

Differential Scanning Calorimetry (DSC) Manual

Introduction

The DSC-2000 machine is used to heat and cool samples at a uniform rate under an inert gas to prevent oxidation. By changing the temperature at a set, uniform rate, the change in the heat capacity of the sample can be plotted either as a function of temperature or time. Samples with different compositions and aging times will respond differently to the applied heat flow. This manual will detail how to run a test on the DSC.

Safety Information

- The DSC machine uses nitrogen as a purge gas while it is running. The nitrogen is kept in pressurized cylinders. Use caution when interacting with the nitrogen tanks in case of a blown seal. Stay away from the metal connection hoses when opening the tank valve.
- The DSC machine is kept in a relatively well-sealed room. If one of the nitrogen tanks leaks, the room may fill with nitrogen. This is an inhalation hazard that can quickly lead to hypoxia and unconsciousness. Never work in the lab alone.

Procedure

This document contains the following sections:

- A. Pre-Run Preparations (4 steps)
- B. File Creation and Storage (6 steps)
- C. Running the Test(12 steps)
- D. Post-Run Operations (5 steps)
- E. Exporting Test Data (16 steps)

Materials

- Pre-Cut, Pre-Weighed DSC Sample
- DSC-2000 Machine
- TA Instruments Program
- Nitrogen Gas
- Analytical Balance
- Tzero Aluminum DSC Pans
- Tweezers
- Flash Drive (for data exportation)

DSC Test

Pre-Run Preparations

1. Open TA Instruments program on lab computer and select the Q2000 machine.
2. Under **Control** in the top left corner of the window, select **Event>>On**.

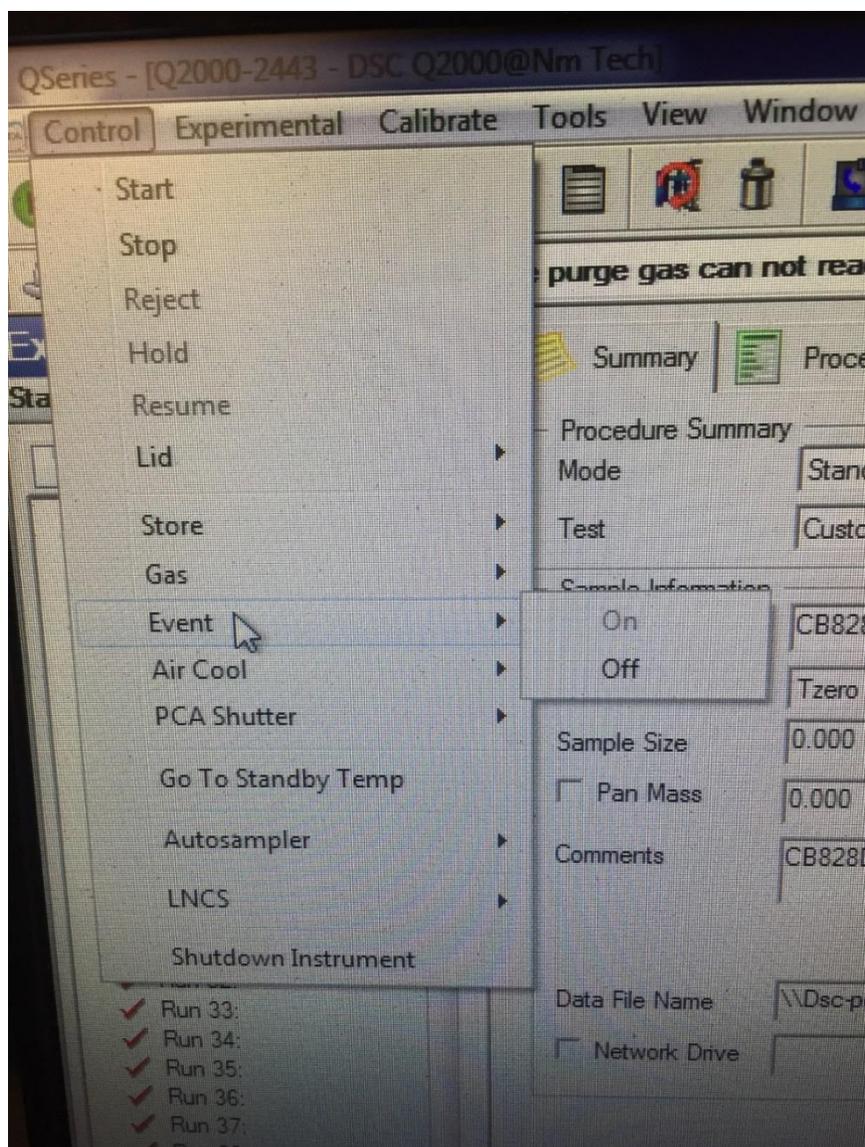


Figure 1. Computer control menu.

