DSC using Q20 instrument

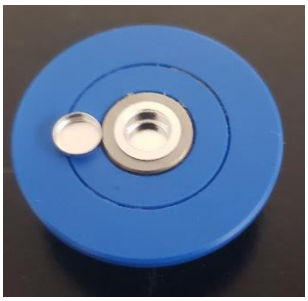
Experimental Considerations

1) Before starting the instrument consider your experiment conditions:
    a. What temperature range are you testing?
        i. Maximum temperature on this system is 550 °C
        ii. Minimum temperature is -90 °C
    b. What scan rate will you be using, will you need different scan rates in different temperature zones?
        i. Depending on your goal temperature there is a maximum cooling rate that can be achieved
        ii. From 550 °C to 300 °C; 100 °C/min
        iii. From 550 °C to 120 °C; 50 °C/min
        iv. From 550 °C to -20 °C; 20 °C/min
        v. From 550 °C to -50 °C; 10 °C/min
        vi. From 550 °C to -75 °C; 5 °C/min
        vii. From 550 °C to -90 °C; 2 °C/min
    c. Will your sample react with the sample pan at the desired temperatures?
        i. If sample reacts with pan this will damage the instrument, **stop here**
        ii. If you're not sure then **stop** and go look up more information
    d. How will your sample break down under heating? Will it decompose to form gaseous by products?
        i. Samples that off gas when heated require special pans and setup
        ii. If you are not sure how the sample will react under heating test on the TGA to determine decomposition temperatures and when weight losses occur is recommended
        iii. If in doubt go to the literature to see how others have handled samples of similar type

Sample loading

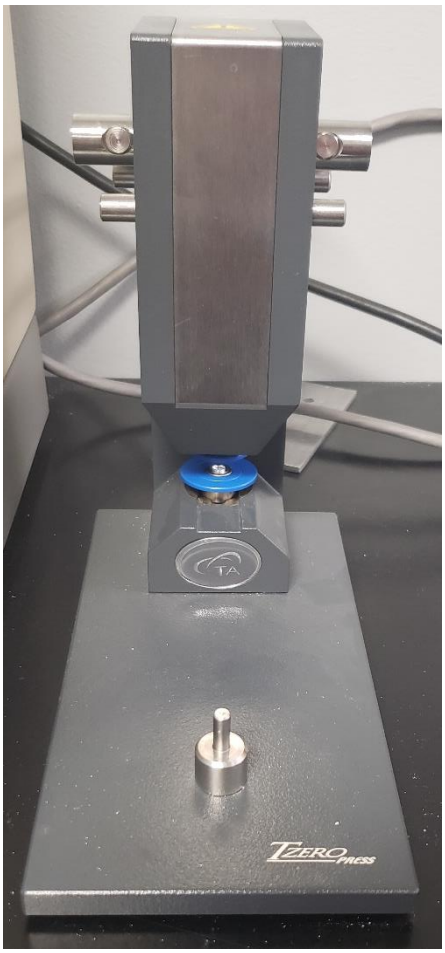
1) Select a pan and a lid for your sample
2) Make sure that you have a reference pan of the **exact** same pan type
3) Place the pan and lid on the blue colored pan holder

4) Put pan/lid/holder setup on analytical balance and zero the balance



5) Carefully load the pan with sample
    a. The sample should lay flat on the bottom of the pan
    b. Make sure there is no sample outside of the inside part of the pan before weighing
6) Check weight of the sample
    a. Mass should be between 1 mg and 20 mg
7) Once appropriate amount of sample is loaded carefully place lid on pan so that it sits level

