

# **EMPA Instructions for Geological Samples**

## **Modified after Johnson Lab Thin Film Instructions**

### **Carbon Coating Procedures**

For Edwards Coating System E306A

1. Clean polished samples with ethanol and wipe clean.
2. Put samples in oven for ~ 1 minute to dry (optional).
3. Put a fresh piece of filter paper and clean brass stud on plate.
4. If necessary, take out carbon rods and sharpen. (Left-side needs to be flat on the end and the right-side needs to be sharpened ~2-3 mm back and then flattened on end.)
5. Put carbon rods back and tighten. (When tightening the right-side, push the spring back and push the carbon rod in, tighten while the spring is loaded so that it will push the carbon rods together.)
6. Place samples on filter paper.
7. Put bell jar in place over samples.
8. Place implosion guard over top of bell jar.
9. Turn off airflow by pushing AIR ADMIT button (light turns off)
10. Turn handle on front of coater counterclockwise to the ROUGHING position.
11. The pressure dial (found at the top-right of the instrument panel) will go up and then will slowly go back down.
12. Once the pressure dial reaches the red arrow, rotate the handle clockwise to the BACKING position, then pull the handle out towards you and continue rotating it clockwise (go slowly so that the needle does not rise above the red arrow) until it is in the OPEN position.
13. Let sit for ~20 minutes.
14. After 20 minutes, SLOWLY turn the rotator knob (top left on instrument panel) until the plate is spinning.
15. Press the LT button.
16. Turn the POWER CONTROL knob (bottom left on panel) until the carbon starts sputtering. (You want to do this slowly to keep it from sputtering too fast.)
17. Let it sputter until the brass stud turns purple (corresponding to a carbon coat  $22\pm 1$  nm). The brass stud will first turn orange, then red and then purple.
18. As soon as the stud turns purple, turn the POWER CONTROL knob back to zero.
19. Turn the rotator knob back to zero to stop the plate from spinning.
20. Turn off LT.
21. Turn handle to BACKING position (counterclockwise).
22. Push AIR ADMIT button to allow air back into the chamber.
23. Remove implosion guard and bell jar.

## **SX-100 Insert/Exchange Samples**

1. Make sure specimen is clean and dry (place sample in oven for ~ 1 minute).
2. Using the KVM switch (located under the monitors) make sure computer 1 (Cameca computer) is selected. (If the KVM switch is “stuck”, press the spacebar on the computer).
3. In the SX CONTROL window click the “Vacuum” tab.
4. Click the “Sample Exchange” button and then click “Yes” to confirm sample exchange.
5. In the SX CONTROL window it will give you a series of directions to follow:
  - a. “Turn gun valve to position 1”: Turn valve clockwise to position 1. (The gun valve is found on top of the instrument toward the back. You will need to pass position 3 to get to position 1). Wait until it says “Airlock Backup Completed”.
  - b. “Open airlock gate valve”: The handle is located just to the left of the viewing window. Push it in, lift it up, and release.
  - c. “Move in/out shuttle”: Make sure the correct direction is selected using the switch on the outside of the shuttle. Move the shuttle all the way in and then all the way out. Depending on which direction is selected, it will either grab or release your sample.
  - d. “Close airlock gate valve”: Turn handle clockwise, push down, and release.
6. Click CONTINUE when the airlock gate valve is closed.
7. To remove your sample, open the airlock door by de-pressing the black button on the side latch.
8. After about 1 minute it will continue to prompt you with directions:
  - a. “Turn gun valve to position 2”: Turn valve counterclockwise to position 2 (You will need to pass position 3).
9. In the SX CONTROL window it will indicate that the vacuum is ready after about a minute or so.

