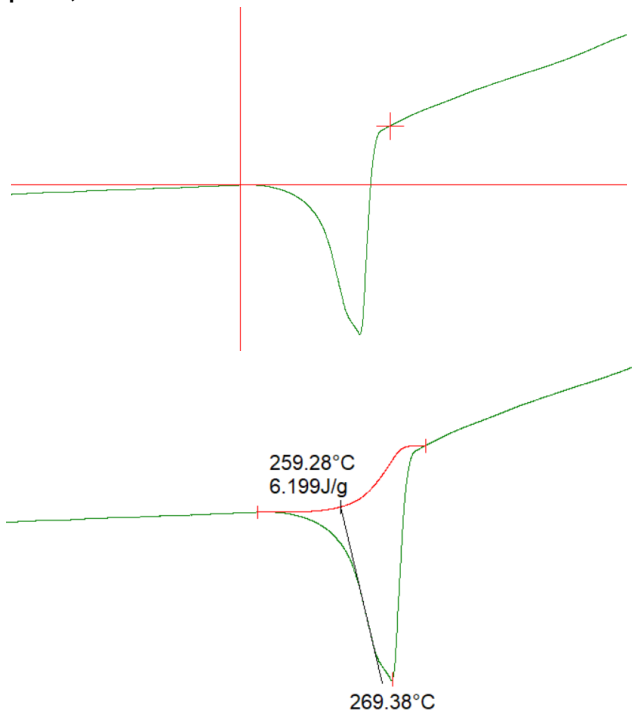
- Click and drag the red “+” symbols to seat each one on either side of the peak. You may alternatively double click the location to set the limit. Ideally, the horizontal lines in each symbol should align with each other and the baseline.



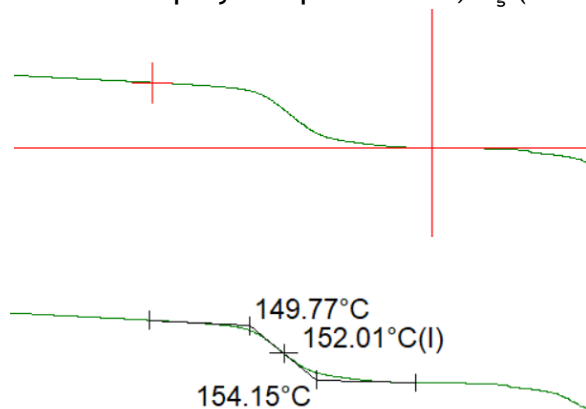
- Hit “enter” to integrate the peak. The peak onset, peak maximum, and peak area will now be labeled on the plot.



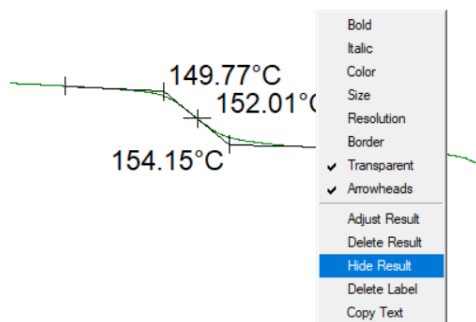
- To integrate a peak with an asymmetrical baseline (baseline undergoes a step up or down before/after the peak), click the “Integrate Peak Sig Horizontal” icon. Set the limits as in step 10, then hit “enter.”



- To identify a glass transition temperature ( $T_g$ ), click the “Glass/Step Transition” icon. Set the limits by clicking/dragging or double clicking. Set the limits as close to the step transition as you can while aligning the horizontal red lines tangent to the baseline. Hit “enter” to display the peak onset,  $T_g$  (black “+”), and endset.