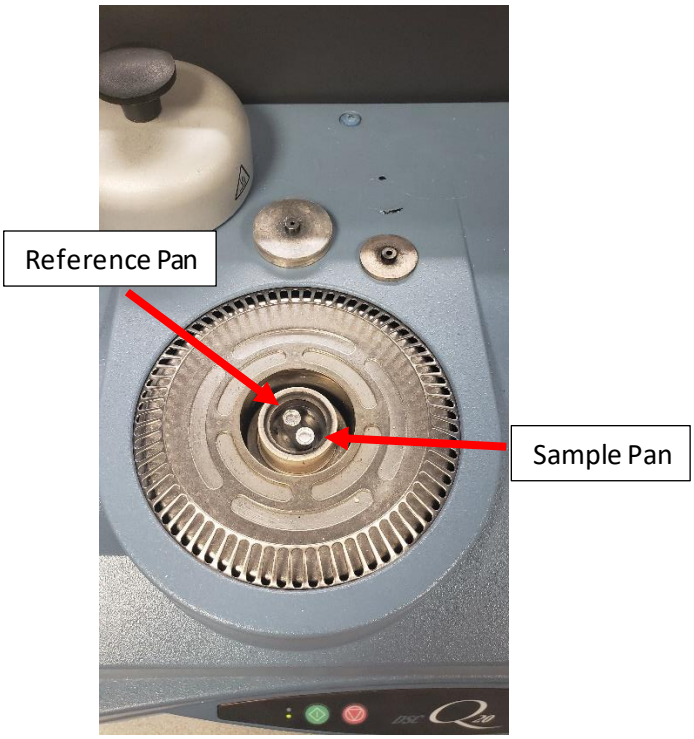
8) Place the pan/lid/pan holder setup in the pan crimper and pull the lever down completely then raise it
    a. The whole setup should sit snuggly in place before you pull the lever



9) Check the pan and lid to see if the lid is fully seated in the pan with no visible sample
10) Weigh the sample after sealing the pan, this is the mass that will be input in the instrument program
11) Load sample plan and reference pan into the sample cell in the instrument
    a. To load pans remove heat cover, shown below

b. Next remove both cell lids using tweezers to handle them

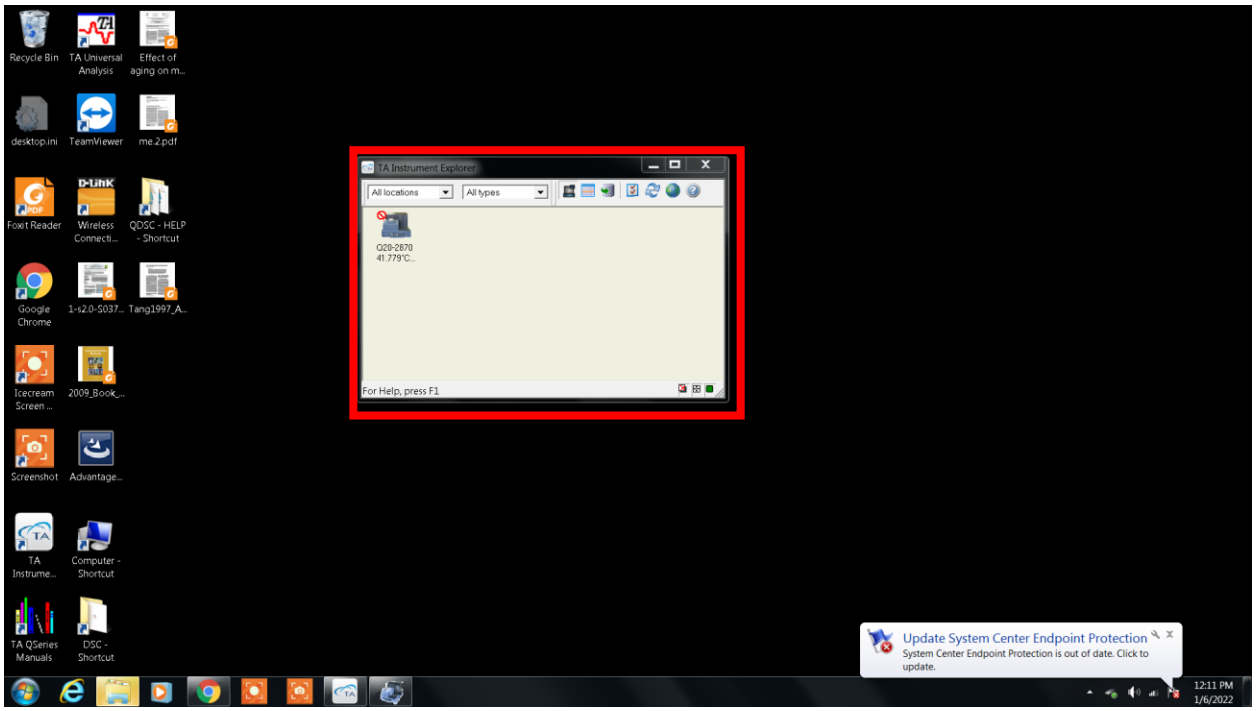
c. The sample pan should be placed on the column closest to you while the reference pan should be on the column towards the back



