

Instructions for using the PerkinElmer DSC-8500

Instrument Summary:

Differential scanning calorimetry (DSC) measures the amount of energy required to heat or cool a sample to a given temperature. This is accomplished by measuring the energy required to heat a “sample pan” containing a material of interest to a given temperature versus that required to heat an empty “reference pan” to the same temperature. DSC provides characteristic information about a material including its: phase changes, crystallization, glass transition, heat of vaporization etc. The PerkinElmer DSC-8500 can be used to analyze these phenomena at temperatures ranging from approximately -80 °C to ~750 °C. These experiments can be conducted during temperature ramping experiments or operation with cyclical periods of heating and cooling.

Perkin Elmer DSC 8500

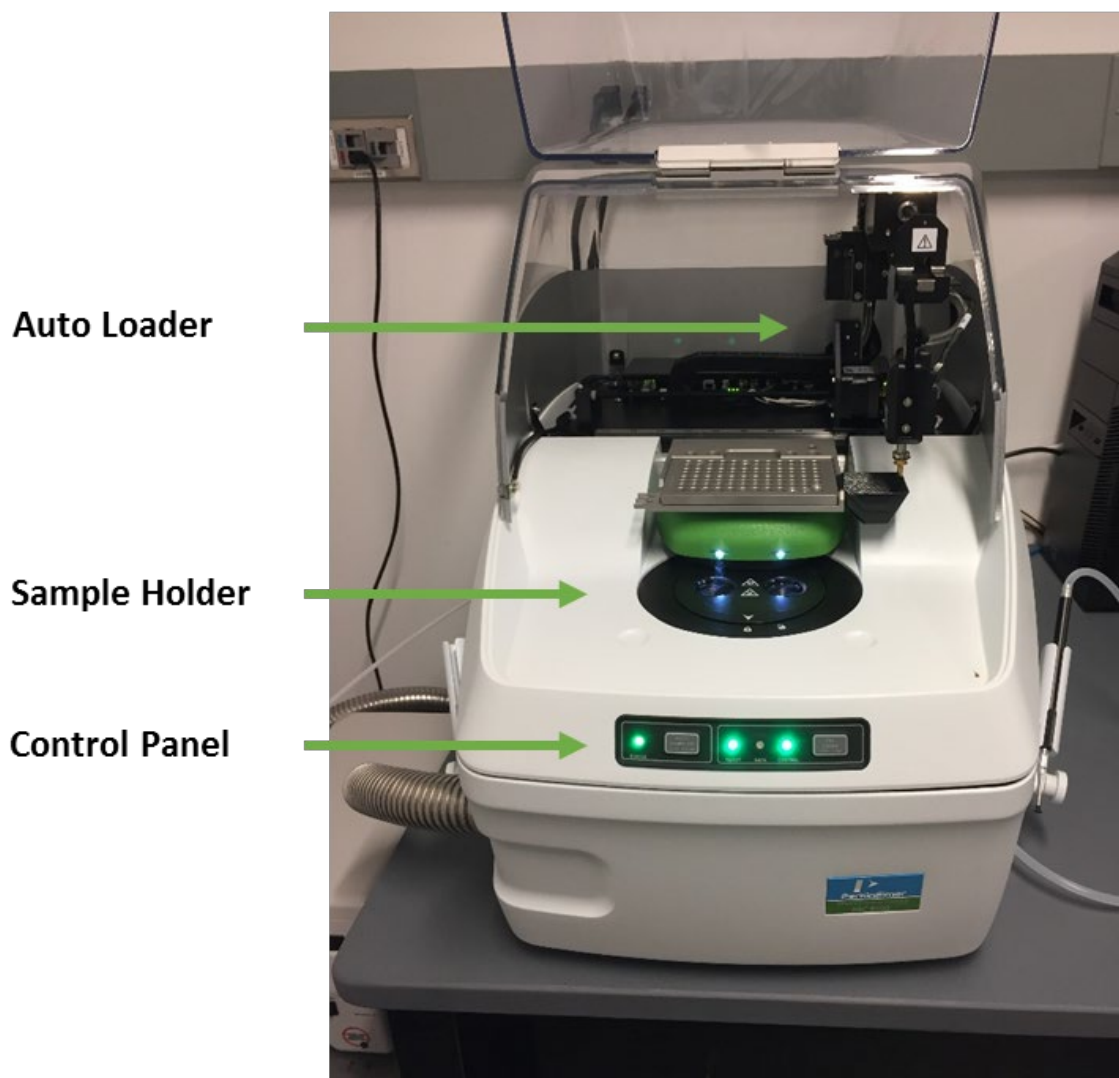


Figure 1. Perkin Elmer DSC 8500.

Safety and Precautions:

- Check the temperature of the sample holder before removing items from the instrument.
- Check the melting point of the sample pan, ensure that your experiment does not exceed the temperature rating of the pan.
- Use a pair of tweezers or the vacupick to remove items from the holder.
- Do not allow loose sample to fall in the sample holder or in the cracks around the holder.

I. Turning on the System:

1. **Turn on the Chiller** – Turn on the main power “Toggle Switch” located on the back of the chiller, on the bottom-left hand side.
2. **Turn on the DSC** – Turn on the main power “Toggle Switch” located on the back of the DSC, on the bottom-right hand side. Wait a few moments for the instrument to turn on.
3. **Open Pyris (Software)** – Click the icon for the DSC unit on the Pyris software banner.
4. **Initialize the Cooling Process** – Click the “Cooler on/off” button (Barrel Icon) on the bottom-right hand side of the Pyris window to start cooling the chiller. The icon will turn blue when the chiller’s cooling process is functioning.



Cooler On/Off “Barrel Icon”

***Note:** The chiller must be turned on 1-2 hours prior to sample analysis in order to allow the temperature to stabilize. ***Warning:** Do not leave the cover open for extended periods of time while the chiller is in operation or ice will collect on the instrument.

