

Differential Scanning Calorimetry

Sample Preparation

1. Using the Sartorius Microbalance, tare an aluminum Tzero pan, place 1-10 mg of sample in the pan, weigh and record mass to 4 significant figures. Be careful to eliminate static electricity on your 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!*

Turning ON the DSC

1. Check that the house nitrogen line is ON (to the right of the DSC computer)



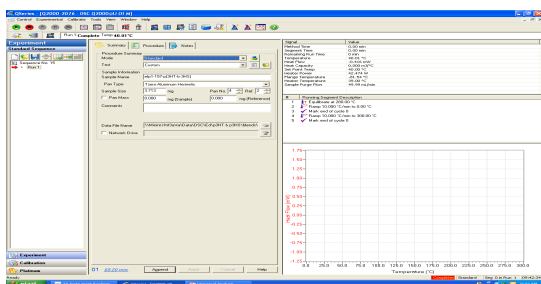
2. Open the TA software by clicking on “TA Instrument Explorer” icon and then “Q-2000”
3. Check that the chiller is powered ON. Turn on the chiller in the TA program by clicking: Control → Event → ON. *Caution: Never turn the cooler on without nitrogen flow! If left on without N₂, ice will build up in the cell and cause very costly damage!*
4. Wait until the Flange Temperature reaches about -80 °C before beginning a run.

Running a Sample

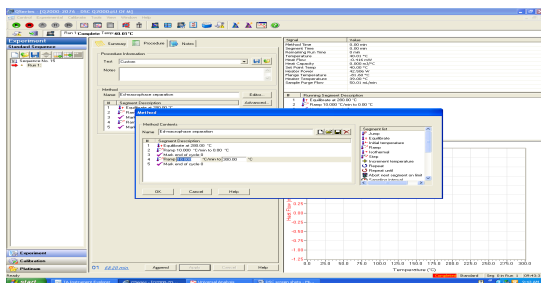
1. Place your sample pan in the auto-sampler, in one of the positions labeled #1-50.
2. Place an empty pan (with sealed lid) into one of the positions labeled **R1-R4** to be used as the reference mass. *Be sure that the pan is the same type as your sample pan.*



3. In the TA program, select the tab which reads “Summary.” Select “standard” mode, Tzero Aluminum Hermetic pan type. Enter your sample name, mass, pan # and ref # (as above). Click on the icon next to “data file name” to choose a directory and filename for this run.



4. In the “Procedure” tab, select a pre-programmed run, or “Custom.” To edit your custom sequence, click “Editor.” You can drag and drop commands in the editor to define temperatures and ramp rates, or isothermal segments in the custom run.



5. Click “Apply” on the bottom of the main screen to save all the parameters for the current run
6. Click the play icon at the upper-left to start the run. The lid will automatically be raised, and the auto-sampler will place your sample and reference pans into the cell.

Processing the Data

1. On the desktop, click the icon.



2. Click File → Open, Click OK.
3. To integrate a melting peak, click:



4. To find a Tg, click:



Shutting down the DSC

1. If someone is planning to use the DSC in the next few days, DO NOT shut down the machine. 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.
2. If nobody plans to use the DSC for the next week or longer, shut down the machine as follows:
 - a) Power down the chiller by clicking Control → Event → OFF in the Q2000 software
 - b) Wait for the flange temperature to equilibrate to ambient temp with the nitrogen still flowing.
 - c) Turn off the house nitrogen line.