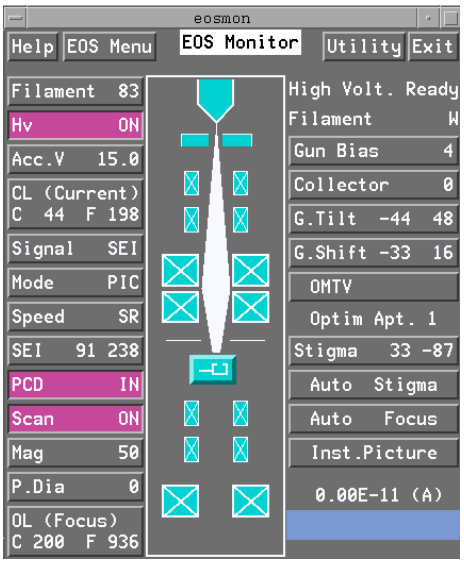
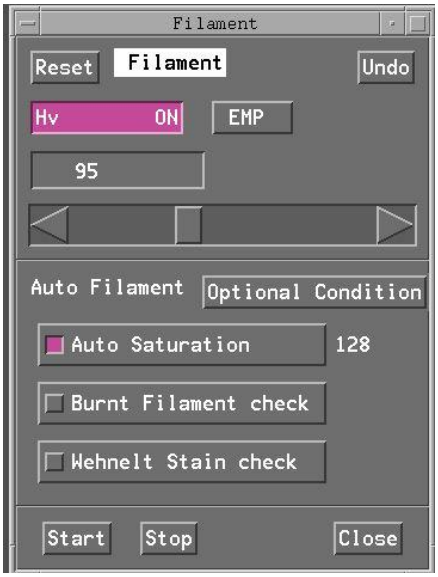
EPMA General Operations

Filament Startup Procedure

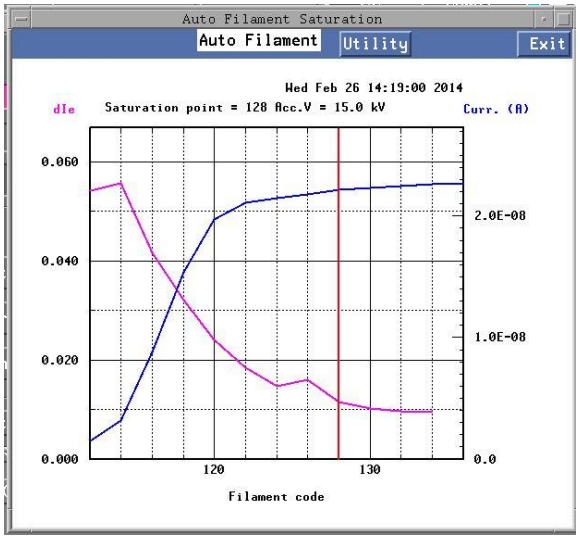
1. Once the EPMA software has been started, it will be necessary to saturate the filament at least one hour prior to operation. To do this, follow the following saturation procedure. NOTE: Filament may already be saturated ahead of remote use; check with FCAEM staff if this is the case. If so, skip these procedures to "imaging start-up and navigation".
    - On the Off-line Menu select "Monitor" and EOS Monitor to initialize the EOS Monitor.



    - At the upper Left, select "Filament" with the current DAC setting shown to initialize the Filament Panel.



- Verify that the Auto Saturation is selected and select “Start”. This will initialize the filament saturation process with a sub panel indicating the saturation curve.



- Once the filament saturation process is completed, simply close both of the filament saturation panels and begin working on the analysis process.
- Once an hour has passed, set the current by selecting “CL (Current) and use the scroll bar-Start with fine to adjust the current to the desired setting.
- Once the analysis is complete, select filament and move the slide bar until the setting reads in the 90’s and complete the standard shutdown procedure.

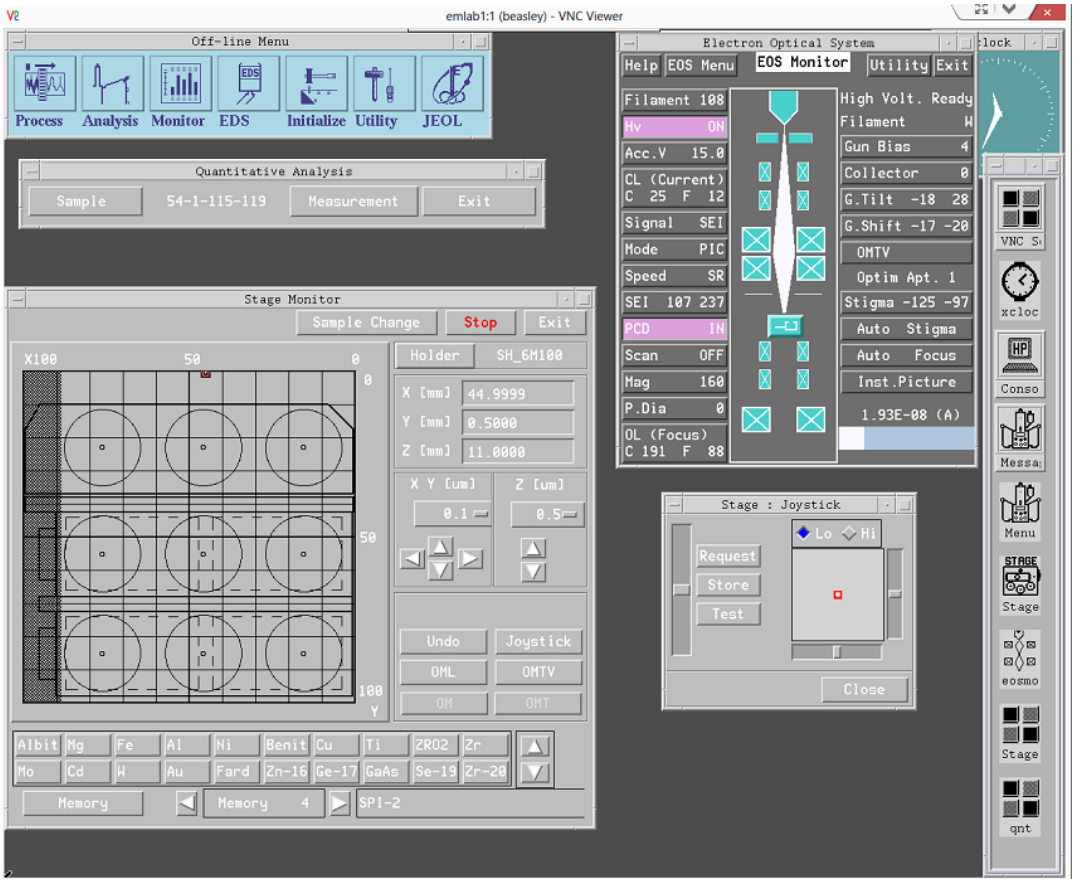
Imaging Start-up Procedure & Navigation

1. The system while operating remotely requires an external video to view the electronic and optical image. To access the images/video, simply input the following IP address in a browser, the suggested browser is Firefox because it gives the user the option of magnifying one of the images. Use IP address **131.94.95.141** to initiate viewer.

2. Now you should have the EPMA Menu operations screen in one window and a browser window to view the optical and electronic image. On the EPMA Menu Under the "Monitor" tab, open EOS Monitor and Stage. The EOS monitor window is a mock-up of the beam path with controls for various microprobe functions; The Stage monitor is a mock-up of the microprobe stage that provides X-Y-Z coordinates for your location and that of samples and standards.

3. Under the Analysis tab, click Quantitative Analysis. The Quantitative Analysis menu appears. This tab/window is where we will set up your analysis of standards and unknown samples. We will come back to this later in the procedures.