II. Sample Preparation and Encapsulation:

1. **Select A Pan Type** – Select the proper pan for your experiment. Ensure that your pan will not melt or form an alloy within the temperature range of your experiment.

***Warning: Pan Melting** – Sample pans can melt at elevated temperatures and severely damage the instrument. Only utilize temperatures at which your sample pan will remain stable. Pans come in a variety of materials (i.e. aluminum, platinum, copper etc.); see the table below in order to pick the proper pan for your experiment.

***Warning: Pan/Sample Alloying** – Your sample pan may interact with your sample during heating to form an alloy. This will negatively affect your experiment and could damage the instrument. See the table below in order to pick the proper pan for your sample and to prevent the formation of an alloy.

***Warning: Pan Rupturing** – Your sample may evolve gases or vapors at elevated temperatures, resulting in high pressures within a sealed sample pan; this could cause the pan to rupture. Select the proper pan for volatile samples.

Table 1. List of sample pan types, temperature range, and material compatibility.

Pan Type	Temperature Range	Part Number	Description
Aluminum	< 500 °C	0219-0041	Used for nonvolatile samples (polymers etc.) for polymer melting, glass transition, and crystallization studies.
Gold	< 700 °C	0219-0042	Used for nonmetals, ceramics, coal, soils. Good for performing anti-oxidant testing, thermoset tests, melting point testing etc.
Copper	< 850 °C	0319-0026	Used for high temperature experiments (i.e. melting point studies) or when aluminum pans are not suitable.
Volatile	< 500 °C	0219-0062	Allows aqueous characterization up to 2 ATM and 100 °C. Used for phase changes, heat of vaporization, heat of fusion, etc.
Alumina	< 700 °C	N519-0180	Used for oxidation studies or when metallic alloying is a concern.

- 2. Weigh and Load your Sample** – Weigh and tare an empty sample pan. Add 0.5-30 mg of sample to the pan and record the weight. In order to improve data collection, ensure that the sample is spread evenly along the base of the pan. For decomposition studies, only use a small amount of sample. Use **gloves** when handling the pans to prevent contamination.

***Note:** There are two general pan types: standard pans and volatile pans. The “**Standard Crimper Press**” (See **Figure 2a**) is used to encapsulate samples in standard pans (See the pan on the left in **Figure 2b**); while volatile pans (See the pan on the right in **Figure 2b**) require the “**Universal Crimper Press**” (See **Figure 3**).

- 3. Crimping Standard Sample Pans** – Pull the lower base of the “**Standard Crimper Press**” down, place the filled sample pan and lid on the cylinder, slowly release the base and allow it to rise, **slowly and gradually press down on the handle** to crimp the pan. Pull the base down and remove the pan from the crimper.
- 4. Crimping Volatile Sample Pans (Optional)** – The pan is first placed in a die assembly kit, then inserted into the crimper, crimped, and the excess metal is sheared from the pan.
 - a. Insert Sample Pan into the Die** – Use tweezers to place the bottom portion of the sample pan in the sample die. Ensure that the pan is sitting flush in the die. Place a lid on top of the pan with the dome side of the lid facing down (See **Figure 3**).
 - b. Place the Die in the Crimper Press** – Place the die and sample pan assembly on the sample table of the “**Universal Crimper Press**”. If your sample is hard, place the cylindrical insert in the base of the die. Rotate the arm of the crimper down to the black stopper knob, hold for 5 seconds, and then slowly rotate the arm back to the open position. Do not allow the arm to swing back when releasing it.

- c. **Shear the Excess Metal off of the Sample Pan** – Remove the die assembly from the crimper and place it on the black “Ejector Knob”. Place the shear ring (cone side facing upwards) over the sample pan. Push down on the shear ring to eject the sample pan from the die and shear off the excess rim.
5. **Prepare a Reference Pan** – Follow the same procedure listed above to create an empty reference pan.

