EPMA Instructions for Thin Film Samples

General guidelines to reading computer related commands:
'Single quote' = menu item, window, or icon
"Double quote" = something you type
<carrots> = button you press on the keyboard
• = flag to set usually comes up as a dot in a circle when chosen
-dashes- = refers to a command given by the computer

☐ = button

Making an EPMA puck
1. Get a puck, 5 min epoxy, toothpicks, gloves, duster, Kim wipes, and your sample
2. Mix epoxy and use a toothpick to put the smallest drop possible on the puck (remember you have 5 minutes until the epoxy dries so don't dawdle)
3. Put your sample, thin film up, on the dot of epoxy
4. Repeat until all samples are on the puck
5. Flip your puck onto a Kim wipe for several minutes to let the epoxy dry
6. When the epoxy on your mixing surface is dry the epoxy on your puck will be dry. It is then OK to take the puck over to the EPMA facility

*****There should be a 1/16" area of unused area on the edge of the puck to accommodate the spacer you will need to use later*****

Scheduling Probe Time
Look at the probe schedules:
http://sweetwater.uoregon.edu/epma
http://sweetwater.uoregon.edu/sx100/

and pick an open day. E-mail John Donovan at donovan@uoregon.edu to request the time targeted.

## Mounting and Exchanging Samples
1. John usually gets in around 9:30 or 10:00 so you should arrive around then to get started unless you don't need these instructions

2. Put carbon paint on two corners of your sample(s) in order to make a conductive pathway from the surface of your sample down to the aluminum puck. You will find the carbon paint in the sample prep area. Use as little paint as possible and clean the brush with the acetone squirt bottle available in the prep area when finished! (Don't forget to wear gloves while doing this)

3. **Non-conductive films or non-conductive substrates must be carbon coated.**

4. Go to the sample prep area and find the sample holder with the 'J' block or the block with the standards you need. The standards themselves may be located in

the drawers around the seating area. They could also be in another sample block, including the one in the probe. The list of available standards is located in the binder that includes each user's runs.

    a. If the standard you need is located in the probe go to the 'Crosstalk' screen

    b. "ARLK" <return or enter> for SX50 or click Sample Exchange button for SX100

    c. <return or enter>

    d. –move gun to the position Crosstalk or PeakSight asks you to-

    e. When 'Crosstalk' or PeakSight tells you that the airlock backup is complete use the black lever on the sample interchange to open the interlock using the arrow out symbol ()→. To remove the sample you must push the sample insertion rod in but not all the way. Then pull the sample insertion rod out again. The sample should come with it. If it doesn't, keep trying. If you are very frustrated, find John.
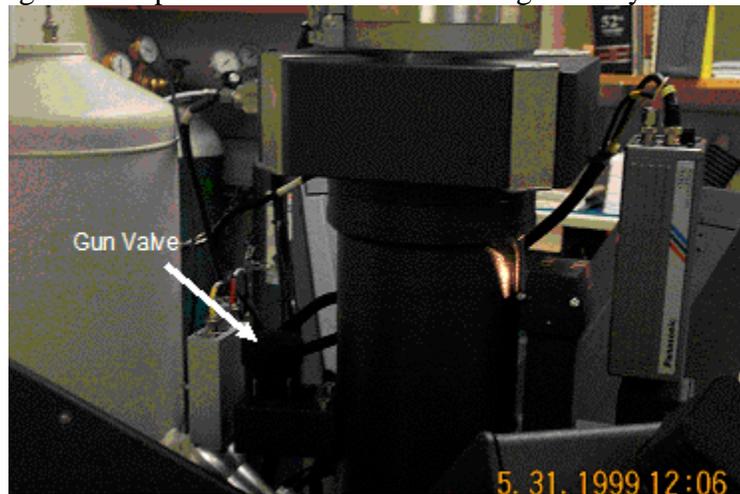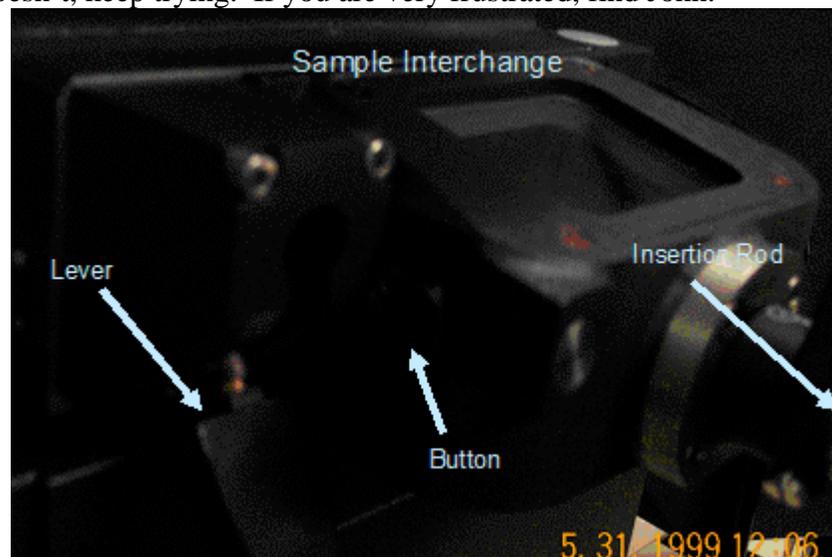

