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Updated on 02/12/2017 by Peter Wang

The following is the Standard Operating Procedure (SOP) for the Mettler Toledo DSC 822e.

All Mettler Toledo instruments are operated using the STARe software v15.00a.

I. General UseMETTLER

1. Double click STARe Software



2. A prompt will appear asking for the User Name and Password. The User Name is **mettler**; there is no password. Hit **OK**.

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- 3. Two windows will open; one controlling each of the attached devices. Open the window controlling the DSC 822e.
- 4. On the left hand portion of the window, select **Routine Editor**.

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- 5. Hit **Reset** in the lower right hand corner of the window.
- 6. If a method needs to be created, select **New** and create a program for the TGA (for specifics on creating a new Method, please refer to the person/s in charge of the instrument). If a method is already created, select it using the **Open** tab.
 - The pans used are 40 μ L aluminum pans with a pin on the bottom. The pin allows for accurate sample placement on the sensor. Each pan contains two parts: the bottom with the pin and the hat.
 - Prior to running a DSC, knowledge of the decomposition temperature, *T_d*, is beneficial. This will allow you to run a method that ramps up to the highest temperature possible without sample degradation. Most runs should not come within 25 °C of the *T_d*.
 - When choosing a method, determine if the method is appropriately made. Runs should mostly look like the following:
 - Dynamic Segment (heating or cooling)
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 - o Dynamic Segment
 - o Isothermal Segment @ end temperature of the dynamic segment for 2-5 m
 - Dynamic Segment back to room temperature
 - When creating a new method/modifying an old method, make sure that the end temperature of one segment matches the start temperature of the next segment.

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- 7. Sample Preparation:
 - 1. Weigh out the sample in an aluminum pan. The weight should be between 3-10 mg.
 - 2. Once weighed out, bring the sample to the press and place the pan in the press holder.
 - 3. Place the hat (top half of the DSC pan) onto the pan. It should sit in fairly easily.
 - 4. Place the press holder in the press and crimp the pan.
 - 5. OPTIONAL: Using the pin in the DSC box, poke a small hole in the top of the pan. You do not need to stab the pan, light force is sufficient in poking the pan.
- 8. Once a method is selected, place a sample name into the field. Sample name should be placed as follows: <u>YYMMDD_NOTEBOOK CODE_DSC</u>

Place the weight of the sample in the box and place the sample into the DSC on the left side. DO NOT TOUCH THE BLUE SENSOR WITH THE TWEEZERS! The position does not matter. Click **Send Experiment**.

- The DSC does not have an auto sampler.
- Make sure the reference pan matches the sample pan. If the sample pan has a hole in the top, the reference pan should also have one. If the sample does not have a hole, the reference should match.

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- 9. Open up the nitrogen line. Make sure the leftmost flowmeter by the DSC is reading between 10 and 50 mL.
- 10. Before opening the liquid nitrogen dewar, check the level. If it is less than a quarter full, ask someone in charge of the instrument for help in how to fill it up. If it is sufficiently full, turn it on
- 11. Click **Start** at the bottom right hand of the window. This will open the experiment on the module. A prompt wil appear in the green bar that says *Going to Insert Temperature*. Click **OK**. A prompt will appear in the green bar that says *Waiting for sample insertion*. Click **OK**. The bar will go red.

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12. When the sample is done running, remove the sample and click OK. DSC samples can be stored and used for multiple runs. Turn off the liquid nitrogen and nitrogen.

Retrieving Old Data before 02/09/2017

1. Click Database at the top part of the software



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- 2. Click Select Database and chose Archieve DB1. Do not manipulate anything else!!!

3. Now you can go back to the **Home** tab and click **Evaluation Window**. Click **File-Open Curve** and select your curve.

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Following the same procedure as step 1-2 and switch the data base back to the original Working DB1.

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