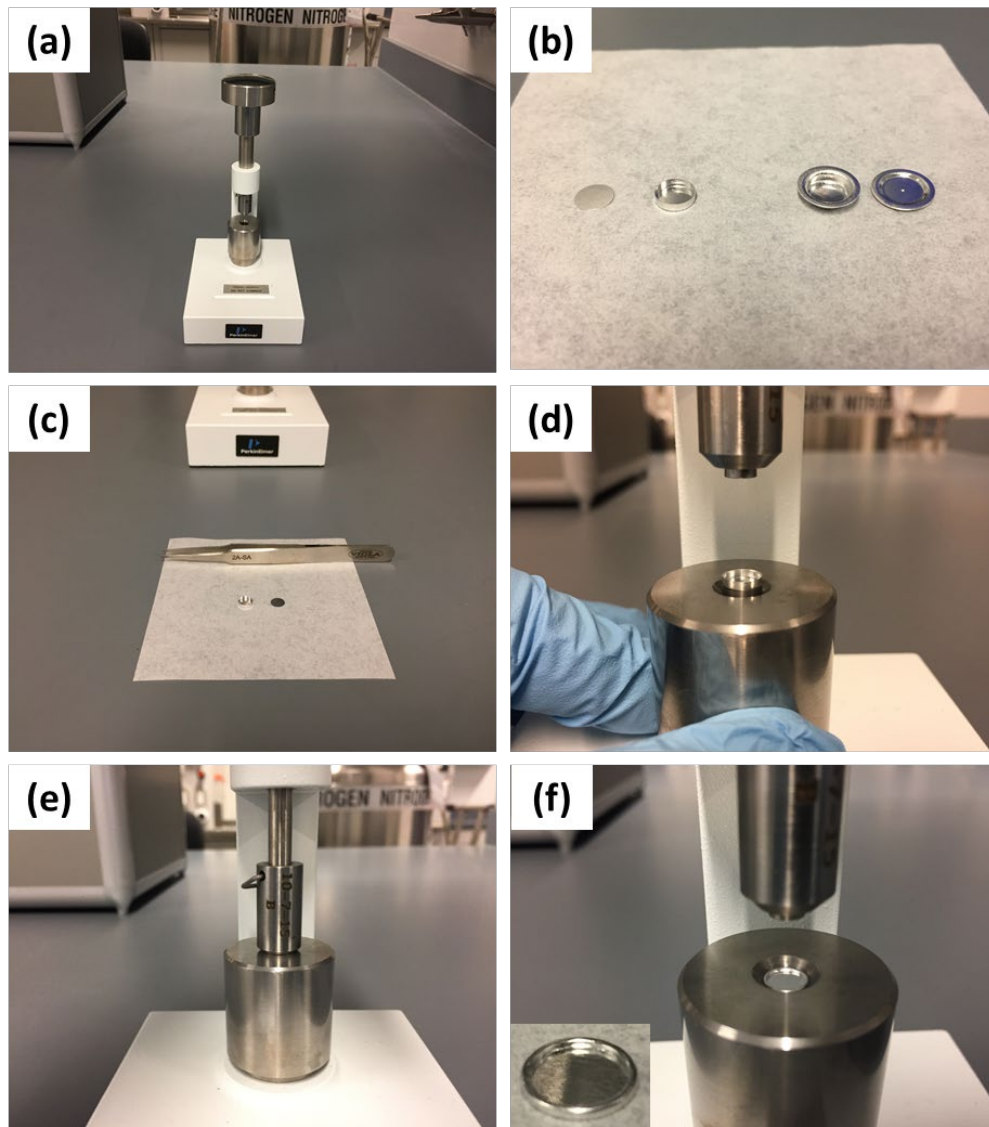


Figure 2. Pan encapsulation with the standard crimper press: (a) the standard crimper press; (b) aluminum pans including: a standard pan (Left) and a volatile pan (Right); (c) loading a standard pan; (d) inserting the pan in the standard crimper press; (e) crimping the pan; (f) removing the crimped pan (insert).

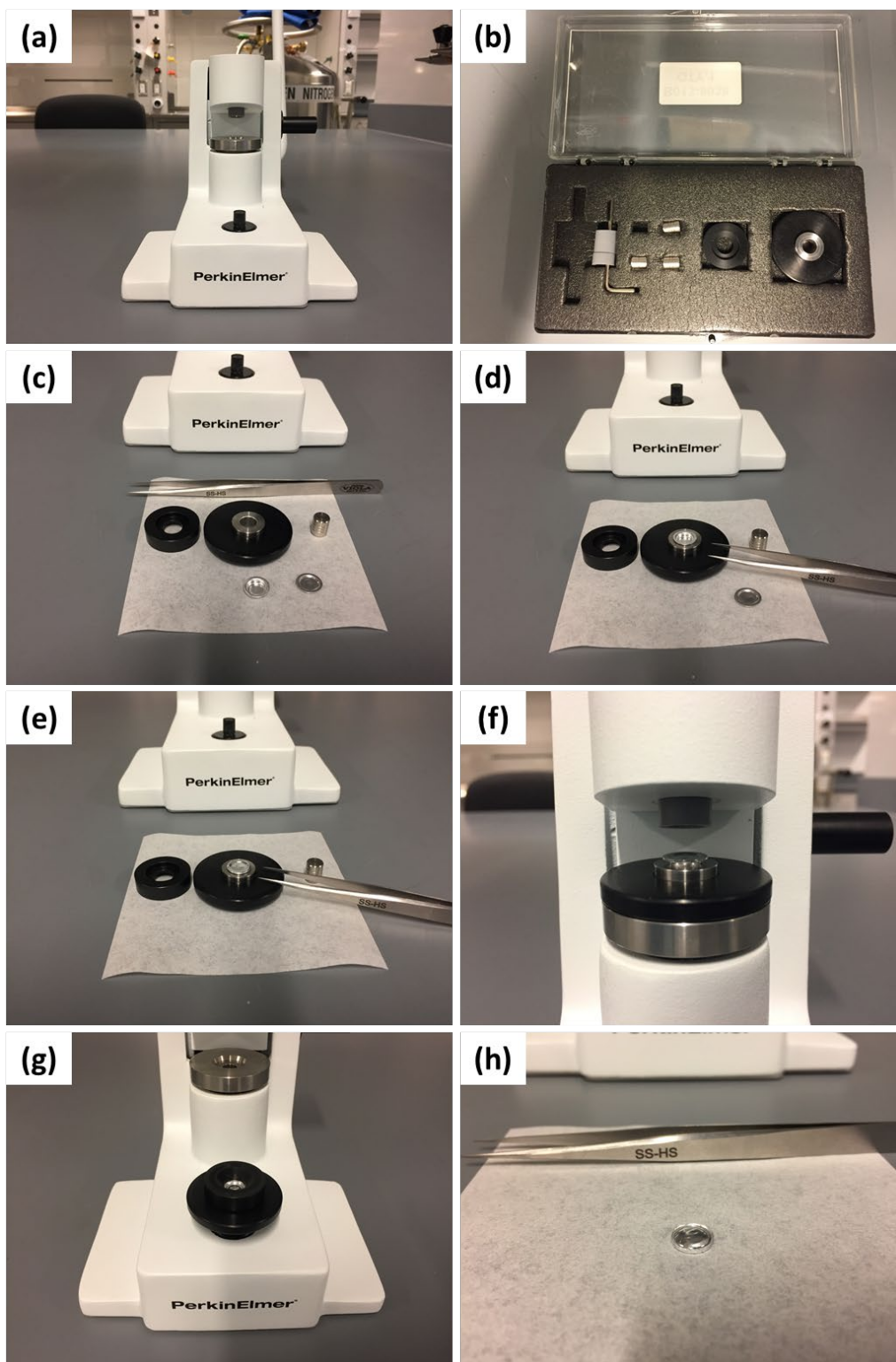


Figure 3. Universal encapsulation equipment: (a) The universal crimper press, (b) the die kit, (c) sample encapsulation components. The sample encapsulation process: (d) insert a sample pan in the die, (e) place a lid on the sample pan, (f) place the die assembly in the crimper press and rotate the lever to the stopper knob, (g) put die assembly on the ejector knob and place the shear ring over the die, (h) push down on the shear ring to eject the encapsulated sample pan.