3. Go to nitrogen tanks and open the valve. The tanks will hiss when they have been opened, and the pressure gauges will show the change in pressure. **Caution: Keep face and body away from the metal connecting hoses in case the seal between them and the tanks breaks.**



Figure 2. Nitrogen Tanks.



Figure 3. Nitrogen tank pressure gauges.

4. Remove the wooden lid over the DSC-2000 machine.



Figure 4. DSC Machine.

File Creation and Storage

1. Obtain a pre-cut, pre-weighed DSC sample. **Note: DSC samples are extremely small and should be stored in the envelope they arrive in. The envelope should have information pertaining to the composition, creator, aging time, and mass of the sample written on it.**

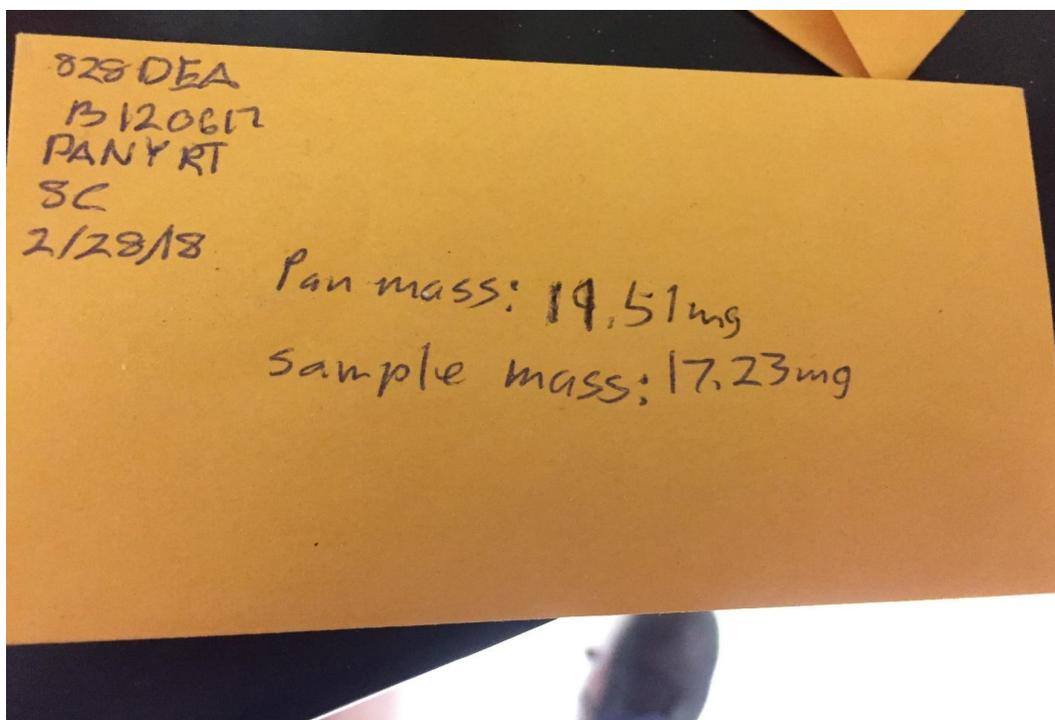


Figure 5. DSC sample envelope

2. In the TA Instruments window, on the leftmost panel, select the button with the folder and the green plus sign. This creates a new file.