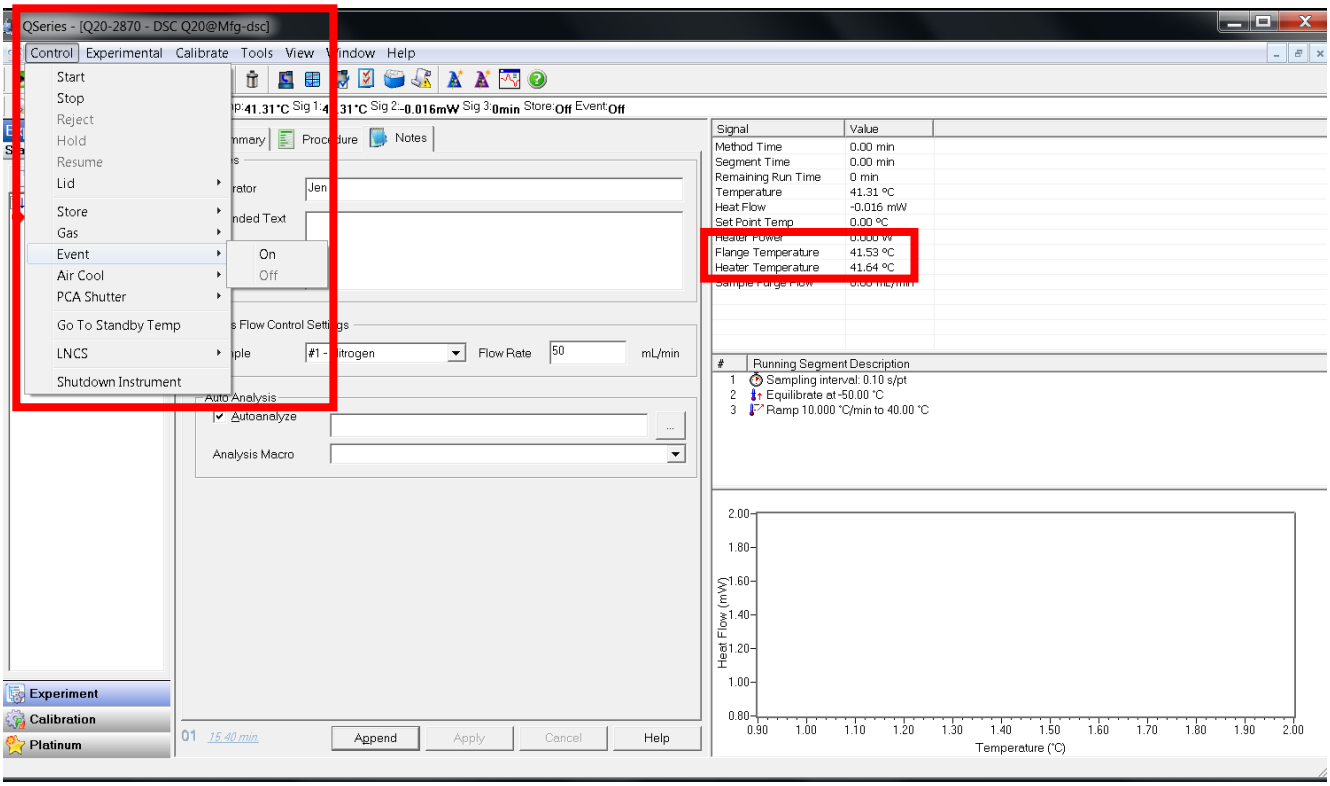
d. Replace all of the cell lids and heat cover before setting up computer system and starting the scan

