Discovery SDT 650

Location of Machine: Advanced Composites Lab, IGRM 1308A

<u>Location of SOP and Machine Operating & Safety Manual</u>: Composites Lab website under resources; Composites Lab TRACS site; and Hardcopy near machine.

Emergency Contact:

- Call 911
- Call EHS & Risk Management at 512-245-3616
- Call Head Lab Technician, Dr. Ray Cook (office 512-245-2050)
- Call Dr. Jitendra S Tate (office 512-245-4872)

Before using this machine:

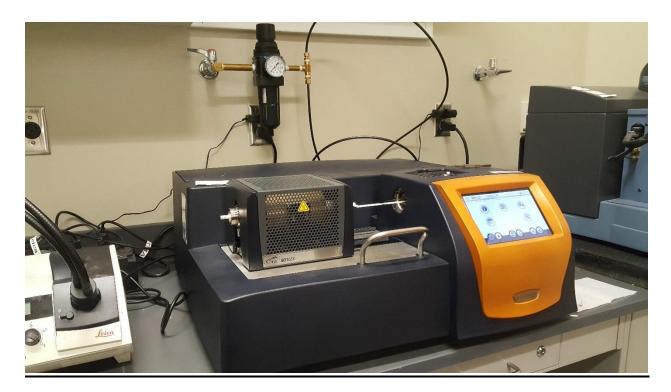
- You must have permission from Dr. Tate.
- You must have received formal training from technician or, trained research student (designated by Dr. Tate) related to machine safety and operation.
- You must read and understand SOP and Machine Operating & Safety Manual.
- You must use this machine under direct supervision of Dr. Tate or, Dr. Cook or, trained research student (designated by Dr. Tate).
- You must have signed "Lab Rules" document with Dr. Tate. This document must be signed every semester fall, spring, and summer (as applicable).
- If you do NOT follow above instructions you will be held responsible for your own safety and damages.

Safety Precautions:

Protective Equipment: Prior to performing this procedure, the following personal protective equipment must be obtained and ready for use: **Gloves, Safety Goggles, and Lab Coat.**

Important Safeguards:

- 1. Make sure to open or close the furnace only after equipment is online (connected to the system).
- 2. Always TARE the equipment before loading the sample. Never TARE the equipment after loading the sample
- 3. Never load sample with the pan still on the cantilever beam.
- 4. Always turn on the compressed air and nitrogen supply before starting the experiment and turn them off after the experiment.



General information

The SDT 650 helps in studying the change in transitions of a polymer under conditions of controlled temperature, time, and atmosphere. In our lab we use SDT 650 with dual horizontal beam design for superior heat flow and weight measurements. SDT 650 uses **TRIOS** software for instrument control, data analysis and reporting. It is a high performance, ease-of-use device that is reliable and can be used in a wide variety of applications, including:

- Glass transition temperature
- Heat capacity
- Weight Loss
- Crystallization transition temperature
- Melt transition temperature
- Stress Relaxation Analysis

Specifications:

Temperature Range (max): Ambient to 1500°C

Temperature Precision: ±0.5°C

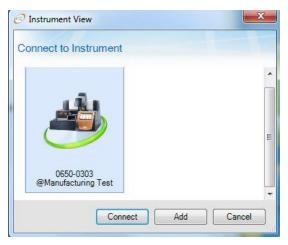
Heating rate (linear): 0.1 to 100°C/min

Calorimetric Accuracy/ Precision: +/- 2%
Heat capacity accuracy: +/- 5%
Sample weight accuracy: 200 mg
Weighing accuracy: +/-0.5%
Weighing Precision: +/-0.1%
Vacuum: 50 µTorr