III. Loading the DSC with your Sample Pan:

1. **Open the DSC's Covers** – Press the “DSC Cover Open/Close” button on the front of the unit to open the green cover. Open the large clear cover by rotating it up and back.
2. **Insert the Pans into the Holders** – Use the “Vacupick” suction tool to place the “Sample Pan” in the left holder and the “Reference Pan” in the right holder.
3. **Place Lids on Sample Holders** – Use the vacupick to place the appropriate lid over the appropriate holder. Always keep the lids oriented in the same direction in order to optimize data collection.
 - L1 – Sample Holder Lid
 - L3 – Reference Holder Lid
4. **Close the DSC's Covers** – Visually inspect the instrument to ensure that no tools or items are in the way prior to closing the cover. Press the “DSC Cover Open/Close” button to close the green cover. Rotate the clear cover back into place to seal the instrument.

***Note:** Only open the covers when the sample holder is at room temperature. This will ensure that the instrument is safe to work with and will prevent excessive accumulation of condensation.

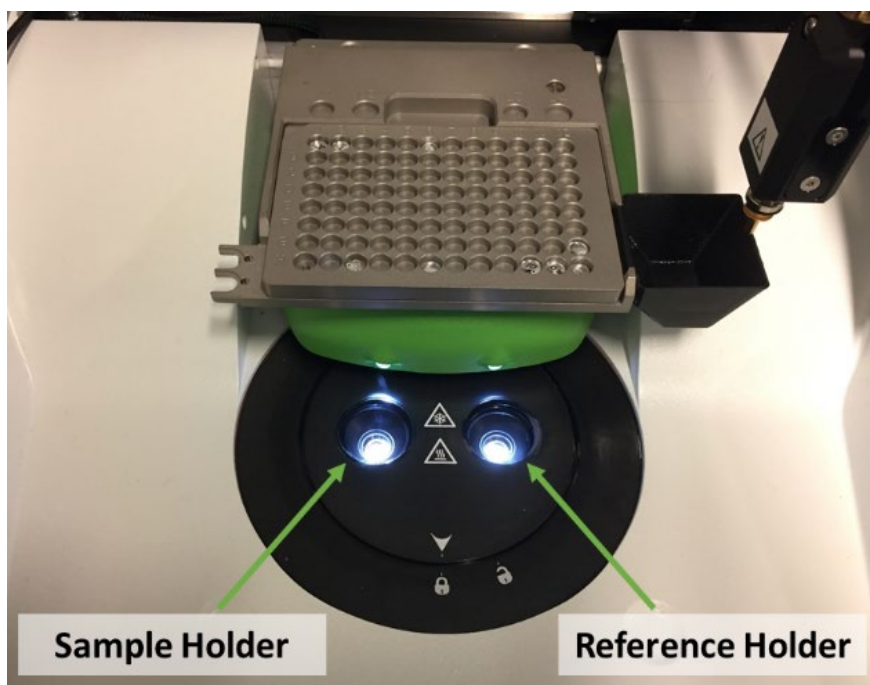



Figure 4. Sample holder positions.

IV. Creating an Experimental Procedure:

- 1. Create a Method** – Click the “**Method Editor**” button:  to create an experimental method. Enter the following sample info in the method editor window:
 - Sample ID
 - Operator ID
 - Comments
 - Weight – ~0.5-30 mg
 - File Name
- 2. Enter “Initial State” Settings** – Click the initial state tab and select your pre-run settings.
 - Common Pre-Run Actions:
 - Switch gas to N₂ (20 mL/min)
 - Action Occurs Immediately
 - Start the Run
 - Action Occurs Immediately
 - Set Initial Temperature (°C)
- 3. Enter “Program” Settings** – Select the program tab and enter the appropriate experimental procedure for your sample. Use the “**Add a step**” and “**Insert a step**” buttons to add experimental procedures.
 - The following steps are available:
 - **Temperature Scan** – Increases temperature at a given rate.
 - **Isothermal** – Holds the temperature at a given value.
 - **Step Scan / Scan Iso.** – Performs a step from T₁ to T₂ and holds at T₂.
 - **Repeat Steps** – Repeats specified steps.
 - Example temperature program conditions for a PETE polymer sample:
 - 1) Hold for 1 minute at 30 °C
 - 2) Heat from 30 °C to 300 °C at 10 °C per minute
 - 3) Hold for 5 minutes at 300 °C
 - 4) Cool to 30 °C at 10 °C per minute
 - 5) End Condition: Go to Starting Temperature
- 4. Check the Procedure** – Select the “**View Program**” tab and ensure that the appropriate settings are selected for your procedure. Make sure that the temperatures are appropriate for your sample and sample pan.
- 5. Start the Experiment** – Click the “**Start/Stop**” button to initiate the experiment.



Start/Stop Button

V. Data Analysis:

Pyris offers several tools for analyzing data collected on the DSC 8500. To open a file for data analysis, click the “Data Analysis” button and select the appropriate file. Several analysis features can be accessed by clicking “Calc” on the toolbar.



Data Analysis Button

Example: Peak integration can be performed by selecting “Calc” and “Peak Area” on the toolbar. The area can be determined by connecting any two points of interest on the graph. The limits can alternately be entered manually in the calculation window.

