SOP 1 – Changing Sample Carrier for Jupiter F1 STA

1. Identify needed sample carrier, our lab carries three different types
    a. DSC
        - This carrier has two sample holders at the top of the alumina stem. Will measure change in heat flow between the sample crucible and a reference crucible along with mass change throughout the heating process.
        - Useful for identifying phase changes through the DSC curve and mass losses.
    b. TG
        - This carrier has the U-shaped pins at the top of the alumina stem. Measures precise mass changes while heating.
        - Will need the other half of the U-shaped sample hanger to use. It is found in the large cabinet underneath the STA computer.
    c. Pin
        - This carrier is used solely for testing sample compatibility with different heat cycles. Used with the alumina platform found in the large cabinet underneath the STA computer.
2. Remove sample carrier you want to replace from the STA.
    a. On the front panel push the lifting device up-arrow button and the button marked safety on the bottom of the right panel at the same time. This will lift the furnace from its position on the platform.
    b. Once completely lifted, rotate the furnace 45 degrees counterclockwise to free up space to lift the sample carrier out. You will feel the furnace “lock” into place.
    c. Wearing gloves, grab the inserted sample carrier by the alumina stem about 2 cm below the crucible holder, gently but firmly lift straight up.
    d. The radiation shield will lift out with the sample carrier.
    e. Have an empty radiation shield container ready on the benchtop. These are the plastic tubes with black lids and green foam at the bottom.
    f. Slowly insert the bottom of the sample carrier into the empty radiation shield tube until the base of the radiation shield fits into the hole in the green foam.
    g. Keep sliding the sample carrier down another few centimeters while the radiation shield stays in place.
    h. Two metal pieces, a ring with a slot, and a flared ring will be on the alumina stem.
    i. Detach these two pieces from one another and remove the slotted ring first and then sliding the flared ring over the bottom of the sample carrier stem.
        - Place these in the sample carrier’s box.
    j. Now the sample carrier can be removed from the radiation shield by lifting up, back through the radiation shield and its container.
    k. Remove the two foam support blocks in the sample carrier’s box and gently insert the alumina stem into the cut slot, about ¼ and ¾ of the way down the stem.
        - Be gentle, stems have been snapped during this step.
    l. Insert sample carrier into box with foam blocks.
3. Insert Sample Carrier into STA

a. While wearing gloves, reverse steps to take new sample carrier out of the container and attach radiation shield.
    - Be careful taking carrier out of the foam blocks, safest way is to remove blocks from the stem, not the stem from the blocks.
b. If furnace is down, lift furnace and rotate to make space.
c. At the bottom of each sample carrier, on the copper-looking section there is a red dot. This dot will be facing towards you as you insert the sample carrier and radiation shield into the STA.
    - This step is very important. If inserted the wrong way, the gold wires in the bottom of the STA can become bent. Also if inserted backwards, the signal given from the STA will be wrong.
d. Slowly insert the sample carrier and radiation shield with the red dot facing you into the sample carrier mount.
e. The radiation shield will sit first, this can help you guide the base of the alumina stem properly.
f. Slowly bring the base of the sample carrier down to the bottom of the mount.
g. Applying gentle pressure, the base should fit into a mount at the bottom.
    - This takes some practice, nothing should be forced, but it does need a gentle amount of pressure to fit onto the slot.
    - Take your time, don't rotate the sample carrier, if you need to, lift out the carrier and radiation shield, realign and try again.
h. Once mounted, if using the DSC sample carrier, the crucible holders will be aligned directly straight in front of you, not off to any side/angle.
i. Move the furnace back into place and lower the furnace onto the sample carrier by pressing the down and safety buttons at the same time.
    - If mounted correctly, the furnace should have no ability to hit the sample carrier or radiation shield on its way down. However the fit is tight and can be nerve-racking so slowly lower and double check it won't snap the sample carrier or radiation shield, especially the first closing after changing the sample carrier.

