Standard Operating Procedure
Differential Scanning Calorimeter (DSC) in POWER Laboratory
Model TA Q-20

John Hodul, February 2019

Description of Process

The DSC measures heat flow associated with thermal transitions of a material such as glass transition, melting point, and crystallization temperature.

Personal Protective Equipment

EYE PROTECTION: Safety glasses
PROTECTIVE CLOTHING: Laboratory coat and nitrile gloves

Sample Preparation Procedure

1. The sample should be thoroughly dried in the solid state to avoid damaging the equipment. Weigh the sample being tested (2-10 mg) in a Tzero hermetic aluminum pan. It is best to place the sample directly in the pans, transferring the sample from the weigh paper to the pan can lead to a loss in material.
2. Place the lid on the pan. Be sure to place the lid with the indention facing down.
3. Put the covered pan in the blue holder. Assure that the lid is flush with the top of the holder.
4. Place the holder under the press. Again assure that the lid is flush with the top of the holder.
5. Pull the handle down, this will seal the pan and lid.
6. Repeat with an empty pan to be used as a reference.

Note that both the pans and the press are located in room 2182 next to the weighing scale.

DSC Procedure

1. Place the sealed pan(s) in the sample tray. Make note of the slot number.
2. Place the reference pan in a reference slot. Make note of the slot number.
3. The reference pan is an empty sealed pan.
    a. Keep the reference pan so that multiple runs can be performed with it.
4. Turn on the power to the cooler. It should be on event mode.
5. Open the software, TA Universal Analysis on the desktop of the computer.
6. In the Control tab, scroll down to event and press ‘On’. This will start the cooler. It may be necessary to press ‘Go to standby temp’ so that the cell does not freeze. The temperature of the cell is shown in the status bar.
7. Turn on the nitrogen gas tank, which should be held at 20 psi and the sample purge flow should be 50 ml min^{-1}. If the sample purge flow (located in the signal-

value box in the upper right corner) shows no flow, it may be necessary to open the smallest black knob on the gas tank.

8. On the program in the center panel named “ ” enter the data for the sample that you are running. You will need to fill out all the information including: sample name, sample weight, sample slot number, reference slot number, and data storage location. You will also have to select an operating procedure.
9. On the second tab, Procedure, you will need to enter the parameters for the operating procedure. Here you can alter the method previously chosen in the first tab. You can also load a standard procedure that you have saved.
    a. Click on Test to adjust the procedure type.
    b. Click on Editor to set the temperatures, ramp rates, holding times, cycle names, etc. Note that the operating range of the equipment is -80 °C to 400 °C.
    c. Click Ok to accept the parameters.
    d. At this time you can save a procedure to be used in further experiments by clicking the floppy disk logo. Give the program and name and select a saving location.
10. On the far right panel, Experiment, you can add runs. You do this by selecting the  button. After a run has been added the appropriate data information should be added. Runs can also be deleted. The order of the runs can also be adjusted on the panel by clicking and dragging the run to the appropriate location.
11. Once all of the sample information has been entered and the runs are in the appropriate order you can begin the program. There will be an arrow next to the run that will begin first. Click  at the top of the window to start. Note once you begin an experiment you cannot change the program.
12. When the program ends, the cooling system will automatically turn off. However, the nitrogen tank needs to be turned off after the test.

<u>Maintenance Procedure</u>

Calibration

1. Make one sample pan containing ~10 mg of indium, which is located in the DSC kit.
2. Load the sample pan along with the reference pan.
3. Turn on the cooling and nitrogen tank similar to the above procedure.
4. In the lower left corner of the program, click on the calibration tab.
5. Change the file name that will be saved. Change the mode to Calibration Enter the weight of the sample.
6. Below the run, it should read ‘Do indium calibration’.
7. Click start. The sample will be heated in two different cycles.
8. After both runs are completed, go to the calibration scroll down menu and click on indium tracking. The results will be displayed along with previous calibrations.

Cleaning

-There is a special brush located in the DSC kit that is intended to clean the cell.

Data Transfer Procedure

The data is processed using an external program. To open the program click TA Universal Analysis on the desktop.

1. Open the file for the sample being processed.
2. When the file is opened all the data collected is presented. If the program used contains multiple loops (to erase the thermal history of a given polymer) you will need to select the appropriate cycle to analyze. This is done by going to Edit tab, clicking cycle list then selecting the specific cycle.
3. The appropriate analysis needs to be selected (i.e. glass transition, melting temperature, etc.). This is done under Analyze tab.
    a. Once the analysis type has been selected, two crosses will appear on the data. The crosses need to be clicked and dragged so that one is on each side of the transition state.
    b. Next left click and select Accept Limits. The temperature for the beginning, middle, and end of the transition will appear on the screen.
4. To export the data to be further processed in excel, matlab,.. etc., go to File, Export Data File, File and Plot Signals, Spreadsheet text file, Click Finish, name the file, and click save.