    f. Put the lever back to its original sealed position (push in, turn clockwise, then push back to the down position)

    g. "pump" <return or enter>

h. Use the button on the side of the interlock to open it and remove the sample holder

5. Put a spacer on the puck then put it in the sample holder and secure it in place with the set screws on the side.

6. If they are not already in, put the standards needed on the block.

7. Pull a sample map from the user log binder and make a drawing of where your sample is on the map. This map will come in later. Also put your name, the lab name, sample designation, and index to be charged on the paper so that the proper grant can be billed for the instrument time.

8. Take the sample holder to the blue oven held at 60ºC and put it in for two minutes. (This outgases any solvent left in the carbon paint and equilibrates the temperature of the sample holder to the approximate temperature the puck will experience in the EPMA).

9. Remove the sample holder from the oven and put it into the interlock. (See above for instructions on removing old samples in the probe)

10. Type "Arlk" <return or enter> for SX50 in CrossTalk window, click Sample Exchange button for SX100 (under Vacuum tab of Peak Sight)

11. Wait for the airlock back-up is complete message then open the interlock using the airlock valve lever and put the sample into the chamber using the sample insertion rod set to arrow in mode →(). Then close the interlock.

12. "pump" <return or enter> for SX50, click "Continue" for SX100

13. Wait approximately five minutes and follow directions on the screen.

14. When the vacuum is ready "mov stag ref" for SX50, or right click small sample map in PeakSight software (left side of screen) and click Move to Reference for SX100

15. Focus the arrow on the left hand side of the view screen using the Z control to the right of the keyboard. Make sure crosshairs are in the center of the cross on the screen. If there is no cross on the screen get help. Something has gone awry.

**** These instructions are only valid if you are not going to take a long time getting your sample ready to go into the instrument. If you are taking too long putting the sample in you will find that there will be instructions appearing in the Crosstalk window. Follow them. You may need to use the orange tab instructions in the user binder if that is the case. The instructions in the Crosstalk window are always right.****

Using the Probe (with an experimental set-up already containing your standards)