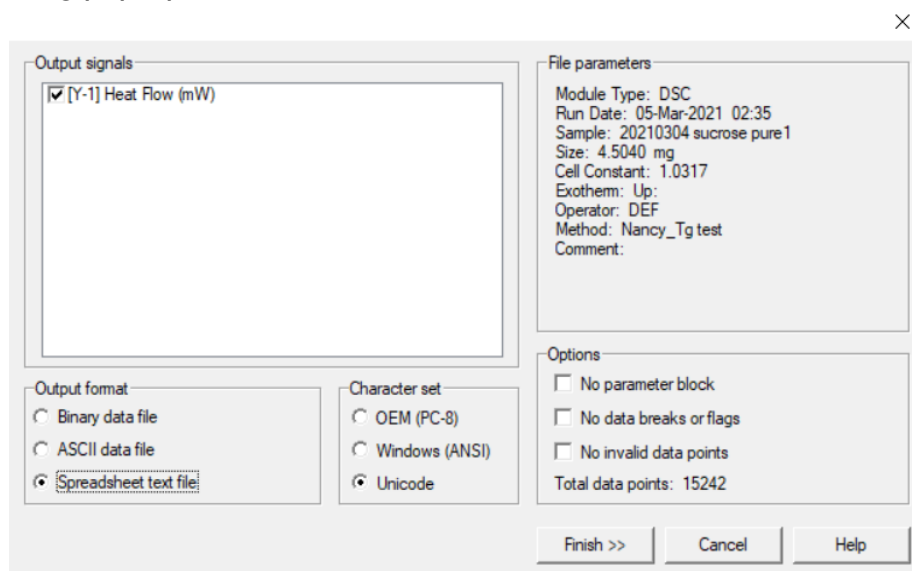
- A thermogram can look cluttered as you integrate peaks. To hide the labels (while keeping the analysis), right click on a given label and click “Hide Result.”



- To export your integrated/labeled peak data to MS Excel, select “View” → “Results” → “Spreadsheet.” A new spreadsheet will open with peak data.



- To export the thermogram trace as a text file, select “File” → “Export” → “Plot Signals Only” and select ASCII or Spreadsheet Text File under “Output Format” in the resulting pop-up window. Then click “finish” and save.

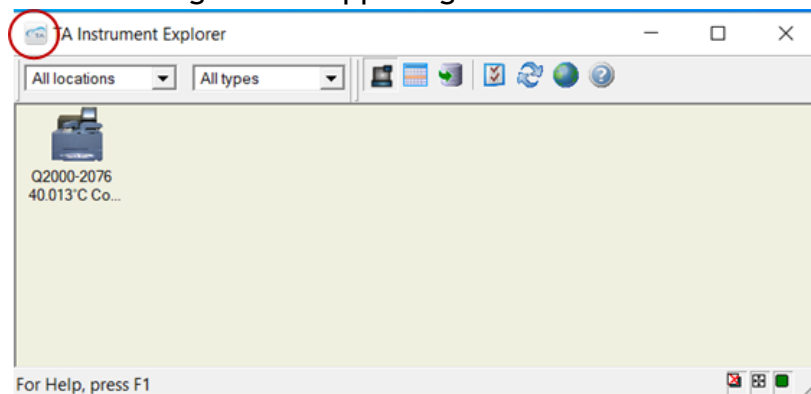


## Shutting down the DSC

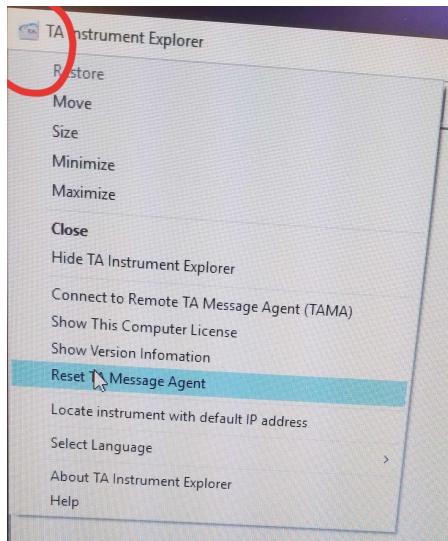
- If someone is planning to use the DSC in the next few days, DO NOT shut down the instrument. The majority of energy consumption is expended in cooling the flange from room temp to -80 °C. Therefore, repeated ON/OFF cycles are not recommended.
- If nobody plans to use the DSC for the next week or longer, shut down the machine as follows:
  - Power down the chiller by clicking Control → Event → OFF in the Q2000 software or by manually flipping the switch on the front of the instrument.
  - Wait for the flange temperature to equilibrate to ambient temp with the nitrogen still flowing.
  - Turn off the house nitrogen line.

