IOWA STATE UNIVERSITY
Chemical Instrumentation Facility

1234 Hach Hall
515-294-5805
www.cif.iastate.edu

Netzsch TGA/DSC – IR/MS Operating Instructions

Revised 09/02/20 B.B.

Location: **1238 Hach Hall**
Contact: **Brett Boote, 1234 Hach Hall**

Safety

All researchers working in 1238 Hach Hall must complete the EH&S course: *"Fire Safety and Extinguisher Training"*. When preparing samples in this room, please wear all appropriate personal protective equipment. Aprons, safety glasses, and rubber gloves are available in 1238A Hach Hall. If solvents are involved, please prepare your sample in room 1238A.

Properly dispose of glass pipettes and plastic sharps in the containers provided. Waste solvents can be disposed of in the waste containers provided in 1238A. All of the computers in this lab have direct links from the desktop to MSDS sheets, the EH&S Laboratory Safety Manual and to the CIF Safety Manual.

Fig.1 Netzsch STA449F1 TGA/DSC System with IR and MS

Introduction

The Netzsch STA449F1 TDA/DSC system simultaneously provides TGA data (Thermogravimetric Analysis) and DSC data (Differential Scanning Calorimetry) simultaneously. In addition, the identity of gases evolved during the heating process can be determined by IR (infrared) or MS (mass spectrometric) detection. The system is fully automated and includes a 20-position sample carousel.

This guide will introduce you to the Netzsch STA449F1 hybrid TGA/DSC-IR/MS system. For further guidance regarding gas analysis, plumbing gas cylinders, calibration procedures, etc, please consult the CIF TGA website.

This guide is presented as an overview and concise flowchart; for some operational details you may need to refer to the appropriate appendix. During your training we will refer extensively to those appendices; undoubtedly during your initial uses of the instrument you will need to refer to them.

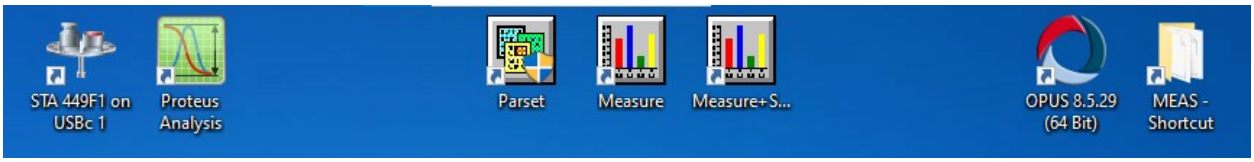
> ***NOTE: The presumption is that you already know a fair bit about the theory and application of DSC and TGA measurements, either from course work, textbooks, or through experience using other TGA-DSC instruments. If that is not the case, then you should NOT proceed with training. First, learn the fundamentals of these measurement techniques and their application to your research project. We provide limited application assistance for this instrument.***

The Desktop

The instrument computer should always be logged on as .*cif*. Do NOT logoff when you are finished- you will use the *LockScreen* program to log on and off the computer.

The notification section of the Taskbar should show the mass spec icon in *green*. When you mouse-over the icon it should display the message: "QMV200 connected". A red icon indicates a broken connection and will require assistance prior to MS data collection.

All programs you will need are at the top of the desktop:



Each program will be introduced throughout this document.

Directories and File Structure

During your training session, a data folder will be established for you at this location:

D:\Data*<your user name>*.

All data files must be stored in this location. Also, you were assigned a three-letter code such as *<bwb->*. This will normally be the initials for your first, middle, and last names, followed by a dash. All data files, method files, and parameter files must be named in this manner. Please append a date code to these filenames using the format *<mmddyyyy>* or similar. For example, *<bwb-01072020>*. For data files, please also add the Autosampler position to the filename (for example, *<bwb-01072020-1>*).

> As part of creating an *autosampler tray* to run your samples, you will direct the TGA/DSC (Proteus) data to your folder. The FTIR (OPUS) data will follow automatically. The mass spec (Aeolos) data will not. It should show up in a common directory at *<D:\Data\mass_spec_data>*. After it is collected you should move it to your folder. Occasionally the mass spec data may seem to be lost. Notify me of the problem.

Setting up a Measurement

You must verify that the initial state of the hardware is correct.

Hardware: Ensure that the correct gas or gases are connected to P1, P2 and Purge ports on the back of the instrument. The regulator(s) should be set to approximately 5 psi. Check the instrument front panel. It must show the same gases and ports as currently plumbed. If not, launch the STA449F1 program BEFORE you begin to write your method or autosampler tray to acquire data. Once the initialization has completed, select the *AFC-Extended (blue and red gas cylinders)* icon from the ribbon. A view will open showing the current selection of gases and flows for the three ports, P1, P2, and Purge. Use the software controls to select the correct gases and to set appropriate flows. Protective should generally be 20 mls/min; P1 and P2 are often set to 20 mls/min each, for a total of 60. Upon exiting the *AFC-Extended* view the gases and flows displayed on the front panel should match those just selected.