Computer Program Setup

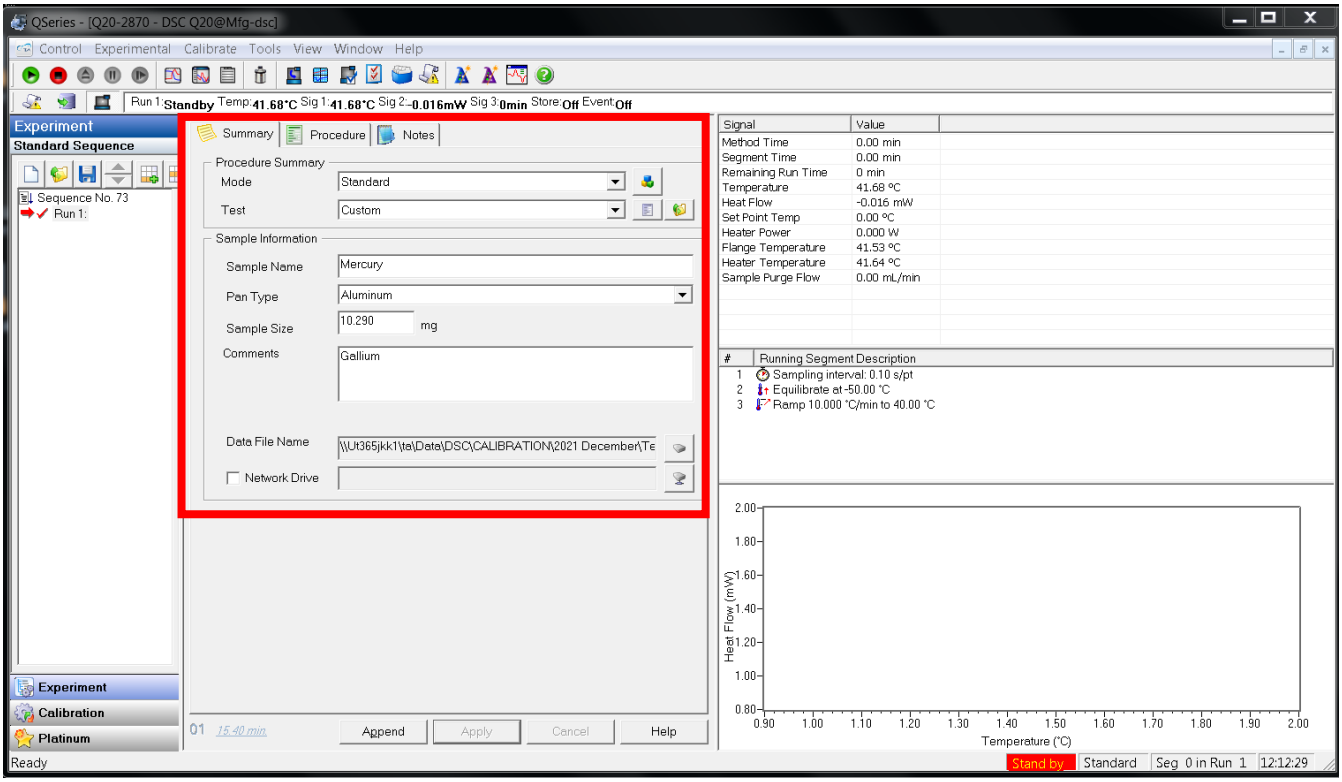
1) Open the N_2 tank attached to the DSC
2) On the instrument computer open the instrument control program by clicking on the instrument icon outlined in red in the image below