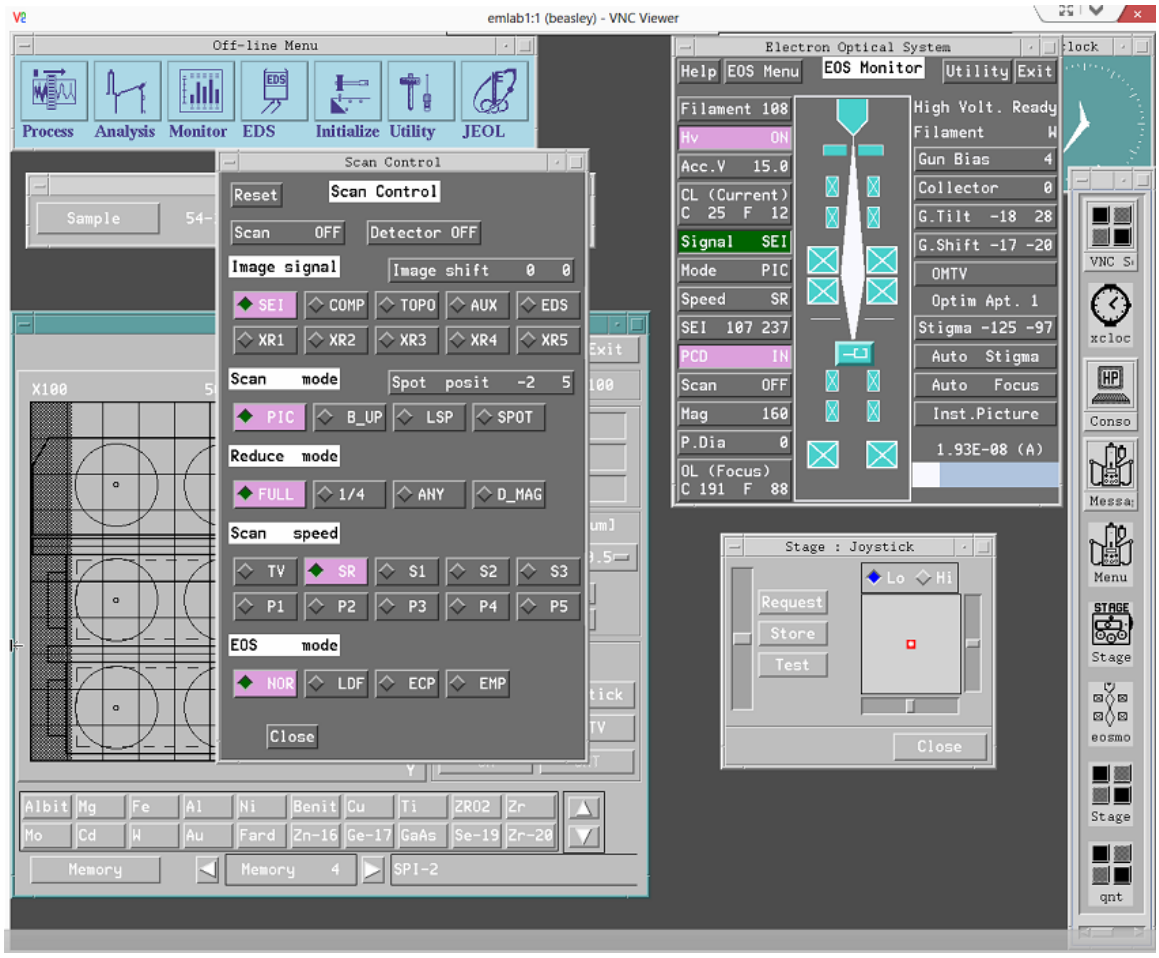
4. On the Stage window, click "**Joystick**", and move the Joystick window to a clear space on the emulator screen. Arrange these windows once they appear so that you have a clear view of each one, ideally without tiling them – this will take up most of the screen (See Below).



This is an example of how to arrange the various windows in order to operate the probe easily. On a separate internet browser window you should have the video feed open.

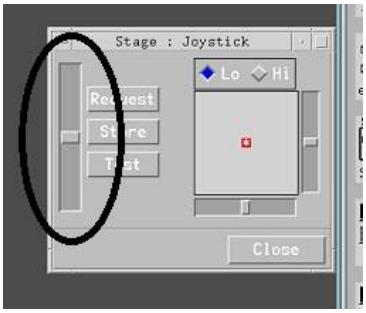
5. It's time to find your sample! On the "Stage Monitor" window, you can double click on the area depicting the sample holder to a specific location where your sample is located. This is a fast form of stage navigation. Most times the staff at FCAEM will provide you with a digital picture of sample locations and even digitize the locations of different samples for easy navigation.
6. Once on your sample or close to it, you can now use the manual joystick window to make minor movements on the stage in order to navigate around on your sample (click and hold the red dot in the middle of that space and move your mouse in any direction). You can select the "Hi" button in this window to increase rate at which the stage moves with the joystick controls. You can also use the scroll bars to the left and bottom of the joystick space to move in a strictly north/south or east/west direction.
7. Once on your sample open the Faraday Cup by clicking on the "**PCD**" button in the EOS monitor window. In your web browser you may see an SEI or BSE image appear. In the EOS monitor you can choose the type of signal/image and scan rate in which to view the sample. Click on "**Signal**" button in the EOS

monitor window and click on “comp” under image signal for BSE (backscattered electron) imaging or SEI for secondary electron imaging. We recommend a TV rate for SEI and BSE mode (click “TV” under scan speed) for navigational purposes. **For remote video feed to work at live speeds, keep magnification equal to or greater than 100X.** The other scan rates give higher pixel density (better image) however they will appear as still images and won’t display live movement if you are navigating.



8. If your SEI image brightness or contrast is incorrect, then adjust that by pressing the “SEI XXX XXX” button on the EOS monitor screen (fourth button down on the left). BSE contrast and brightness can only be changed locally so contact FCAEM staff for adjustments. If you continue to have problems with contrast and brightness with BSE and/or SEI imaging, don’t hesitate to contact the FCAEM staff. They will gladly set up your imaging for the days use.
9. If your SEI or BSE image is out of focus you can use the “OL Focus” button at the bottom of the EOS monitor to make adjustments.
10. It is vital that your optical view is in focus. Turn on the “Optical Microscopic Telescopic View” or “OMTV” button on the right side of the EOS Monitor

screen. You should see another image turn on in your internet browser window. To put the optics into focus, click on the scroll bar on the left of the joystick controls (see picture). You will know it's in focus when the cross hairs come into view and intersect in the middle of the image. **Any time you navigate around to different spots in a sample or move to a different sample you must adjust the optical focus. Having your OMTV in focus ensures that the beam is striking the exact spot for analysis.**



11. You can zoom in and out of your sample by clicking on the "Mag" button on the EOS Monitor screen and adjust accordingly. There are "quick" magnification buttons that will instantaneously zoom your view closer to you sample.

EDS Measurement Procedures (Mineral/Phase Identification)

So, you've got a mineral grain identified, and you want to get a bead on what it might be. The backscatter shading is (*light/dark*) which can give you some information on its makeup, but the shading is relative – you set this on the backscatter detector as needed. So, what we need to do is take advantage of the other major product of interactions between the electron beam and the sample: the emission of X-rays that are characteristic of the elements in the materials analyzed. Electron microprobes have two instruments that do this: a Wavelength Dispersive X-ray Spectrometer (WDS) and an Energy Dispersive Xray Spectrometer (EDS). For quick-and-dirty non-quantitative examinations, we use the EDS spectrometer.

