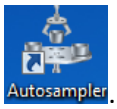
Acquiring Evolved Gas Data with DSC/TGA Measurement (ver.3)

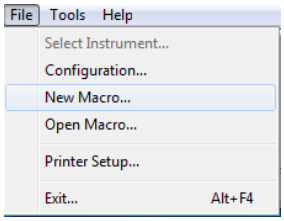
Autosampler macros can include the acquisition of IR data, mass spec data, or both. The steps involved include:

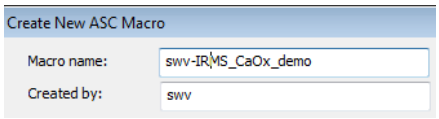
1. Enabling FTIR and/or mass spec acquisition within the instrument definition part of the macro.
2. Including FTIR acquisition in the segment editor part of the macro.
3. Configuring and testing the FTIR before running the Autosampler macro.
4. Configuring the appropriate MS parameter set before running the Autosampler macro.
5. Enabling the MS trigger as part of running the Autosampler macro.
6. Post run data processing of FTIR data using OPUS-3D software. [Not part of this tutorial]
7. Post run processing of mass spec data using Proteus software. [Not part of this tutorial]

The example used in this tutorial is calcium oxalate. The FTIR data will include the standard mid-IR spectral range from 4000-650 wavenumbers, at 4 wavenumber resolution. The mass spec data will be "bargraph" scans from m/z 10-100. For a more complete discussion of FTIR and MS protocols, consult other CIF tutorials or the manufacturer documents. ***This tutorial assumes you have had basic OPUS (FTIR) training!!***

The macro developed in this tutorial will not include running a baseline correction sample, nor will it include Evac and Fill cycles. *NOTE: These should both normally be included in proper research sample macros.* The furnace will be programmed from 40-870 C at 20 K/min.

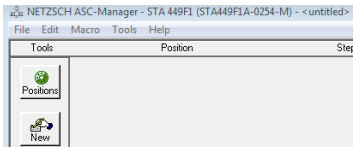
1. Select 

.
2. Select File -> New Macro 

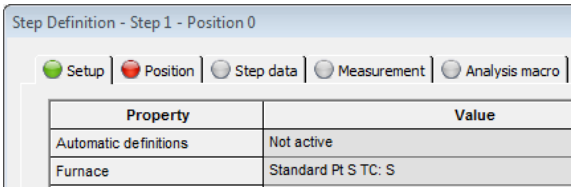
3. Name the macro. 

Be sure to use your assigned three-letter code (your initials) and a *mmddyy* date code, e.g. *swv-081215. "Created by"* should be your netid, *e.g. sveysey*

4. Set the appropriate Property Values in the Definition view. For this example we use:
    - Al2O3 (alumina) crucibles;
    - The reference is in position 19;
    - Special instrument control = FTIR;
    - FTIR definition will run the OPUS TGA.XPM program;
    - OPUS data files will be stored in the same location as the Proteus data;
    - QuadStar trigger support (MS) is enabled;
    - STC and TC calibration are both ON;
    - MFC gases are set to argon for PG, P1, and P2.

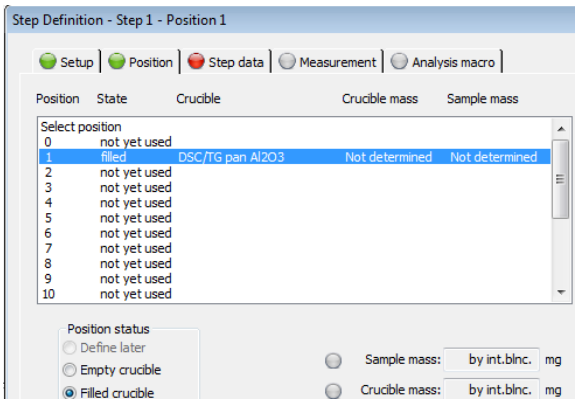
5. Select OK. This opens the ASC-Manager view. 

