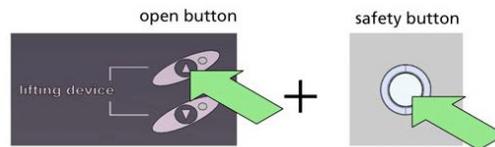# Standard Operating Procedure for STA 449F3

1. Make sure instrument is idle and temperature of the furnace is near room temperature (25-30 C) as indicated in the front panel of the device. Login in NUCore to have the screen turned on

2. To load your sample, move the furnace into the upper end position by pressing and holding simultaneously the "Open" button in front of the unit and the "Safety" button on the right side of the instrument. Keep holding the two buttons until the furnace has stopped moving

3. In the upper end position, the furnace can be swung out by 30° to the left (parking position) which gives you more room to mount your crucible

4. Measure the mass of your sample using the microbalance located next to the CHNS instrument (on your right)

5. Using tweezers, gently place your sample crucible onto the sample carrier head (sample in front/reference back). If you use loose powder directly into the TGA crucible, remove the TGA crucible from the carrier without a tweezer. Sample carrier sits on a very sensitive micro-balance, please be careful when you (un)mount your sample. With the default carrier, you can measure simultaneously TGA and DTA. If you need to measure DSC/TGA, please talk to a staff

6. Lower the furnace by swinging the furnace back to the normal centered position, and by pressing simultaneously the "Close" button and "Safety" button. Hold both buttons until the furnace has stopped moving
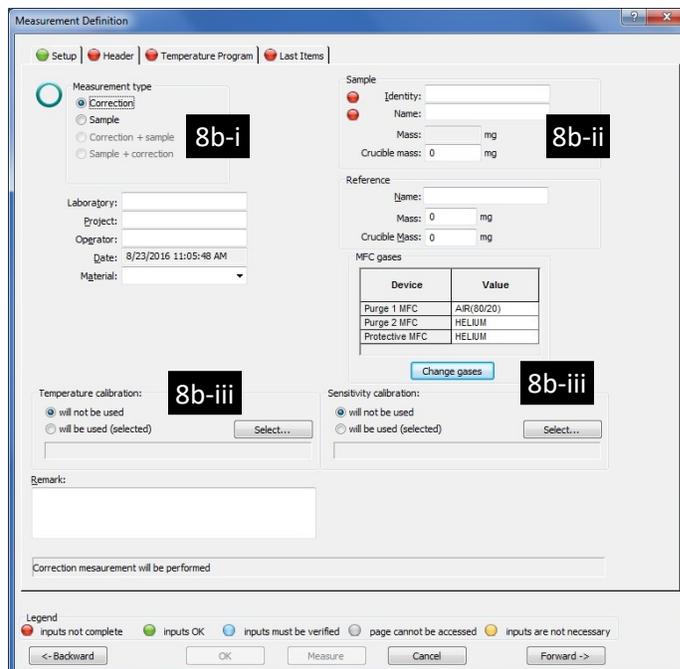
7. Start the STA 449F3 measurement program (icon on the desktop)

8. To create a new measurement or baseline:

    a. Go to 'File' menu and select 'New'. The 'Measurement Definition' window opens

    b. On the 'Header' tab of the 'Measurement Definition' window (red indicators next to each tab name or textboxes mark fields that need information by the user)


    i. Select the right measurement type: 'Correction', 'Sample', or combination of these two types. 'Correction' files are provided for some standard temperature programs. In case you want to perform a non-standard new measurement, it is highly recommended before measuring your sample, to run a baseline 'Correction' with your empty crucibles under the same conditions (i.e., Temperature Profile) you'd use for your sample. Uncorrected measurements may have overestimated weight values of ~ 0.2 mg under He or ~ 0.8 mg under air flow

8/24/2016

ii. Give values to the 'Identity' (e.g., lab book code), 'Name' (composition), and 'Sample mass' (disabled for a 'Correction' measurement) fields. All other information on the tab is optional.