1. Open up the EDS window by hitting EDS on the EMPA Menu and selecting EDS from the dropdown list, which opens the EDS window, a graphic interface that is basically a plot of energy versus wavelength for the generated X-rays. (see image below)

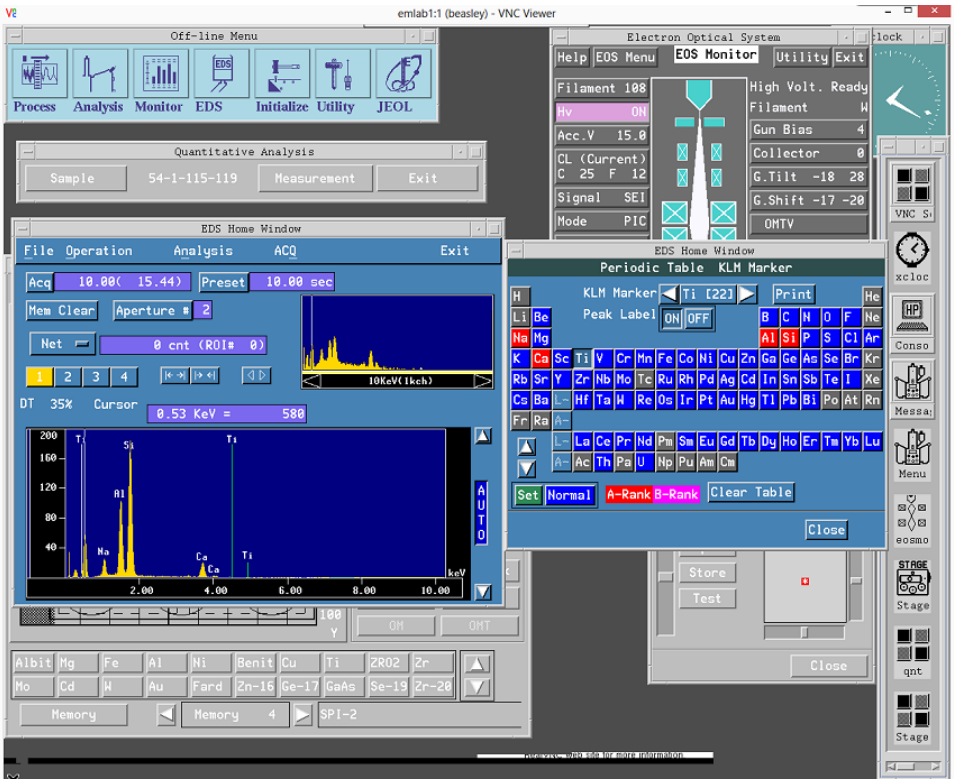
The EDS spectrometer makes use of a particular kind of X-ray detector (called a Silicon Drift detector) that allows it to look at the entire X-ray spectrum simultaneously. What we'll get back is a scan of all the X-ray wavelengths the sample is emitting, and we can then match these to different elements based on their X-ray profiles.

2. Turn SCAN off on the EOS monitor window. Make sure you're in optical focus via OMTV. Hit ACQ on the EDS window; let it run for a few seconds to generate the scan.

"Now that we have X-ray peaks, how do we tell what is what?"

3. Open Periodic Table on the EDS window: the periodic table appears. Pick several elements in the low Z range to see where their peaks are; iteratively match up the peaks with the right elements. Identify with the class the elements that are in the mineral being analyzed.

Note that the program identifies different kinds of X-ray peaks for different elements: for low Z elements (most of the common ones) we get Kα peaks; but for some of the heavier elements (Zr, Ce, Th, etc), we see Lα peaks. What the program is doing is picking based on the most intense X-ray peaks for each element. Note also that on the EDS spectrometer, there is not a simple relationship between peak height and concentration: in fact, as Z goes up, the peak intensity at a given concentration level of an element goes down, and this trend is distinct for the Kα, Kβ, and Lα peaks. So the EDS spectrometer can really only be used as a chemical identification tool of sorts: it can tell you what elements are in the sample, but it can't quantify them. For that we need a different spectrometer.



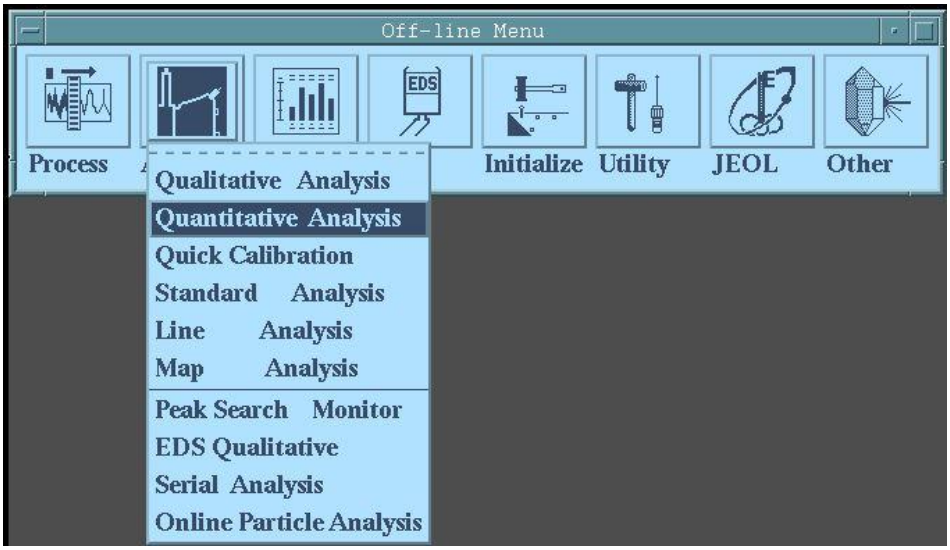
Typical view of EDS window and periodic table for element identification.

Quantitative Analysis

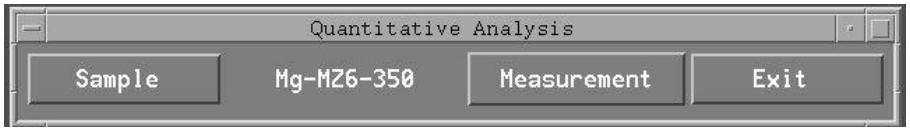
The Wavelength Dispersive Spectrometer (WDS) works on the principle of X-ray diffraction: X-rays entering the spectrometer hit an analyzing crystal, which then diffracts a small subset of them, representing a particular element, at a particular angle of diffraction into a Faraday Cup detector.

This JEOL microprobe has five different WDS spectrometers, which can each measure one element at a time. Each of the spectrometers has three different analyzing crystals, which are rotated into place for analyzing different elements. Different analyzing crystals are good for different Z elements: TAP is good at low atomic numbers, while LiF and PET are better at high atomic numbers. The instrument will default to the "best" crystal for any element chosen, but you can adjust this manually if you want (we won't do that today!).

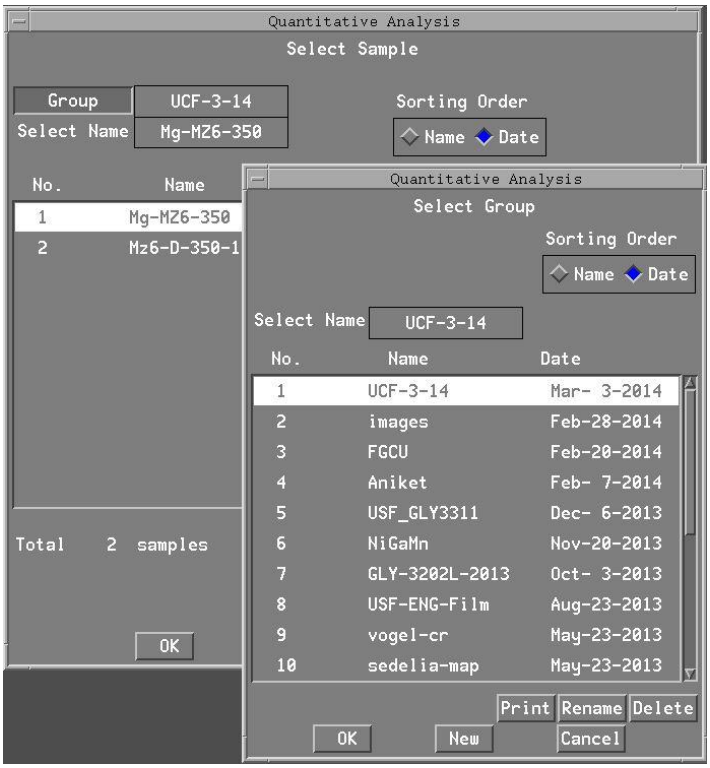
1) Prepare the element list for analysis before initiating the quantitative analysis software based on the material phase of interest.
2) On the Off-line menu, select **Analysis** and then **Quantitative Analysis**.



3) On the Quantitative Analysis menu, select **Sample** and verify that the Group created for your use is selected.

