

Netzsch TGA/DSC – IR/MS Operating Instructions

Revised 08/12/2015 S.V.

Location: 1238 Hach Hall
Contact: Steve Veysey, 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



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Additional Resources

| | |
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| Basic Data Processing Tutorial | <i>(07/03/14)</i> |
| Acquiring Evolved Gas Data with DSC/TGA Measurement | <i>(revised 08/12/15)</i> |
| Using OPUS to Process Evolved Gas IR Data | <i>(revised 08/12/15)</i> |
| Using Proteus and Dispsav to Process Evolved Gas MS Data | <i>(revised 08/12/15)</i> |
| Creating Instrument Calibrations | <i>(revised 08/12/15)</i> |

Introduction

The Netzsch STA449F1 TGA/DSC system simultaneously provides TGA data (Thermo Gravimetric Analysis) and DSC data (Differential Scanning Calorimetry) in one analysis. 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.

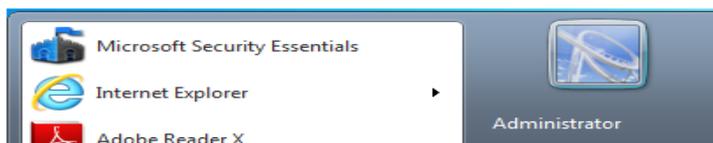
This guide will introduce you to the Netzsch STA449F1 hybrid TGA/DSC/IR/MS system. It represents a consolidation of the training materials written in 2014. Updated versions of those original documents are attached as Appendices.

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's not the case, then you should NOT proceed with training. First, learn the fundamentals of these measurement techniques and their application to your particular research project. We do not provide application assistance for this instrument.

The Desktop

The computer should always be logged on as Administrator. Do NOT logoff when you are finished.



The program section of the Taskbar should only have Word and Snag-It present. Close other programs before beginning your session.



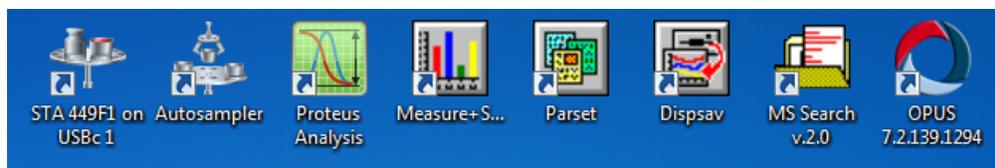
The notification section of the Taskbar should show the mass spec icon. When you mouse-over the icon it should display the message: "QMV200 connected".



The four software packages involved in acquiring and processing TGA/DSC-IR/MS data are:

- Proteus -DSC/TGA control, acquisition and processing
- Aeolos -MS control, acquisition and processing
- NIST -MS library and search software
- OPUS -FTIR control, acquisition and processing

Icons for the most commonly used programs within these packages are on the desktop:



Directories and File Structure

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

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

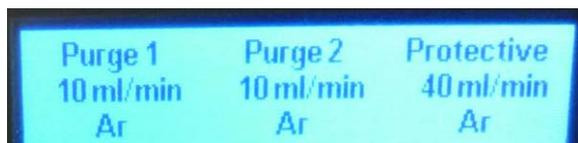
All data files must be stored in this location. Also, you were assigned a three letter code such as *<swv->*. This will normally be the initials for your first, middle, and last names, followed by a dash. All data files, method files, parameter files, and macros must be named in this manner. I **STRONGLY** suggest that you append a date code to these filenames using the format *<mmddyy>*. For example, *<swv-060115>*. For data files, I **STRONGLY** suggest that you also add the Autosampler position to the filename. For example, *<swv-060115-1>* would be the correct filename for the sample in position “1” of the carousel. The “0” position is reserved for baselines acquired with an empty crucible. That filename would be *<swv-060115-0>*.

As part of creating a macro to run your samples, you will direct the TGA/DSC (Proteus) data to your folder. The FTIR (OPUS) data will follow automatically. The Aeolos (mass spec) 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. Use a simple Windows 7 Search to find it. Notify me of the problem.

At the Beginning...

You must verify that the initial state of the hardware and Desktop is correct. You must start a Word document with the current date, saved in the *D:\Errors* folder. Snag-It must be running.

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 5psi. Check the instrument front panel. It must show the same gases and ports as currently plumbed. If not, corrective action will need to be taken from within the STA449F1 program **BEFORE** you begin to write your macro to acquire data.



Front Panel View

Software: The only Desktop programs running should be **Word** and **Snag-It**. Open **Word** and start a new document with the current date. Immediately “Save As” *D:\Errors*<yyyy-mm-dd>**. For every occurrence of an error, questionable system response, or other software issue, use *<Print Screen>* to invoke **Snag-It**. Paste the captured region into the date-coded **Word** document. If you do not do this, I will not be able to assist you with difficulties you might be having.

Mass Spec state: (1) Mouse-over the MS icon in the taskbar notification area. It must say “QMV200 connected”; if not, please contact me. (In this situation you can acquire DSC/TGA data and IR data, but not mass-spec data.) (2) Be sure you have a proper parameter set for acquiring the mass spec data. Use the PARSET program if you need to create or modify a MS parameter set. PARSET can also be used to browse the contents of your parameter sets so that you will be able to select the correct one when the *acquire-data* macro calls for a selection. Close PARSET before proceeding.

FTIR State: OPUS may be started at any time prior to the execution of the macro. Add liquid nitrogen (LN2) to the Tensor MCT detector. Use the desktop icon to start the OPUS program. Log in as *user= Administrator; password= OPUS; workspace = midIR_FullAccess*. Respond OK to the license validation prompt. The main OPUS view will open.

- Choose *Measure -> Chromatography*.
- Load the TGA.XPM parameter set. Ignore warnings about validation, but if a “*detector not ready*” warning is present, it usually means you haven’t filled the MCT dewar with LN2.
- Select the *Check Signal* tab. Within a few seconds you should see an interferogram; amplitude >14,000.
- Select “*Accept and Exit*”. Do NOT close the program, but you can reposition or resize the window as necessary.

OPUS is now ready



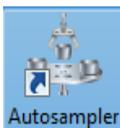
Using STA449F1 program

This program controls all functions of the DSC/TGA and Autosampler. It will eventually be called by the macro you execute to acquire data on your samples. Usually you do not need to enter this program prior to writing or executing your macro, but there are two situations that may require action before writing your macro. You can also resolve occasional instrument configuration issues using this program.

Hardware panel display doesn’t match gases plumbed: The gases actually connected to the P1, P2 and Purge ports on the back of the instrument must match those displayed on the front panel. The gas flows displayed must have numerical values for each port that will be used. From the ribbon bar, select the *AFC-Extended* icon. 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 40 mls/min; P1 and P2 are often set to 10 mls/min each, for a total of 20. Upon exiting the *AFC-Extended* view the gases and flows displayed on the front panel should match those just selected.

Status of sample or reference on the sample carrier is unknown: 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!

