

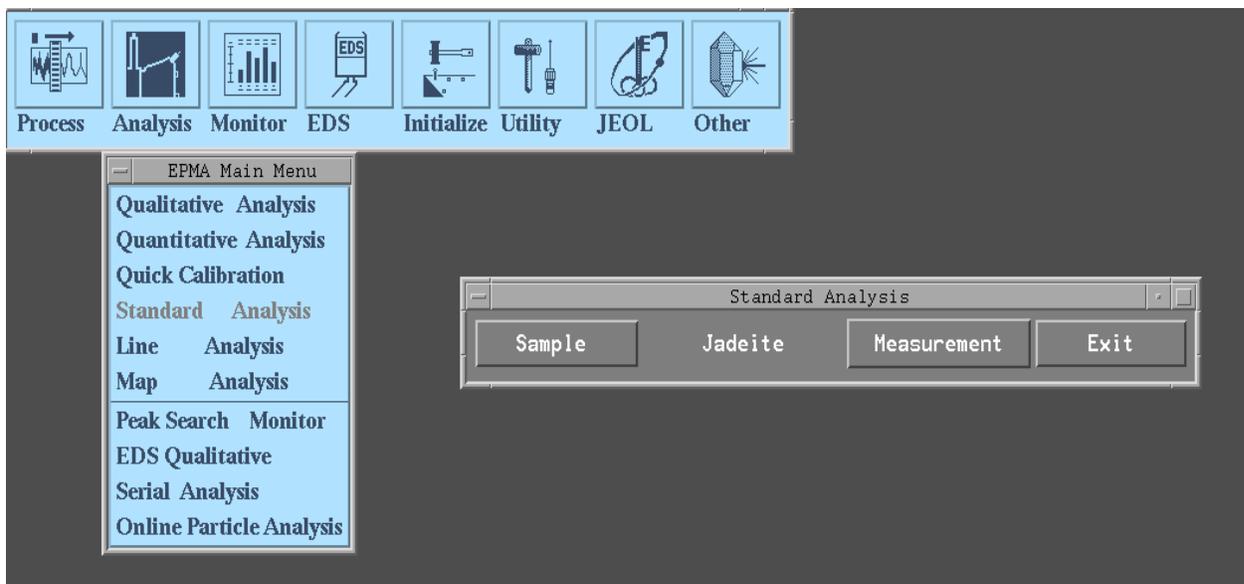
Standardization

Standardization is the most important operation after the instrument is calibrated. The process for standardization has a number of important factors associated with it. The first step is to identify the composition of the material in question if possible. If it is not possible visually or by having the data provided to you, it may be necessary to analyze the specimen with either EDS or WDS qualitative analysis. In many instances, the material to be analyzed is either synthetic or mineral so identification is relatively simple by use of optical means or knowing the synthesis process.