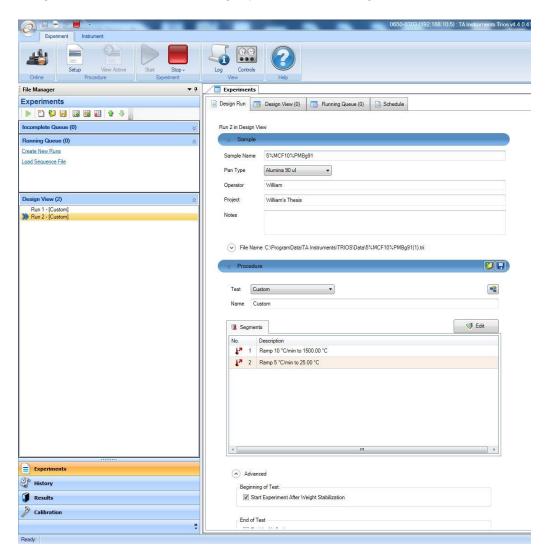
Standard Operating Procedure:

1. Locate the TRIOS icon on the desktop and click on it to get the following

screen.



2. Select the **connect** option to link the SDT 650 equipment with the TRIOS software. Clicking on the connect button will get you to the following screen.



3. By clicking on the **setup** option under **experiment** tab.

4. The left panel of the TRIOS software; has options of:

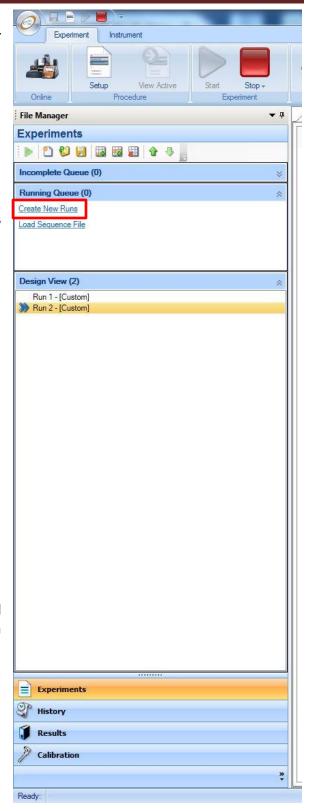
Experiments,

History,

Results and

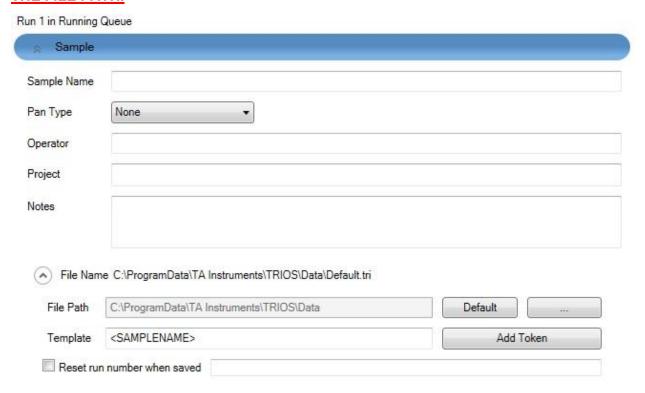
Calibrations.

5. Clicking on the **Experiments** tab, would open up a dialog box, in which one can design the runs.

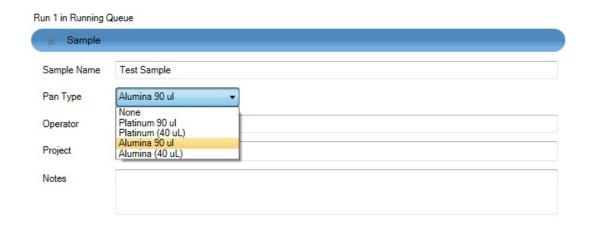


Designing a run:

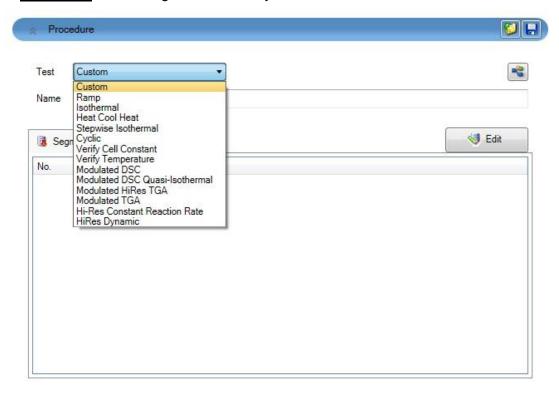
- 1. Clicking on the <u>Create New Runs</u> will display the dialog box as shown in the screen below in which you can enter all the details related to the experiment run.
- 2. The files will be saved in a default location and **NO ONE IS ALLOWED TO CHANGE THE FILE PATH.**