Resolving instrument configuration issues: Generally, all aspects of the instrument configuration can be set while creating your macro. However, if any difficulties or inconsistencies arise in the macro program, use <File -> Instrument Setup...> in the STA449F1 program. Check that all of the settings on the Setup Tab make sense; make them consistent with the setup choices you are trying to make in the Autosampler macro program. *NOTE: This step should NOT be necessary unless a problem arises.*

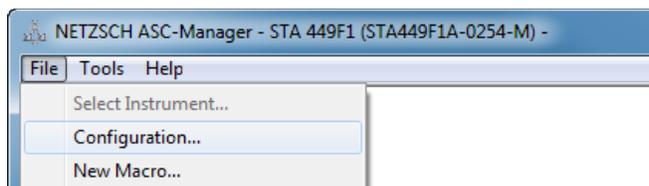


Writing and Executing a Macro

The Autosampler program should be used to set up and acquire all data on all samples. These samples could be for baseline correction (empty crucible), furnace calibrations (a series of standards with known melt/freeze characteristics), or your research samples. Program setup is similar in all three cases.

Configuration

After entering *Autosampler* (identified as NETZSCH ASC Manager), select *File -> Configuration*. This will allow you to make reasonable choices for global parameter settings to be used for all of the samples you intend to put in the macro sequence.



Modifications and/or choices for each property are accessed via drop-downs in the Value fields, or by using the more involved dialog buttons in the right column of the table. Only those properties that might require modification are discussed below.

| Property | Value | |
|--------------------------|---|------------------------------|
| Automatic definitions | Not active | Modify automatic definitions |
| Furnace | Standard Pt S TC: S | |
| Sample carrier | DSC/TG Octo S TC: S | |
| Measurement mode (*) (°) | DSC/TG | |
| Crucible (*) | DSC/TG pan Al2O3 (... 1700 °C) | Help on crucible selection |
| Reference crucible (*) | 19: DSC/TG pan Al2O3 | Reference manager |
| Start criteria | 7.5 K, Heat.: (20 K/min,30 min), Cool.: (50 K/min,300 min) | Modify start criteria |
| ASC start conditions | Standard start: Insertion: +10 K, delay: 001:00 mm:ss. Standby start: Threshold: 10 K, start delay: 01:00 mm:ss. Additional delay time in adjustment: 30 seconds. | Modify ASC start conditions |

Measurement mode: Always leave as DSC/TG.

Crucible: Select the correct crucible type! At the moment we have alumina (Al₂O₃), aluminum, platinum, and platinum-rhodium with Al₂O₃ lining. Use the “Help on crucible selection” button to access compatibility information.

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!!!

Reference crucible: Must be set to match the sample crucible. Select from the drop-down. *NOTE: If there is a mismatch, the software may display a warning and request permission to match crucibles. Verify, and allow it to make the correct choice.*

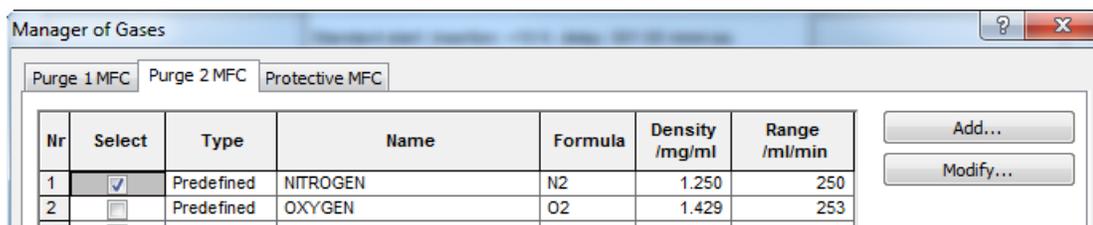
Special instrument control: Select FTIR from the pull-down. This simply defines the ability of the software to communicate with attached instruments and reserves your right to choose while *writing* the macro program.

QuadStar trigger support: Leave ON. This defines the ability of the software to communicate with the mass spectrometer and reserves your right to choose later during *execution* of the macro.

STC: Leave ON unless acquiring standard samples for furnace temp/sens calibrations.

TC calibration (20 C): Leave ON. I don't recall what this actually does.

MFC gases: If these do not match the gases and ports you plumbed and then verified on the front panel display (possibly using *MFC-Extended* in *STA449F1 Program* to resolve conflicts), you must make those corrections now using the *Modify MFC gases* dialog button. You will not be allowed to change the selection of ports or gases later in the macro development. *NOTE: Actual gas flows are set later in the macro.*

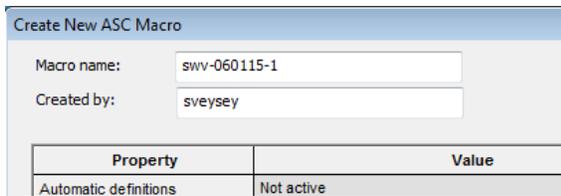


Weighing mode: Typically left as “Pre-weighing of all magazine positions”. This means it will use the internal balance.

Removal temp: Defines the cool-down temperature before the furnace will open and the robot will remove the crucible. Normal guidance is to set it 10-15 degrees above the start temperature for your samples.

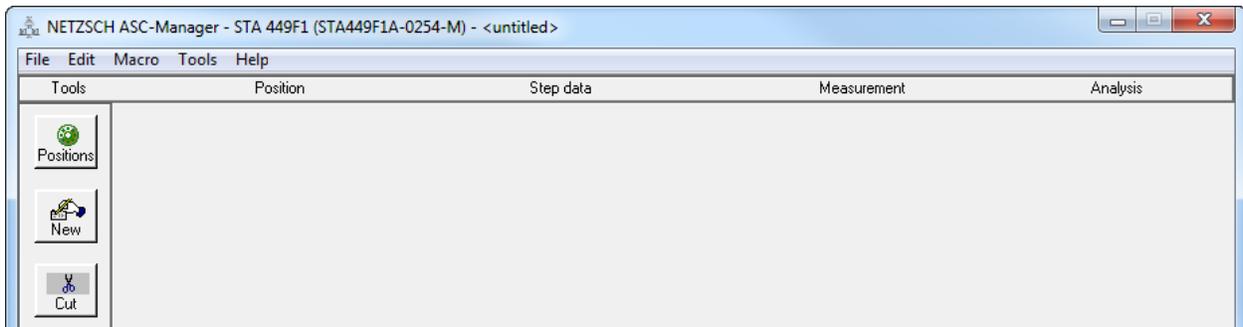
Create New Macro

Select **New Macro** from the **File** pull-down. This will open the now-familiar Configuration dialog, and allow you to name the Macro. “Owner” must always be your username. Macro name must be your assigned three-letter code with *mmdyy* appended, and possibly an incremental number if you write more than one macro on the same day: e.g. *swv-060115-1*.



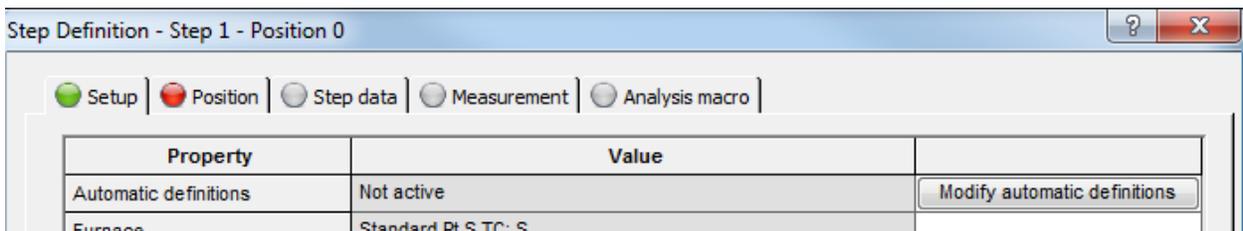
Verify and/or modify any of the properties in the configuration, then select OK.