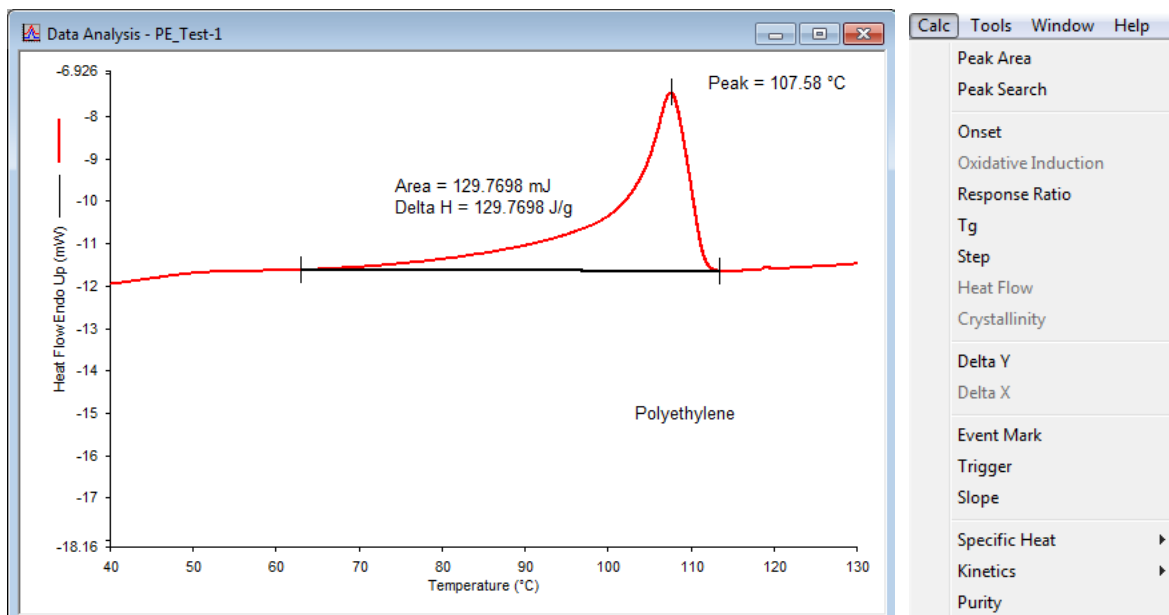
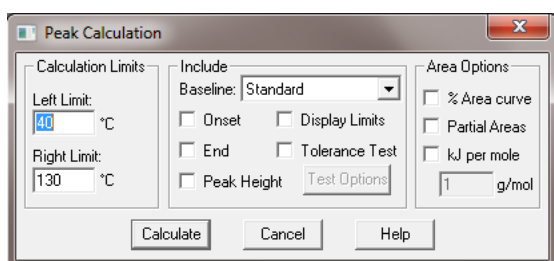


Figure 5. Integrating the area under the melting point of a polyethylene sample.

Troubleshooting Signal Intensity:

- **Signal Sensitivity/Intensity** – To improve signal sensitivity/intensity, run a larger amount of sample at a faster heating rate; this is at the cost of reduced accuracy/resolution.
- **Peak Resolution/Accuracy** – To improve resolution, run a smaller amount of sample at a slower rate of heating; this is at a cost of reduced sensitivity/intensity.

VI. Analyzing a Standard Reference Material:

Material standards with known melting points can be used to check the accuracy of the instrument when utilizing a given ramp rate. To determine the accuracy of your experimental procedure, select a material with a known melting point within the range of your procedure.

- 1. Select a Reference Material** – Indium and Lead are common reference materials.
 - **Indium Melting Point** – 156.6 °C – Analyze from ~ 130-170 °C.
 - **Lead Melting Point** – 327.47 °C – Analyze from ~ 300-350 °C.

***Note:** Do not use zinc as reference material as it is susceptible to alloying.
- 2. Open a Standard Method** – Click “File”, then “Open Method”, and select a standard.
- 3. Edit the Procedure** – Change the settings to match your rate of temperature ramping (i.e. 10 °C per minute). Save as a new file.
- 4. Start the Experiment** – Bring the instrument to the starting temperature and allow it to equilibrate. Then select “Start/Stop” to begin the experiment.
- 5. Compare Melting Point** – Compare the measured “onset melting point” to the published value, determine the accuracy.
- 6. Return the Standards** – Return the standards to their appropriate vials.


VII. Shut-Down Procedure:

- 1. Open the DSC’s Covers** – Press the “DSC Cover Open/Close” button on the front of the unit to open the green cover. Open the large clear cover by rotating it up and back.
- 2. Remove Lids from Holders** – Use the vacupick to return the lids to their appropriate locations. Always keep the lids oriented in the same direction.
 - **L1 – Sample Holder Lid**
 - **L3 – Reference Holder Lid**
- 3. Remove Pans from the Holders** – Use the “Vacupick” suction tool to remove the pans.
- 4. Close the DSC’s Covers** – Press the “DSC Cover Open/Close” button to close the green cover. Rotate the clear cover back into place to seal the instrument.
- 5. Turn off the Chiller** – Press the blue “Cooler On/Off” icon to turn off the chiller from the PC, allow the system to vent for several hours before turning off the DSC.
- 6. Turn off the DSC** – Flip the toggle switch on the back of the instrument to the off position.
- 7. Log-Off the Instrument** – Close the Pyris software. Remember to log off the instrument.

VIII. Autosampler (Optional):

The **autosampler** can be used to mechanically load and unload samples from the sample tray to the holder. The 8x12 tray holds up to 96 samples with each position designated by a letter, ranging from A-H, and a number from 1-12. The robotic arm allows controlled loading, unloading, and operation of the DSC within the enclosed hood. This facilitates atmospheric control and can be used to limit humidity within the DSC's enclosure for prolonged analysis of sensitive samples.