 The SDT 650 is calibrated for both alumina and platinum type pans. The Advanced Composite Laboratory uses <u>Alumina 90 ul pans as reference and sample holder</u>. Under pan type <u>always select Alumina 90 ul</u>.



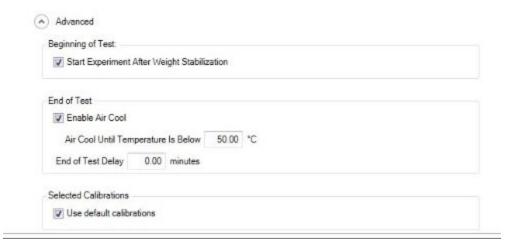
4. After selecting the pan type and entering the information, go to the next option of **procedure** for creating conditions for your desired run.



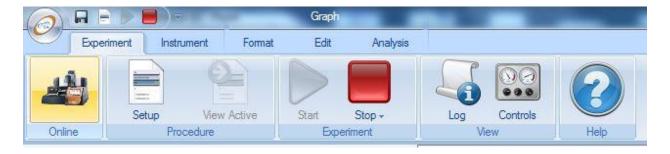
- 5. The <u>Test</u> drop down menu gives you variety of options to choose from for testing of polymers. Select a test type and give a name for the procedure. Based on the type of test chosen, the segments are going to load-up in the following sections.
- 6. If you select <u>custom</u>, you will have the freedom to determine the conditions of test. Clicking on <u>edit</u>, will give you options to <u>drag and drop</u> into the segment box.



- 7. Click on <u>apply</u> once after you design the parameters. You can save the procedure for your future runs.
- 8. The TRIOS also gives the following options and Advanced Composites Laboratory uses all the options for consistency.



9. Once after this step, you can click on the <u>START</u> button on the top under the <u>EXPERIMENT</u> tab. Once after the run is complete and the <u>temperature of the furnace</u> is <u>below 50 °C</u>, click on <u>STOP</u>.

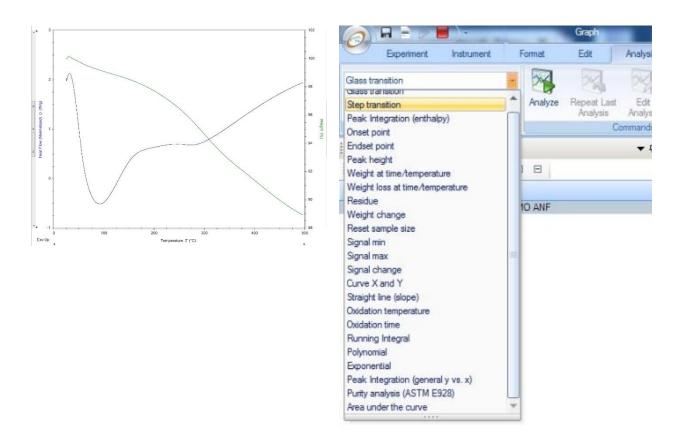


10. The <u>INSTRUMENT</u> tab gives you the options of opening and closing the furnace before and after the run. <u>NEVER USE THE SHUT DOWN BUTTON</u>. Similar options for operating <u>FURNACE</u> and <u>TARING</u> are on the console of the equipment.



Post-test Analysis:

1. A typical plot of the DSC and TGA curve is as shown, with **Normalized heat flow** plot on the **Y1 Axis**, **Weight percentage** on the **Y2 axis** and **Temperature** on the **X axis**.