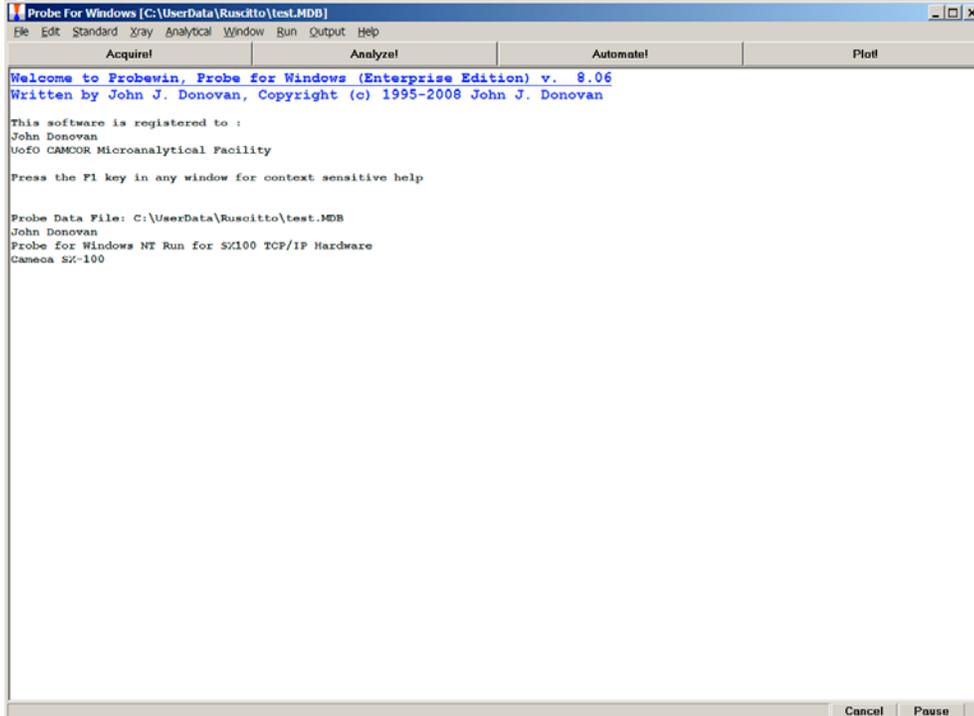
## **Resetting Stage Reference Point:**

1. Switch to the CAMECA computer using the KVM.
2. Right-click on the “Position” window (lower left of display) and choose “Move to Reference”. This will move the stage to what the computer thinks is the reference point.
3. Use the joystick and Z-button to focus on the bright spot near the center of the cross etched into the sample holder. Right-click on the “Position” window and choose “Update Reference”, then “OK”.

## Setting up the Probe for a Run:

This guide is intended for a quick-start and assumes that a setup has already been created and just needs to be loaded.

### 1. Open Probewin.exe (Probe for Windows)



2. When prompted “Do you want to interface to the microprobe” click ‘YES’
3. Create a new probe run by selecting File >> New.
4. 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.

File Information				
File Name	C:\UserData\Ruscitto\test.MDB			
Version	8.06	Type	PROBE	OK
User	John Donovan	Cancel		
Title	Probe for Windows NT Run for SX100 TCP/IP Hardware			
Department	CAMCOR			
Account #	Group	MicroAnalytical Facility		
Description	Cameca SX-100			
Date Created	11/14/2008 9:50:57 AM	Date Modified	11/14/2008 9:50:56 AM	
Last Updated	11/14/2008 9:50:57 AM			

- Next, load an old file setup for the same elemental standards you wish to run by pushing 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. Then click OK again.

SP1	SP2	SP3	SP4	SP5	X	Y	Z
38490.0	81480.0	41175.0	24068.0	48077.0	-15663.	-9204.0	-28.000
1-TAP	2-LPET	3-LLIF	4-PET	5-LIF	Faraday		
30.00	20.00	5.00	5.00	45.00	1.00		
1533.	2059.	498.	611.	310902.	49.9282		

- When prompted "Do you want to use the old standard intensities," DO NOT USE THE STANDARD INTENSITIES FROM LAST TIME.

7. Load the column conditions using the 'Probewin' log window menu and choosing Window >> Load Column Conditions. Sort the files by DATE and choose the most recent one. Change the analytical conditions by selecting Window >> Analytical Conditions, set your values and save.
8. Check the electron beam position and focus before running any standards:
  - a. Use the KVM switch to access the CAMECA computer.
  - b. In the SX100 CONTROL Window, set beam size to 0  $\mu\text{m}$ . Return to the regular computer using the KVM switch.
  - c. Move stage to MgO Standard (so that you can see a fluorescing dot).
  - d. Use screwdrivers to align fluorescing dot within circle on Optical Scope monitor (make sure you are qualified to do this or ask John).
  - e. Focus with reflected light on sample surface
  - f. Focus with SEM onto sample surface
  - g. Select Window >> Save Column Conditions to File.
9. In the 'Probewin' window go to Automate!. Then choose Unknowns and if there are samples left from a previous run choose Delete Selected Samples and Yes to All.

