Operation Manual for the TA Instruments DSC Q-100 and Q-20:

Temperature Range: -90°C – 400°C
Sample Weight: 5 mg- 20 mg.

Each lab is responsible for supplying their own pans.

Pan selection: Aluminum pans are most commonly used. Non-hermetically crimped pans are fine for most samples (Perkin Elmer Part #0219-0041). Hermetically sealed pans have an airtight seal and can be used for volatile liquids, samples that sublime or for aqueous solutions (TA Instruments Part #900793.901 and 900794.901). Copper pans are available for higher temperature runs.

Sample Preparation: Tare the empty pan on the balance. Remove the pan from the balance and add your sample to the pan. Weigh the sample and record the weight. Generally, 10 to 20 mg of sample should be used for Tg measurements and 5-10 mg for melting transitions. The sample should be as flat as possible so that there is good thermal contact between the sample and the pan. For solid samples, use the flattest surface. Powdered samples should be spread evenly across the bottom of the pan. Place a lid on top of the pan and sample and crimp the pan according to instructions from your training. After crimping the bottom of the pan should be flat and the sides rolled down over the cover. An empty pan should also be prepared for use as a reference.

Put your samples aside while you input your data information and procedure.

On the computer: click on the appropriate instrument on the bottom of the screen to open the experimental view page. Click on the **SUMMARY** tab on the computer screen to input sample information. Input the **SAMPLE** name and **WEIGHT**. Create a new folder to store your data files. Your data will be saved in a data file. You must make a new folder. To do so, click on the icon next to **DATA FILE NAME** (looks like the pages of an open book). Click on the UP icon until you find **DATA**, then click on the **NEW FOLDER** icon. Name the folder (your name), click to open it and then enter your sample name. Your data will be automatically saved in your folder as the "data name.001". The numbering will automatically increase (.002, etc) if you do not change the sample name with each new sample.

Click on the **PROCEDURE** tab to write your method. To the left hand side of the screen there is a heading **SEQUENCE**. Select the **PAGE** icon to clear the current sequence. It will ask you if you want to save it…select **NO**. Now click on the **EDITOR** command in the center of the screen. Again, select the **PAGE** icon to clear the current method.

The method and the temperature range that is used are dependent upon the limits of the DSC (-90°C – 400°C) and the sample. In general, good results are obtained when a heating rate of 10-20°C/min is used. For measurement of glass transition temperature a heating rate of 20°C/min (10°C/min-20°C/min are acceptable) is used. For melting point determination a rate of 10°C/min is used though rates of 5°C/min-10°C/min are acceptable.

The starting temperature should be at least 2 minutes below the transition of interest. For example, if the glass transition temperature is expected to be 40°C and the heating rate is 20°C/min, the starting temperature should be at least 0°C. The starting temperature should be held isothermally for at least 2 minutes before beginning the scan. This ensures a good baseline.

The final temperature should allow completion of the transition of interest but should not be above the degradation temperature of the sample.

It is often desirable to erase the thermal history of a sample. This is done by heating the sample through the transitions of interest, allowing it to cool at a given rate, and then re-heating the sample. The sample should be held above the melting temperature for approximately 5 minutes to ensure complete melting of the crystals. A sample that is quench-cooled may not have time to recrystallize and may result in a larger more defined Tg upon reheating. A sample that is cooled slowly will have more time to recrystallize.

To write a method, double click the options to the right. The instrument is kept in stand-by mode at 40°C. In general the sample should be placed in the DSC cell when the temperature is at 20°C. A good general procedure to use:

INITIAL TEMPERATURE 20°C.
EQUILIBRATE at a starting temperature (as low as -90C)
ISOTHERMAL 2 minutes
RAMP 5°C/min-20°C/min to your final temperature (maximum 400°C but not above the degradation temperature).
ISOTHERMAL 5 minutes
MARK END OF CYCLE 1
RAMP 5°C/min -20°C/min to your starting temperature
EQUILIBRATE at starting temperature
ISOTHERMAL 2 min
MARK END OF CYCLE 2
RAMP 5°C/min -20C/min to final temperature
MARK END OF CYCLE 3

Click on the **SAVE METHOD** icon and save your method in your folder. (The icon looks like a hard disk).

Click on the **NOTES** tab. Put your name next to "**OPERATOR**". Input the **type of pan** you are using (standard aluminum or hermetic). (For the Q-20, the pan type is on the summary page). Check that the **purge gas** is nitrogen or argon and that the **flow rate** is 50 ml/min.

1. Now click on the **APPLY** command at the bottom of the screen.

2. Click on the **GREEN** arrow at the top of the screen to start the run. When the temperature is at the initial temperature you may open the cell. There are 3 lids which you must remove. Place the empty reference pan in the back and the sample pan in the front. Replace the 3 lids.

3. **IMPORTANT**: When the instrument is in the ready state you must click on the **GREEN** arrow again to continue the run.

4. You may stop a sequence using the red buttons on the top of the screen. You may modify your procedure at any time by clicking on **EXPERIMENTAL** and then **MODIFY PROCEDURE**. You may change conditions as well as add or delete steps from here.

5. You may observe real-time data in Universal Analysis. Open UA from the bottom of the screen by clicking on **UNIVERSAL ANALYSIS**. Open the data files and find your sample. It will open and continue to update the data as it runs. Data is automatically saved at the end of the run.

6. See Laura for help with data analysis and interpretation.