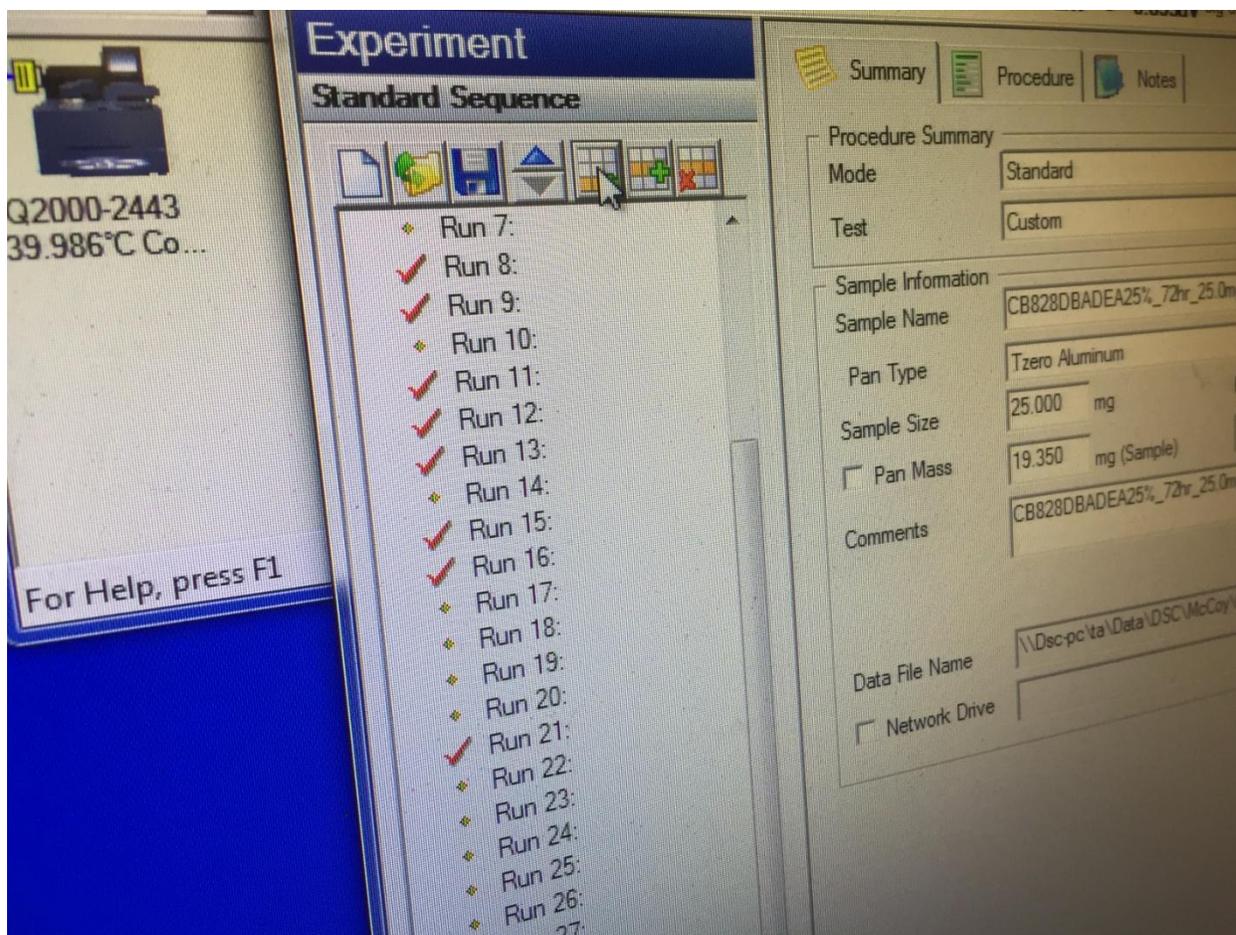


Figure 6. DSC Run Panel

3. In the central panel, under the **Summary** tab, in the box labeled **Sample Name**, type in the sample information. Include the initials of the creator, the composition of the sample, the time the sample was aged, and its mass. Each piece of information should be separated with underscores.