1. Close everything except for 'Crosstalk' on the screen
2. Open 'Probewin'
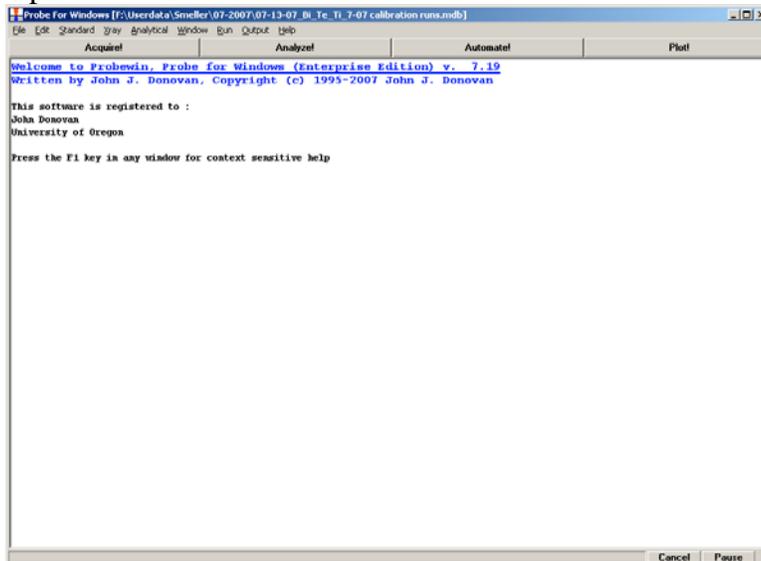
3. When prompted "Do you want to interface to the microprobe" click 'YES'
4. 'File' then 'New'
5. Name the file appropriately for your activity and put the file in a folder that makes sense to you. You may also wish to change the sample identifications and who is operating the probe.
6. You will now load an old file set-up for the same elemental standards you are running by going to □Acquire!. In the new 'Acquire!' window go to □New Sample, □Load File Set-up, then navigate to the old file (use the most recent file with your standards) and click □OK. This will establish the sample setup and standards you wish to run include. Then click □OK again.
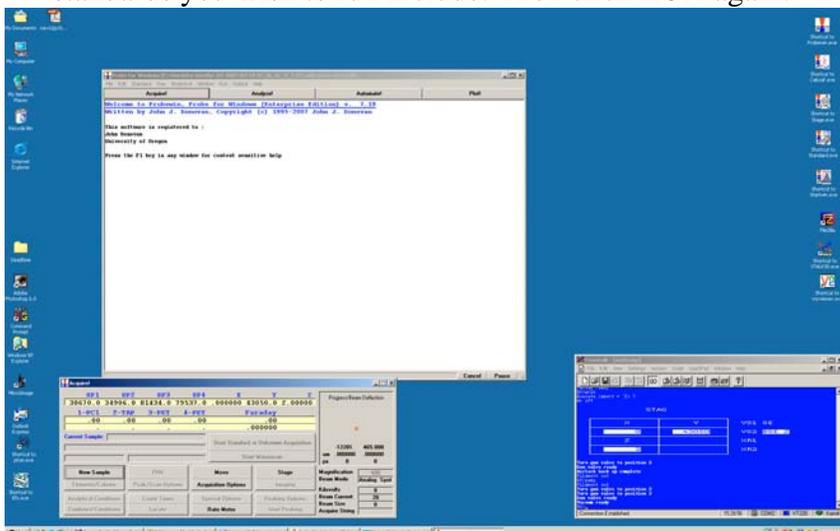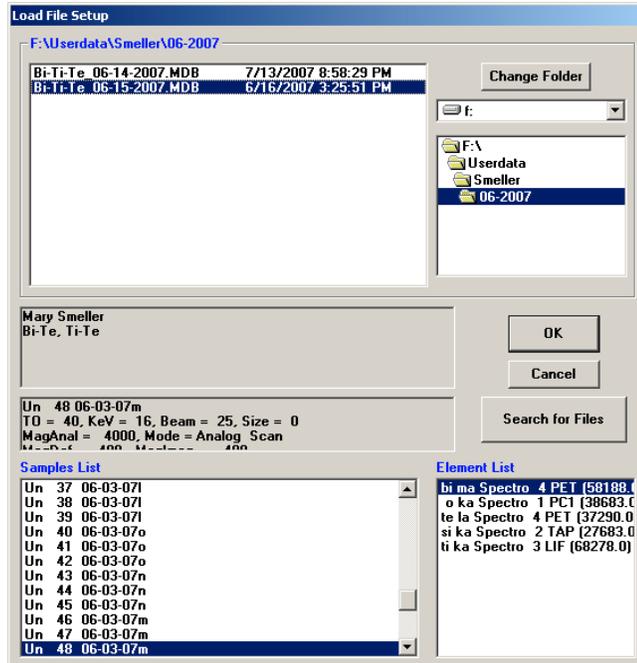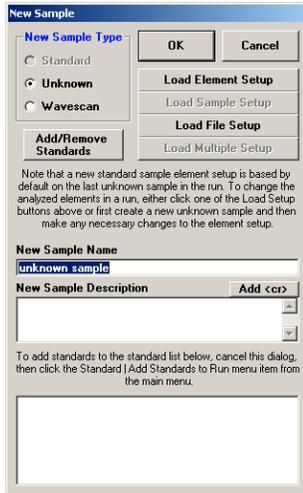
7. When prompted "Do you want to use the old standard intensities," DO NOT USE THE STANDARD INTENSITIES FROM LAST TIME.
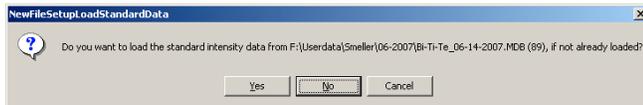
8. In the 'New Sample window', type in the one of the voltages you intend to use in your run (10keV is common). You should see the standard samples in grey located in the bottom white box in the New Sample window if you have added the file setup correctly. If you are unsure of which voltages you want, you can model your sample to find out what might work using Stratagem. Get some help in order to do this. Pick as many energies as you need but make sure that is at least three. You will be modeling those three energies in order to find the best compositional fit for the data.
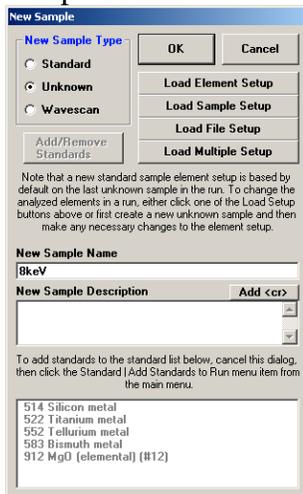
9. In the 'Probewin' window go to ☐Automate!. Then choose ⦁Unknowns and if there are samples left from the previous run choose ☐Delete Selected Samples and ☐Yes to All.