After selecting OK, the macro editor page will open. This page is used to create the specific experiment configuration and program for each position in the autosampler.

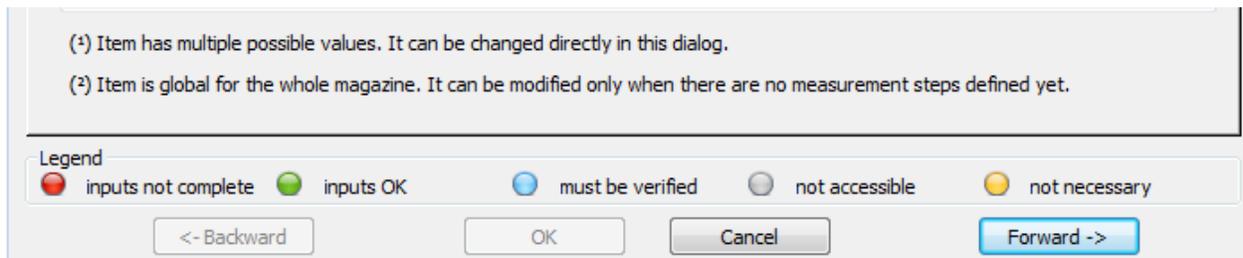


Do not use the *Positions* button; it is not helpful in most cases. Instead, start by selecting the *New*

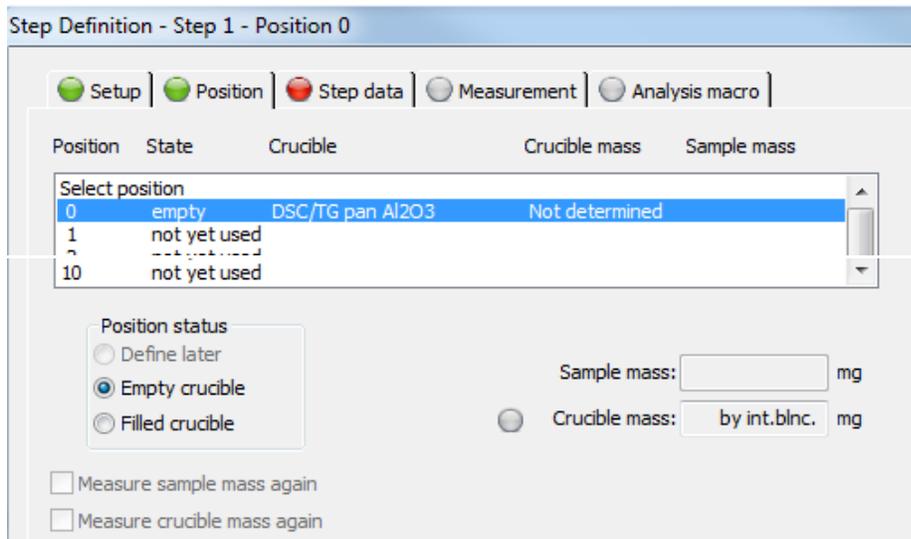
button  to begin the **Step definition**. Note the various tabs.



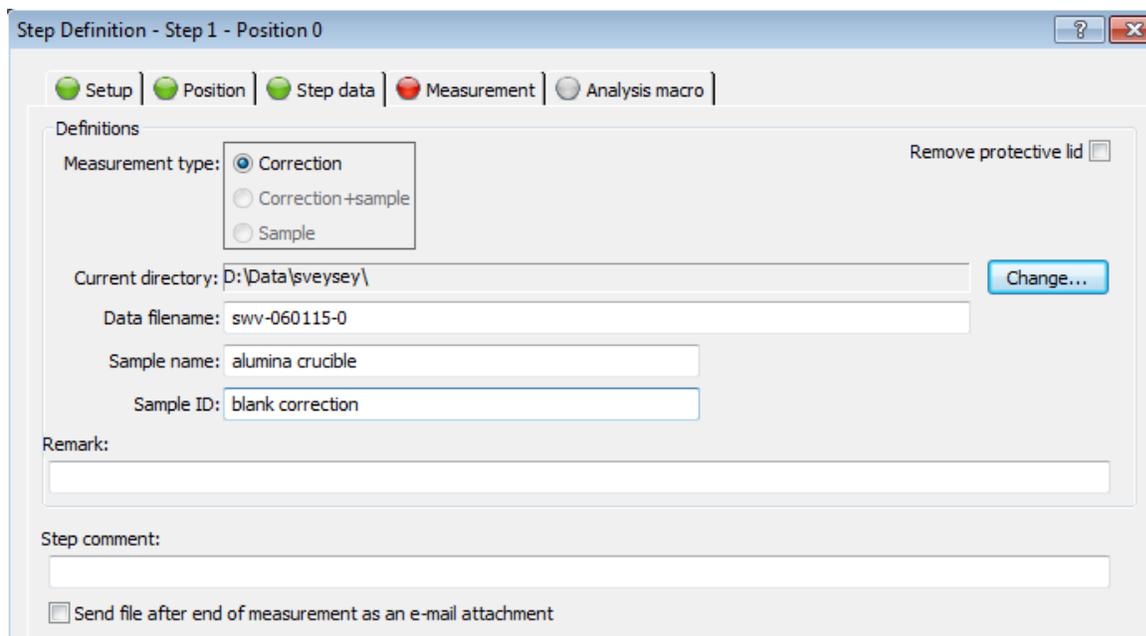
Carefully check all of the items on the Setup tab. Note that most of them cannot be changed when you are this far into creating the macro. You would need to start over. Only when a tab is green (all required entries have been made) can you move “Forward” to the next tab.



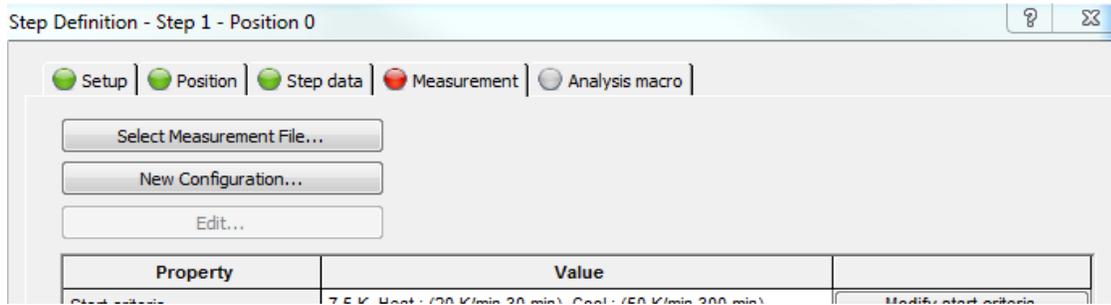
For position 0, select **Empty crucible** to run a baseline correction sample. This is highly recommended, but not essential. It is possible to create a sample definition that uses a pre-existing baseline correction file, and it is possible to acquire a baseline correction file later and then apply it to the data during processing. Both of those choices require some advanced skill with the software. Consult with me.



Advance to the **Step data** tab. For position 0 already defined as an empty crucible, the software grays out choices other than *Correction*. In this example, the correction algorithm will measure the empty crucibles in position 0 as sample, and 19 as reference. Change the data path as necessary to your folder on the D drive <D:\Data\Sample name and *Sample Id* fields must contain text, but filling in *Remark* and *Step comment* is optional.