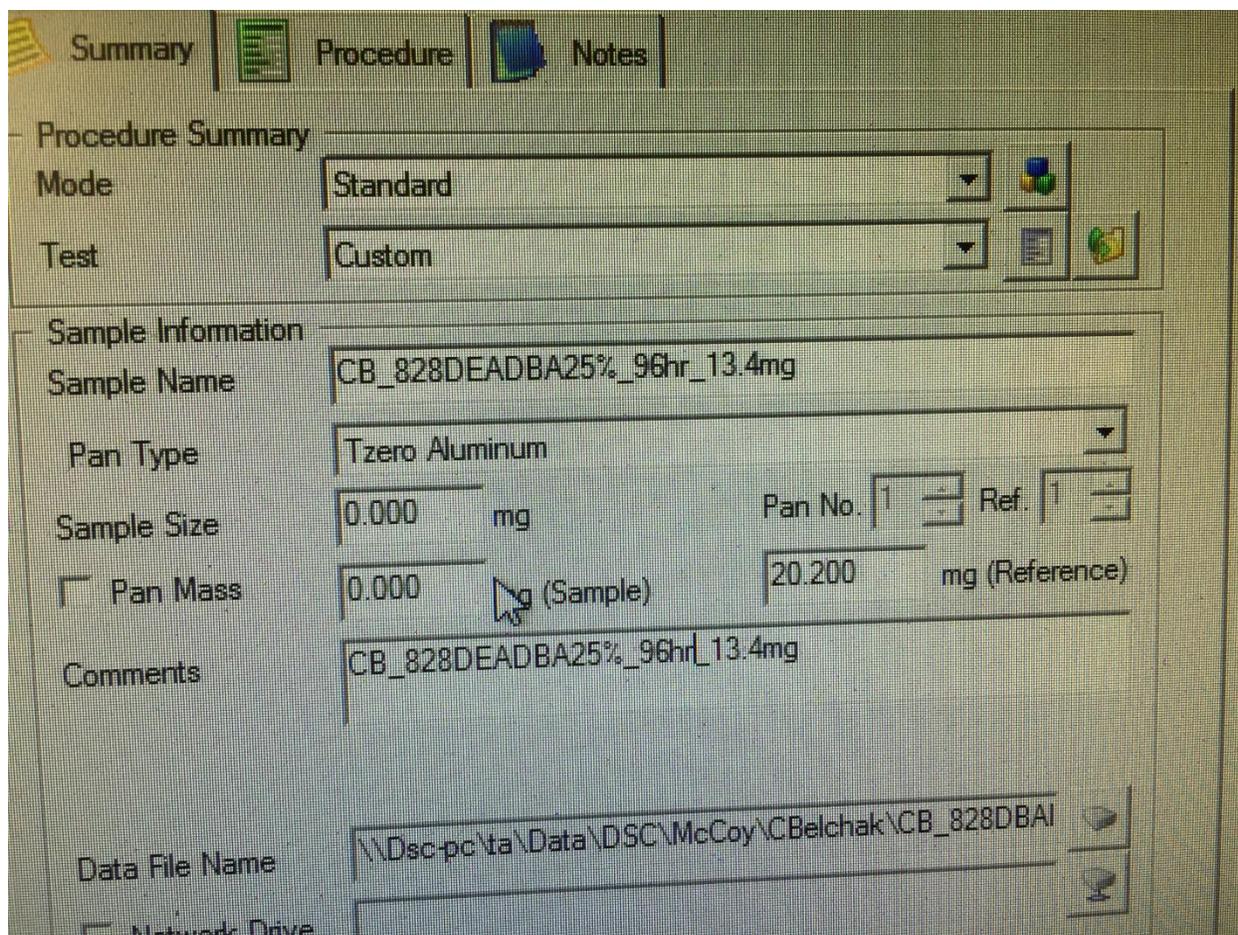


Figure 7. Sample Information Panel

4. Copy the sample information and paste it into the **Comments** box of the **Summary** tab, the **Notes** box under the **Procedure** tab, and in the **Extended Test** box under the **Notes** tab.
5. In the **Data File Name** box under the **Summary** tab, click the button to the right of the box.
6. Navigate the save directory to match the initials of the sample creator under the **McCoy** file and open the file.