6. Select "New". This opens the Step Definition dialog with multiple tabs.

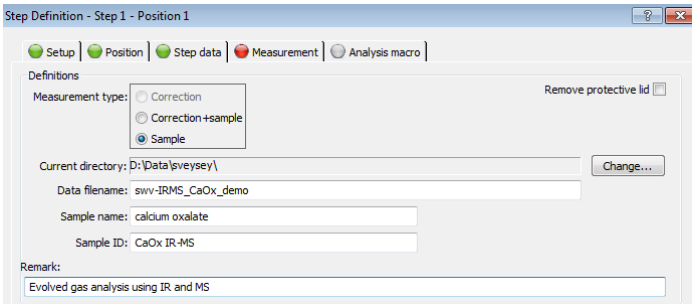


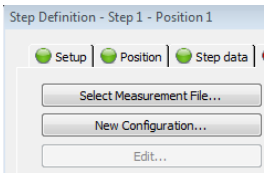
7. Verify the Property Values on the Setup tab. This is an opportunity to correct any mistakes you made in the Definition view. Select Forward  to advance to the Position tab.

8. Since we are not running a baseline correction, position 0 will be left as "unused". Select "positon 1" and define it as a "filled crucible". Note that we have specified that masses will be determined by the internal balance. Select "Forward" to advance to the Step Data tab. Note that it is identified as Step 1 – Position 1.

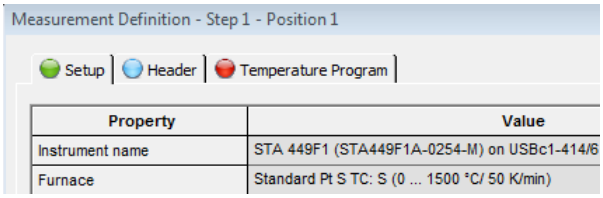


9. Change the path to your directory; assign a filename starting with your three-initial code; enter sample name, sample ID, and remarks. Select "Forward" to go to the Measurement tab.



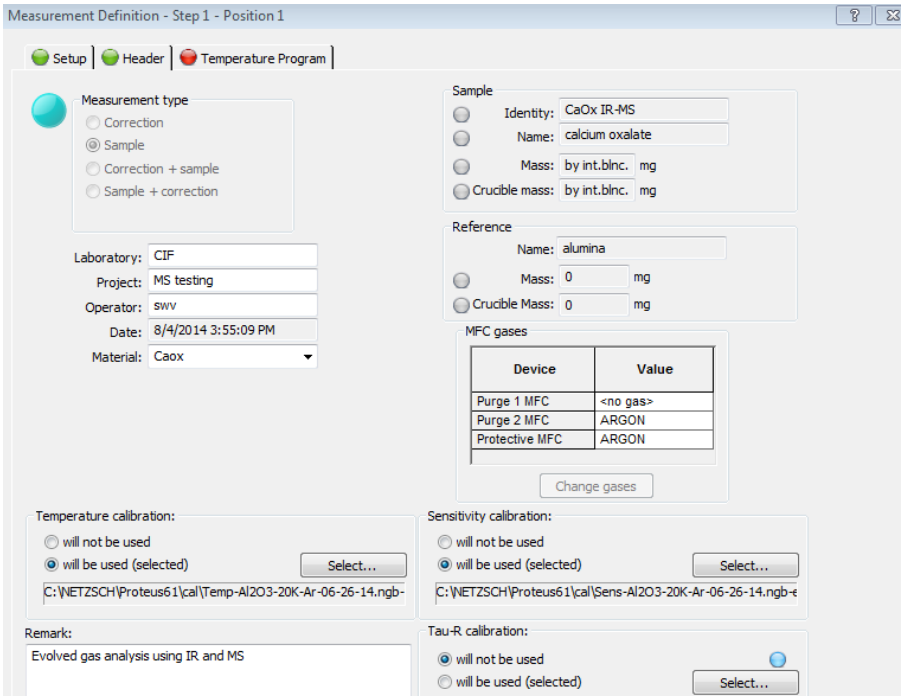
10. Select *New Configuration*. 

This opens the Measurement Definition dialog for Step 1 – Position 1, with three tabs that need to be completed.