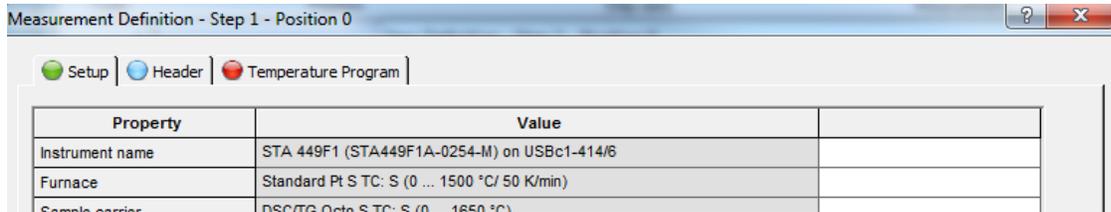


Advance to the **Measurement** tab.

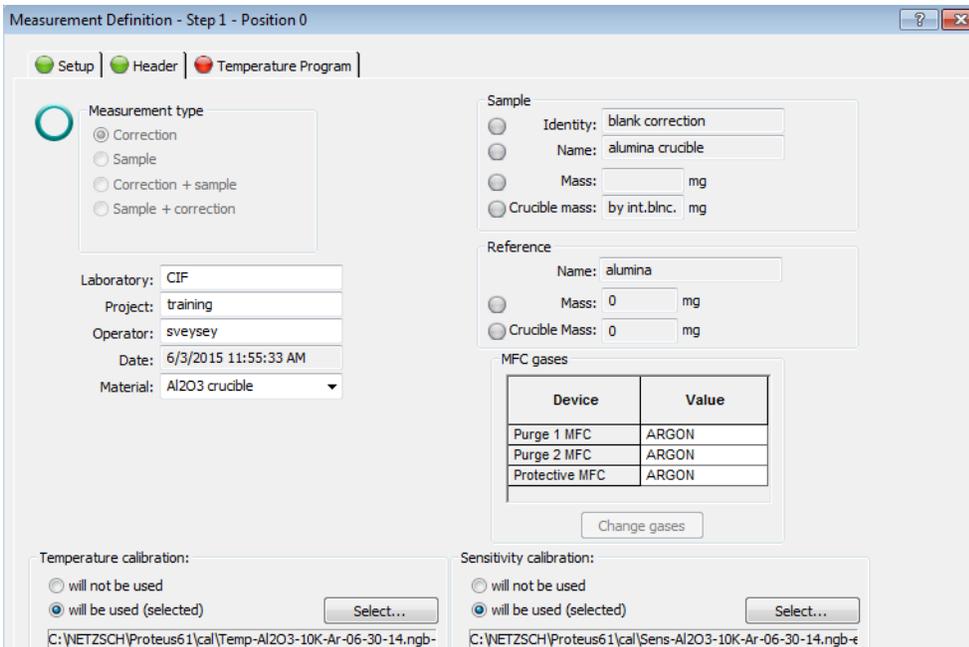


If you have previous data you wish to mirror, you can **Select Measurement File**, and the software will pull the appropriate parameters from the meta-data for that file. Be aware that you will not be able to change anything, and you might encounter a problem if you try to pull in measurement parameters that do not match the instrument definition selections you have just made in the creation of the macro.

When in doubt, select **New Configuration**. The Setup tab will again be displayed. Note that you cannot make changes now.

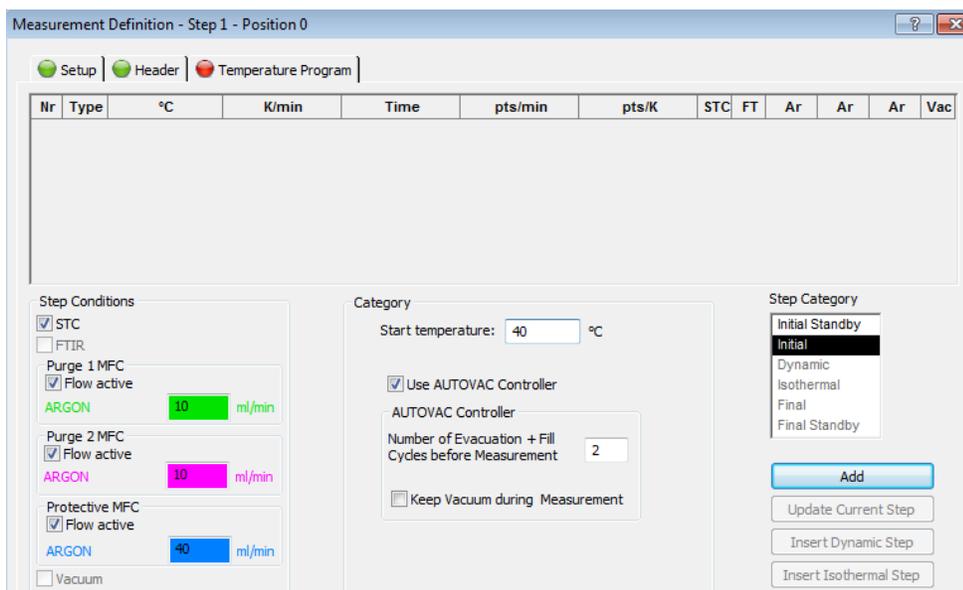


Select **Forward** to go to the **Header** tab.



Identify the Laboratory (your research group), and the Project. Select *Temperature* and *Sensitivity* calibrations corresponding to the (i) purge gas type, (ii) crucible type, and (iii) furnace program rate. If a calibration does not exist for your combination of crucible, temperature-rate, and purge gas, consult with me.

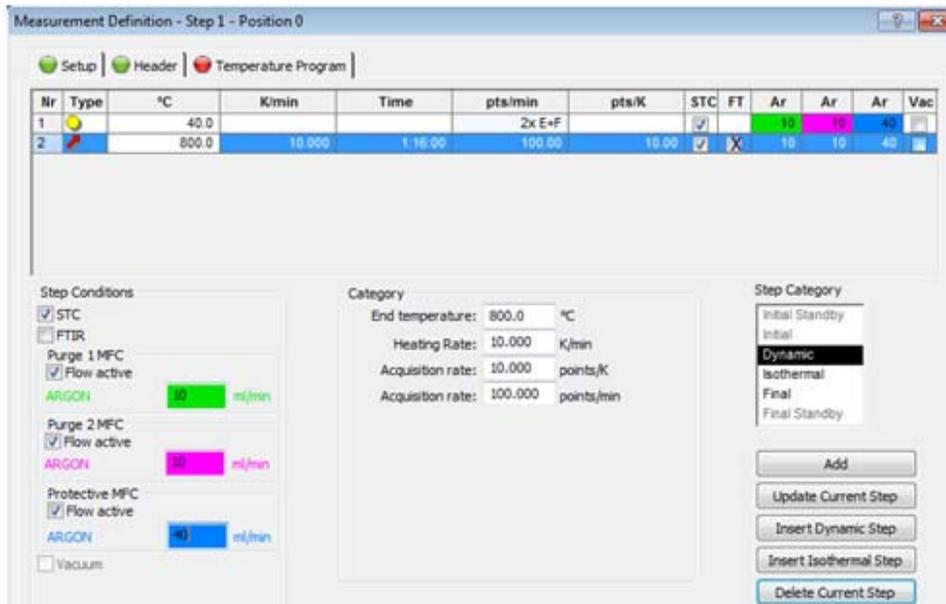
Select **Forward** to move to the **Temperature Program** tab. Create a temperature program with multiple steps, starting with “*Initial*”, then “*Dynamic*” and/or “*Isothermal*”, and ending with “*Final*”. When creating the “*Initial*” step, you will also be allowed to select purge flows, and the use of the autovac controller. *NOTE: When idle, the furnace seems to rest at about 40 C; you cannot start the program much lower than that.*