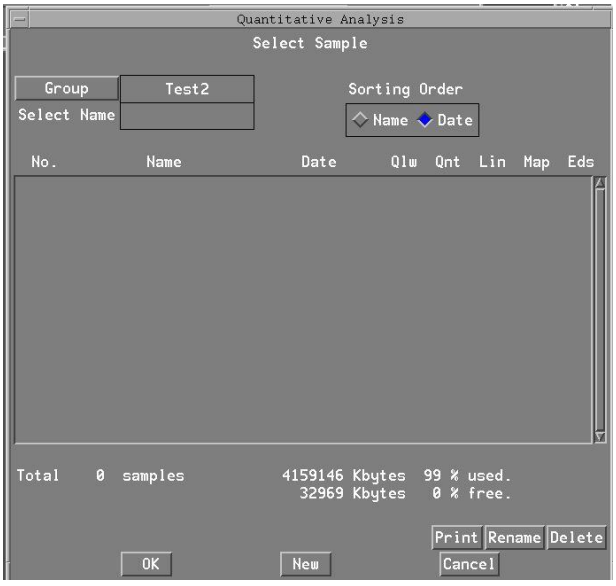


4) To create a new Group, on the Quant menu select Sample to initialize the sample select menu.



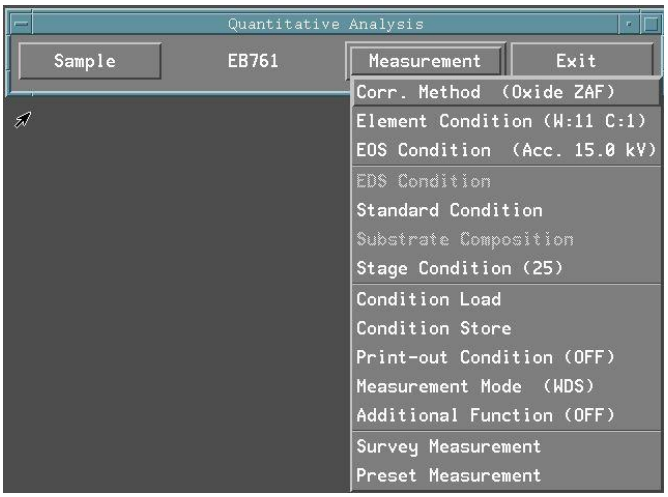
5) On "**Select Group**" at the bottom select "**New**" and with the curser in the typing box, type the group name, special characters include the dash and underscore, select OK to complete.

6) At this point an empty box will appear, select "**New**" again and place the cursor in the type box and name the specimen to be analyzed.



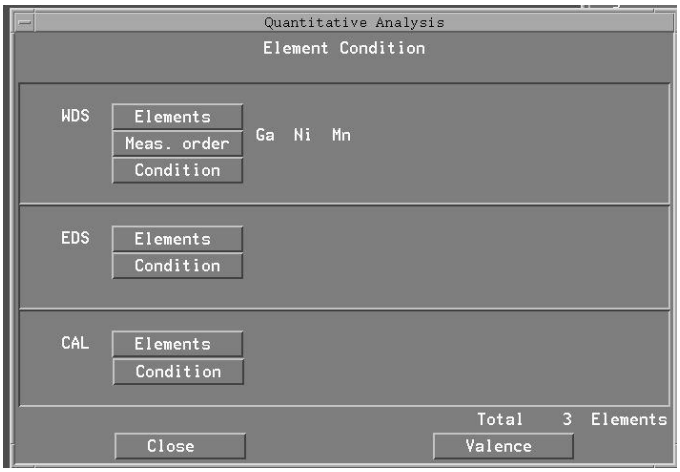
7) On the Quantitative Analysis menu select "**Measure"**, a pull down menu will appear with the quantitative analysis commands, verify that Oxide or metal is

selected and ZAF or Phi Rho Z are assigned in the matrix correction method box depending on the type of materials to be measured.

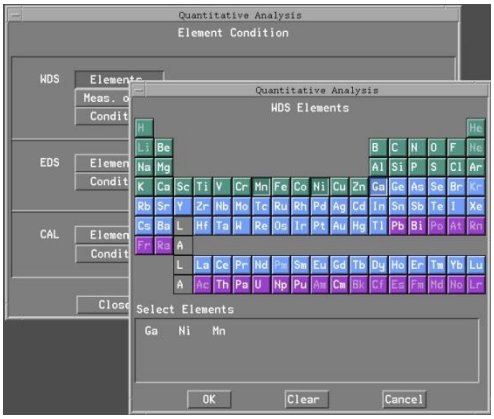


The microprobe has a data correction and calibration system called the ZAF system: Z (for atomic number effects – X-ray intensities decline with increasing atomic number), A (for absorbance – some emitted X-rays are absorbed by the materials being hit with the beam), and F (for Fluorescence: some materials emit secondary X-rays related to the primary X-rays passing through them).

8) Once the element list is known, select all the elements and assign the element to the crystal and spectrometer to be used for the analysis.
   a) Under the "**Measurement"** menu, select "**Element conditions"** and "**Elements"** to view the periodic table to select elements.



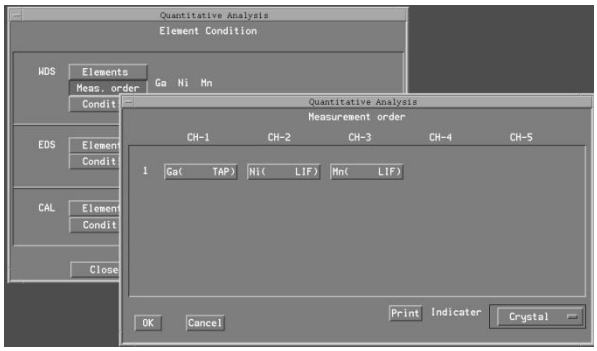
   b) Select the elements in the order to be analyzed, by placing the curser on the element and selecting it, select only one of each elements to be analyzed, no duplication in Quantitative Analysis. If an incorrect element is selected, simply highlight the element with the curser and select "**Clear"**.



c) Once the list is correct, select "**OK"** to exit.

9) The selected elements must be assigned to a spectrometer and crystal, select "**Measurement"** and "**Measurement Order"** to initialize the measurement order menu.

a) The computer will assign locations arbitrarily; the user will need to reassign the elements to the correct spectrometer and crystal assignments based on the list that was created earlier.

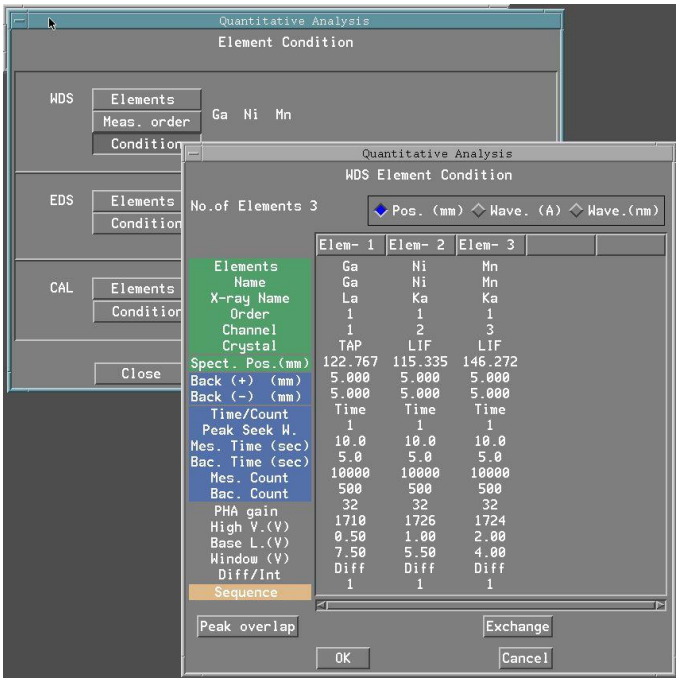


b) With the curser, select the element by holding the left mouse button down and slide each element to the proper spectrometer location. Unavailable spectrometers will white out indicating that the spectrometer does not have either the correct crystal available or that the correct energy line conditions were created in the conditions folder. If a crystal is needed to be changed, right click on the element and a crystal selection will appear, select the crystal to be used or verify that the crystal that is present is the correct one to be used. Please note that located on the lower right hand corner is a box (**Crystal**) that will allow the user to select different modes of information about the crystals on the spectrometers. If there are more than two passes for any spectrometer, it is advisable to select this mode and select pos [mm] to get the crystal positions and verify that the collection order has all of the crystal in a sequential line. This keeps the spectrometers from wasting time by moving past element positions and returning to a position that was passed to get to a higher range element.

c) Once the assignments are completed select OK but do not leave the "**Element Condition Menu**".