1. **Load Samples in the Auto Loader Tray** – Load your samples in the auto loading tray and record the position of each sample.

2. **Operate the Autosampler** – Press the “**Autosampler Control**” button:  to open the control window. The experiment can be directly run from the desktop using the autosampler's robotic arm to load and unload the furnace.

- **Sample Location** – Load or unload a sample pan from a given tray location.
- **Reference Location** – Load or unload a reference pan from a given tray location.

3. **Run the Method Editor** – Use the “**Method Editor**” to analyze each sample. Return analyzed samples to either their original position or the trash bin when finished.

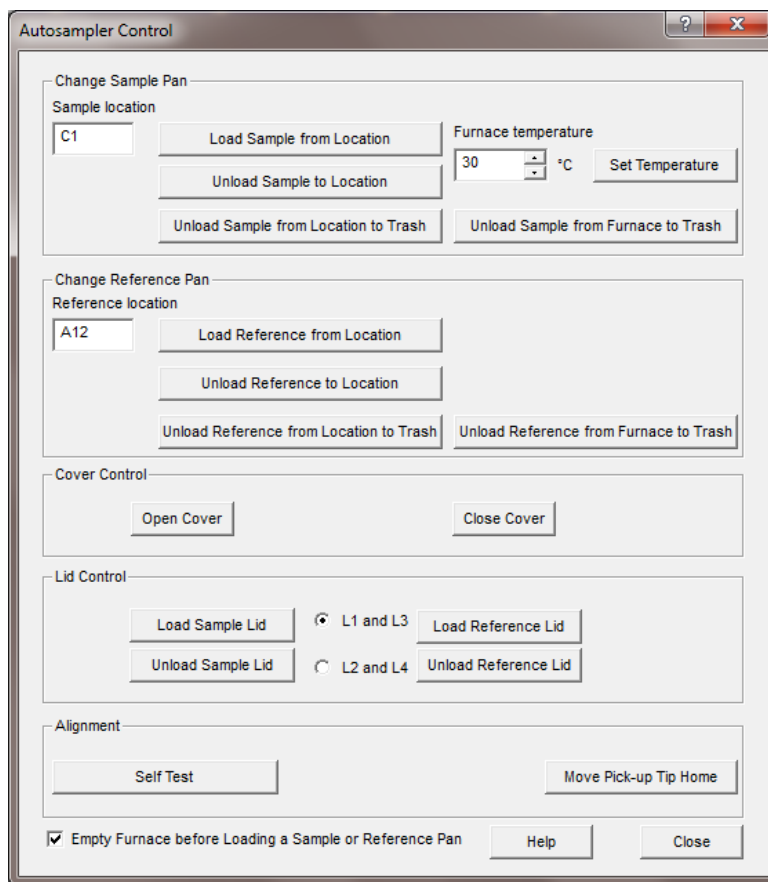



Figure 6. Autosampler control window.

IX. Programming a Play List for the Autosampler with Pyris Player (Optional):

The autosampler can be programmed to automatically analyze multiple samples in series using Pyris Player. To automatically analyze samples with the autosampler, create a “Play List” by selecting the “Pyris Player” button.



1. **Load Samples in the Auto Loader Tray** – Load your samples in the auto loading tray and record the position of each sample.

2. **Start Pyris Player** – Press the “Pyris Player” button:  to open the “Player Editor” window. Insert appropriate information related to the Lot ID and any comments related to the composition of your sample in the Setup tab.

3. **Edit a Play List** – Add desired steps for analysis using the “add a step button”. Then click on each step and select the appropriate information related to each step.

- a. **Prepare a Sample** – Have the instrument load a reference pan by designating the location of your reference pan in the tray and the location of the reference lid.

- b. **Sample Group** – Create a sample group using the: “add a step button”. Name the group and select the desired method name you would like to utilize. Then click on each sample and enter in the sample’s ID, weight, operator ID, comments, sample location, lid location (i.e. L1), and create a file name.

4. **Start the Play List** – Use the “Play”, “Stop”, or “Pause” buttons:   to start and stop the experiment. You can also press the pause button on the front of the instrument. The instrument will continue running until all samples have been analyzed.

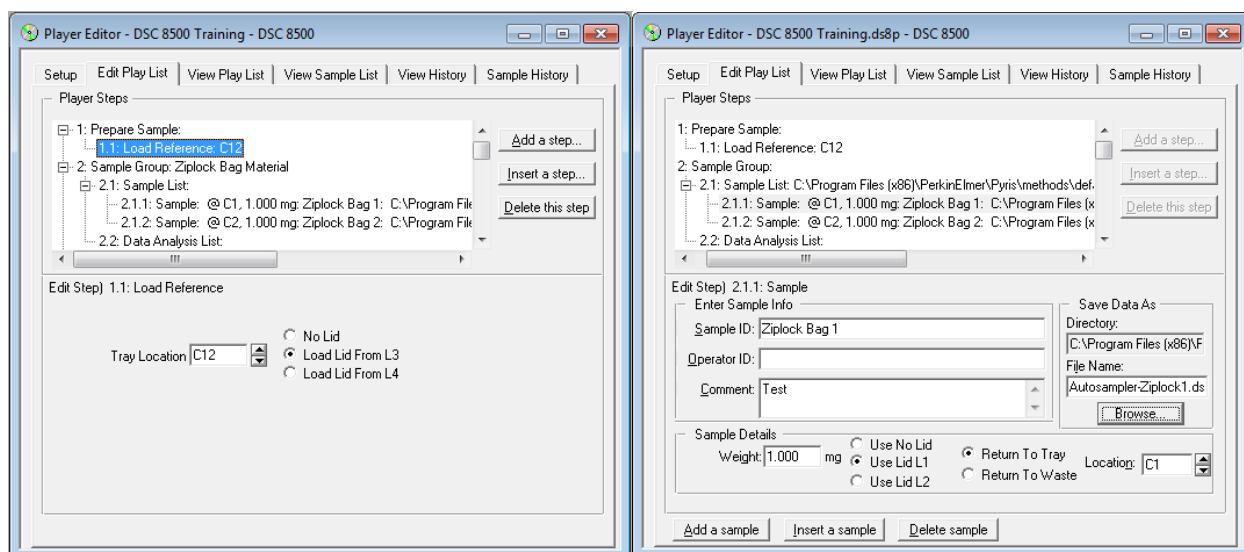


Figure 7. The Pyris Player Editor’s menu options and creation of an example “Play List”.