Name	Date modified	Type	Size
CB828DBADEA25%_24hr_10.2mg	3/19/2018 10:48 AM	2MG File	
CB828DBADEA25%_72hr_25.0mg	3/9/2018 10:54 AM	0MG File	
CB828DBADEA25%_72hr_25.0mh	3/9/2018 10:55 AM	0MH File	
CB828DBADEA25%_72hr_25.0mi	3/9/2018 12:22 PM	0MI File	

File name: CB_828DBADEA25%_96hr_13.4mg

Figure 8. File save directory

Running the Test

1. Under the **Summary** tab, enter the mass of the sample in the **Sample Size** box.

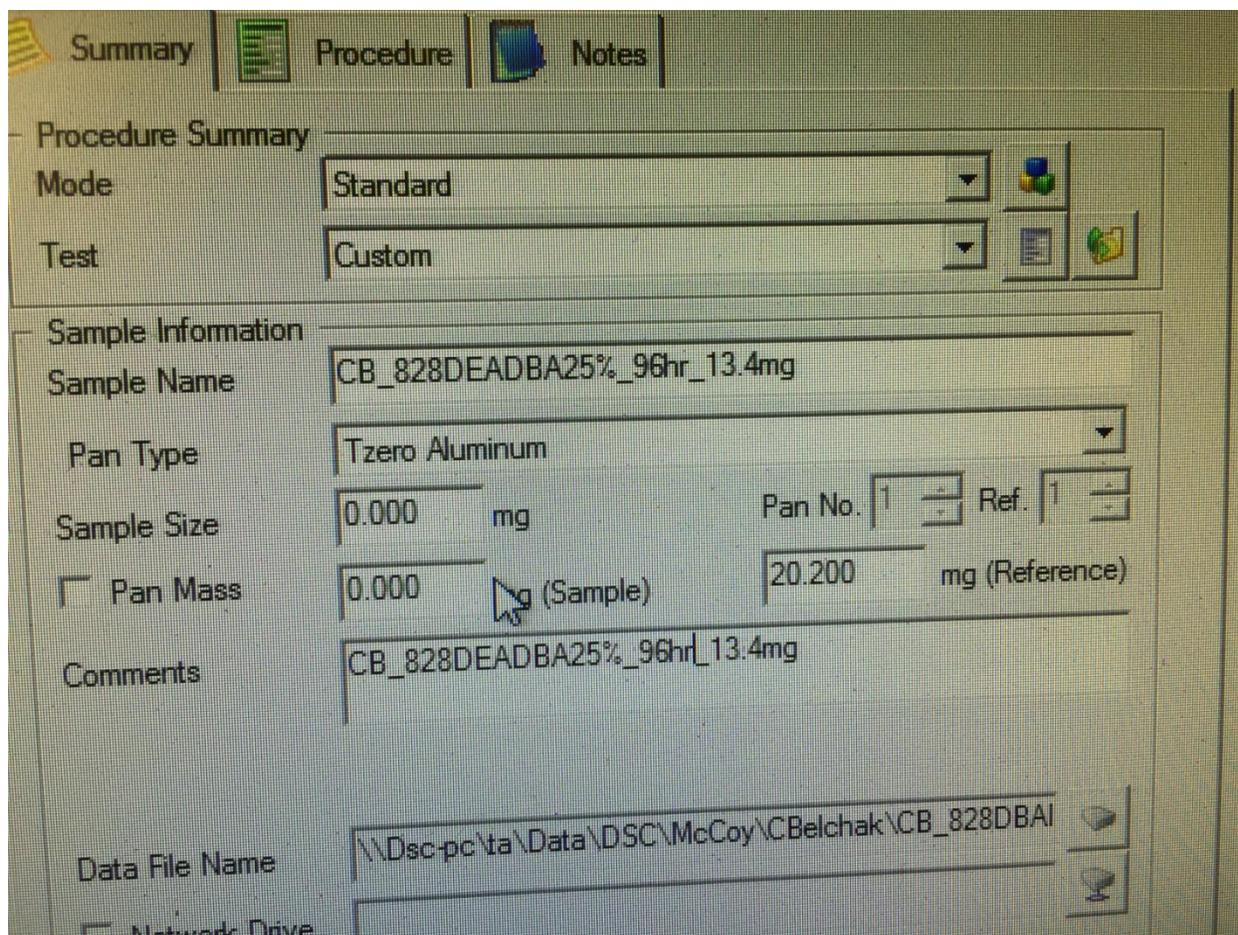


Figure 8. Sample Information Panel

2. Weigh a Tzero Aluminum DSC Pan on the analytical balance. Record the mass of the pan in the **Pan Mass** box. **Note: Do not touch the pan with your fingers. Doing so will affect the quality of the run. Use a tweezers to manipulate the pan instead**
3. Open the sample envelope and get the DSC sample from it. **Note: Do not touch the DSC sample with your fingers. Doing so will affect the quality of the data. Use the tweezers to manipulate the DSC sample.**
4. Place the DSC sample in the Tzero Pan.