In the event of errors: If at any time you notice a strange response or error state, please proceed accordingly: open **Word** and then use a snipping tool (WinKey+Shift+S) to capture the software/error window. Paste the captured region into the date-coded **Word** document. This will allow staff to keep records on problems with the setup and assist all users in the long run with overall system stability.

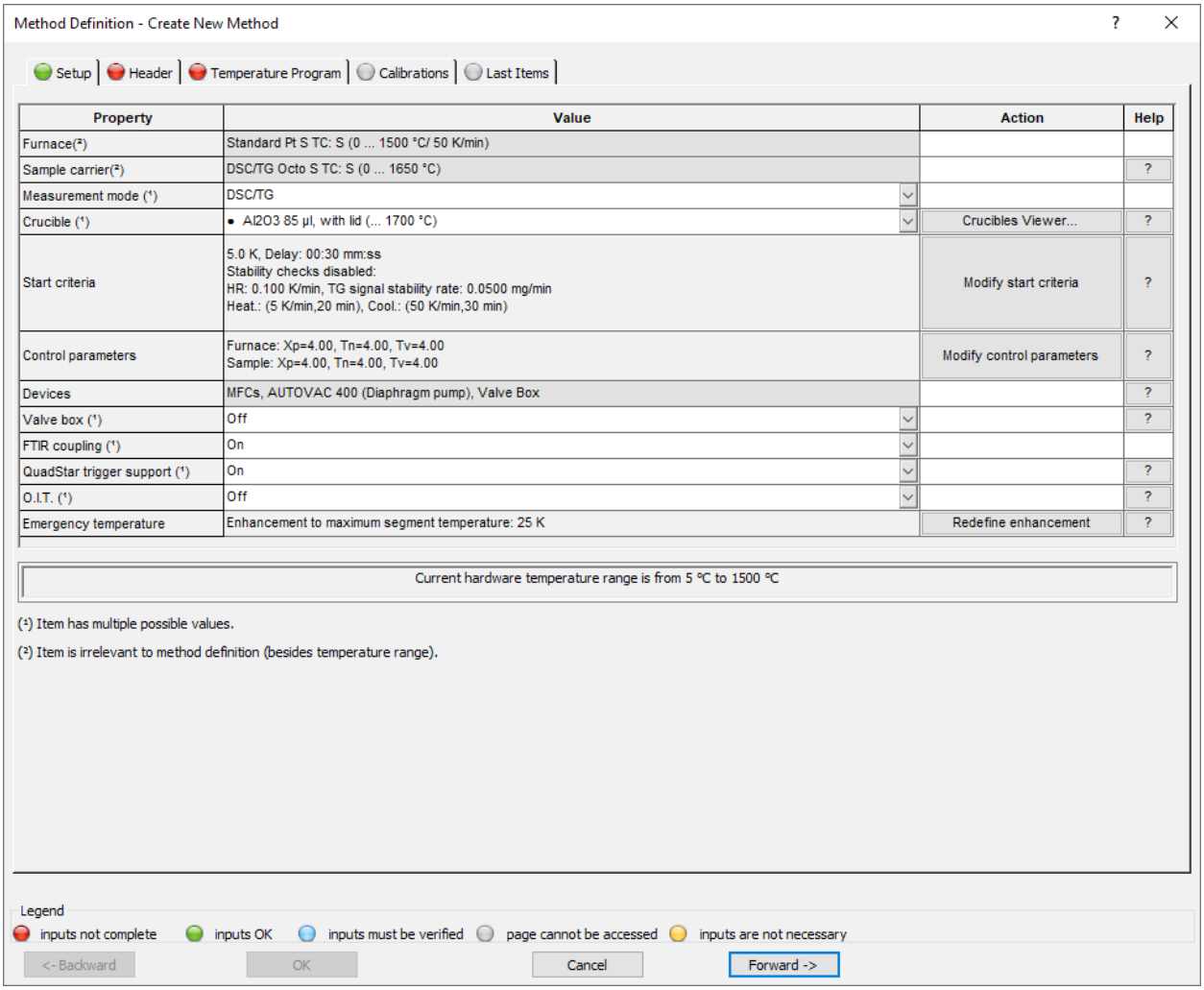
Using the STA449F1 program

This program controls all functions of the TGA/DSC and contains the autosampler mode once an appropriate method for your samples is programmed. A good first step is to double check the status of the furnace to guarantee proper operation.