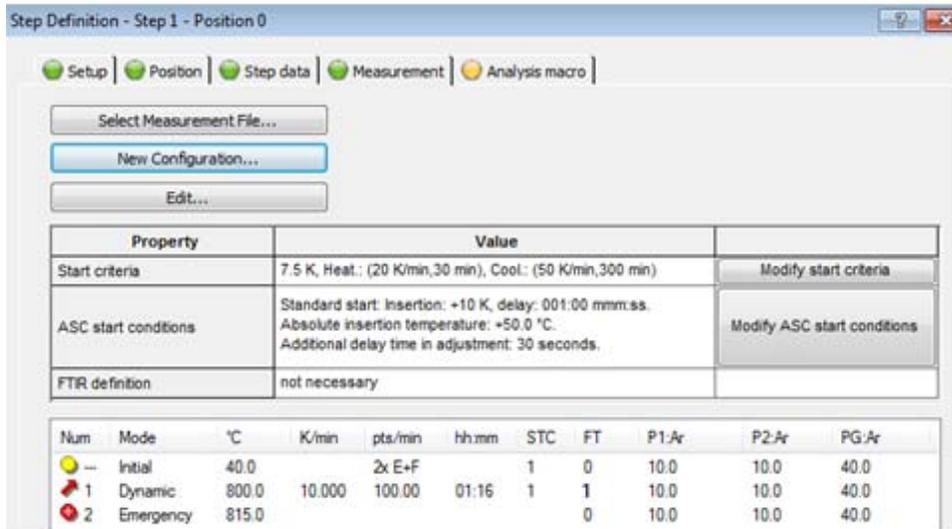
Press Add  when you have the correct settings selected.



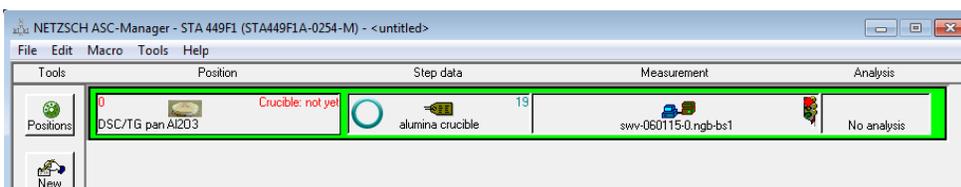
*NOTE: It can be beneficial to create an Isothermal step for 10-15 minutes at the initial temperature with a higher flow rate of PG, P1 and P2. This can be used in lieu of using “Evac and Fill” cycles. Consult me for the latest consensus advice. Then create and **Add** a Dynamic step with the proper purge gas and flows, an end temperature, and a program rate matching the Temp/Sens calibration files selected in the previous step. If you wished to obtain FTIR data, check the FTIR box for all of the dynamic and isothermal segments of your program. **Add** a Final segment to the program with an emergency temperature of 15-25 degrees above your end temperature. Use 15 for slower rates, and 25 for fast rates. 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, but you may include one for the last sample in your queue.*



Selecting **OK** will bring up a summary of the Step Definition you've created. Selecting **Forward** would advance to the optional Analysis Macro tab. Most people are not using analysis macros.



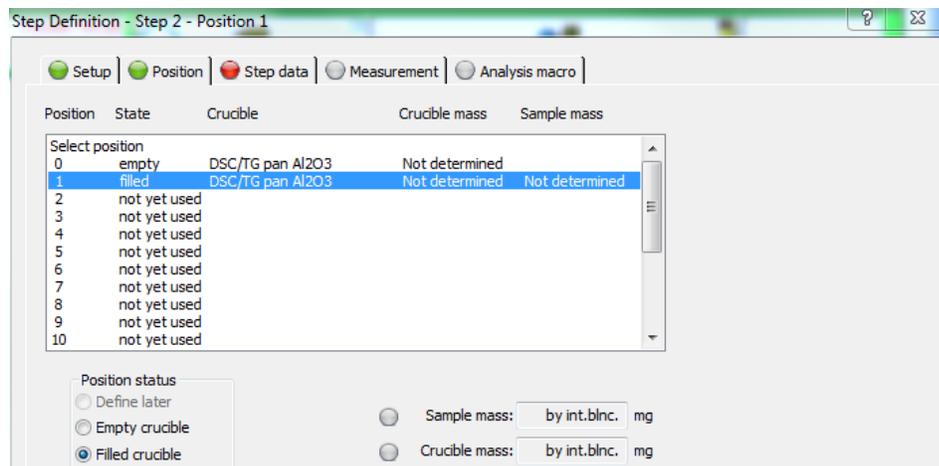
Select **OK** to complete this step definition for position 0 of the carousel. The ASC Manager will show:



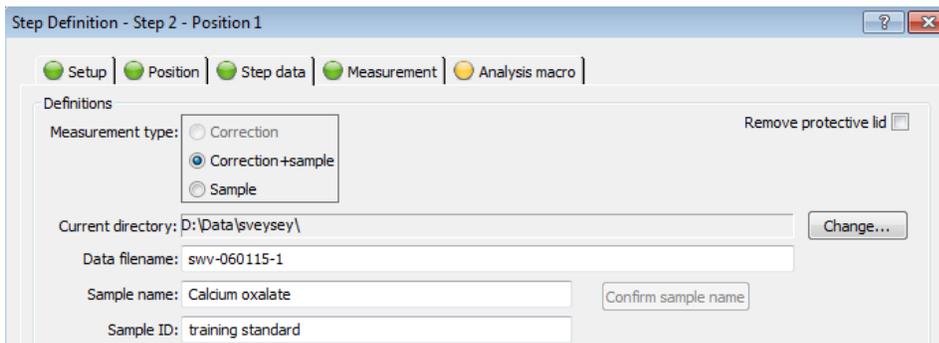
Use **Copy** and **Paste** to add the step for position 1, and then edit it appropriately.