Standardization process

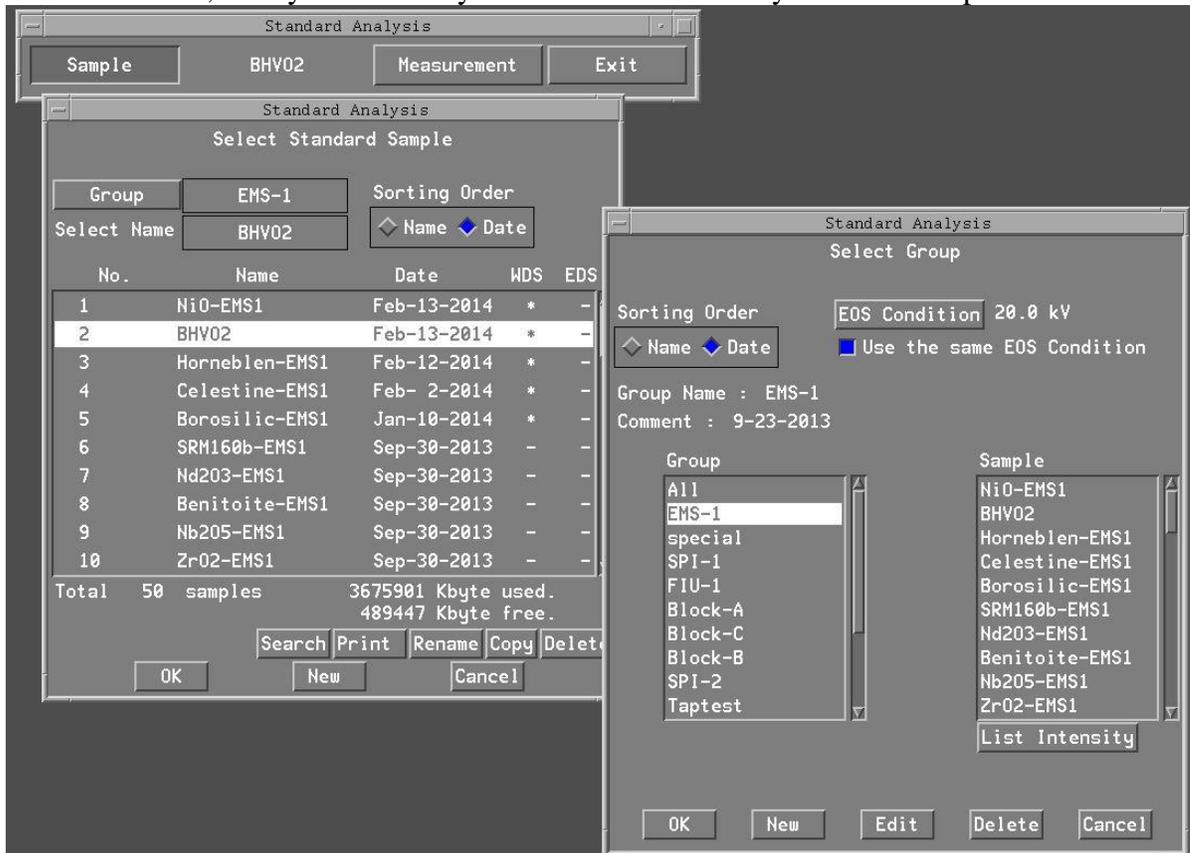
Section 1-initial set up

- On the EPMA Menu, select **Analysis** to get the pull down menu and scroll down to select **Standard Analysis**.