10. In the 'Automate!' window, click on ⦁Standards to make sure you have the correct standard block files in the computer. If it does not include your standards, get John to show you how to find the correct standards file to load.

11. Now select the standards you will be using for your experiments (hold the ctrl key while clicking on each one) and look to the right hand column where you will choose ⦁Confirm Standard Positions and ⦁Peak Spectrometers. Then click the ☐Peaking button. This will bring up another window, click on ⦁ROM based and ⦁Acquire Automated PHA Scan After Peaking. Once these options are selected click ☐OK followed by ☐Yes. Look in the 'CrossTalk' window to see if the beam is on. If it is on, you will see a series of amperage readings measuring the beam. If it isn't, use the 'Quickpad' and hit the ☐HV15 button.
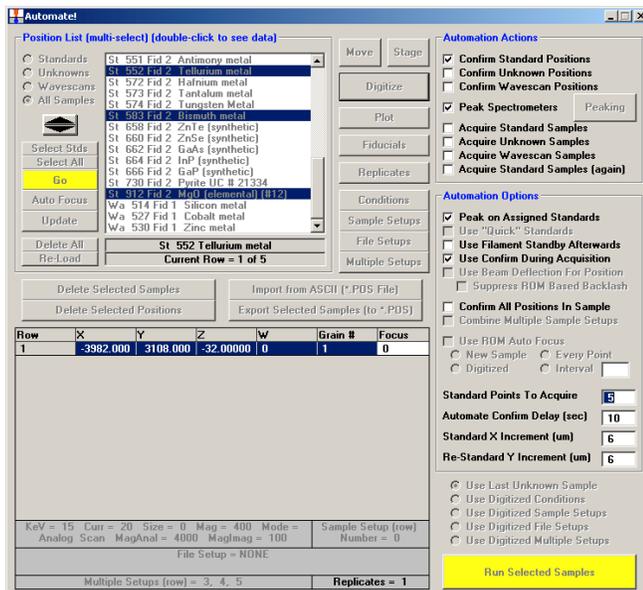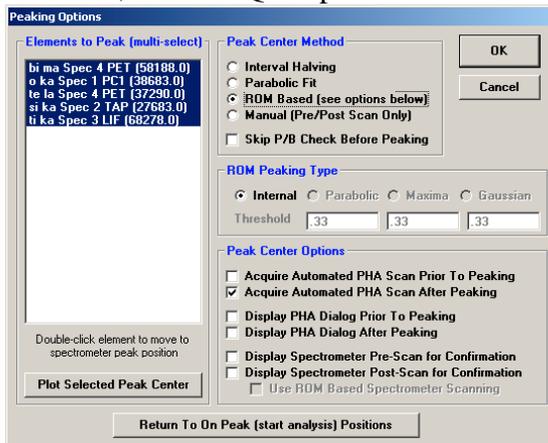
12. In the 'Automate!' Window, click on ☐Run Selected Samples followed by ☐Yes. This will run a series of scans that will allow you to confirm (and refocus) the location of the standard on the block you are using, find the maximum intensity of the peak, take a scan of the peak itself, and compare what your intensity value was before and after you peaked the spectrometer. The focus and location can be changed using the dials labeled X, Y, and Z to the right of the keyboard. You should see a black arrow superimposed on the left hand side of the screen. If the arrow is in focus, so are you on the sample. Once the position on the standard is established click on ☐OK in the countdown window that comes up when it goes to the standard.
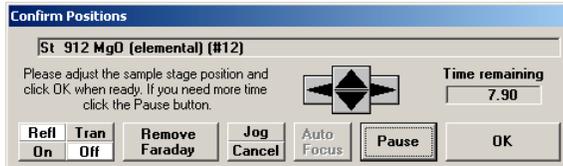