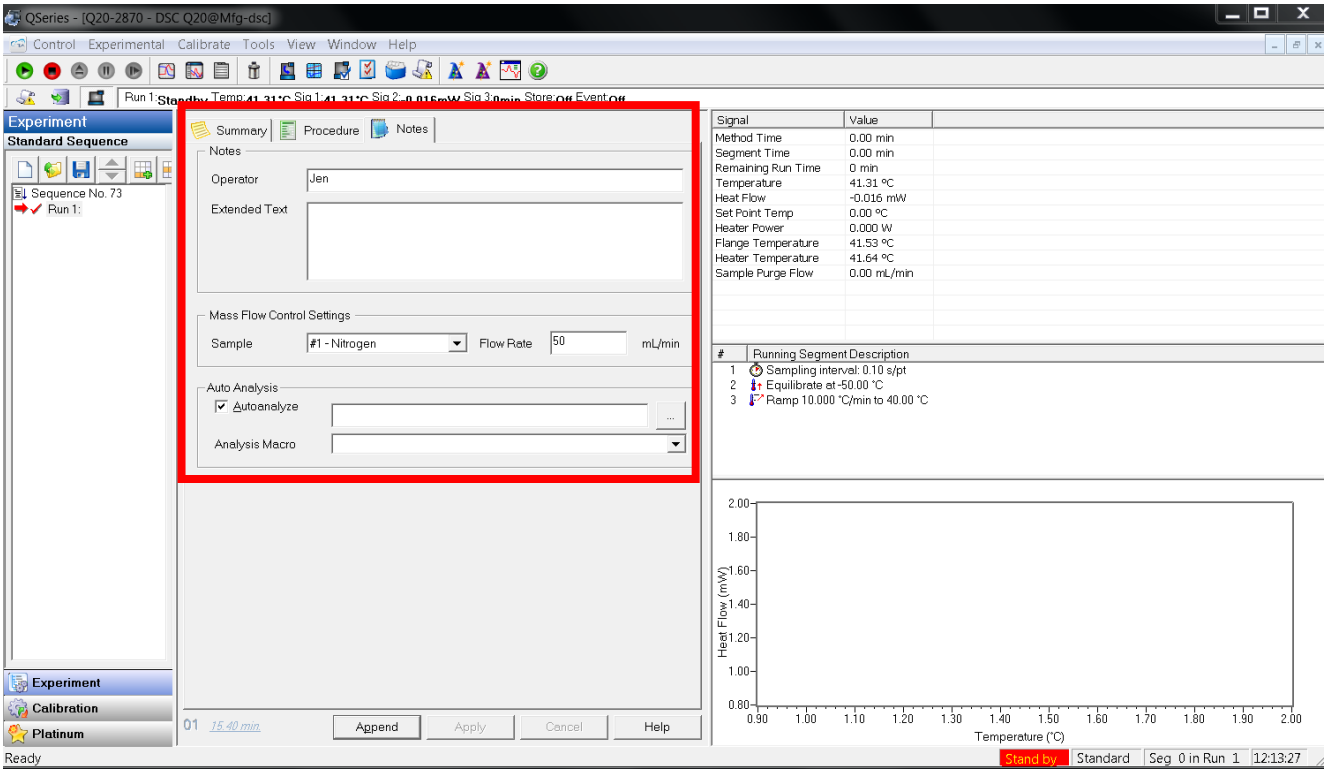
3) In the instrument program go to **Control** select **Event** and click **On** to turn on the cooling system
    a. Do not start any experiment until the Flange temperature reaches below -70 °C
    b. Flange temperature is displayed in the table on the right hand side of the screen