Status of sample or reference on the sample carrier: If there is any doubt about the current status of samples and/or references loaded on the sample carrier inside the furnace, use the *ASC Diagnosis* icon on the ribbon bar to open the ASC Test view. Use the appropriate commands on this view to lift the furnace and determine if the actual sample carrier state matches what the program believes is the state. Use the program commands to fix any inconsistencies. Do NOT add or remove crucibles manually from the sample carrier! If anything seems out of the ordinary, document it and notify CIF staff.

Generating a Method

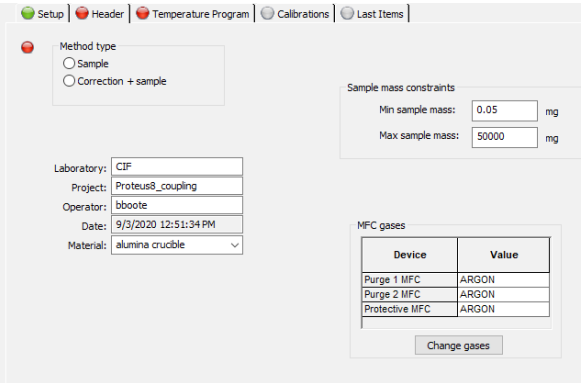
New to Proteus 8 is the use of **method** files, which is a way to program a furnace temperature profile, gas flows, and attached instrumentation much like the first steps of building a macro in Proteus 6. In this way, you can construct a method, then apply that method to as many samples as you like, including coming back and running similar samples at a later date. When you launch *Method→Create new Method…* you will see a window like this:



Here select the crucible type, and whether to collect FTIR and MS (Quadstar trigger support) with your run. The rest of the settings should be left as they are.

WARNING!!! Selection of the appropriate crucible type is critical to safe operation of the instrument. You will be held accountable for damage caused by wrong choices!!!

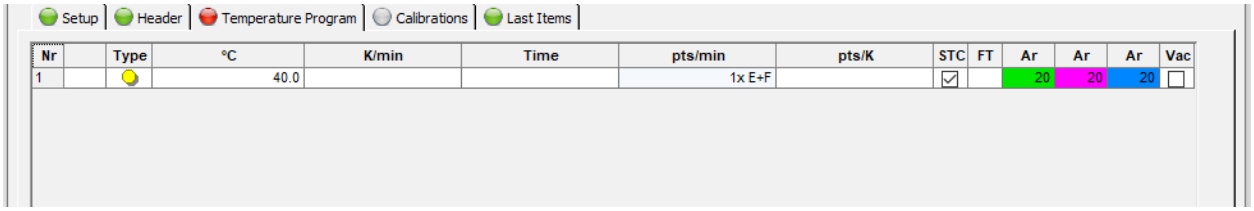
Advancing forward to the *Header* tab you'll be able to enter your lab, project, and material, as well as whether to include a correction (blank crucible run) and constraints on the sample mass if you'd like.



Select *Forward* to move to the *Temperature Program* tab. Create a temperature program with multiple steps, starting with *Initial*, then *Dynamic* and/or *Isothermal*, and ending with *Final*. When creating the *Initial* step, you will also be allowed to select purge flows, and the use of the autovac controller. *NOTE: When idle, the furnace seems to rest at about 38°C; you cannot start the program much lower than that.*

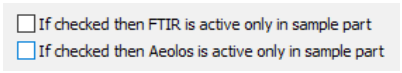
> *Performing an evacuate and fill cycle is recommended any time the protective purge gas is changed during the configuration in order to prevent leakage of the former purge gas into the furnace.*

Press *Add* when you have the correct settings selected.