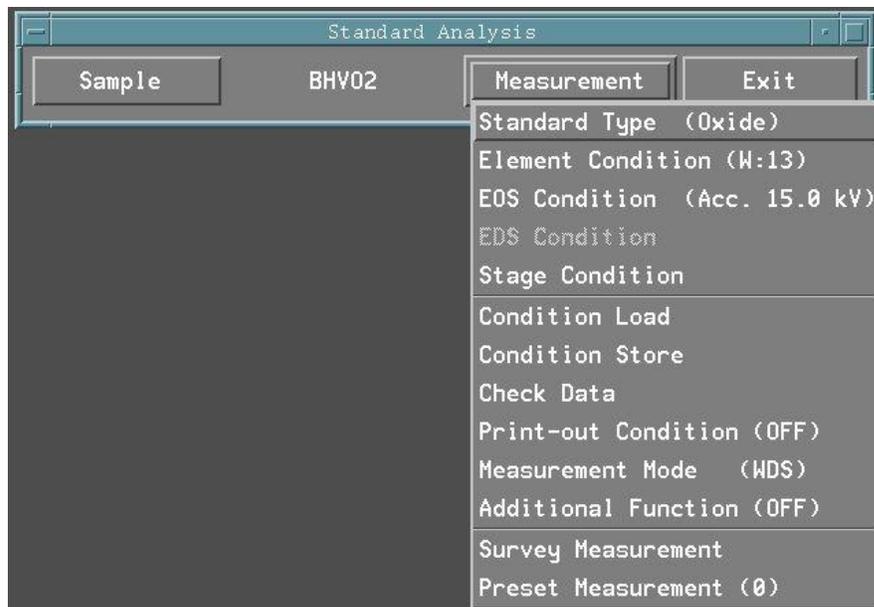


- Select Sample to choose the standard to work on, this selection will provide the “Select Standard Sample” menu for the selected group.
 - As a preliminary step, discuss the analytical needs so the available standard mount can be loaded on the stage while unknown samples to be analyzed are being loaded.
 - If the desired standard is not contained in the current selected group, select “Group” on the panel which will initiate a sub panel with a selection of all of the standards available at FCAEM.
 - Verify where the actual standard mount is located on the stage for measurement and calibration.
 - Find the location of the standard of interest standard map provided on the website of the standard mount to set up the calibration process for any of the standards needed

for the reference elements to be analyzed during the “Quantitative Analysis” software, it may be necessary to use the EDS to verify the elements present.

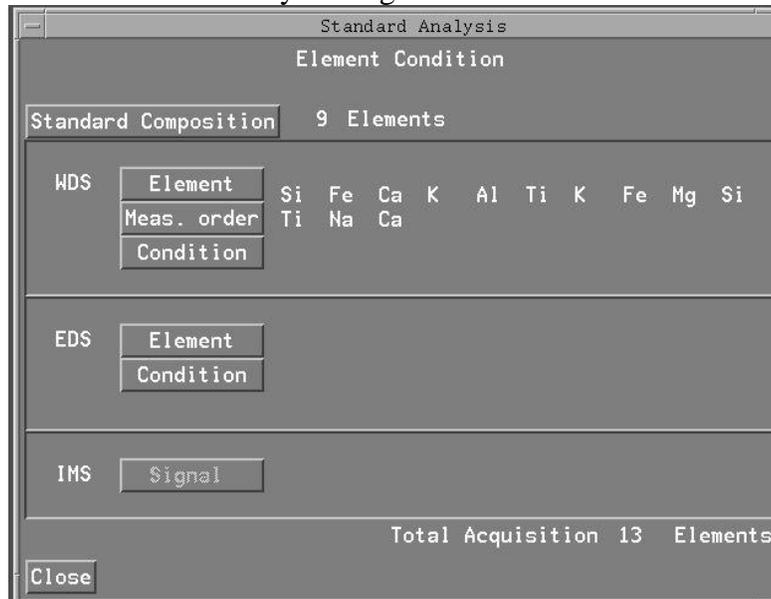


- Once the standard is selected and ready for analysis, select **Measurement** and observe the selections available.
 - The process for Standard Analysis is typical of most operations in the JEOL software package, begin at the top and work your way down to the bottom.



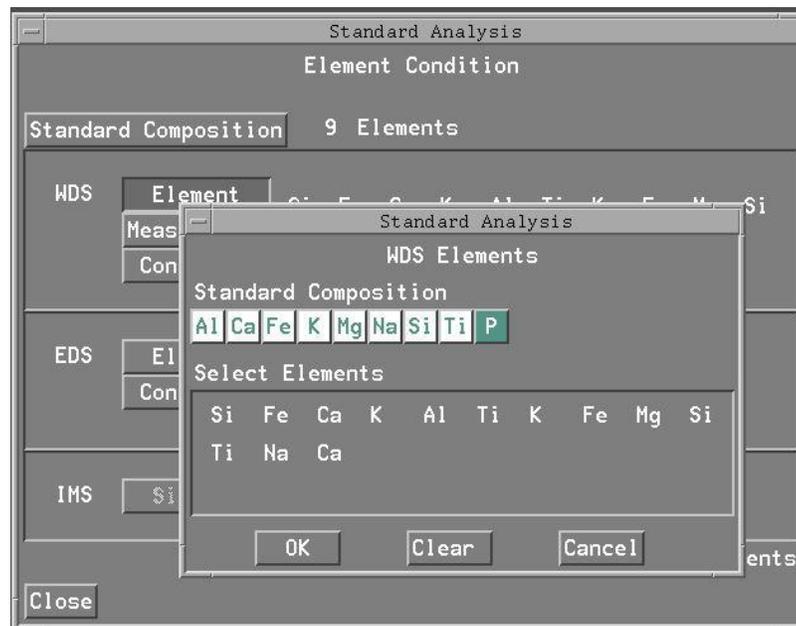
- First verify that the Standard type matches the type of analysis being performed, metal or oxide.