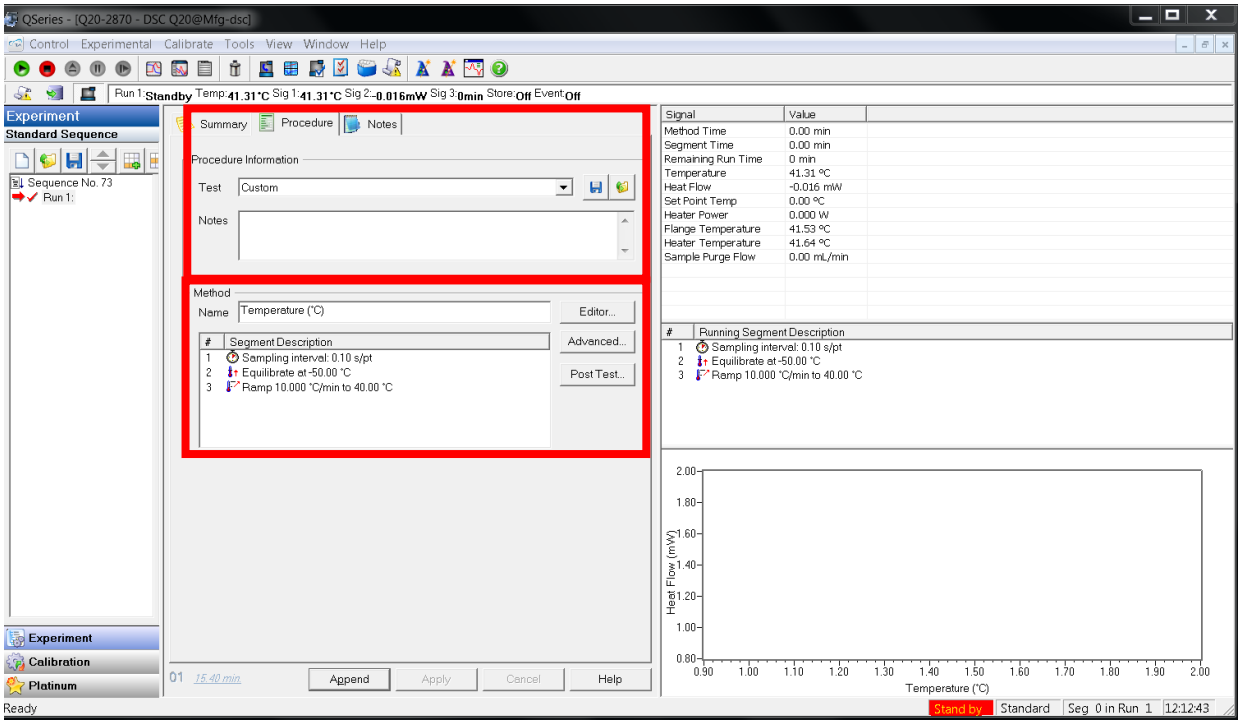
4) While the flange is decreasing set up experimental conditions
5) Go to the Summary Tab
    a. Under **Mode** select **Standard**
    b. Under **Test** select **Custom**
    c. Under **Sample Information** fill in the sample name, select the pan type, input sample mass in mg from before and add any comments in the comments section
        i. If you are using the supplied DSC pans select Aluminum
    d. Next to **Data File Name** click on the button with the box on it to open the file save menu
    e. In this menu select the folder you want to save in and fill in the file name then click save



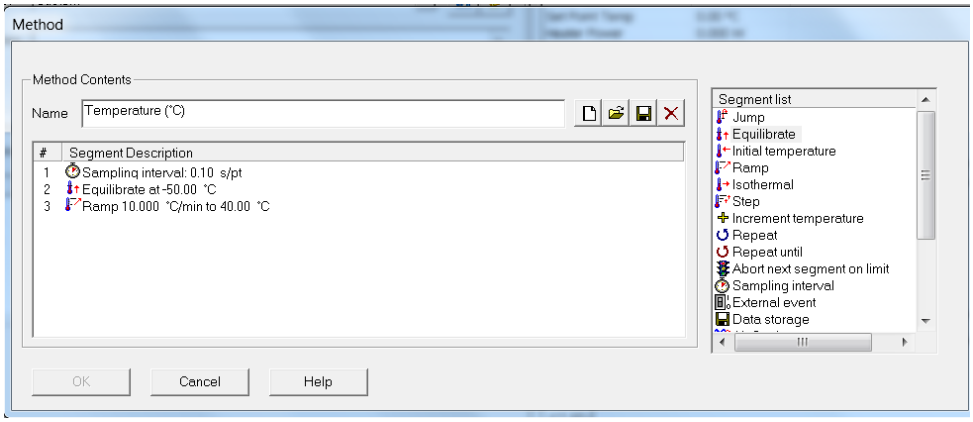
6) Go to the Notes tab
    a. In the operator field type in your name
    b. In the purge gas section select the gas you will be using (N_2 or air)
    c. The Flow rate should be kept at 50 mL/min



7) Go to the Procedure tab
    a. In the drop down next to **Test** select **Custom**
    b. In the notes section you can fill in any information about your test that you would like to have
    c. Under **Method** click on the **Editor** button to change the method