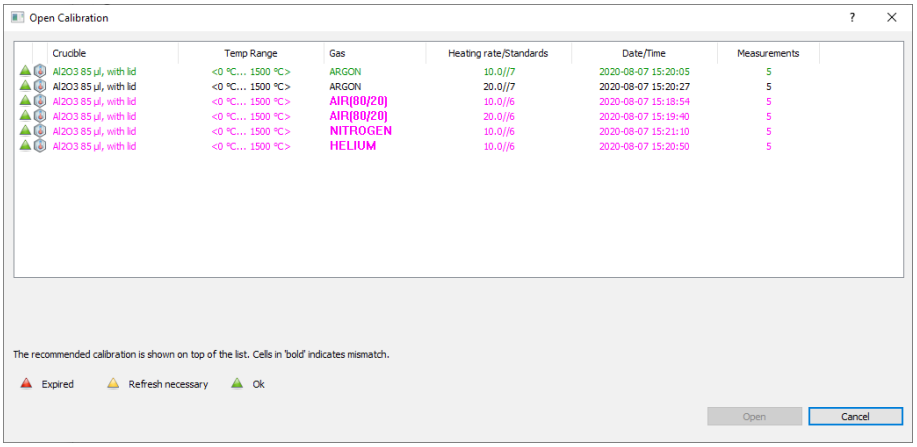


If you wish to obtain FTIR data, check the FTIR box for all the dynamic and isothermal segments of your program. Add a *Final* segment to the program with an emergency temperature of ~25 degrees above your end temperature. If you make a mistake, select and edit the segment, then press *Update Current Step*. It is not necessary to add a *Final Standby* step.

NOTE: At the lower part of the window there are options to collect FTIR and Aeolos data "only in sample part"--do **not** check these boxes as the connection to the other instruments will not function correctly.



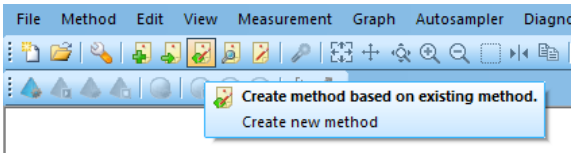
Select *Forward* to advance to the *Calibrations* tab. Here you will select temperature and Heat flow calibrations that best match your purge gas mixture. From *Select…* in the temperature calibration area, the following view comes up:



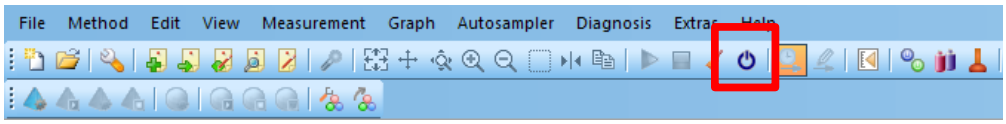
Here you can see the system tries to select the best calibration for your selected crucible type, gases and heating rates (in this case alumina, argon, and 10K/min). Select the calibration for both temp and heat flow (DSC scaling), and disregard the Tau-R calibration. Please contact Brett if you need a calibration not shown in the options (i.e. aluminum crucibles, non-standard heating rates).

Once you advance to *Last Items* and hit *Okay*, give your method a name. I would prefer you to use your 3 letter prefix and date, but you may add content to the name such as a sample type or hints about gases/etc (i.e. *bwb-09012020-oxalate-Ar-10K* or something similar). For now, save the methods in the default *Methods* folder.

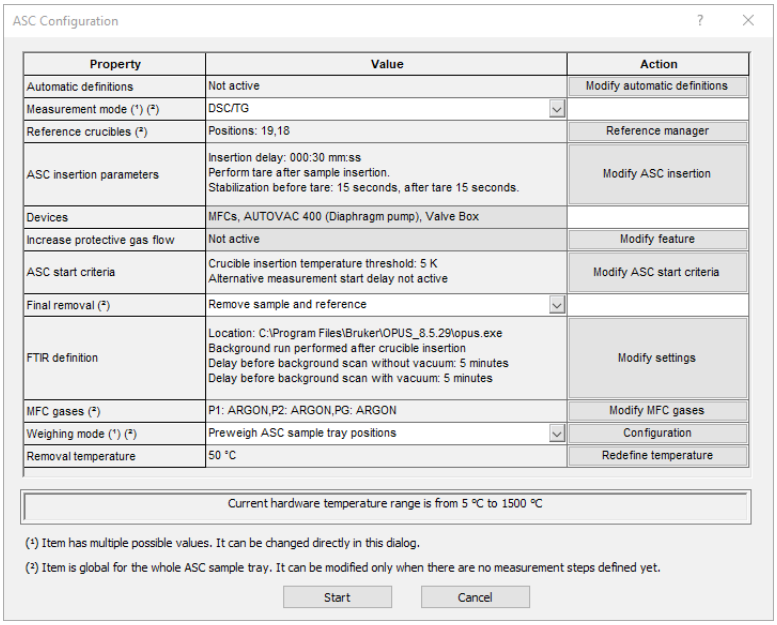
Once methods are entered, new methods can be created starting with a previous method as a template using *Create Method based on Existing* or *Modify method* options in the top ribbon.