iii. Load optional calibration files for temperature and sensitivity if you have any, i.e., when you measure your sample in an evacuated quartz ampoule, temperature may be slightly different then the temperature read by the thermocouple in the base of the ceramic crucible, therefore a calibration file must be loaded

c. Go to the 'Temperature Program' tab (or click on the 'Forward ->' button) where you'll build the temperature profile for your measurement


   i. On the right side of the windows, under 'Step Category', select 'Initial' and give a 'Start temperature' value of something close (± 7 degrees) to the actual temperature of the furnace shown in the front panel of the instrument. The 'Use AUTOVAC Controller' option must be unticked
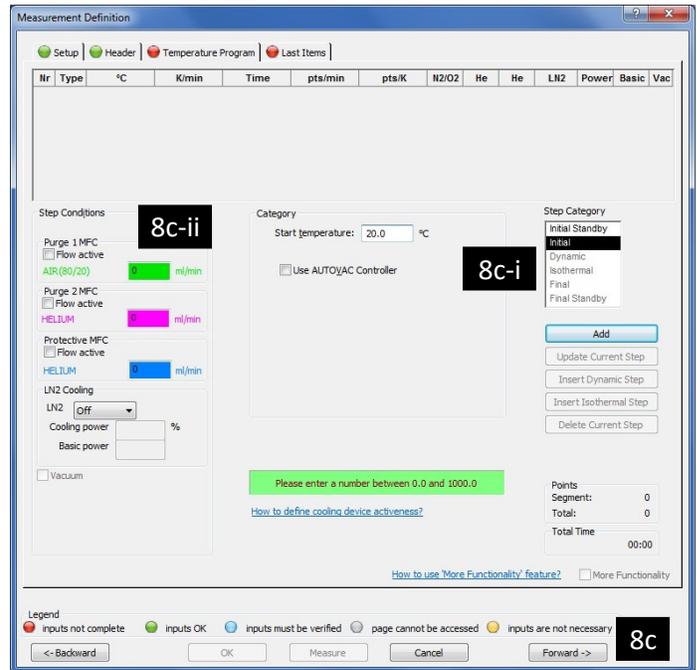
   ii. On the left side of the window, under 'Step Conditions':

      o Set the 'Protective MFC' (blue textbox) Helium flow to 25 ml/min. This gas flows internally through the instrument and it is independent from the gas environment your sample sees in the furnace (which you'll set in the next step)

      o If you want to use Helium as the gas in contact with your sample, set the 'Purge 2 MFC' (purple textbox) to 50 ml/min. In case you want to use air, set the 'Purge 1 MFC' (green textbox) to 50 ml/min instead

      o Set 'LN2 Cooling' to 'Off' or untick the 'Cooling' option if pressurized air is connected

   iii. Click on the 'Add' button at the right side of the window to add the 'Initial' step into the 'Temperature Program'. There can be only one 'Initial' step in the program

   iv. Select the next step in the program, either 'Dynamic' or 'Isothermal'

      o Set the 'End Temperature' and 'Rate' to the desired values

      o Make sure the gas flows are set to the intended values. 'Protective MFC' must be always at 25 ml/min

      o For a cooling 'Dynamic' step at a rate > 5C/min you have to select the 'Cooling' option (pressurized air)

      o Click on the 'Add' button to add the 'Dynamic' or 'Isothermal' step. Step will be added in the list under the 'Initial' step

   v. Keep building the 'Temperature Program' with all the steps you need for your measurement. You can tick on the 'More Functionality' box to see an overview plot of your temperature profile

8/24/2016

vi. Add the 'Final' step in your program. This step is preset by the software to 25 degrees above the maximum temperature of your program as an overheating safety step

   d. Go to the 'Last Item' tab and set the file name where the data will be stored. Always save into your group folder

   e. Click on the 'Forward ->' button to start the temperature program. If you click on the 'OK' button instead, follow step 9