10) Once the element selection is completed it will be necessary to select the "**Condition**" to verify the spectrometer conditions.

a) Select "**Condition**" to initialize the WDS Element Condition Menu.



b) At the bottom of the box, verify that the all of the elements appear to have been calibrated by verifying that the Diff/INT reads Diff and the bias ranges aren't maximum. If there is a problem, select the element(s) in questions and verify that it is not simply a read problem and that once selected the elements on the table update. If there is no update, it may be necessary to calibrate the problem element which is done at the center.

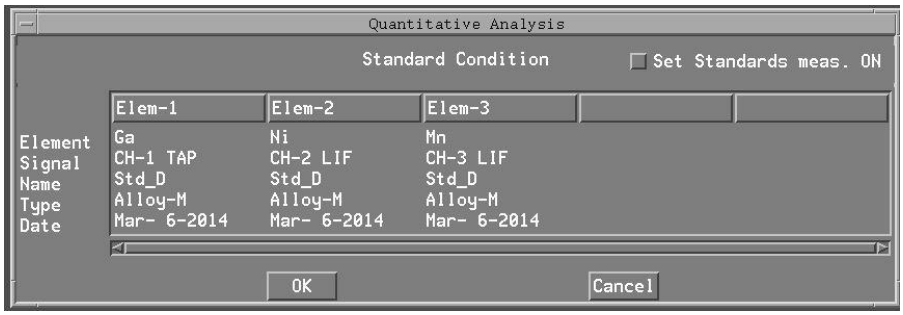
c) Verify that the peak and background times are set to a reasonable level, this is determined by the concentration of the element, typically higher concentration elements are 10 seconds on peak and 5 seconds on the + and – background settings.

d) Verify that the background in both Back (+ and -) are properly set, the "**Peak overlap**" button is used to confirm the status of the elemental overlap, present or not. The adjustment of the background will move the location of the background collection point in millimeters along the wavelength collection axis left and right of the peak. A qualitative analysis may be needed for the more complicated collection to assist in the bkg positioning.

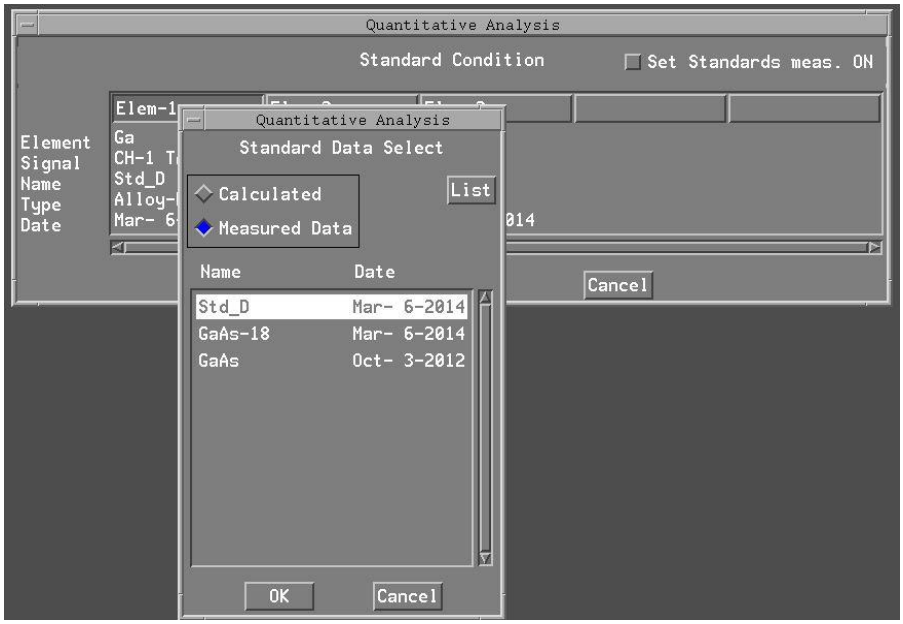
e) Once the process is complete click "**close"** to close the window and ok to complete the operation. Please note that all changes are set once the OK button is selected, if you select cancel, all operation will be made null and void.

11) Close the Element Condition menu to complete this process.

12) Select "**Measurement"** and **"Standard Conditions"** to initialize the standard selection menu.



13) Select any of the Elem-X to see the available standards for the element being analyzed to select the desirable standard.



The calibration protocol is complicated and (fortunately) automated, but it depends critically on a good choice of standards. The rule of thumb when doing microprobe standardization is: compare apples to apples whenever possible. So, if we know we have feldspars in our rock, we make sure to use feldspar standards for Si, Al, Ca, Na, and even K; same for pyroxene, olivine, garnet, etc. The problem is that you can't easily do this for every element in every mineral – ultimately you have to make some compromises (like for example, running Si in all the minerals off of a feldspar standard, Ca off a pyroxene standard, Al off of feldspar, etc.) The challenge is getting the best set of standard choices, which can be difficult for challenging samples.

14) Select the standard calibrated for the analysis to be performed, for a standard to be available, it must be collected at the same Kv with the same energy peak, K-alpha

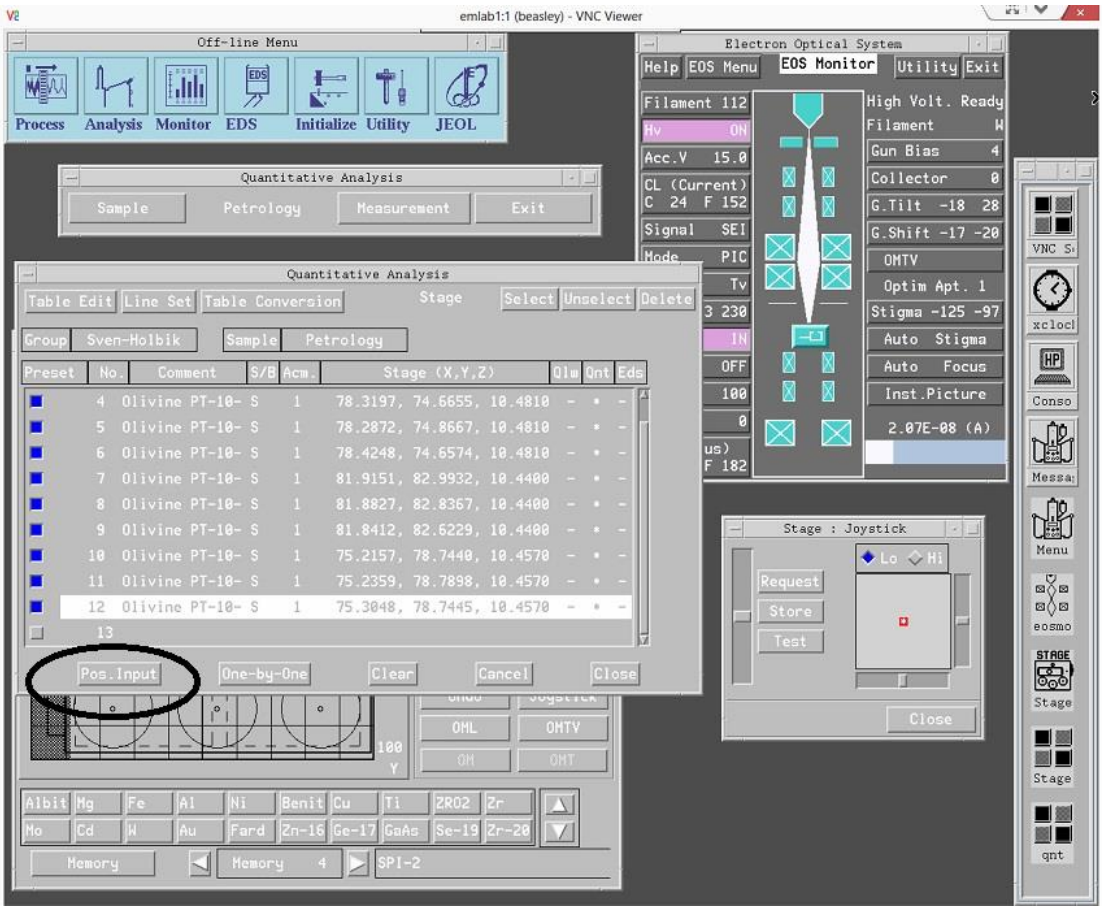
or beta, L- alpha or beta, etc. and correct spectrometer, if any of the conditions is not met and the desired standard will not be present. If a standard is not available, it is possible to calculate the element in "calculated" mode which will not use collected spectra against the unknown.