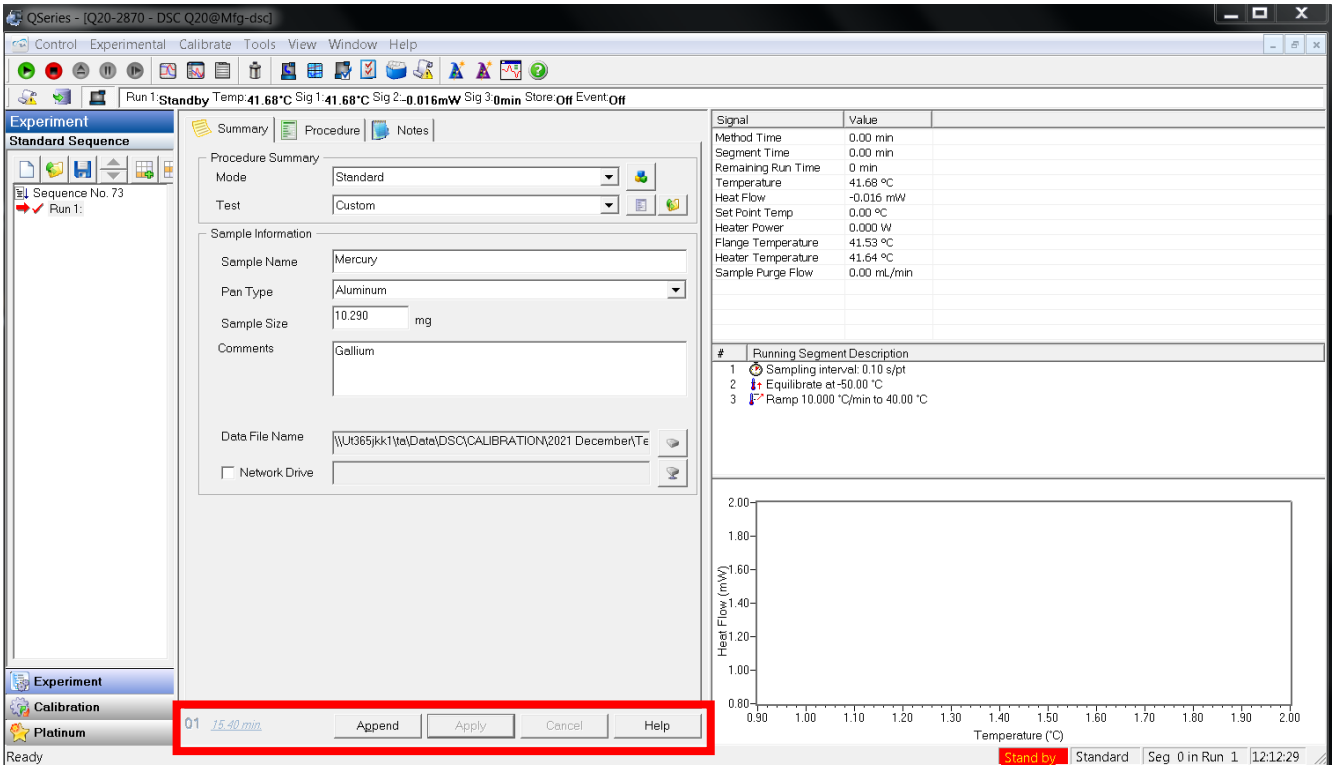


d. This will open the method editor window shown below



e. Click on the **New Method** button (White sheet icon) to start a new method
f. Click on and drag the segment types you want to use in your experiment into the white box on the left
g. Most common segments used are:
    i. **Ramp**: Changes furnace temperature at a given rate in °C/min to a specified temperature
    ii. **Isothermal:** Holds furnace at last set temperature for a specified amount of time in minutes
    iii. **Equilibrate**: Heats or cools the system as quickly as possible to a particular temperature and then holds until the temperature stabilizes

8) Once you have confirmed that all method and data file information has been properly input click on **Apply** in the bottom left side of the screen



Starting scan

1) Check that sample pan and reference pan are properly loaded in the instrument cell
2) Check that the desired method is properly loaded and does not exceed the max temperature of 550 °C
3) Check that the Flange Temperature is below -70 °C
4) If all of these are set up then start the run by clicking the green play button

Post Experiment tear down

1) Once then scan is complete the sample pan can be removed
    a. If you are doing multiple samples then replace the finished sample with the next sample and start that scan
    b. If you are done with your experiments then just remove your sample pan and replace the cell covers and heat shield
2) When you are done with the instrument turn off the cooling system
    a. Got to **Control** then **Event** and click **Off**
    b. You will hear the cooling system shut off
3) Leave the N_2 gas tank open until the Flange Temperature warms up to higher than 10 °C
    a. This is to prevent forming ice in the sample cell
    b. If ice forms in the cell then it will have to be baked out and recalibrated resulting in the instrument being down for several days