Appendix

Pyris Software

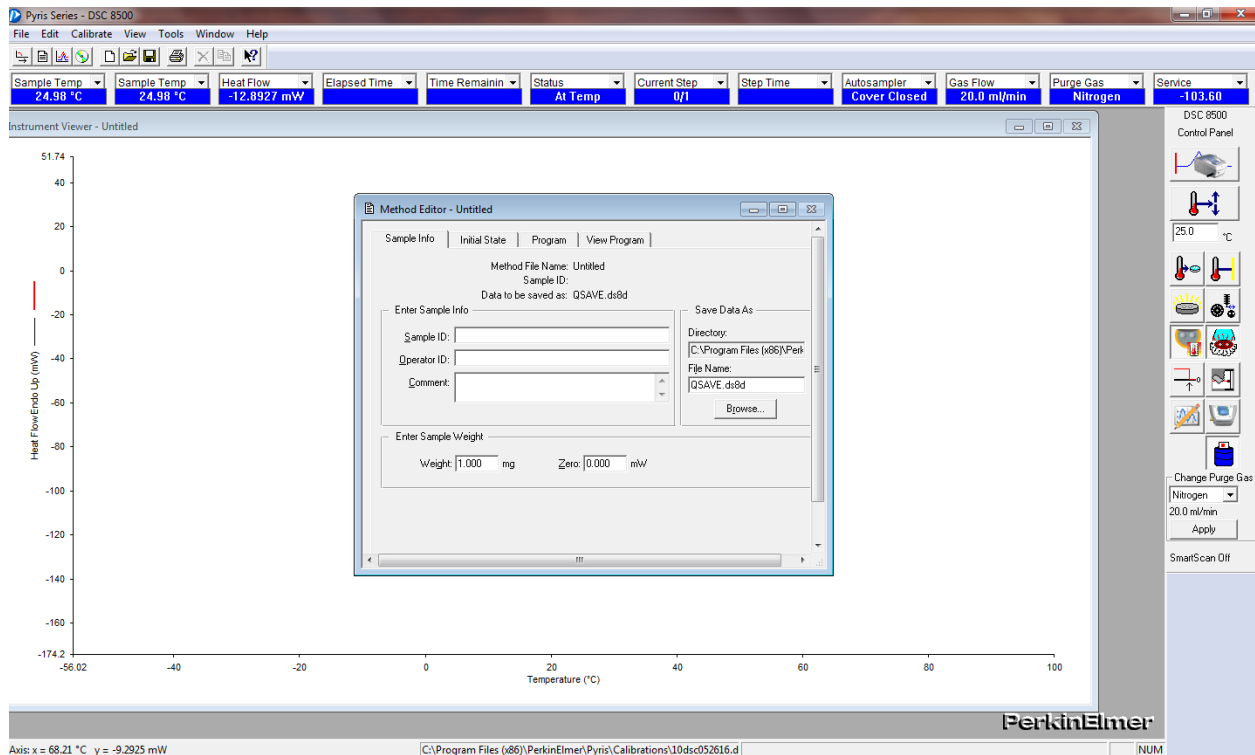
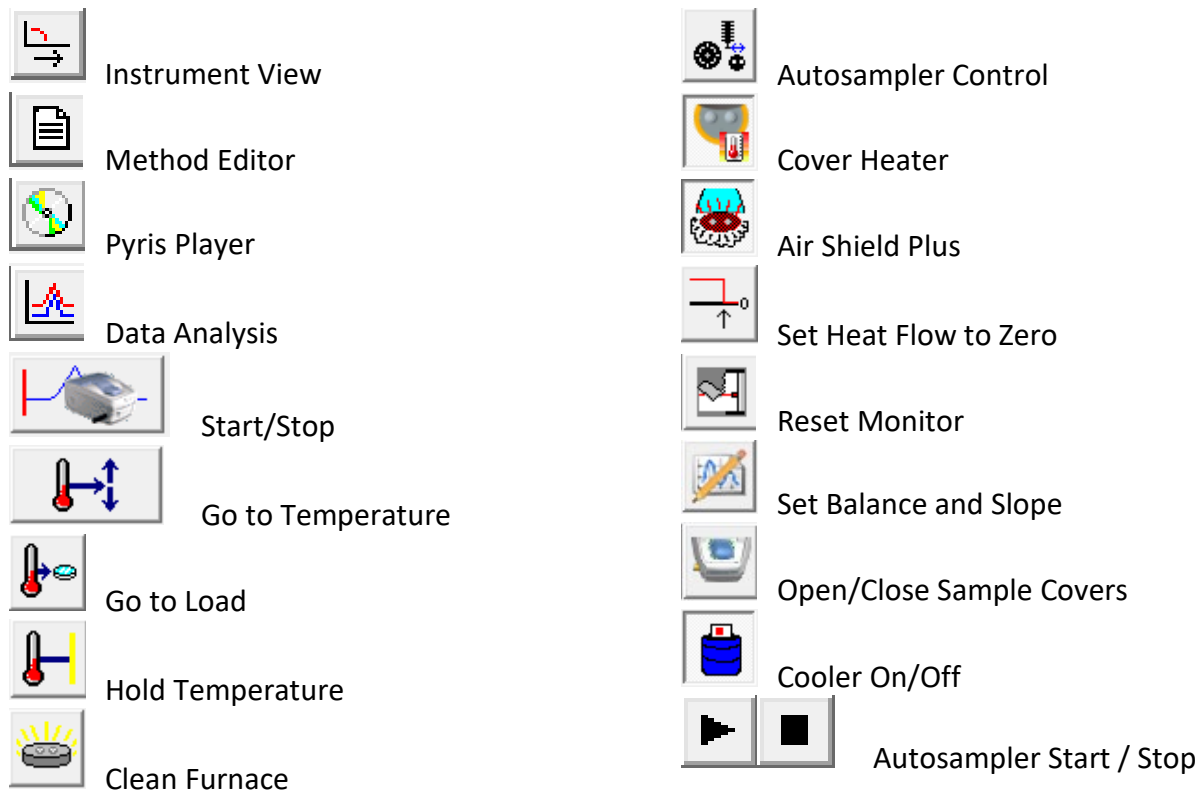