Information about the peak intensities will be located in the 'Probewin' window. If there are any major changes in your peaked values, both on or off peak, you should get help from John. Also check to make sure you only have one peak in the PHA scan when you peak the spectrometer. If there is more than one get John. This should be done before every run.
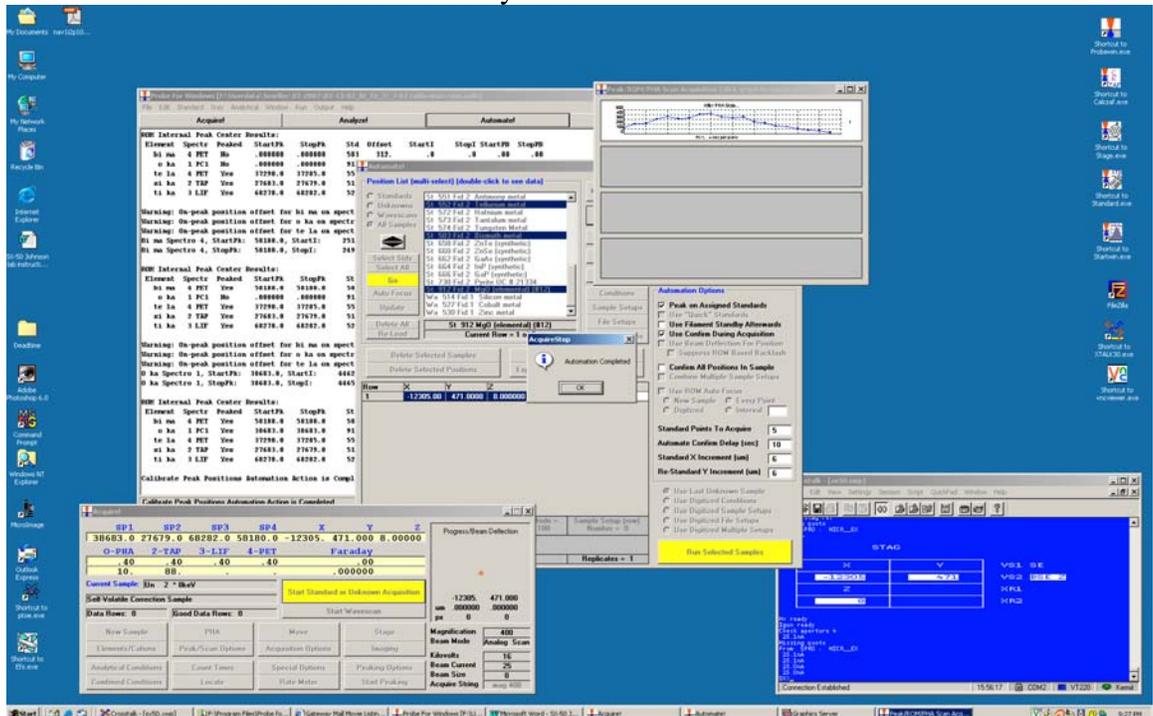

13. Go back to the 'Automate!' Window and click on ☐Digitize. You will now put digital positions in for the computer to reference. Because these samples are thin films you will be taking several data points in a line and summarizing the information you learn from then before sending the information into a modeling program to learn composition.

14. In the 'Digitize Sample Positions' window choose •Unknowns and put a sample name in where you see   -Unknown Sample- or a previous sample name you

typed in and click □Create a new Unknown or Wavescan then click on the □Stage button to bring up the 'Stage Map' window.
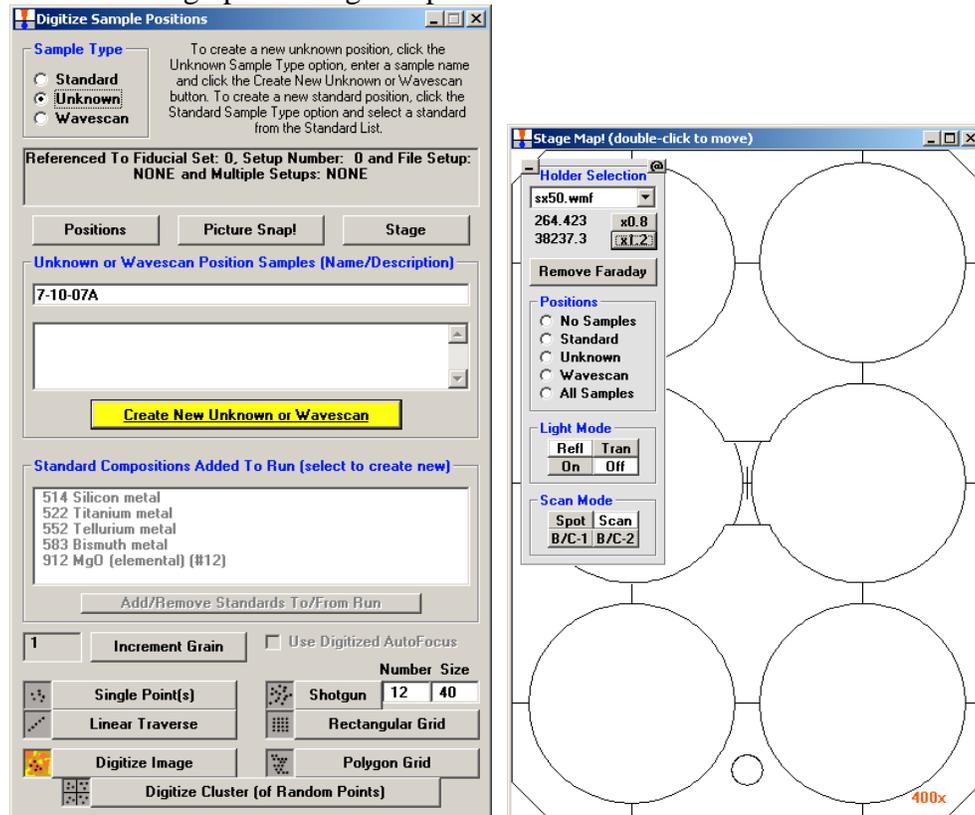
15. In the 'Stage Map' window double click on where you think your sample is (based on the drawing you made earlier), focus the arrow on the viewing screen and find a region of the sample that is clean of dust or rough spots. This is done using the X and Y controls to the right of the keyboard in conjunction with Z. You may need to refocus the arrow when you go from one side of the sample to the other.