5. On the computer, under **Control>>Lid**, select **Open**. **Note: The lid should be kept closed as much as possible to avoid contamination. Perform steps 6 and 7 as quickly as possible.**

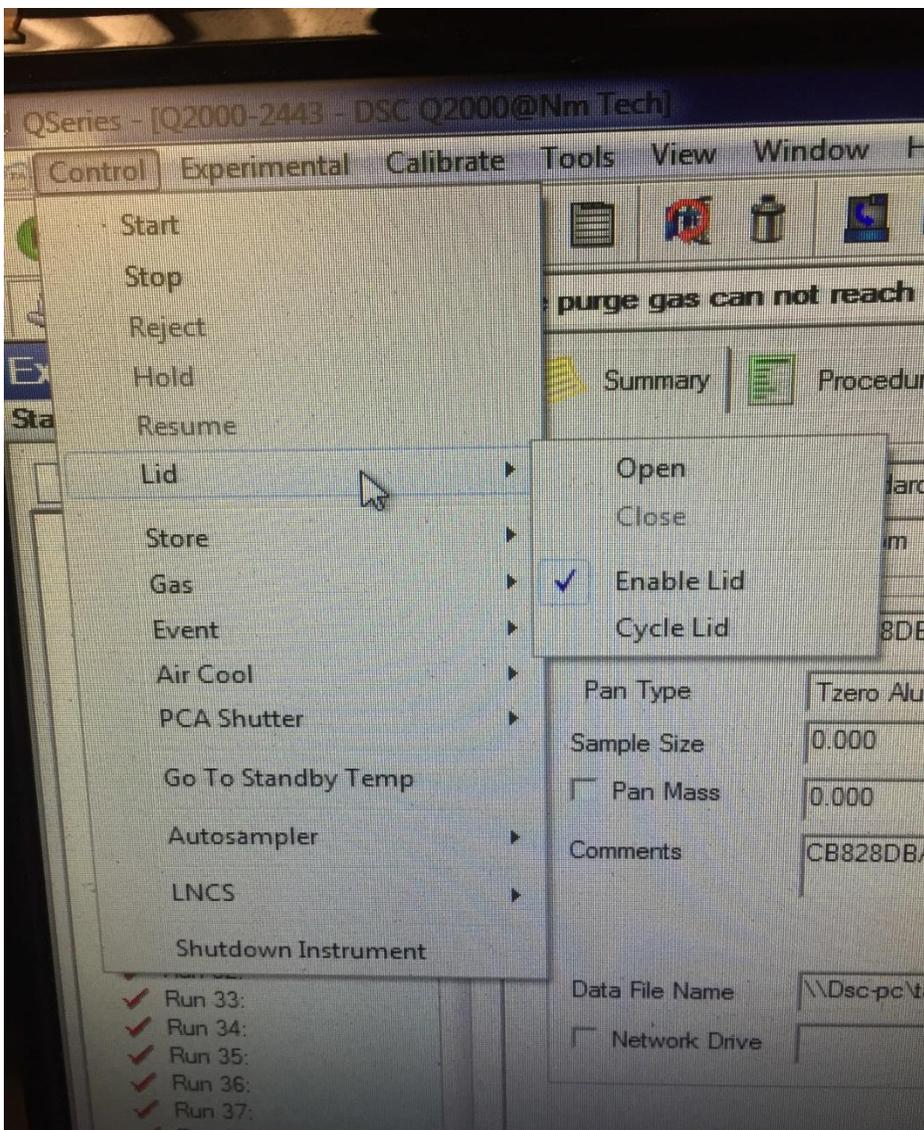


Figure 9. Lid Control Menu

6. Once the DSC lid is open, place the pan and DSC sample on the open pedestal inside the analysis chamber. **Note: There is a Reference pan in the analysis chamber. Do not remove it from its pedestal.**



Figure 10. DSC sample chamber with sample pan and reference pan.