- Next, select **Element Condition**, this will initialize a panel with the element selection, WDS selection and crystal alignment.



Element

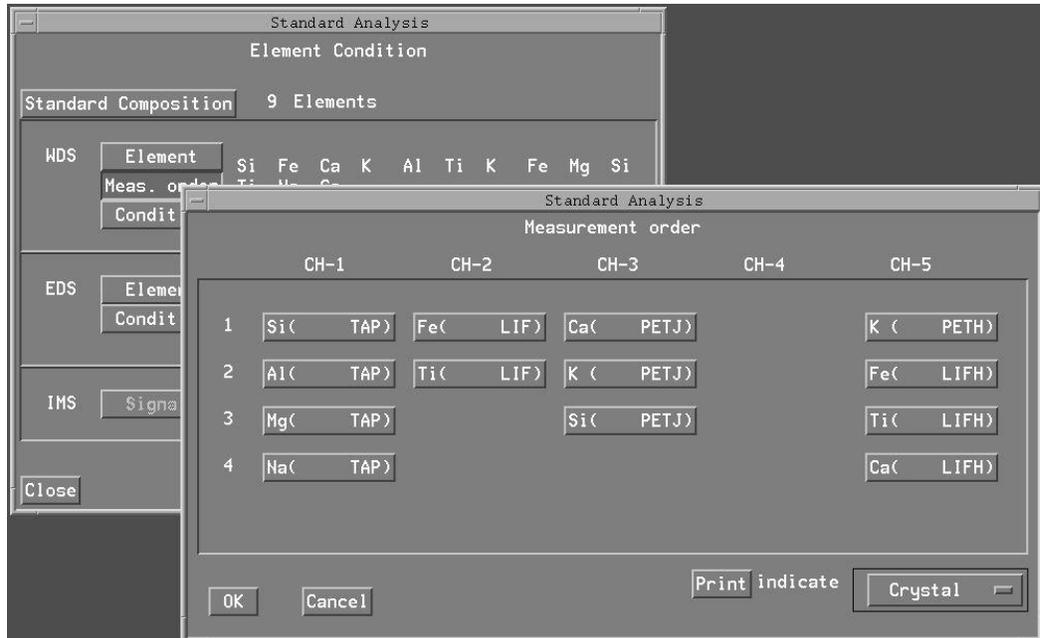
- Begin with Element to choose the elements required for the current analysis. Below is a typical geological selection.



- For standard analysis, an element may be repeated on additional spectrometers because of analysis management to minimize collection time, additionally, some elements may be analyzed on multiple crystal type.
- Once the elements of interest are selected, select OK to move to the next process.

Measurement order

- From the Element Condition panel, select Meas. Order to initialize the “Measurement Order” panel.