15) Select **"<u>Measurement</u>"** and **"<u>Additional Functions</u>"** to verify that Peak Search- (Always), Background Measurement- (Always) and Asynchronous Measurements are all selected.

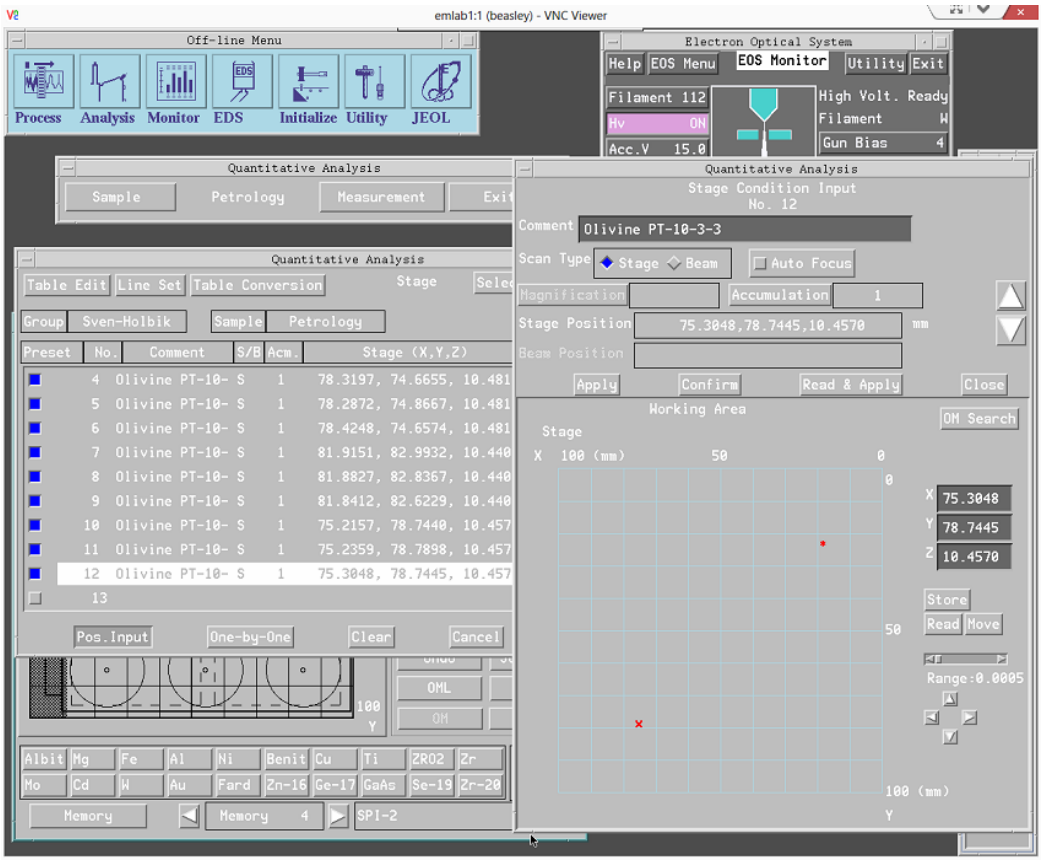
This concludes the preliminary settings; so now that we have standards we like for the run, we should try measuring something!

16) Once the stage is on the sample to be measured, select **"<u>Measurement</u>"** and **"<u>Stage Condition</u>"**.

17) Select **"<u>Position Input</u>"** (Pos. Input) to initialize the stage control and position selection menu. In this menus is where we select points on the sample to analyze.



A large window appears with a space for sample identification, the X-Y-Z location of the point, and a mock-up of the stage showing the position. What we do to choose a grain to analyze is center it under the beam (not the crosshairs on the window!) and then save its X-Y-Z position for measurement.



Center a grain in the Backscatter image window using the X-Y focus on the Joystick. If required do this at several different magnifications.

18) To change Magnification, hit "**MAG**" on the EOS Monitor window, and change magnification using the preset buttons or the slidebar.

19) Turn on "**OMTV**" on the EOS Monitor Window – note that the backscatter image washes out as the optical microscope window lights up.

20) Move off any holes or cracks observed in the optical microscope image with the X-Y focus on the Joystick (if needed turn off OMTV to check and see if the grain is still suitably centered in Backscatter mode!)

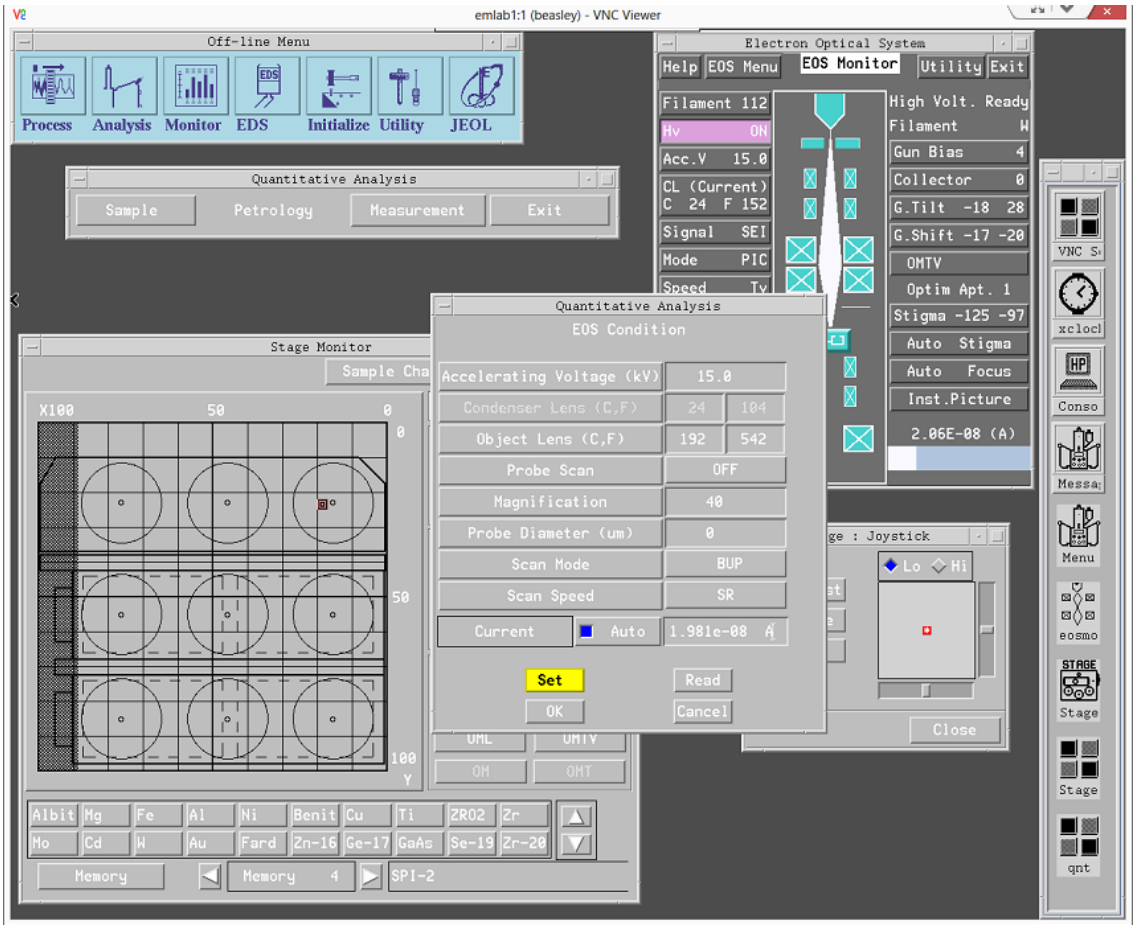
21) Focus in the Z direction using the slidebar on the Joystick, until you can resolve the crosshairs in the window clearly on the OMTV screen (**remember your electron beam position!)**

22) On the Position Input window, hit "**Read and Apply**" to save the position, and close the position input window.