9. To start your temperature profile, go to the 'Measurement' menu and select 'Start Measurement' (or click on the 'Play' button on the icon bar)

10. The 'Start Measurement' window opens. The 'Start' button will be enabled only when the temperature difference between your setting in the 'Initial' step and current temperature in the furnace is <7.5 degrees. Re-adjust the temperature in your 'Initial' step to bring the difference within the expected threshold
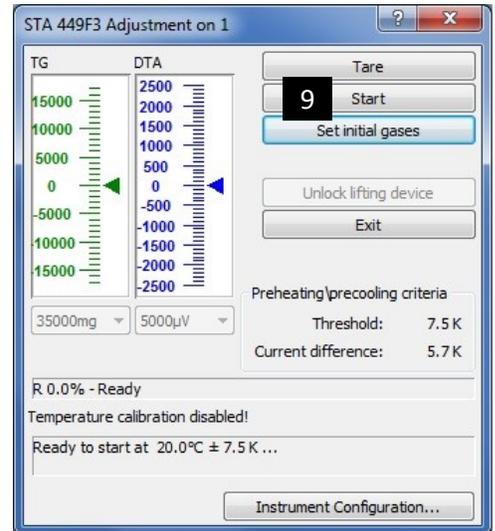
11. Click on the 'Start' button to start your temperature profile

12. During measurement, you can see a step-by-step progress of your program by selecting 'View/Edit Running Measurement Program' under the 'Measurement' menu. If you need edit the running program (e.g., extend the isothermal time or increase the target temperature of a step), change the corresponding values on the window and press 'Apply'
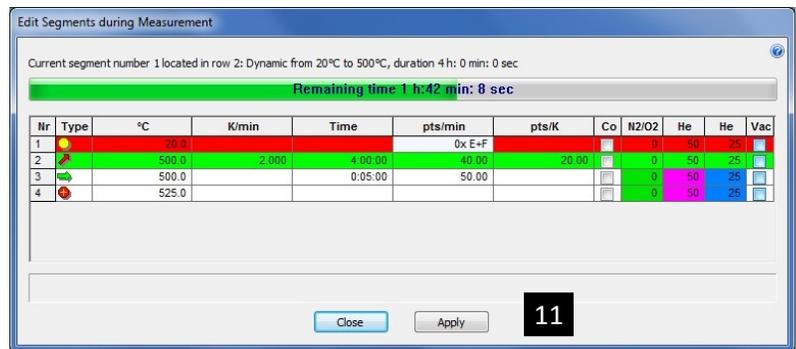
13. To stop/cancel the measurement, click on the 'Stop Measurement' under the 'Measurement' menu. Software will ask you for a confirmation and for saving or not the data collected so far

14. To convert your data to an ASCII file:

   a. Start the 'Proteus Analysis' software (icon on desktop)

   b. Load your file

   c. Press on the 'T/t' icon (X-Time / X-Temperature) to plot the data as a function of temperature

   d. Select either the TGA or DTA curve in the plot by clicking on it

   e. Select 'Export Data' under the 'Extras' menu

   f. On the menu above your plot:

      i. Tick 'Full range'

      ii. Tick 'All' in the 'Signal' section

      iii. Select 'All' in the 'File' section in case you have multiple files opened and you need to convert all of them

3

  iv. Click on the 'Change' button and confirm that 'CSV' is selected, click on 'OK'

  v. Click on the 'Export' button to save into a *.csv file

  vi. Set the file name and make sure you are saving into your group folder

15. When measurement is done:

 a. Remove your sample and lower the furnace as described in step 2, 3, 5, and 6

 b. Clean the ceramic crucible (e.g., soak it in a solvent that dissolves your material and its residue)

 c. Dry the crucible in the oven located across the STA

 d. Remove any vials, weighing paper, etc. you may have left in the room

 e. Logoff from NUCore