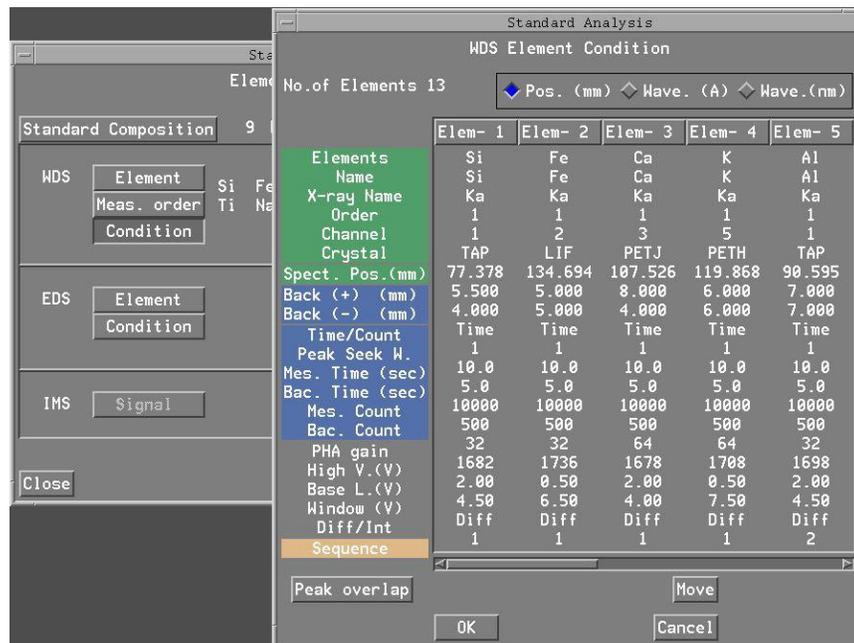


- This panel shows the element arrangement on each of the spectrometers; elements can be moved to appropriate spectrometers with crystals capable of x-ray collection.
- At the bottom right is an indicate panel to select the form the elements are displayed, crystal, wavelength in mm, angstroms or nanometers.
- Use crystal to select the crystal to be used for the element in question then wavelength in mm to arrange the order of collection which should be in consecutive order to reduce collection time.
- Try to reduce crystal flipping by selecting the like crystal selection on each spectrometer where possible as seen above.

Condition

- Condition is where the data of each standard is stored. Operations like change collection times, adjusting background points and all of the spectrometer settings can be viewed or changed when necessary. Care must be given in this area as peak data can be adjusted and cause incorrect analysis to occur.
- When selected, all of the elements which are to be analyzed will be listed here, verify by using the slide bar located near the bottom and make sure all of the elements are in “Diff”. Any element that does not have this can be selected at the top and will possible have the diff in this screen and if it is not or if any of the data disagrees with the initial screen, call the FIU manager.

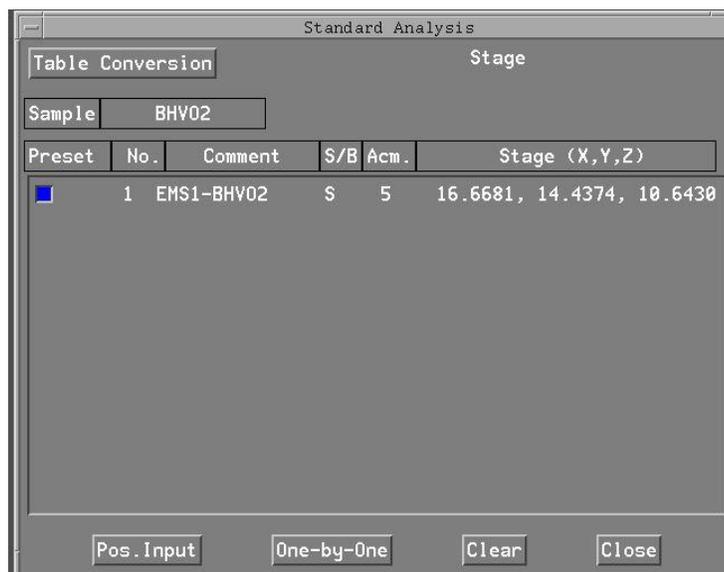
- This panel provides one additional service in that it will detect issues of peak overlap. By selecting this process, it can be shown if the background has interference peaks as well as the element peak. Some of the peaks can be adjusted on the background while major peaks cannot. If necessary, peak deconvolution software can be utilized to compensate for this type of interference if necessary.