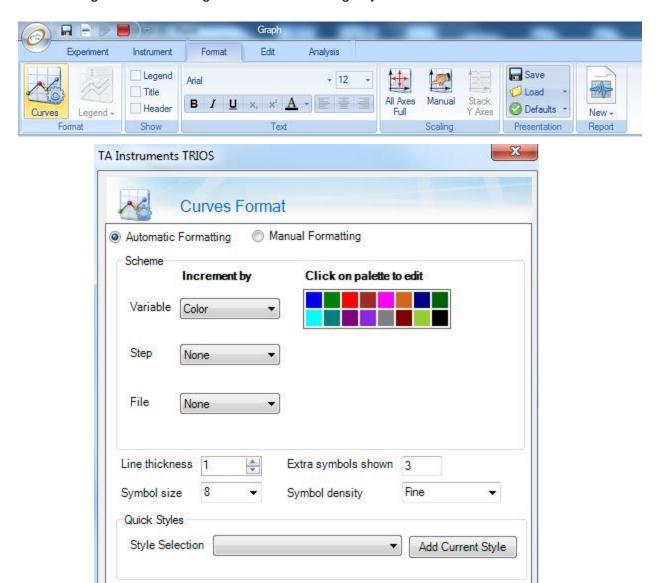
- You can select the region of your interest on the curve and analyze a variety of phase transitions, which is provided under the **ANALYSIS** tab. Select your <u>region of interest</u> on the curve, go to <u>analysis</u> tab, select the <u>transition</u> of interest and click on <u>analyze</u>.
- 3. The **ANALYSIS** tab also provides a number of options of finding the derivatives and adjusting the curves.



Set As Default

Preview

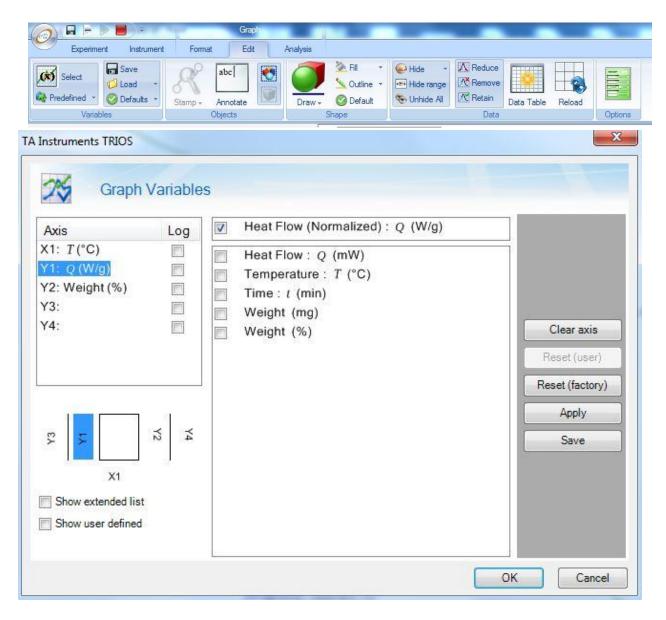
4. **FORMAT** tab gives various options to edit the text on the graphs, creating legends, scaling and color coding the curves according to your interest.



5. **EDIT** tab gives options selecting graph variables, insert/use different shapes and editing the data. Under the variables option we can select from a different options for the axes of the graph.

OK

Cancel

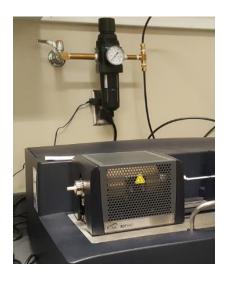


6. By clicking on the <u>TRIOS icon on the top left</u>, you can save the data into a PDF file or any other format by selecting <u>"Save As.."</u> You can also export your curves in the form of <u>Images</u> or export data into an <u>excel file</u>, by selecting option <u>"Export".</u>



Shut-Down Procedure:

- 1. Make sure you click on the red **STOP** button before turning off the Nitrogen supply.
- 2. Turn off the **COMPRESSED AIR** supply and the **NITROGEN AIR** supply.





- 3. Then you can **EXIT** the application.
- 4. Do not attempt to **CLOSE OR OPEN** the **FURNACE** once after you **EXIT** the application.