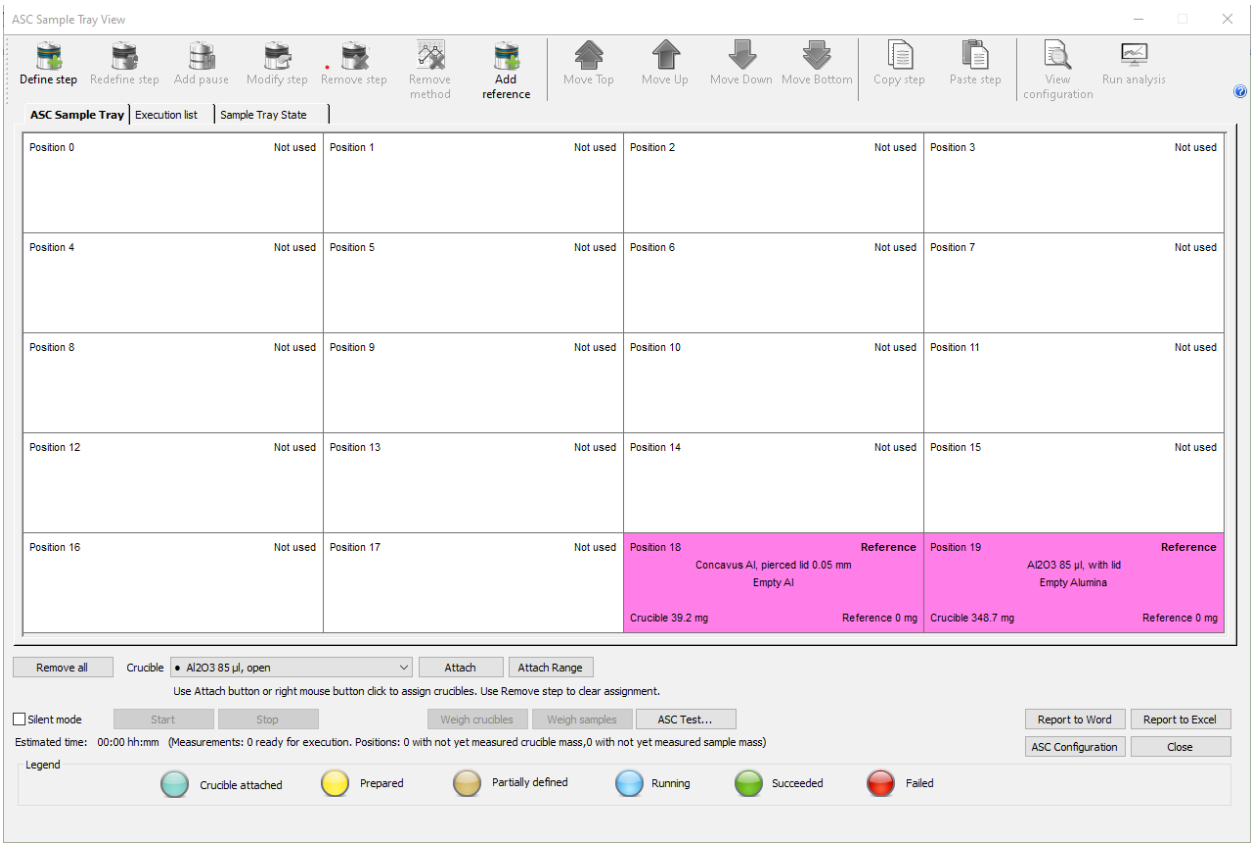
With a method defined, we can begin setup for *Autosampler mode*, which takes the place of the separate autosampler program we used to set up for Proteus 6. Before autosampler mode can be enabled, the instrument *Setpoint* needs to be turned on. This can be done from the top ribbon, under *Help*. *Setpoint* basically is an active standby mode, where the furnace proceeds to a set temperature during any down time during or after sample runs.



With *Setpoint* enabled, we can start Autosampler mode. Do so from the *Autosampler* menu entry, which brings up a dialog box for the ASC configuration:



None of these entries need to be changed unless you plan to manually weigh your crucibles and samples to save time. If so, change the weighing mode to *manual input*. Once you are finished, press *Start* to bring up an image of the autosampler tray and another window showing step definitions based on ASC positions. Now we can use the method file we wrote earlier and input steps for our samples. You will see a reference aluminum and alumina crucible in positions 18 and 19, with defined masses. Everyone can use these empty references since they are pre-measured. The step definition view looks like this:



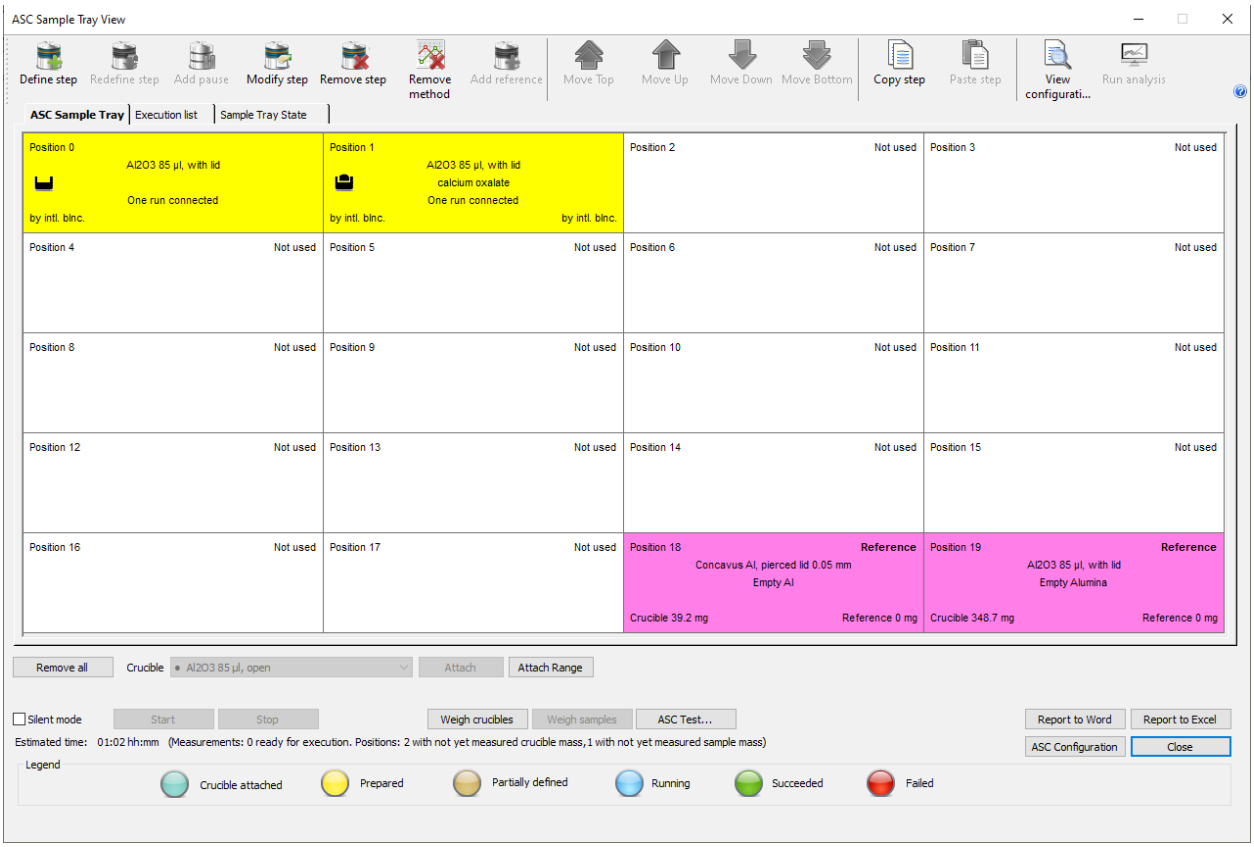
Select *Position 0* and the *Define Step* option will become active. Select *Define Step* and a list of available methods will come up, with suitable methods shown in green. A method is deemed suitable if the gases are correct and a reference is available for the crucible type.

Choosing your method will open the *Measurement Definition* window, where you will select *Correction* since the first run is typically the blank. Then fill in the sample *Identity* and *Name* blocks, as well as a *Filename*. Remember the filename conventions, which are your three letter code followed by the date, followed by the autosampler position (i.e. *bwb-01072020-0*) You will also direct the program to your *D:\DATA\...* folder during this step.

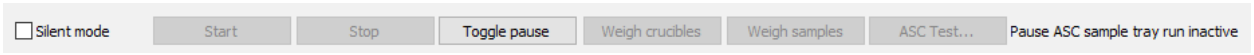
NOTE: In the event you are manually entering masses, they would also be entered on this step.

Once these fields are entered, click *Add* to send the step to the Sample Tray view. You'll see the first position turn yellow, indicating a run has been prepared there. Now you may add subsequent steps from Position 1 onward, using the same method file and giving new names to each sample you add.

Here is an example ASC tray, showing a correction and a sample run.