Figure 8. Pyris software window layout.



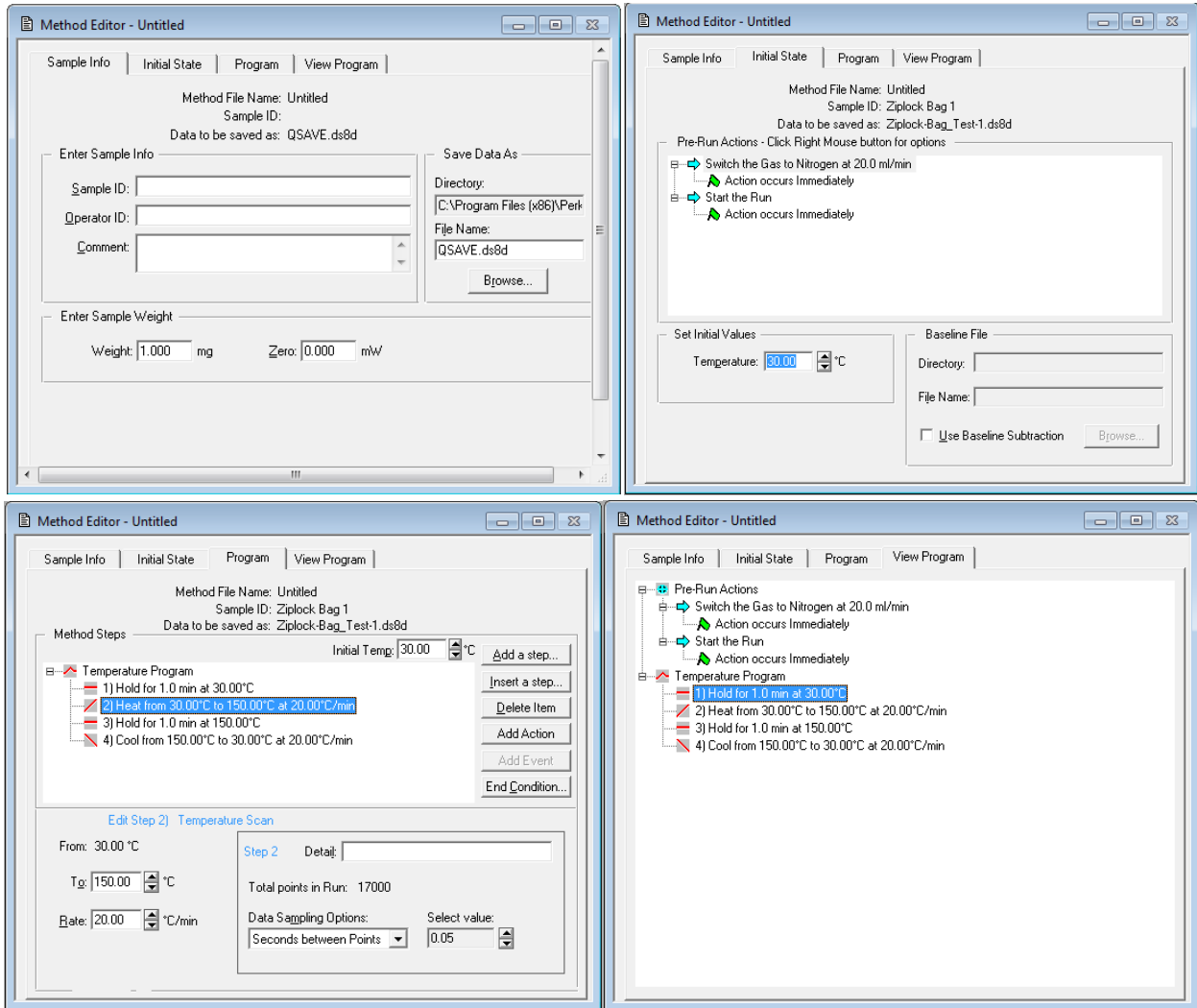


Figure 9. Method Editor window tabs.



Figure 10. Instrument Viewer tabs – allow control of x and y-axis during analysis.

Safety requirements

The laboratory in room ACEE 027B is a wet chemistry lab, in which some researchers may handle hazardous substances. The lab safety requires that users wear **eye protection** (safety goggles or glasses with side shields), **lab coats**, and **gloves** while using equipment in this room.

Emergency Information:

Medical Emergencies: **Contact 911 and Public Safety (609) 258-1000**

Room / facility emergencies: **Contact Public Safety (609) 258-1000**

Issues related to the instrument:

1. **Contact IAC Staff.**
2. **If unsure, leave system as is.**
3. **Try to power down the system.**

Audible/Siren Emergency Alerts:

Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

Nan Yao: Office (609) 258-6394; Cell (908) 922-2236 Email: nyao@princeton.edu

Denis Potapenko: Office (609) 258-7956; Cell (718) 551-6810 Email: denisp@princeton.edu

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