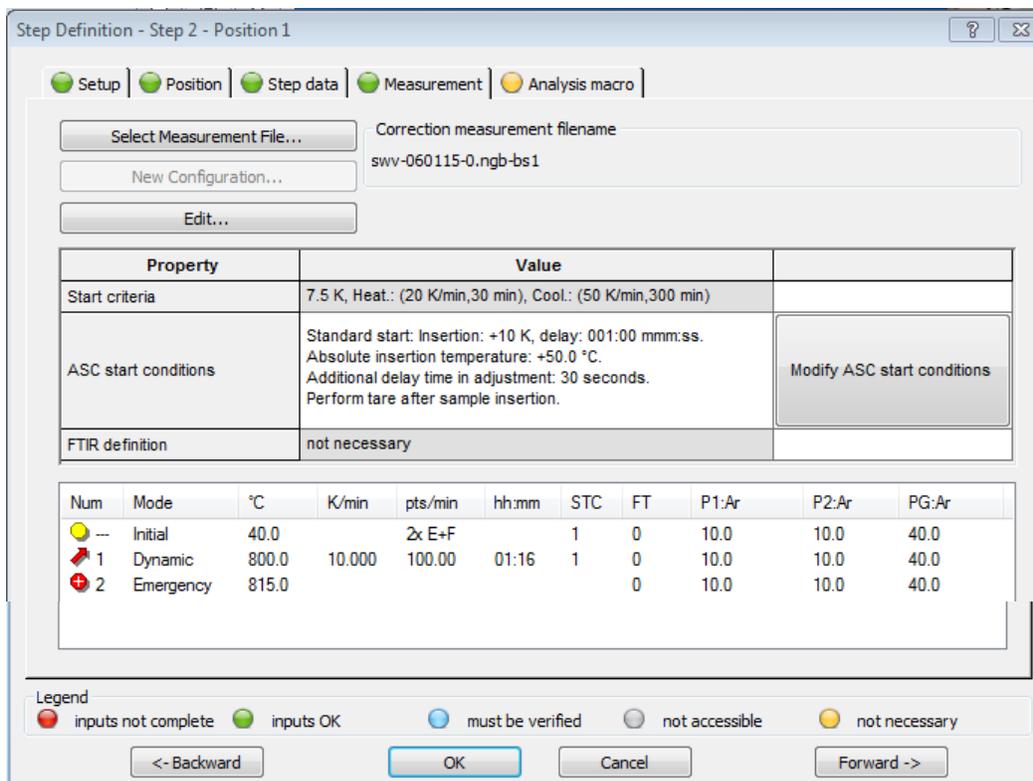
In the Step Definition dialog, select position 1. Set the position status as *filled crucible*. Sample mass and crucible mass should still show “*by int.blnc.*”



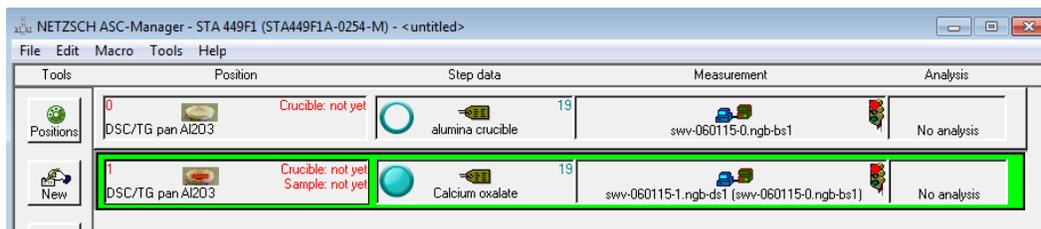
Select **Forward** and enter the Step data information for the sample in position 1, including *Data filename*, *Sample name*, and *Sample ID*.



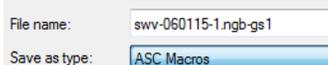
Select **Forward** to advance to the Measurement tab. The blank (Position 0) should be run exactly the same way as the remaining samples, so there should be no changes required here.



Select **OK** to add **Step 2** to the sample queue. Continue using **Copy** and **Paste**, followed by edits on the **Position** and **Step Data** tabs until there are entries in the queue for all of your samples.

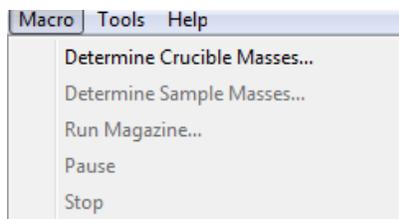


From the **File** pull-down, choose **Save**. The dialog box presented should show the macro name you assigned at the beginning.

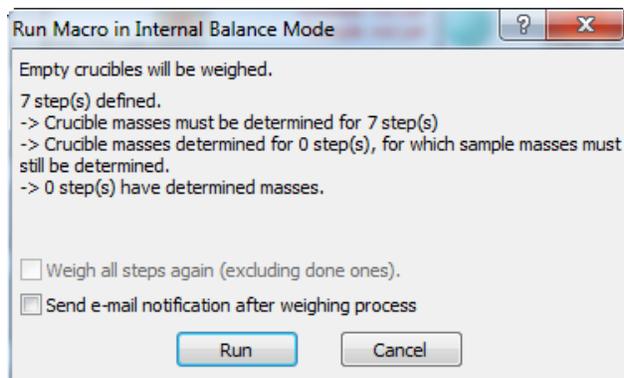


Running the New Macro

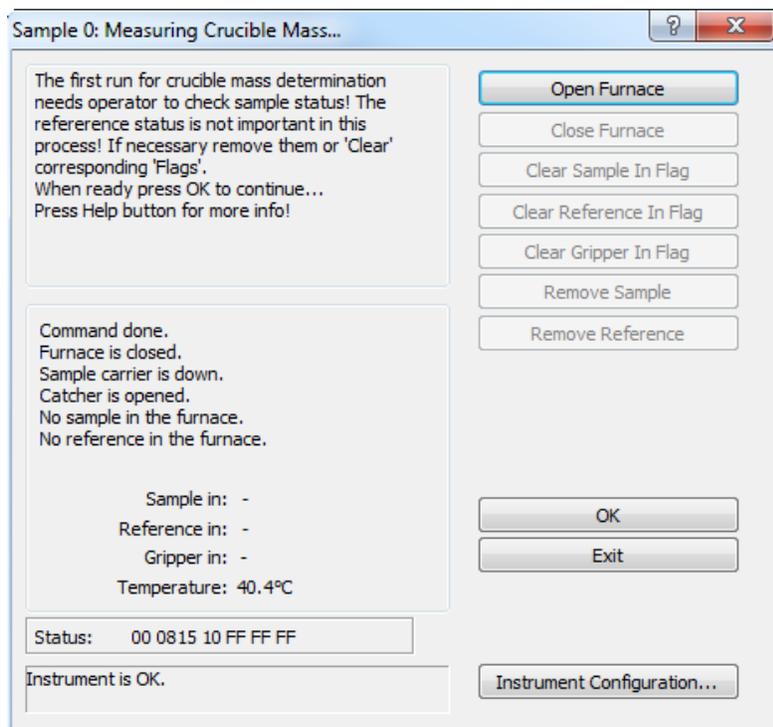
The Macro pull-down will show several choices. At this stage, all will be grayed out except “*Determine Crucible Masses...*” Once that task is completed, “*Determine Sample Masses...*” will be available. After that task, “*Run Magazine*” will be available.



Both of the weighing steps will open a dialog box similar to the one below. Note that “*e-mail notification*” is not enabled on our system at the moment.



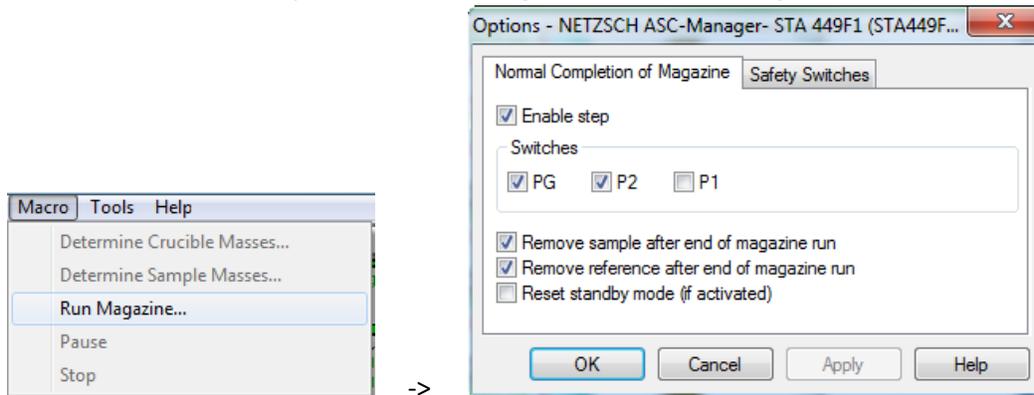
Each step in the macro execution (*Determine crucible masses, Determine sample masses, Run magazine*) will prompt you with a display similar to the one below to verify furnace status before proceeding.