16. In the "Digitize Sample Positions' window click on □Linear Traverse. Follow the line of clean sample you have then click □Update Stop in the 'Linear Traverse' window. You will need to enter the number of points or steps in that line you wish to study. Most people use between 6 and 8 steps, while ensuring that the steps are ~100μm apart. After the number of steps has been filled in once for the experiment it will continue to appear for the following samples. When you are satisfied click □OK.

17. Repeat steps 14 – 16 until all of your unknown samples have been entered into the file setup.

18. In the 'Stage Map' window choose to show the locations of the unknowns to double check that you are not looking at the same sample twice or missing a piece that you wish to study.

19. Ask John to come in and review your spectrometer conditions (count times on each of the spectrometers and count times on and off peak) before moving forward. If you want to make changes at this point it will be easier than if you proceed to the next step.

20. Go back to the 'Acquire!' window and choose □New Sample, type a new sample name in and click □OK. Then add the other energy(ies) you wish to run at one at a time using the same process.
21. In 'Probewin' open the □Analyze! window. In the 'Analyze!' window, click on the each energy then go to □Conditions and make sure that the energy of the sample matches the label it carries. Make sure you are not on the •Analog Line Scan but instead are in •Analog Spot mode. Once you have corrected your voltages, select all of the energies and □Add to Set-up. Then click on □OK.
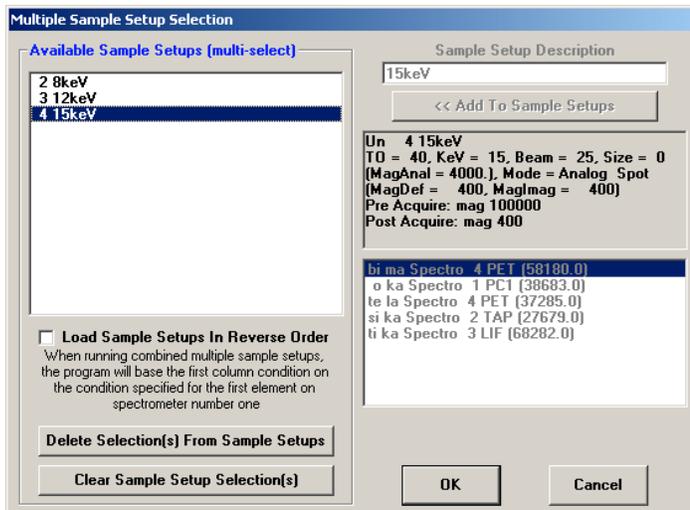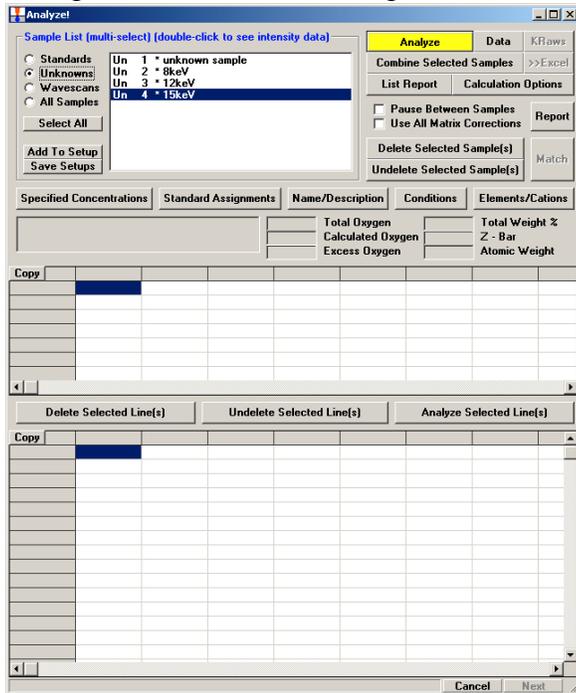
22. In the 'Automate!' window choose the •All Samples flag. Then hit the □Select Stds button and establish that you picked the correct standards for your samples. Then holding down the <control> button choose the unknowns you wish to