SOP 2 – Running a DSC sample in the Jupiter F1 STA

1) Choosing crucible material
   a) This is arguably the most important step in the sample running process for the STA. If the wrong crucible is chosen, the sample carrier and furnace could be damaged.
   b) Things to take into account are the purpose of the experiment, temperature range, and interactions of the material.
      i) The most commonly used crucible in our lab is alumina.
      ii) Alumina crucibles have a large temperature range (1600C) and have few reactions with the metals we have been working with. However, they are not sealed, therefore if you need to contain a gas, we have a hermetic sealing press that can be used with gold crucibles.
      iii) For low temperature applications and organic materials, aluminum crucibles are useful, and our lab contains plenty.
      iv) Research to make sure your sample is compatible, there are many different types of crucibles available to order.
   c) You will need at a minimum two of the same crucibles to run an experiment, ideally three to keep the sample, reference, and blank samples all separate.
   d) Clean the crucibles sonically and ideally heat them up in the furnace to the highest temperature you will be testing them at before starting the sample process. This ensures no contaminants to possibly alter your data.
2) To obtain one measurement for DSC, two different runs need to done.
   a) The baseline run and then the sample run.
      i) The baseline run is done using two blank, identical crucibles. This run will have the exact same temperature profile as the sample run.
      ii) This baseline subtracts the instrument error out of the sample run. Without this, the data collected would be useless.
3) Weigh out crucibles and sample masses.
   a) Blank Crucible, Reference Crucible, Sample Crucible empty, and Sample Crucible + Sample.
4) Open furnace using step 2a from SOP 1.
5) Place reference crucible in the furthest away crucible holder on the sample carrier.
6) Place blank crucible in the closer crucible holder on the sample carrier.
7) Close the furnace.
8) Open the STA software on the computer to the left of the STA.
   a) The correct software is NETZSCH-Proteus-61, there is an -80 version, don't use this.
      i) A USB error may come up when opening the software. Just click through, it is fine.
9) Turn off any gas flow into the furnace.
   a) In the software, click the icon with the red and blue bottle on the top menu that says "MFC gases extended"
   b) Slide down any modifiers to 0ml/min. The Purge 1 and Protective gases should be the only ones running.
   c) The Protective very rarely gets to 0 ml/min. If a high vacuum is needed, turn off the gas manually on the inlet in the back of the STA by closing one of the many valves and purge the tubes by turning up the MFC. Once the MFC starts to go down by the lack of argon in

the tubes, turn down the gas MFC to 0. This should then get the protective gas inflow to 0ml/min.

10) Vacuum Pump and purge the furnace to create an argon atmosphere.
    a) At the top of the furnace there is a black valve.
    b) Close the valve by rotating it 90 degrees clockwise.
    c) Remove gas release tube from beaker of water to the left of the STA.
        i) Although not completely necessary, this step gets rid of any chance you have of opening the valve while the furnace is under vacuum and sucking water into the furnace, ruining the electronics.
11) Turn on fore pump.
    a) To the left of the STA on the counter directly to the right of the computer there are two boxes. The box on top is the vacuum controller. To turn on the fore pump, flip green switch up.
12) Open up furnace to vacuum valve.
    a) On the back-left corner of the STA platform there is a turbo-pump with a circular black valve on the left side. Turn the black valve counterclockwise to open the valve. Open this slowly, especially if your sample is a powder. It can cause turbulence within the furnace, spilling your sample.
    b) You will hear the evacuation beginning
    c) On the STA's front panel you will see a vacuum gauge going from 0-100%. It is all relative but 0 is no vacuum/atmospheric pressure and 100% is high vacuum.
    d) The fore pump by itself should be able to reach a high 90% vacuum.
13) Turn on turbo pump
    a) On the front panel of the vacuum controller, there will be a pressure reading, once the pressure reads …E-1mbar, the turbo pump can be turned on.
    b) To turn on the turbo pump, push the furthest to the right of the four black buttons on the vacuum controller.
    c) The blinking green light should then turn on.
    d) The turbo pump takes a while to spin up, wait for a few minutes and write down the vacuum pressure reached. Should be in the …E-4 scale. Possibly more if you are willing to wait a long time.
14) Once desired vacuum is reached. Turn off turbo pump, close the black circular vacuum valve and turn off the fore pump.
15) Fill the furnace with Argon.
    a) Now that the furnace and sample is under vacuum, argon needs to be introduced to get rid of the vacuum and provide an inert atmosphere for heating.
    b) Turn up the gas inlet flow in the same MFC gases extended menu on the software and wait until the vacuum percentage disappears.
    c) To be safe, you can wait until the front panel of the STA reads OVERPRESSURE.
16) Open gas outlet and let equilibrate
    a) Open the black valve on the top of the furnace to let the overpressure of argon escape, and place the end of the outlet tube back into the beaker, doublechecking to make sure bubbles are coming out of the end and you are not sucking up water into the furnace.
    b) Let the overpressure gas escape to let the mass balance equilibrate. The 4th digit on the balance reading on the front panel of the STA should only go up and down by 3. Don't start a run if the balance isn't this stable.

17) Set up sample program on the STA software
    a) Open up: File-New-Correction
    b) Insert the proper parameters, making sure the correct sample carrier and crucible are chosen. -next
    c) Correctly label your sample crucible, either blank or sample, and insert crucible and sample masses if needed.
    d) Write short description of the experiment.
    e) Choose Temperature and Sensitivity calibration from file. Use latest file. -forward
    f) Insert temperature program. Usual temperature ramp rates depend on need. If looking for small phase changes, consider a slow ramp rate (5C/min). If looking for general melting point 20C/min should be fine. General Protective gas flow rate is 30ml/min and Purge 1 gas flow rate is 20 ml/min – forward-finish.
18) Set beginning flow rates, double check equilibration
19) Click Start.
20) Once run is finished, wait for furnace to cool to a reasonable temperature and then replace blank sample crucible with crucible with the sample.
21) Use same Pump/Purge procedure as above.
22) To use the previous run as a baseline use File-open-"Previous Saved Baseline"
    a) This will import the previous run's settings into the software.
23) Choose "Sample + Correction" when identifying the type of measurement.
24) Insert sample and sample crucible mass
    a) Everything else should be the same.
    b) Changing temperature program will make the baseline invalid.
25) Measurement is ready to be taken.