7. On the computer, under **Control>>Lid**, select **Close**.
8. On the computer, on the leftmost panel, select the arrow. Select the space to the left of the latest run to move the arrow there.
9. Start the run. This can be done by double-clicking the arrow, clicking the green play button on the top left corner, or selecting **Control>>Run**.

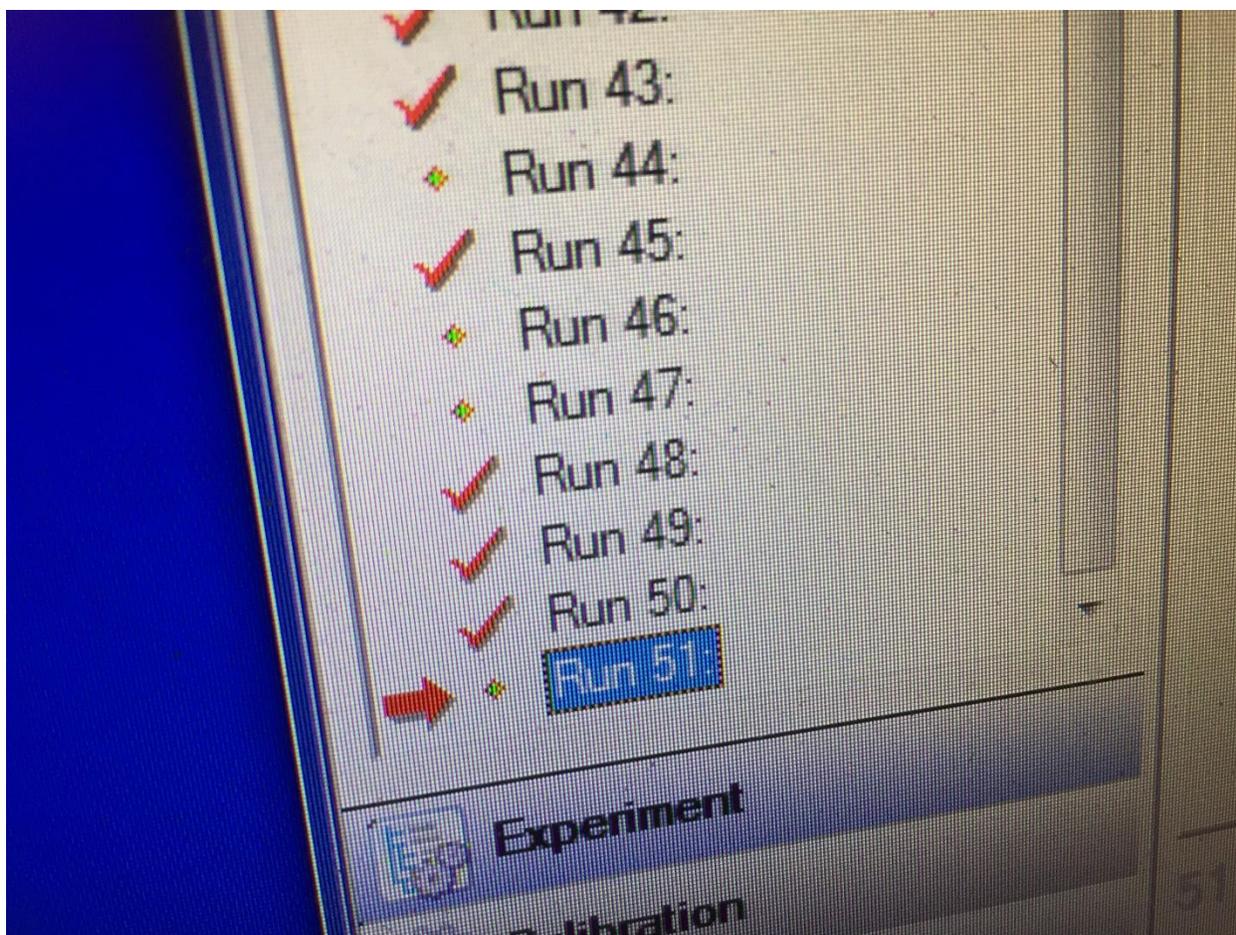


Figure 11. Sample Information Panel

10. Replace the wooden cover over the DSC machine.
11. Place the sample envelope near the computer.
12. As a final check, on the left panel on the TA Instruments window, there should be a green stick figure running in place to show that the run is underway.