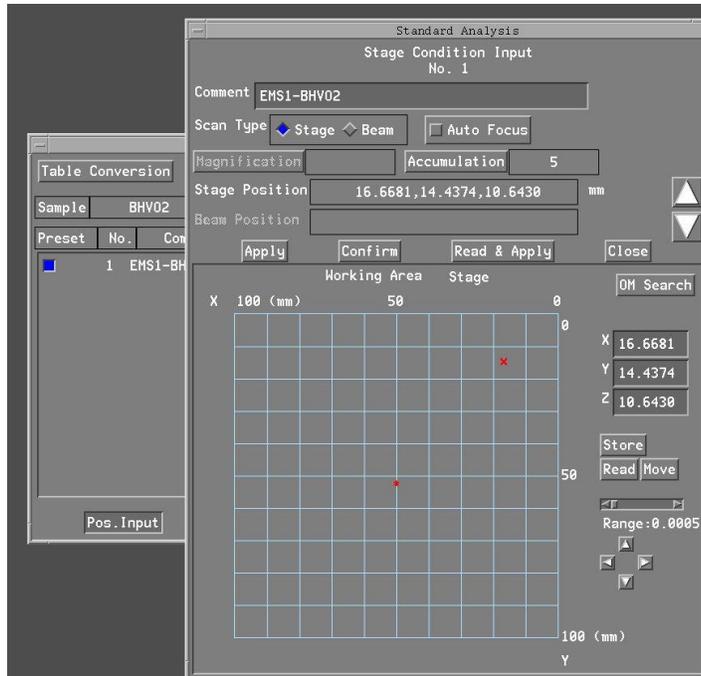
- Once this process is complete, the standard materials must be digitized so that the spectrum information can be collected and verified.

Stage Digitization

- Under “**Measurement**” select “**Stage Condition**” to initialize the stage digitizing software.



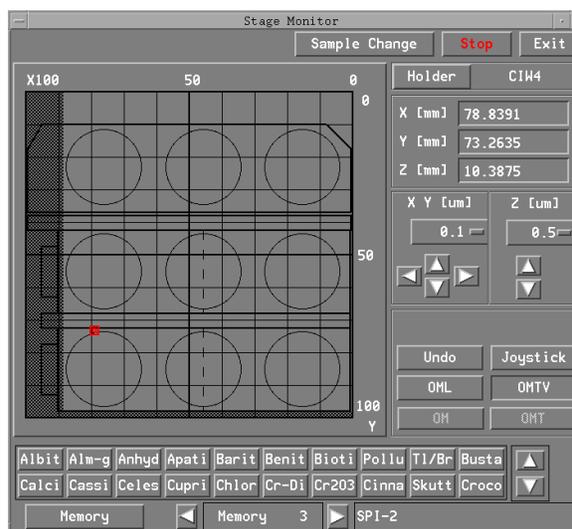
- From this screen, select “Pos. Input” to initialize the stage control panel which controls the points selected for analysis.



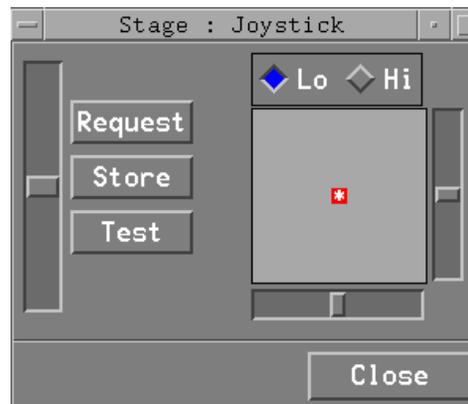
- The following procedure must be completed before the digitizing process can be completed.