Read the text on this dialog page carefully. If the previous instrument session completed correctly, no sample or reference will be on the sample carrier. If you have any doubt that the status reported on this page is correct, use the ASC command button *Open Furnace* to take a look. This will then activate the other ASC commands in the list so you can take necessary corrective action. Finish by using *Close Furnace*, then selecting OK.

After the empty crucibles have been weighed, put the appropriate amount of sample in each crucible and execute the *Determine Sample Masses* step of the macro. After each of the weighing steps the software will show the weights and ask if you want to print the weights or save as a PDF file. That's your choice.

When crucibles and samples have been weighed, choose *Run Magazine* from the Macro pull-down.

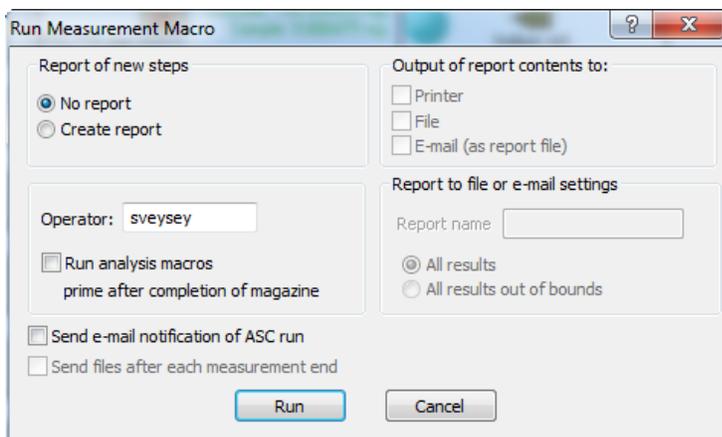


At this point the *Options* dialogue will be presented. This allows users to specify the purge state the instrument should be left in if the sample magazine runs correctly, or if it should fail or be interrupted. It should not be necessary to change these defaults. Just select OK.

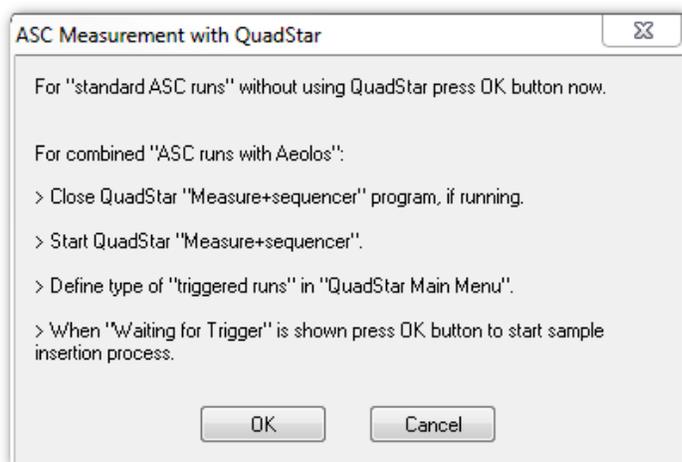
The *Show Macro State* dialog will be presented. This allows you to verify the samples in the magazine that will be run, and will also be useful if a run is interrupted and needs to be restarted later.

| Step | Position | Sample Name | Ref. Pos. | Crucible Mass /mg | Mass /mg |
|------|----------|-----------------|-----------|-------------------|-----------|
| 1 | 0 | Al2O3 crucibles | 19 | 159.118050 | ----- |
| 2 | 1 | Indium std | 19 | 156.456425 | 9.866475 |
| 3 | 2 | Tin std | 19 | 154.011600 | 8.543600 |
| 4 | 3 | Bismuth std | 19 | 155.262625 | 6.689900 |
| 5 | 4 | Zinc std | 19 | 156.550425 | 10.469825 |
| 6 | 5 | Aluminum std | 19 | 157.040925 | 10.215700 |
| 7 | 6 | Gold std | 19 | 155.367850 | 13.320475 |

At this point you will be presented with the *Run Measurement Macro* dialog. Select "Create report" to be saved as a file. This report summarizes Autosampler actions and settings, and errors. It has nothing to do with your data. *NOTE: In the image below, "no report" was selected.*



Select *Run*. Because Quadstar trigger support was enabled in your macro instrument definition, you will now be presented with the *Measurement with QuadStar* choice dialog box, as shown below.



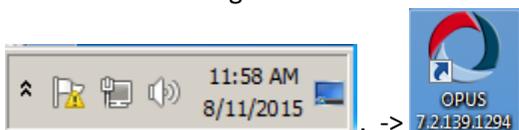
STOP!!! Do not select OK!!!

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

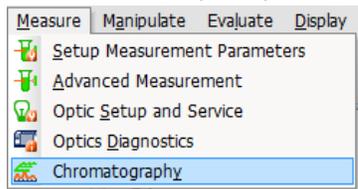
Preparing for IR Measurements

Fill the MCT dewar with liquid nitrogen, if not done so earlier. Hold time is about 2.5 hours.

Use the Windows 7 tool that minimizes programs and shows the desktop. It is the small icon always located at the far right of the notification taskbar. Then start OPUS.

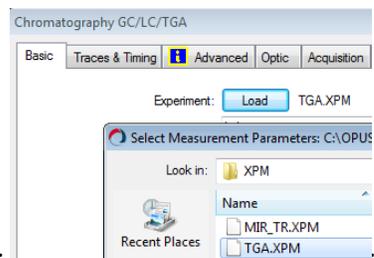


This will bring up the OPUS Login view. Log in as *user= Administrator; password= OPUS; workspace = midIR_FullAccess*. Respond OK to the license validation prompt. The main OPUS view will open.



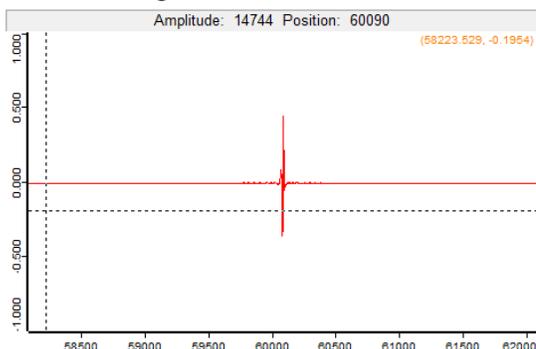
Select *Measure -> Chromatography*. The GC/LC/TGA parameters view will open.

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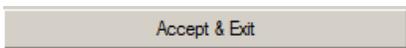


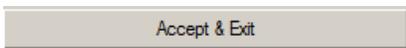
On the "Basic" parameters tab, choose *Load -> TGA.XPM*.

Select the "Check Signal" tab. You should see an interferogram with amplitude greater than 14000



counts.



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

Preparing for Mass Spec Measurements

Use the Windows 7 tool that minimizes programs and shows the desktop. The two mass spec programs you will use are *Parset* and *Measure+Sequencer*.