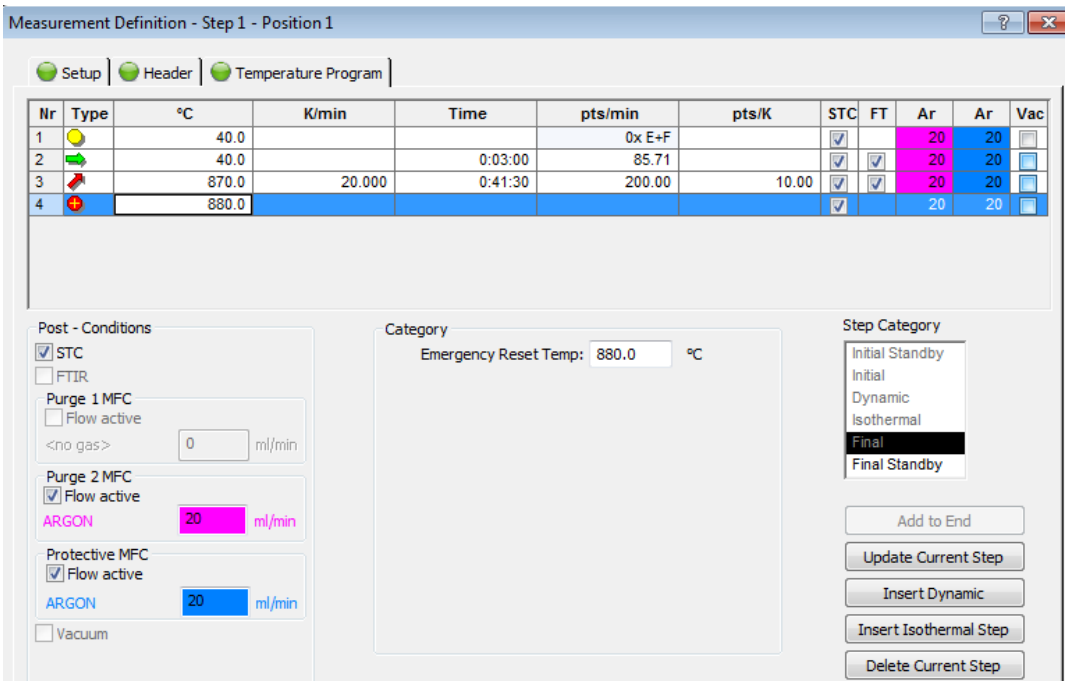


Again, verify the property value settings on the Setup tab. You won't be able to change them here though. If there are mistakes you will need to start over. Select Forward to advance to the Header tab. Sample info cannot be changed, but you can add group/project info and you can select whether calibrations can be used. You can also edit the "remarks" field. Then select Forward to move to the Temperature Program tab.

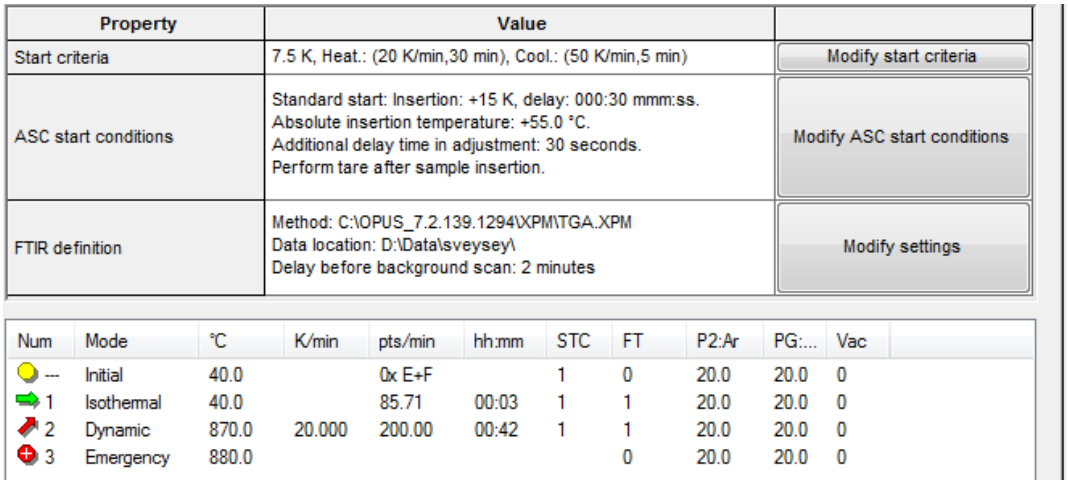
<P1 MFC should also be Ar>



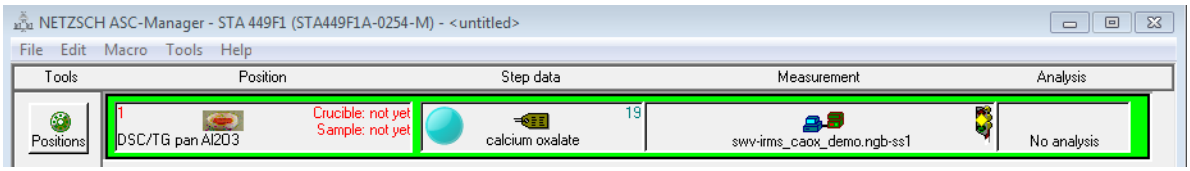
11. Create a program that starts at 40 C, includes at least a five minute isothermal step, and then programs to 870 C at 20 K per minute. In all steps the argon protective flow should be set to 20ml/min; P1 and P2 should each be set to 10 for the temperature program segment. AUTOVAC will not be used. *However, note that 1-2 Autovac Evac/Fill cycles <u>should</u> be used for your research samples.* Be sure that FTIR is enabled.