Post-Run Operations

1. Remove the wooden DSC machine cover.
2. Select **Control>>Lid>>Open**.

3. Remove the pan with the DSC sample from the analysis chamber. Replace the DSC sample back into its original envelope.
4. Select **Control>>Lid>>Close**
5. Select **Control>>Event>>Off.**

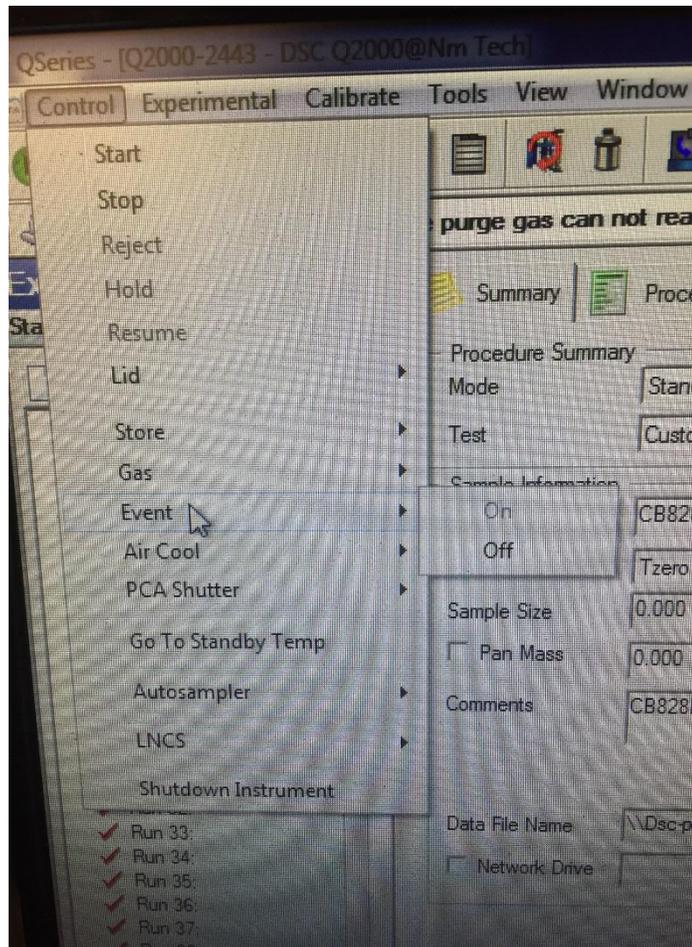


Figure 12. Event control panel

Exporting Test Data

1. Insert a flash drive into the DSC computer tower.
2. Open the “TA Universal Analysis” program.

3. Go to the “File” tab at the top left of the TA Universal Analysis window. Click “Open File”. This will open a file selection dialog box. Navigate to the desired .rar file. Once you select a file, dialog box will open in the program.
4. Within the dialog box, set “Exotherm” to “Down”.
5. Under “Signals”, and check the signal box which contains data. If more than one signal has data, set “Signals” to “Nothing”.
6. Click “Ok”. A graph should appear in the program window.
7. Change the X Axis of the graph to temperature by right clicking on the plot, going to “X Axis”, and selecting “Temperature”. Set the time ranges to the following:
“First Heat”: 10-23
“Second Heat”: 48-60
8. The graph should now resemble an "S"-like curve. If the data time limit is too large, then there will be steep drops on either side of the ramp. Reduce the time in order to get the desired data set. If the data time limit is too short, then the ramp will terminate on one or both ends before it levels off. Increase the time in order to get the desired data set.
9. Once the desired data set has been isolated, go to “File”, “Export Data File” and then “File Signals Only”.
10. An export dialog box will appear. Under the “Character Set” box, check “Windows (ANSI)”.
11. Under “Options”, check all three boxes: “No parameter block”, “No data breaks or flags” and “No invalid data points”.

12. Name and save the data file.
13. Open Windows Explorer, located on the taskbar.
14. Go to “Recent Places” and select your recently saved data.
15. Copy the data files onto a flash drive.(there should be one RAR file that the macs cannot open and a number of csv files that you created in this procedure)
16. Close all windows, and safely eject your flash drive.