measure. In the picture below note that Multiple Setups = NONE in the bottom line of the Automate! Wintow. Now, go to the □Multiple Setups button. Select the voltages and click □OK then □Yes. Once you have applied the Multiple Setups the Multiple Setups = NONE should change to something like Multiple Setups = 1, 2, 3.
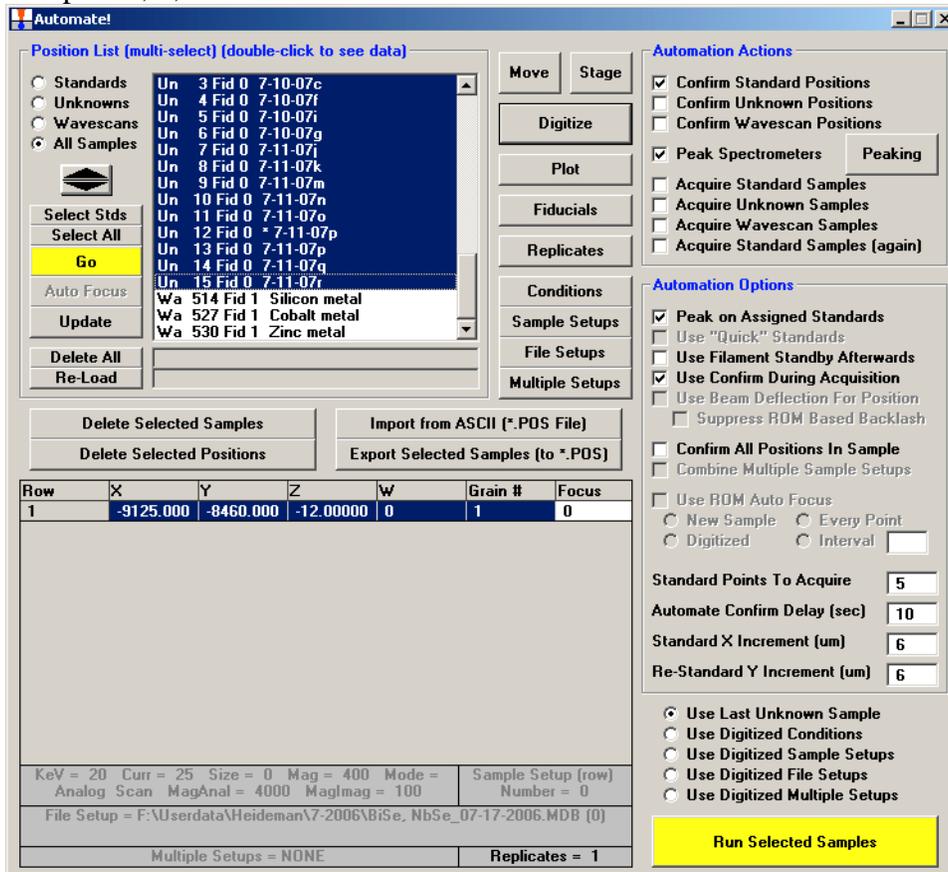
23. Click the following flags: •Confirm Standard Positions, •Acquire Standard Samples, •Acquire Unknown Samples, •Acquire Standards Again, and •Use Filament Standby Mode Afterwards, •Use Confirm During Acquisition, and •Use Digitized Multiple Setups. See picture on the next page for confirmation.

Automate!