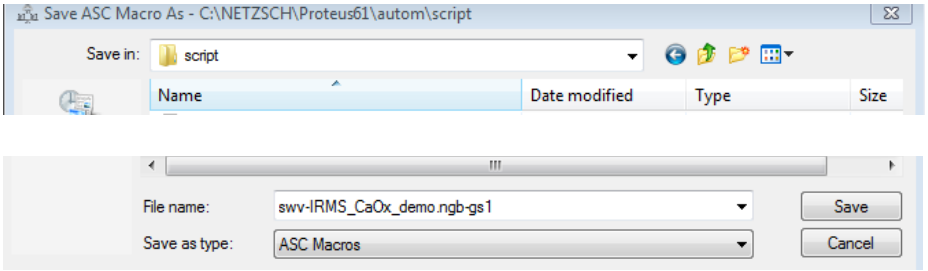
Select Forward to see a summary of the complete step definition. *[Note incorrect values]*



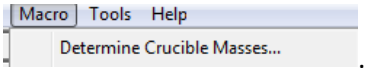
Don't bother with the Analysis macro tab. Select OK. You will be returned to the ASC Manager view and the Position 1 measurement program will be present, with the "yellow light" flashing.



12. Note that even though you have "named" the macro, it has not yet been saved! Choose *File -> Save.* This opens the "Save ASC Macro As" view.

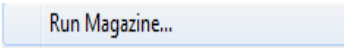


By default, the macro will be saved in the *C: NETZSCH\Proteus61\autom\Script* folder with the name you previously defined for it. Select Save.

13. From the ASC-Manager view, select *Macro -> Determine Crucible Masses*. Be sure that you have empty crucibles in positions 1 (sample) and 19 (reference) 

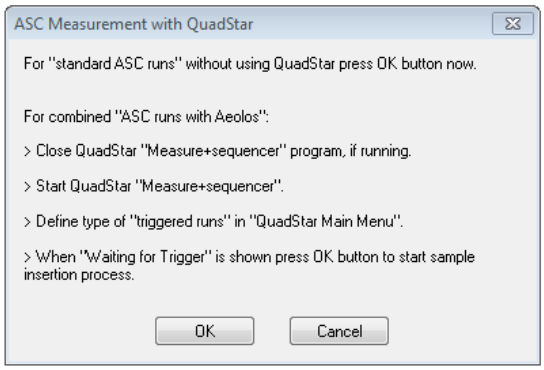
. As usual, you will need to respond to the furnace status dialog before the autosampler will perform the weighing operations. *NOTE: You do not need to deal with either FTIR or MS status until the empty crucibles and the filled crucibles have been weighed.*

14. Place a small amount of CaOx (2-5mg) in the sample crucible. From the ASC-Manager view, select *Macro -> Determine Sample Masses.* Again, verify furnace status and respond OK on the dialog.

15. From the "NETZSCH ASC-Manager" program, select Macro -> Run magazine. 

    - Confirm that step 1 position 1 will be analyzed;
    - Enable Create Report to be saved as a file;
    - Select Run;
    - Select OK on the *Normal Completion / Safety Switches* dialogs.

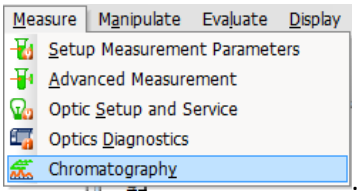
    You will now be presented with this view:

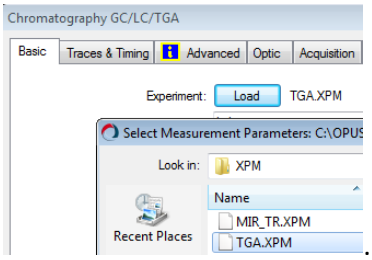


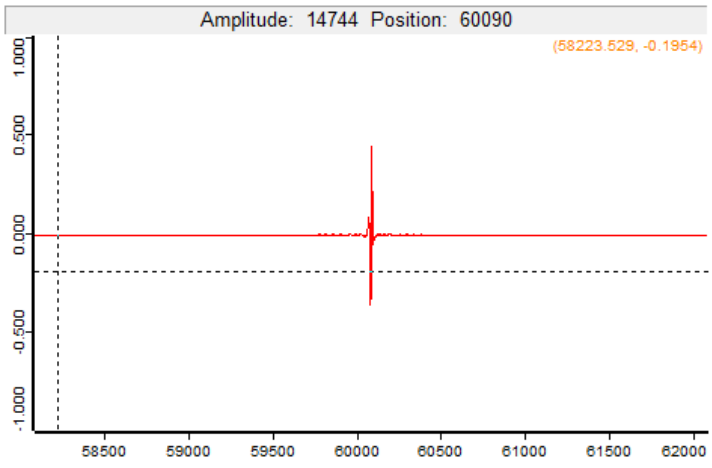
STOP!!! AT THIS POINT YOU MUST CONFIGURE THE FTIR AND THE MS!!!
DO NOT SELECT OK!!!

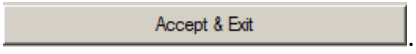
<u>SETTING UP THE FTIR (Steps 16 – 18)</u>

16. Add liquid nitrogen to the Tensor MCT detector using the protocol you have been shown during training. 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.

17. Select Measure -> Chromatography. 

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

Then select the "Check Signal" tab. You should see an interferogram with amplitude greater than 14000 counts (6 mm aperture). 

Then from the Basic tab, select 

. *Do not minimize or close OPUS!*

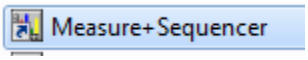
<u>SETTING UP THE MASS SPEC (Steps 19 – 23)</u>

Be sure there are no Aeolos programs running. If there are, exit from them!

19. The Aeolos programs used will be *Parset* and *Measure+Sequencer*. The icons are on the desktop. For this tutorial, we will use a simple, pre-defined mass spec parameter set to acquire bargraph data from m/z 10

to m/z 100. We will be using the "Measure +Sequencer" program to couple the Proteus and Aeolos acquisitions.

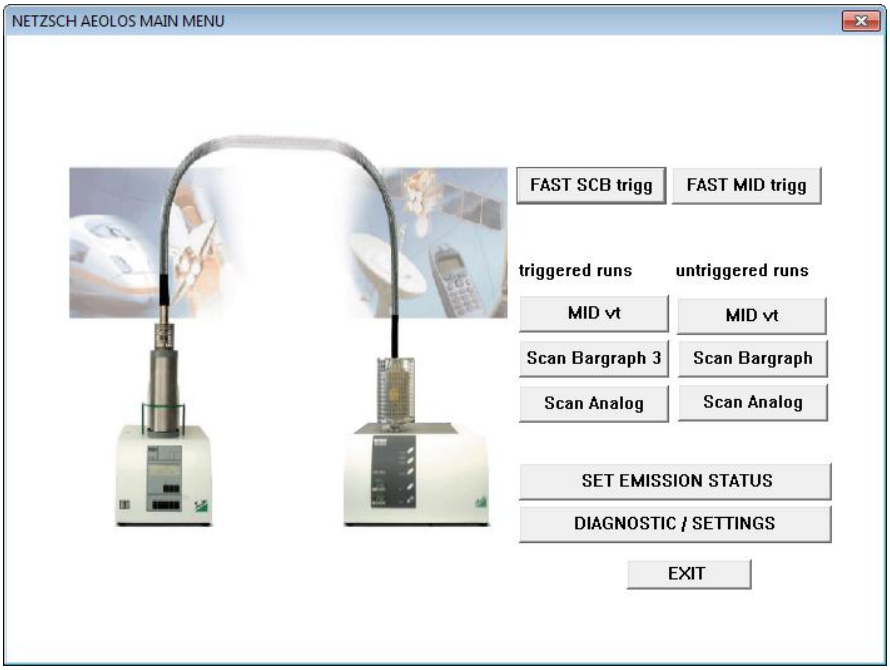
Note: More complicated bargraph or MID parameter sets can be created using the "Parset" program. These should be saved in the Aeolos parameter set directory, C: Aeolos_III\qs32bit\Par\ <filename>, and can be used at any time. Start the filename with your designated three initials!!