23) Repeat Step 18 to 22 until enough points are digitized and close the Position Input menu and the Stage menu.

Now that you have points chosen, we need to do the final preparations for analysis. The one thing that changes quickly over time is the current level of the electron beam, but we can calibrate this to some degree by setting it to precisely 20 nanoamps at the beginning of the run, which we do under EOS Condition.

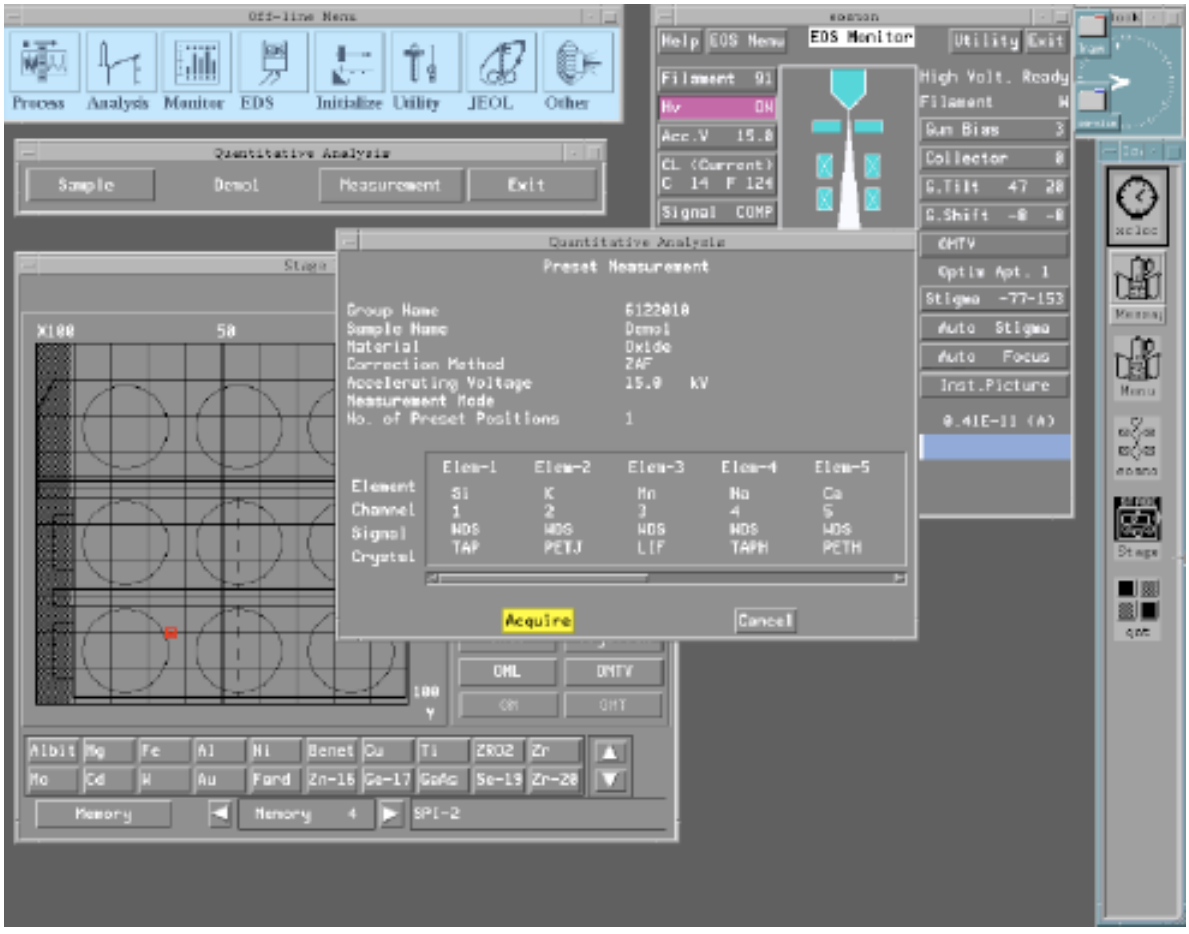
24) On the EOS Monitor window, turn "**Scan OFF"**, noting that this sets the beam to its most focused and energetic condition.
25) On the Quantitative Analysis menu, Open "**EOS Condition"**; a new window will appear recording the modes of the instrument. Move this window by clicking and holding on its header, so you can see it and the EOS Monitor window simultaneously.
26) Check the current level at the lower right on the EOS Monitor window. If it is different from 2.00 x 10-8 amp:
27) Click **CL** (Current Level) on the EOS monitor window. A new window appears with a coarse and fine adjustment slidebars.
28) Adjust the Fine adjustment slidebar to change the current to 2.00 x10-8 amps, as read on the EOS Monitor window, and close the CL window.
29) On the EOS Condition window, hit **"Read"** to save the current level at 2.00 x 10-8 amps, and close the window.



Now that we have points selected and the current level set, we are finally ready to do Wavelength Dispersive Spectrometer measurements!

31) Open "**Preset Measurement"** off of the "**Measurement"** dropdown menu in the Quantitative Analysis window. A new window appears, listing measurement parameters.

32) Hit the yellow "**ACQUIRE"** button. Two new black-background windows appear, one listing the points being analyzed, and the other recording the data as it is collected. Depending on how many points are chosen, this may take a few minutes per point. When data collection is completed, you'll see **MEASUREMENT END** in small print in the left-hand analysis window. Usually the elemental readout from the last analysis will also appear here.


Off-line Menu
Process
Analysis
Monitor
EDS
Initialize
Utility
JEOL
Other
Quantitative Analysis
Sample
Demo1
Measurement
Exit
Help
EDS Menu
EDS Monitor
Utility
Exit
Filament 91
Hv ON
Acc.V 15.0
CL (Current)
C 14 F 124
Signal COMP
High Volt. Ready
Filament W
Gun Bias 3
Collector 0
C.Tilt 47 28
C.Shift -0 -0
CMTV
Optim Apt. 1
Stigma -77-153
Auto Stigma
Auto Focus
Inst.Picture
0.41E-11 (A)
Quantitative Analysis
Preset Measurement
Group Name 6122010
Sample Name Demo1
Material Oxide
Correction Method ZAF
Accelerating Voltage 15.0 kV
Measurement Mode
No. of Preset Positions 1
Elem-1 Elem-2 Elem-3 Elem-4 Elem-5
Element Si K Mn Na Ca
Channel 1 2 3 4 5
Signal WDS WDS WDS WDS WDS
Crystal TAP PETJ LIF TAPH PETH
Acquire
Cancel
X100
50
100
Y
OML
OMTV
Albit Mg Fe Al Ni Benet Cu Ti ZRO2 Zr
Mo Cd W Au Ford Zn-16 Ge-17 GaAs Se-19 Zr-20
Memory
Memory 4
SPI-2

Downloading data from the Probe

Generating text files of microprobe results for data download:

1. From the EPMA Menu, choose Process. The Process menu appears.

2. Select Quantitative Analysis from the Process menu. A small window appears, with a couple option buttons.

3. Choose Summary. The Summary menu appears.

4. Ensure that the sample listed in the menu is the one you wish to compile data for. If not: Choose Sample. A window appears identifying Groups (folders) and samples in that Group.

5. Choose Group (a pull-down menu of Group folders appears) select your group, and close the menu. If a message appears asking if you want to keep the measurement conditions, select "Yes".

6. The samples in your Group should now be listed in the Sample window. Choose your sample (say Yes to the measurement condition message if it appears) and close the window.

7. Select Summary from the Summary menu. Three options appear; choose Summary again. A window appears that includes a listing for all points analyzed for your sample, and two sets of command buttons for different data reporting options. I typically choose Oxide wt. % in the top set, and Spreadsheet in the bottom one, but you can tailor the data report to your needs.