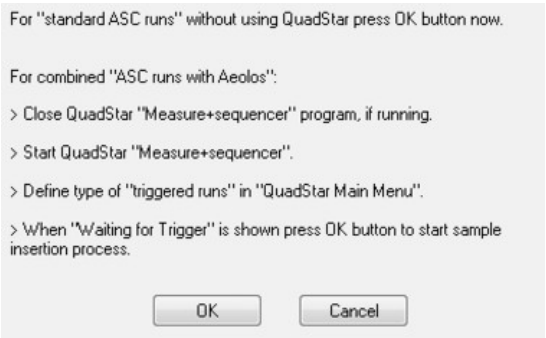


Once your samples are all assigned to positions, select *Weigh crucibles* to measure dry masses of each crucible. As before, the system will prompt you to check the furnace status prior to taking the measurement. If you have previously verified the furnace, continue with *OK*.



The system will proceed to weigh the crucibles empty. When this step completes, fill the crucibles with your samples and select *Weigh samples*. During the sample weighing step is a good time to fill the FTIR detector with LN_2 while you wait, so the detector is cool by the time the run starts.

Once this step is complete, the run is ready to begin. Launch and prepare OPUS if you are collecting FTIR data. Press the *Start* button, and if Quadstar support (MS data) is selected, you'll be presented with this window.



STOP!!! Do not immediately select OK!!!

> *NOTE: This is the only point at which you should start the Aeolos "**Measure + Sequencer**" program if you wish to acquire mass spec data. Also start "**OPUS**" for FTIR data collection, if not started earlier.*

For detailed help setting up the FTIR and MS data collections, see their respective sections at the end of this document.

Monitoring the Progress of the Sequence

After selecting *OK*, the system will insert the relevant reference crucible and the first sample/ blank. No action is necessary; the process is automatic from this point onward. If Evac&Fill cycles were selected, you will also see messages associated with those functions.

Eventually, Proteus will try to connect with OPUS and various messages will flash. Within a minute or two the OPUS *Chrom* real-time display should open. All the while, the Aeolos view will still be flashing *"Wait for start signal from the instrument"*. Finally, the mass spec measurement should also start.

You will observe mass spectra accumulating in the Quadstar, FTIR data accumulating in OPUS, and DSC/TGA data accumulating in the Proteus *STA449F1* view. Keep in mind the MS signal of analytes is quite low compared to the purge gas; it is normal to only see purge gas signals from this view.

Congratulations! You have begun acquiring your TGA/DSC-IR/MS data set.

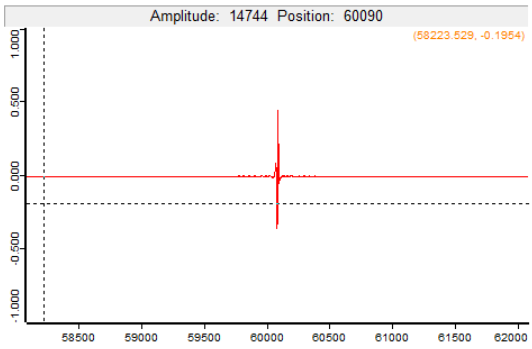
Preparing for IR Measurements

Fill the TGA-IR dewar with liquid nitrogen, if not done so earlier. Hold time is about 4.5 hours.

Use the Windows 10 tool that minimizes programs and shows the desktop (or WinKey + D). It is the small icon always located at the far right of the notification taskbar. Then start OPUS. This will bring up the OPUS Login view.

Log in as *user= Administrator*; *password= OPUS*; *workspace = Tensor37_FullAccess*. Respond OK to the license validation prompt. The main OPUS view will open. On the "*Basic"* parameters tab, choose *Load -> TGA.XPM*. .

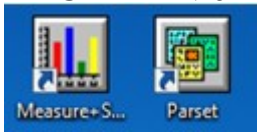
Select the "Check Signal" tab. You should see an interferogram with amplitude of at least 16000 counts.



Then from the Basic tab, select *Accept & Exit*. No further action is necessary. You may resize, reposition, or minimize OPUS, but do not close the program!