Position List (multi-select) (double-click to see data)

- ○ Standards
- ○ Unknowns
- ○ Wavescans
- ● All Samples

Un   3 Fid 0 7-10-07c
Un   4 Fid 0 7-10-07f
Un   5 Fid 0 7-10-07i
Un   6 Fid 0 7-10-07g
Un   7 Fid 0 7-11-07j
Un   8 Fid 0 7-11-07k
Un   9 Fid 0 7-11-07m
Un 10 Fid 0 7-11-07n
Un 11 Fid 0 7-11-07o
Un 12 Fid 0 * 7-11-07p
Un 13 Fid 0 7-11-07p
Un 14 Fid 0 7-11-07q
Un 15 Fid 0 7-11-07r
Wa 514 Fid 1 Silicon metal
Wa 527 Fid 1 Cobalt metal
Wa 530 Fid 1 Zinc metal

Select Stds
Select All
Go
Auto Focus
Update
Delete All
Re-Load

Move | Stage
Digitize
Plot
Fiducials
Replicates
Conditions
Sample Setups
File Setups
Multiple Setups

Delete Selected Samples | Import from ASCII (*.POS File)
Delete Selected Positions | Export Selected Samples (to *.POS)

Row	X	Y	Z	W	Grain #	Focus
1	12913.00	21516.00	81.00000	0	1	0
2	12983.40	21740.40	81.80000	0	1	0
3	13053.80	21964.80	82.60001	0	1	0
4	13124.20	22189.20	83.40001	0	1	0
5	13194.60	22413.60	84.20001	0	1	0
6	13265.00	22638.00	85.00002	0	1	0

KeV = 16   Curr = 25   Size = 0   Mag = 400   Mode =
Analog Scan   MagAnal = 4000   MagImag = 400

Sample Setup (row)
Number = 0

File Setup = NONE

Multiple Setups (row) = 2, 3, 4 | Replicates = 1

Automation Actions
- ☑ Confirm Standard Positions
- ☐ Confirm Unknown Positions
- ☐ Confirm Wavescan Positions
- ☐ Peak Spectrometers   Peaking
- ☑ Acquire Standard Samples
- ☑ Acquire Unknown Samples
- ☐ Acquire Wavescan Samples
- ☑ Acquire Standard Samples (again)

Automation Options
- ☑ Peak on Assigned Standards
- ☐ Use "Quick" Standards
- ☑ Use Filament Standby Afterwards
- ☑ Use Confirm During Acquisition
- ☐ Use Beam Deflection For Position
- ☐ Suppress ROM Based Backlash
- ☐ Confirm All Positions In Sample
- ☐ Combine Multiple Sample Setups
- ☐ Use ROM Auto Focus
  - ○ New Sample   ○ Every Point
  - ○ Digitized   ○ Interval [   ]

Standard Points To Acquire [5]
Automate Confirm Delay (sec) [10]
Standard X Increment (um) [6]
Re-Standard Y Increment (um) [6]

- ○ Use Last Unknown Sample
- ○ Use Digitized Conditions
- ○ Use Digitized Sample Setups
- ○ Use Digitized File Setups
- ● Use Digitized Multiple Setups

Run Selected Samples

24. Double check that everything is just so and click on □Run Selected Samples. This will bring up a time estimate.   Click □YES and the run will start.
25. The standard positions will be checked again. Make sure they are still in focus.

Establishing a new file setup

If you are not running an old file setup because you are using a new set of standards for your new compound, you will need to do the following with John's help:

1. Wavescans to check for interferences between the elements in your sample. This will also help establish where you will be measuring on and off peak intensities.
2. Do a simulation of the samples you will be running in Stratagem to pick the best operating voltages.
3. Establish which spectrometers should be used for which standards. You will want to avoid crystal flipping. Put in proper count times for what you are doing.
4. New file setups should be saved once you have finally made them.

Common Problems

The filament is not on when you go to do a scan. The gun is usually off when the instrument is idle and always off after a sample exchange.

    a. Go to the 'Crosstalk' window and find the 'QuickPad' window above it that comes has a teal background. Use the ☐HV15 button. 'Crosstalk' should tell you that the Igun is ready, then finally Setup ready.

    b. If this does not turn the filament on and start sending information to the 'Crosstalk' window then get John to help.

2. The sample holder is difficult to get out of the instrument when you go to exchange for your new sample holder.

    a. The holder has a clicking mechanism that locks it into place on the exchange device. If you push in too far you disengage the hold of the mechanism. When you go to pull the sample out don't put the exchange rod in the entire way. Just go until you feel resistance. This should click the mechanism so you can pull the sample holder out.

    b. Do not force this process. If you are having trouble, get John to help.

3. You accidentally put two linear traverses on the same unknown sample or skipped a sample you meant to run.

    a. Use the 'Stage Map' window displaying the unknowns to show where all of your linear traverses are. Figure out which numbers correspond to the unknowns you want.

    b. Go to the 'Automate!' window and double click on the sample you need to alter. The linear positions you have digitized should come up in the box below the sample position list. Choose the duplicates and click on ☐Delete Selected Points then ☐OK.

    c. If you have skipped one go to the 'Automate!' window and choose the sample you wish to alter then go back to the 'Digitize Sample Positions' window and choose ☐Linear Traverse. You can now go about business as normal and add the points you want.