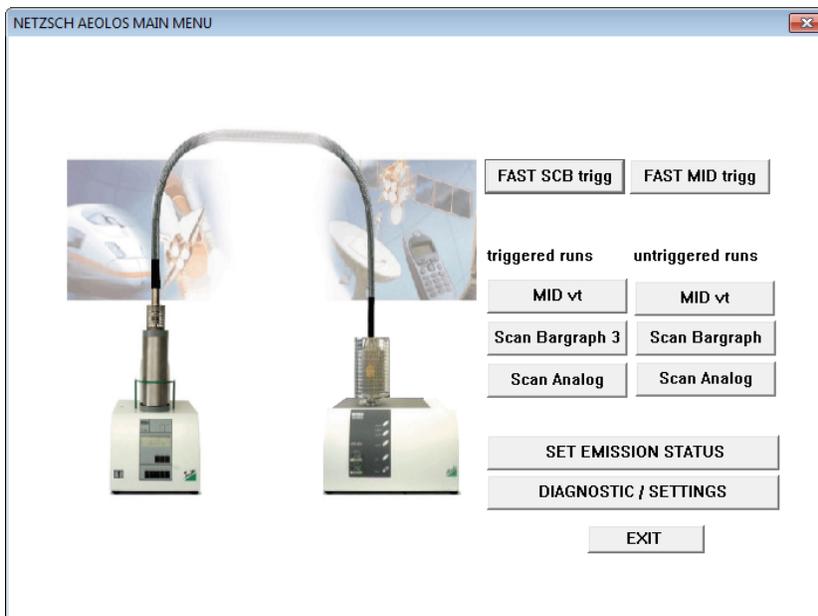
Open *Parset* if you need to create or modify the mass spec parameters you wish to use, or if you need to check which of your parameter sets is appropriate for the measurements you are about to do. You will need to browse and select the proper parameter set as part of running *Measure+Sequencer*, but 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. swv-001). Most people start investigations using “Scan Bargraph” parameter sets, but you should understand the advantages of eventually using “MID” parameter sets.

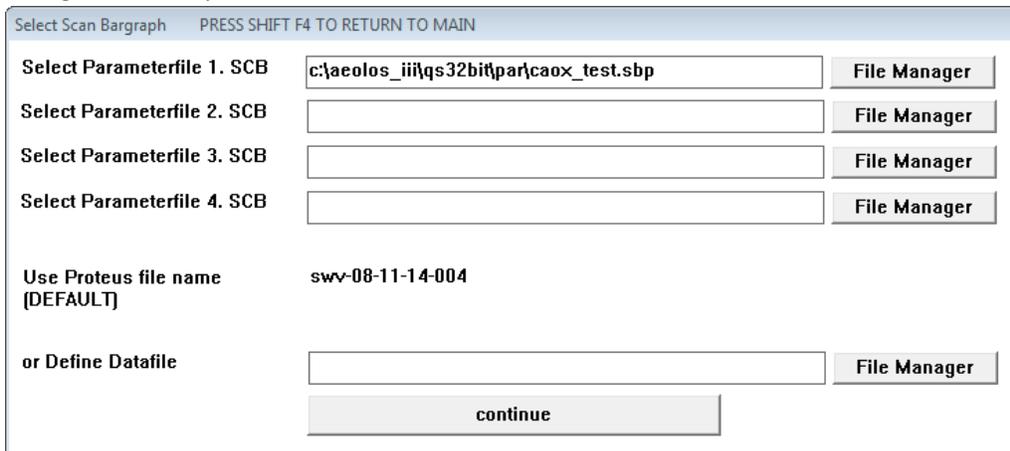
If you opened *Parset*, be sure to exit from that program. Then start *Measure+Sequencer*. The TurboPumpControl view will flash briefly as the program turns on the ion gage and the filament. Next, a table will appear; it will be filled within 10-20 seconds with vacuum readings.

| Measurement Number: 0 | | | | Process | Process | Process | Process |
|-----------------------|--------|-------|------|------------|------------|------------|------------|
| Nbr | Type | Ident | Unit | 5:11:56 PM | 5:11:56 PM | 5:11:56 PM | 5:11:57 PM |
| 0 | F.Var. | gf[0] | | 9.700E-06 | 9.780E-06 | 9.780E-06 | 9.780E-06 |
| 1 | | | | | | | |

Within one minute the Aeolos Main Menu Autosampler Mode view will appear.



Normally you will choose “Scan Bargraph 3” **Scan Bargraph 3** from the three 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. swv-001). *NOTE: The image above shows a special set of parameters selected for running calcium oxalate test samples. You would be selecting one of your own parameter sets.*

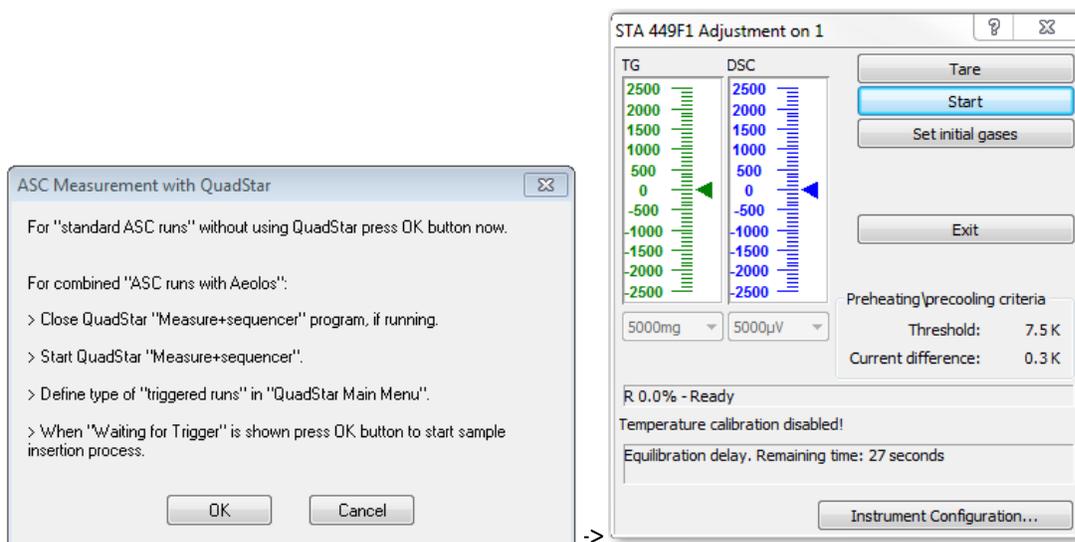
Note that the “Use Proteus file name” field should be pointing to the exact filename you specified for the first sample when you created your macro. If done correctly, the Proteus (TGA/DSC), OPUS (FTIR) and Aeolos (MS) filenames will all be the same. The Proteus and OPUS files will be located in your user folder on the D drive, *D:\data\<username>*. The Aeolos files will always be in the communal directory, *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 MS 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!



Monitoring the Progress of the Sequence

After selecting “OK”, you may see a *STA449F1 Adjustment* view. No action is necessary! If Evac&Fill cycles were selected, you will also see messages associated with those functions. No action is necessary!

Eventually, Proteus will try to connect with OPUS. Various messages will flash. Once the connection is made, the OPUS message “*baseline measurement in progress*” should appear. 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 *Scan-Bargraph* view, FTIR data accumulating in the OPUS *Chrom* view, and DSC/TGA data accumulating in the Proteus *STA449F1* view!!!