8. At the bottom of the window, choose Type Out. A black-background window appears in which your sample data is tabulated.

9. At the top of the window, select Save. A small window appears listing the directory, menu, and file of the saved file – the name of the file is always "Summary.tx", which is a default. I recommend changing the filename to a more recognizable title.

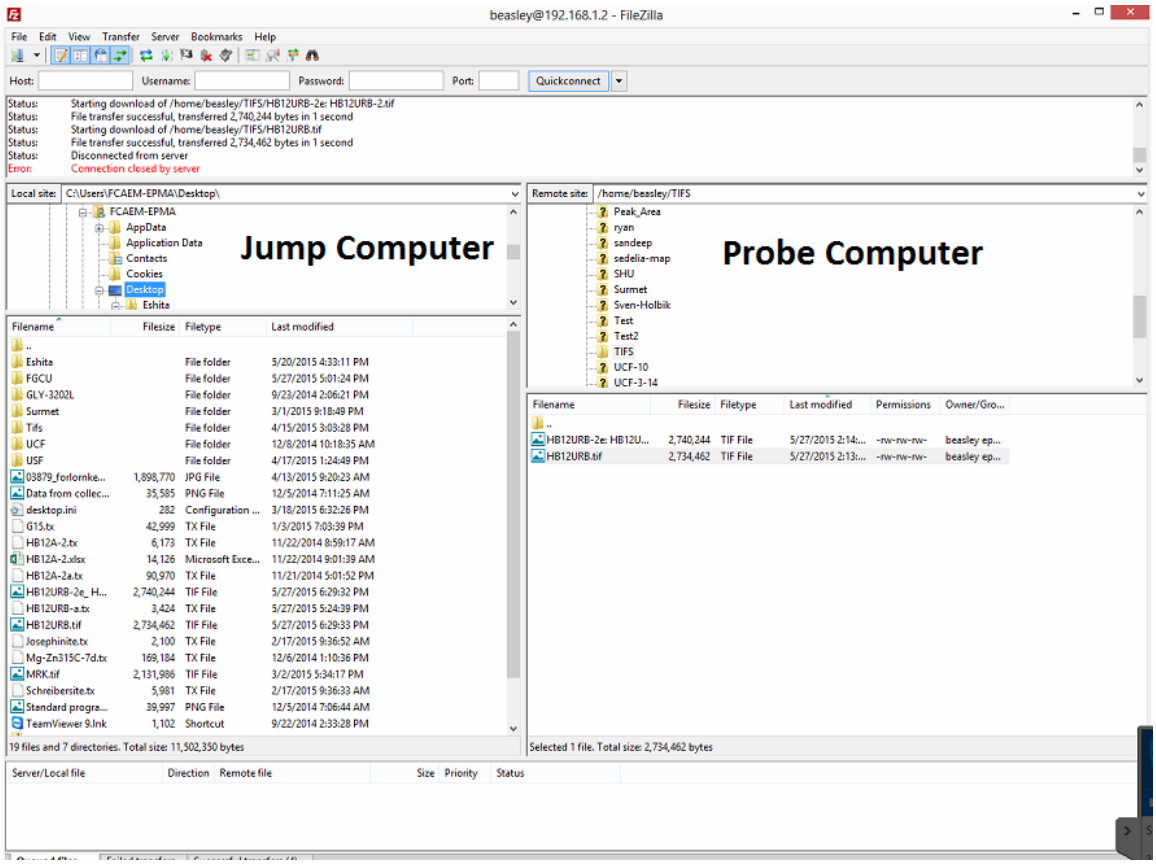
10. Select "OK" on this window to save the file, and close all the windows.

At this point you have generated a text file summarizing the data you collected on the **Probe computer**. The next set of instructions shows you how to extract the probe generated text file to the **Jump Computer** and ultimately to your computer.

1. To download data (to your own computer/possession) once saved in the quantitative files, it is recommended to use Filezilla to retrieve the data from the "Jump Computer". This is the computer you use TeamViewer to connect to. So on the desktop of the jump computer you should see a "FileZilla" icon, click on it to open the FTTP file sharing program.

2. Once FileZilla is open, input the host username and password picture below (the password is "tb100187").

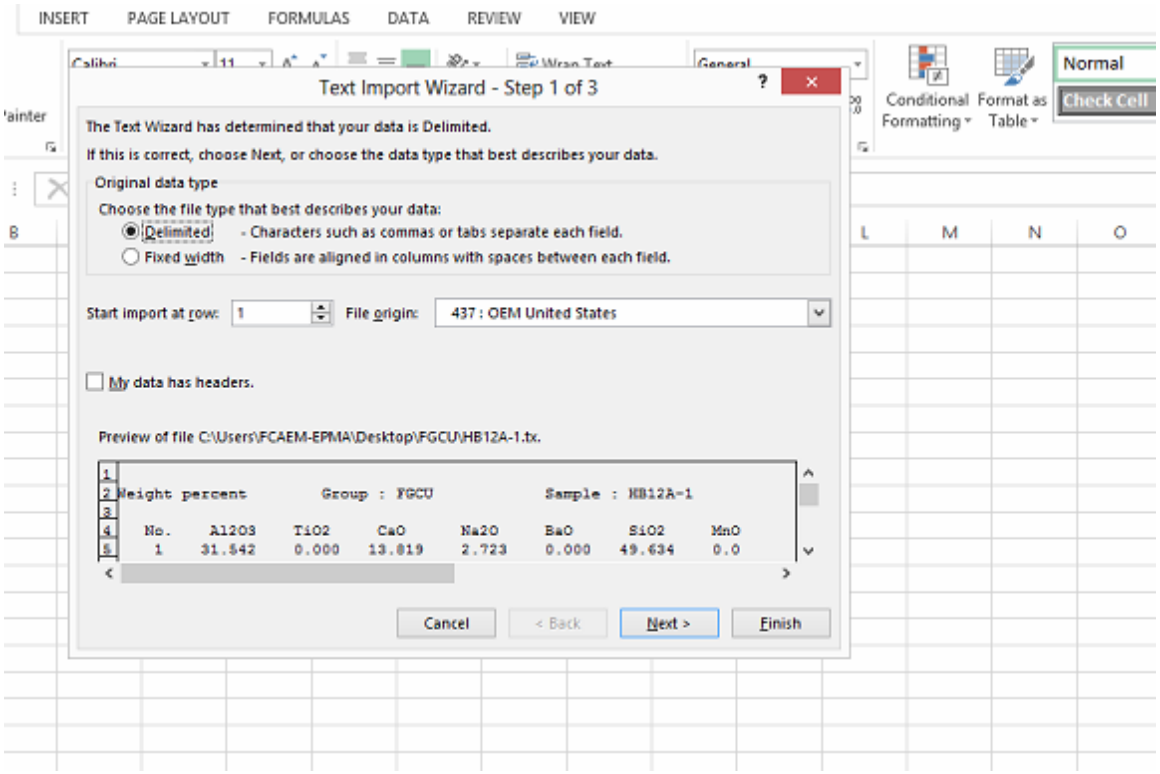
3. Now that you have established a Probe computer to Jump Computer connection, the file directory on the left hand side if the Jump Computer and the file directory on the right hand side is the Probe computer (see image below). Now find the text files that you previously generated using the probe software and copy them over to the Jump Computer. I suggest creating a folder for yourself on the Desktop or My Documents so that you can easily find where you saved your data and create a space where you will continue to save data from future collections.



To disconnect simple select Server and select disconnect. For future connection, simple select the pull down menu next to the Quickconnect icon and select beasley@emlab1.fiu.edu to restore connection.

4. Now that your text files and possibly images are on the Jump Computer, you can e-mail this data to yourself using the an internet browser on the Jump Computer or use a data uploading service like Dropbox to upload your data to a server which you can access from your work/home computer. The data is should now be in your possession! Enjoy!

5. If you want to load you text files into Excel, open Excel and open your .tx file. The "text import wizard" window will pop up. Select "Delimited" on step 1 and hit "next".



6. On step two of the "text import wizard", check the "Tab" delimiters and then click "finish". Your data should appear organized in the Excel sheet.