Preparing for Mass Spec Measurements

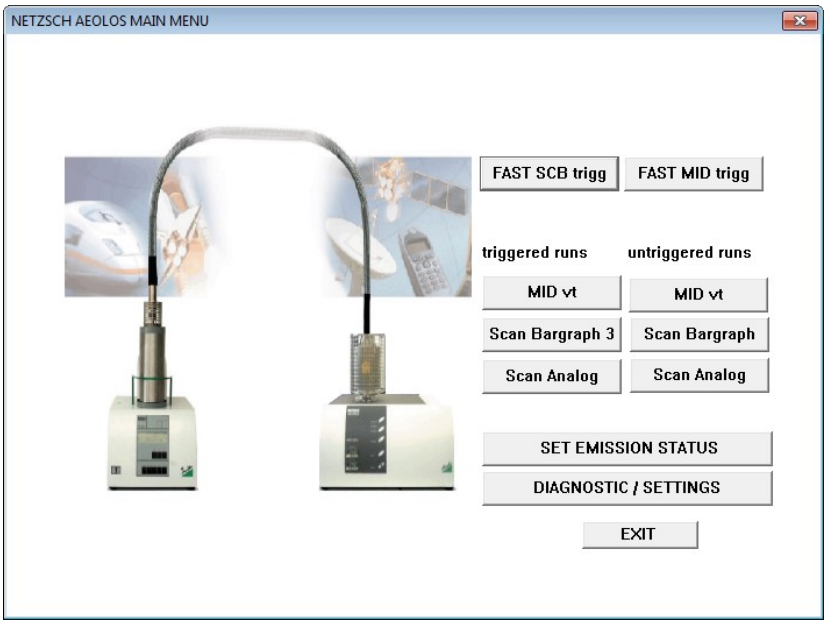
Press **Windows Key+D** to minimize programs and show the desktop. The two mass spec programs you will use are *Measure+Sequencer* and *Parset*. If you need standard conditions (m/z range 10-200), you may use *bwb-001.sbp*.



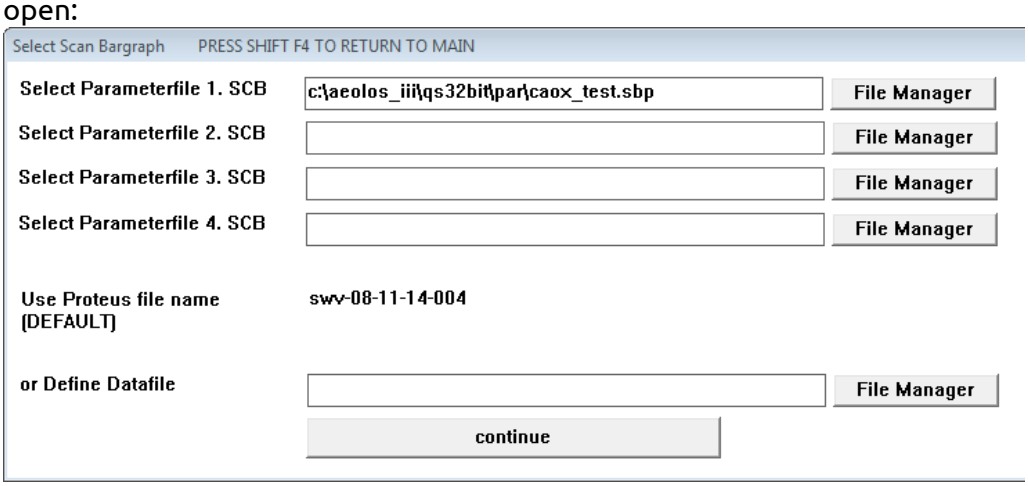
Open *Parset* if you need to create or modify the mass spec parameters you wish to use, or if you need to check which of your parameter sets is appropriate for the measurements you are about to do. You will need to browse and select the proper parameter set as part of running *Measure+Sequencer*, but you won't be able to view the contents at that time. Your parameter sets must start with your assigned three initials and a dash (e.g. *bwb-001*). Most people start investigations using "*Scan Bargraph*" parameter sets, but you should understand the advantages of eventually using "*MID*" parameter sets.

With a suitable parameters file, open *Measure+Sequencer*. The TurboPumpControl view will flash briefly as the program turns on the ion gauge and the filament. Next, a table will appear; it will be filled within 10-20 seconds with vacuum readings.

Within one minute the Aeolos Main Menu view will appear, with a picture of the instrument.



Normally you will choose *Scan Bargraph 3* from the triggered run selections. A dialog view will open:



You must use the File Manager button associated with "*Select Parameterfile 1.SCB*" to select your parameter set (e.g. *bwb-001*). Note that the "*Use Proteus file name*" field should be pointing to the exact filename you specified for the first sample when you created your macro (this should happen automatically). If done correctly, the Proteus (TGA/DSC), OPUS (FTIR) and Aeolos (MS) filenames will all be the same. The Proteus and OPUS files will save in your user folder on the D drive, *D:\data\<username>*. The Aeolos files will always save in *D:\data*

mass_spec_data. You should move the mass spec data into your user folder after the run is complete.

NOTE: Do NOT use the "Define Datafile" feature!!

Select *continue*. You should now see a message flashing at the bottom-left of the program view alerting you that the QuadStar MSD is waiting for a remote trigger.

Both OPUS and Aeolos are now ready to communicate properly with the ASC macro you are attempting to run. You may now select OK from the *ASC Measurement with Quadstar* dialog.