Optical Focus

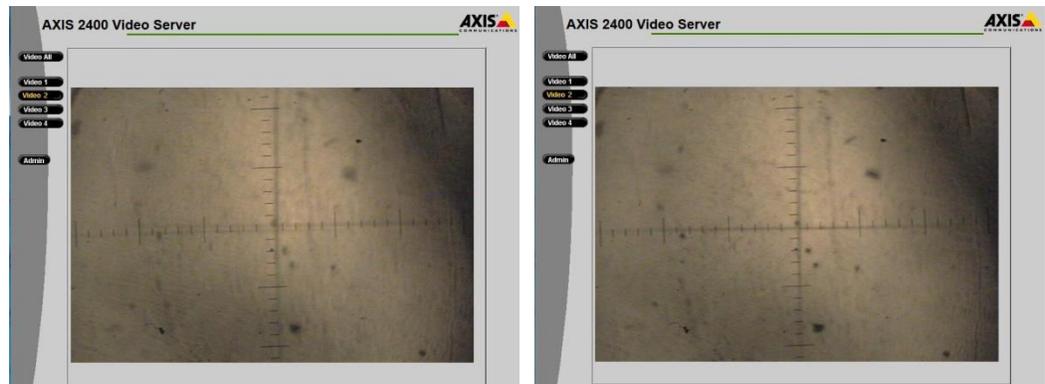
- Use the OMTV located on the EOS Monitor around the middle of the right side to control the lamp for focus of the optical microscope.
- It is extremely important that the optical microscope be focused for each point to be digitized. The controls for this are located on the Stage Monitor found under the Off-Line Menu under Monitor and Stage Monitor.



- Located on the right hand side toward the middle is the link to the “Joystick” used to control the stage operations.
- When selected, the Joystick screen will be small with the controls to operate the X and Y movement with the joystick and a slide bar located on the left side to control the “Z” which controls the optical focus.



- On the standard to be analyzed, using the optical focus must be in optical focus for good standard results, a set of crosshairs will help correct for parallax issues.



- The optical plain will show the flatness of the material being analyzed, if there is a flatness issue, it will be seen on the outer edges of the image and can affect the data if the crosshairs at the center cannot be focused.

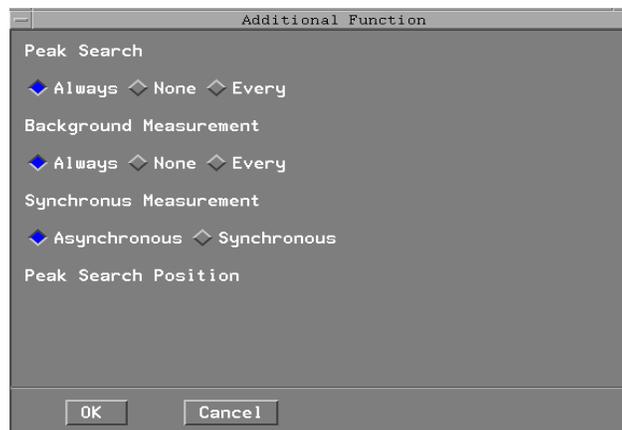
Digitizing process continued

- Once the optical is complete, continue with the digitizing process on the Stage Condition Input screen.
- On a point to be digitized and in optical focus, select “Read”, the screen points should align to one point and select “Store”.
- Verify that on Accumulation there is at least 5 in the number box, this will be then number of points collected for Standard Analysis. (Standard analysis differs from Quantitative analysis generally in this area, Standard Analysis averages standard data points while it is typical for Quantitative Analysis to look at points individually.)
- From this point select “Confirm” to digitize how ever many points that are selected in the Accumulation box.

- When “Confirm” is selected, move the stage to each location using the joystick control in the X or Y or both direction by confirming optical focus for each point and using the joystick “Store” button to store the locations, i.e. move to the first spot, select Confirm and then Store, move to another location and repeat the process until all of the points are stored at which time the stage will return to the original position.
- Upon completion, close the Stage Condition Input Screen at which time a Conformation box will appear to ask if the positions are OK to store, select OK if satisfied with the stored points.
- Close the final Stage control screen and continue.