## Troubleshooting Common Errors

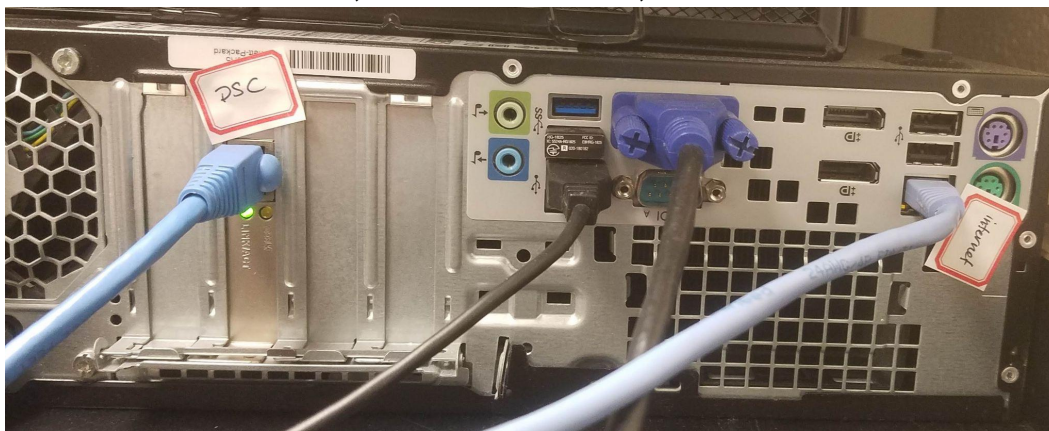
1. Many errors can be resolved by restarting the instrument.
  - a. Push in the thin peg on the lower back right of the instrument
2. “The requested operation requires elevation”
  - a. Ignore this, click “OK”
3. “Sample purge gas cannot reach specified flow rate. (626)”
  - a. Check that the N2 gas gauge reads ~20 psi (behind the HT-GPC)
  - b. Check that there aren't any kinks in the tubing
  - c. Turn the instrument off/on
  - d. If necessary, remove the tubing from the back of the instrument and confirm gas flow.
  - e. Check the cell standby “temperature” (listed in upper right corner of software screen). Standby temp should be 40 °C. If it is much lower (e.g., -90° C), then the purge gas port has likely frozen up and no longer flows. The freezing would be that you have gotten some moisture inside the cell and if someone presses the stop button on the DSC, the standby temp is cancelled and the cell heater is turned off. If the RCS is on then, your cell temp will drop to as low as possible causing any moisture to freeze up.
    - i. Always keep the RCS in “Event” mode and use the Control menu to toggle “event” on or off.
    - ii. From your test drop down menu, you should select “Cell Cooler Conditioning” and run that. This should thaw your cell out and you should see flow again. It will also help dry any moisture that has built up in the cell as well as the cooling head.
4. “The connection to the instrument cannot be made because it is currently offline” or “another user is currently logged in” preventing you from connecting to the instrument.
  - a. Click the TA logo in the upper right of the Instrumental Analysis window



- a.
  - b. Select “Reset TA Message Agent” from the dropdown menu



- c. Try connecting to the instrument again
- d. If the instrument still won't connect, confirm that the ethernet connecting cables for the DSC and the internet are in the correct ports on the computer tower
  - i. Unplug/replug every the DSC cable on both the computer and instrument, restart instrument, restart software.



5. "Power supply impedance error"
  - a. Can occur when starting up the instrument. Ignore this if you're still able to run your samples.

## Resources

### TA Applications Notes

TA Instruments has a collection of "[Applications Notes](#)" with practical procedures and theory to learn how to do common DSC experiments.

## TA Quick Start Videos

TA Instruments has a collection of [videos](#) to learn the basics of how to use their instruments and software. Production quality is low but info quality is high!

- [Universal Analysis Quick Start Video](#) - this one is really helpful if you're new to the software
- [Universal Analysis Advanced Video](#) - good for learning in-depth tricks
- [Modulated Temperature \(DSC\) Video](#) - good for learning MDSC basics

## Other References

There are pdfs on the group drive with info about DSC theory, application, and interpretation.

- [Interpreting DSC Data \(from USCB\)](#)
- [How to Interpret Unexpected Transitions in DSC](#)
- [Differential Scanning Calorimetry](#) - comprehensive book chapter
  - Menczel, J. D.; Judovits, L.; Prime, R. B.; Bair, H. E.; Reading, M.; Swier, S. Differential Scanning Calorimetry (DSC). In *Thermal Analysis of Polymers: Fundamentals and Applications*; Menczel, J. D. and Prime, R. B. Eds.; John Wiley & Sons Inc.: Hoboken, NJ, 2004; pp 2–239.

## Service Help

24 h TA service helpline: (302) 427-4050 - they're actually very helpful!