Now select Measure plus Sequencer. 

from the suite of Aeolos programs. The TurboPumpControl view will flash briefly as the program turns on the ion gage and the filament. Next, a table will appear and 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							

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

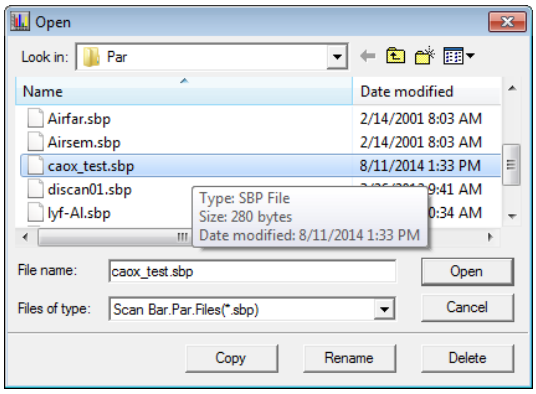


We will be executing a triggered run using the *Scan Bargraph 3* function. Do <u>not</u> use *FAST SCB trigg*. Choose "*Scan Bargraph 3*" 

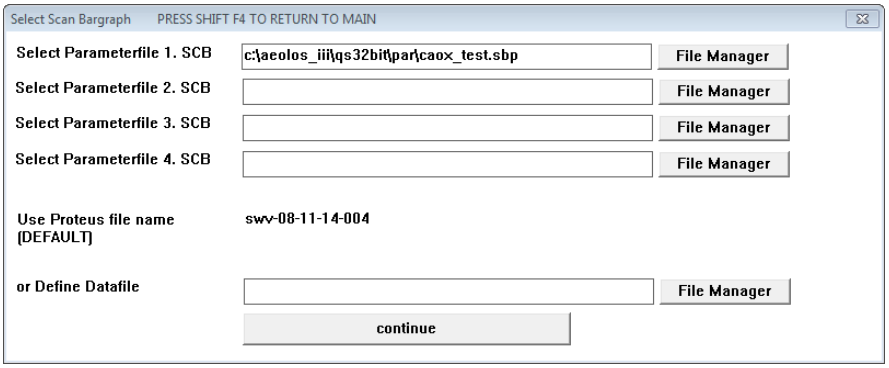
from the three triggered run selections.

21. Use the File Manager 

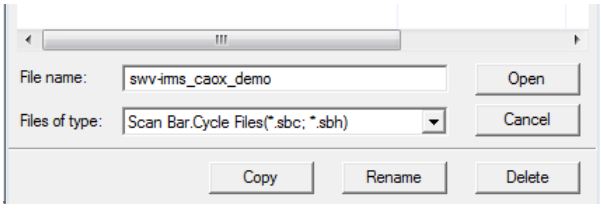
button associated with "*Select Parameterfile 1.SCB*" to select the *CaOx_test* set of parameters. [<u>Your</u> parameter sets would start with your assigned initials, e.g. swv-001]



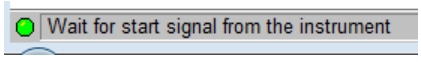
The *Select Bargraph* View should look like this:



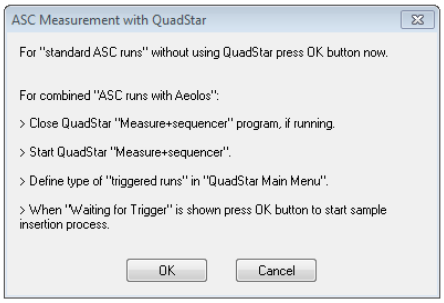
22. **Do NOT use Define Datafile!** *"Use Proteus file name"* should already be pointing to the correct file; the name you choose in your macro for the Proteus data.



23. Now select Continue. 

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

24. **At this point, select OK from the "ASC Measurement with Quadstar" dialog.**



25. Proteus will first connect with OPUS. Various messages will flash. NOTE: A “connection countdown” dialog may appear; this usually means Proteus is having trouble connecting and will probably fail at the end of the countdown. **[We have not seen that issue for quite some time.]**

26. Once the connection is made, the OPUS message “baseline measurement in progress” should appear. Within a minute, 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 and various traces in the Proteus display will also appear.

    You will mass spectra accumulating in the Quadstar view, FTIR data accumulating in the OPUS Chrom view, and DSC/TGA data accumulating in the Proteus STA449F1 view!!!

    HOORAY!!!