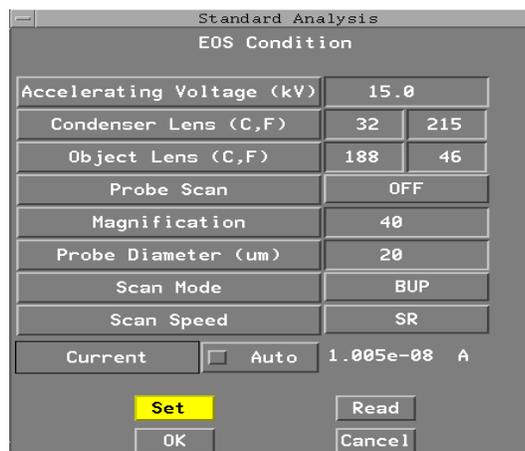
Last Steps to begin analysis

- Skip to **Additional Functions** and select, verify all modes set to Always, select OK.

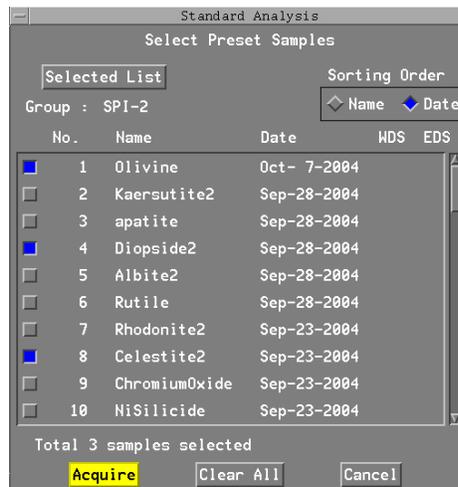


Repeat this process for each standard to be analyzed

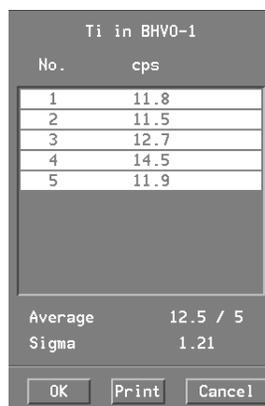
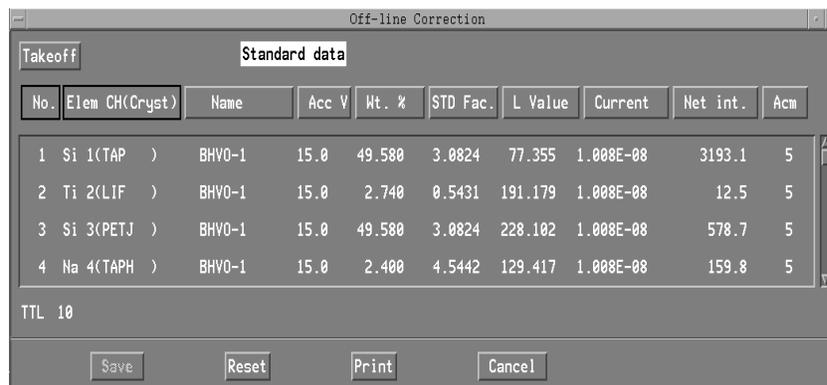
- When the last standard is completed, for all standards to have spectra collected, select EOS Condition, verify that the Scan Control has the correct configuration to run standard analysis and that the beam current is at the right current and the EOS conditions are correct and select Read. Verify that the readings update and close.
- For each of the standards to be analyzed set the EOS conditions making sure the current and column conditions are correct and save for each standard to be analyzed.



- When the column conditions are set, select **Preset Measurement**, and select all standards that are to be used and select **Acquire** to start the collection process.



- After the collection process is complete, it is important to verify that the data is reasonable.
- Select **Measurement** and **Check Data**. Select **Acm** to get the statistical data and verify that it is acceptable, good data is generally good between .01 to 40.
- If the data is considered poor where results are erratic, repeat the standardization process on the effected standards with new data point locations.



At this point, the standards are ready for use, close all standard related screens and select Quantitative Analysis software and confirm the standards by analyzing important primary standards against themselves to verify reliability.