The screenshot shows the Automate! software interface. The main window is titled 'Automate!' and contains several sections:

- Position List (multi-select) (double-click to see data):** A list of standards and samples with a scroll bar. The current selection is 'St 358 Fid 1 Diopside (Chesterman)'. Below the list are buttons for 'Select Stds', 'Select All', 'Go', 'Auto Focus', 'Update', 'Delete All', and 'Re-Load'.
- Automation Actions:** A panel with buttons for 'Move', 'Stage', 'Digitize', 'Plot', 'Fiducials', 'Replicates', 'Conditions', 'Sample Setups', 'File Setups', and 'Multiple Setups'. It also contains checkboxes for 'Confirm Standard Positions', 'Confirm Unknown Positions', 'Confirm Wavescan Positions', 'Peak Spectrometers', 'Acquire Standard Samples', 'Acquire Unknown Samples', 'Acquire Wavescan Samples', and 'Acquire Standard Samples (again)'. A 'Peaking' button is also present.
- Automation Options:** A panel with checkboxes for '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', and 'Use ROM Auto Focus'. It also has radio buttons for 'New Sample' and 'Every Point', and a 'Digitized' radio button with an 'Interval' of 5.
- Table:** A table with columns: Row, X, Y, Z, W, Grain #, Focus. The first row is highlighted: Row 1, X: -21902.65, Y: 2985.453, Z: -26.70525, W: 0, Grain #: 1, Focus: 0.
- Buttons:** 'Delete Selected Samples', 'Delete Selected Positions', 'Import from ASCII (\*.POS File)', and 'Export Selected Samples (to \*.POS)'.
- Parameters:** KeV = 15, Curr = 50, Size = 10, Mag = 2524, Mode = Analog Spot, Sample Setup (row) Number = 0, MagAnal = 8000, MagMag = 1250, File Setup = NONE, Multiple Setups = NONE, Replicates = 1.
- Run Selected Samples:** A large yellow button at the bottom right.

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**.

**Peaking Options**

**Elements to Peak (multi-select)**

- si ka Spec 2 LPET (81480.0)
- mg ka Spec 1 TAP (38490.0)
- fe ka Spec 5 LIF (48077.0)
- ca ka Spec 2 LPET (38431.0)
- ni ka Spec 3 LLIF (41175.0)
- mn ka Spec 4 PET (24068.0)

Double-click element to move to spectrometer peak position

**Peak Center Method**

- Interval Halving
- Parabolic Fit
- ROM Based (see options below)
- Manual (Pre/Post Scan Only)

Skip P/B Check Before Peaking

**ROM Peaking Type**

- Internal
- Parabolic
- Maxima
- Gaussian

Threshold

**Peak Center Options**

- Acquire Automated PHA Scan Prior To Peaking
- Acquire Automated PHA Scan After Peaking
- Display PHA Dialog Prior To Peaking
- Display PHA Dialog After Peaking
- Display Spectrometer Pre-Scan for Confirmation
- Display Spectrometer Post-Scan for Confirmation
- Use ROM Based Scanning for Pre/Post Scan

**OK** **Cancel**

**Plot Selected Peak Center**

**Return To On Peak (start analysis) Positions**

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. Once the position on the standard is established click on **OK** in the Confirm window that comes up when it goes to the standard.

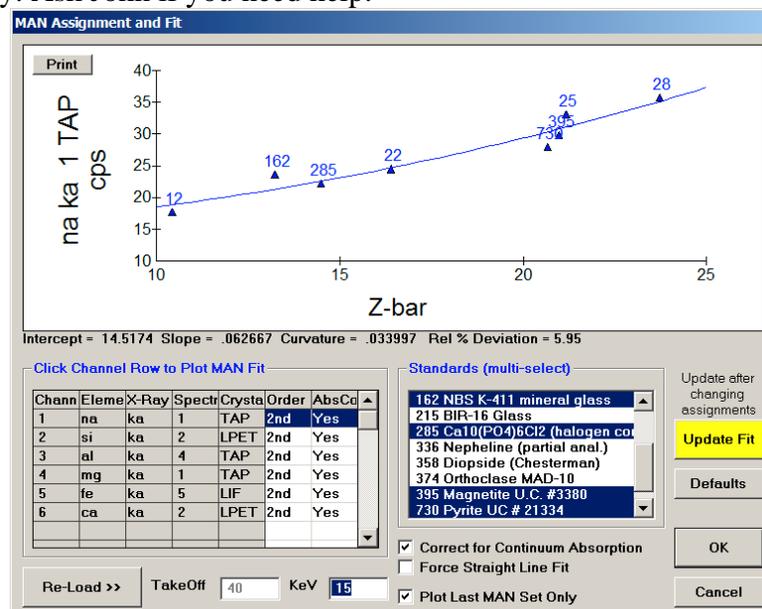


Information about the peak intensities will be located in the 'Probewin' log window. If there are any major changes in your peaked values, both on or off peak, you should get help from John. Also check to make sure you only have one peak in the PHA scan when you peak the spectrometer. If there is more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

13. 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.

## Running Selected Standards

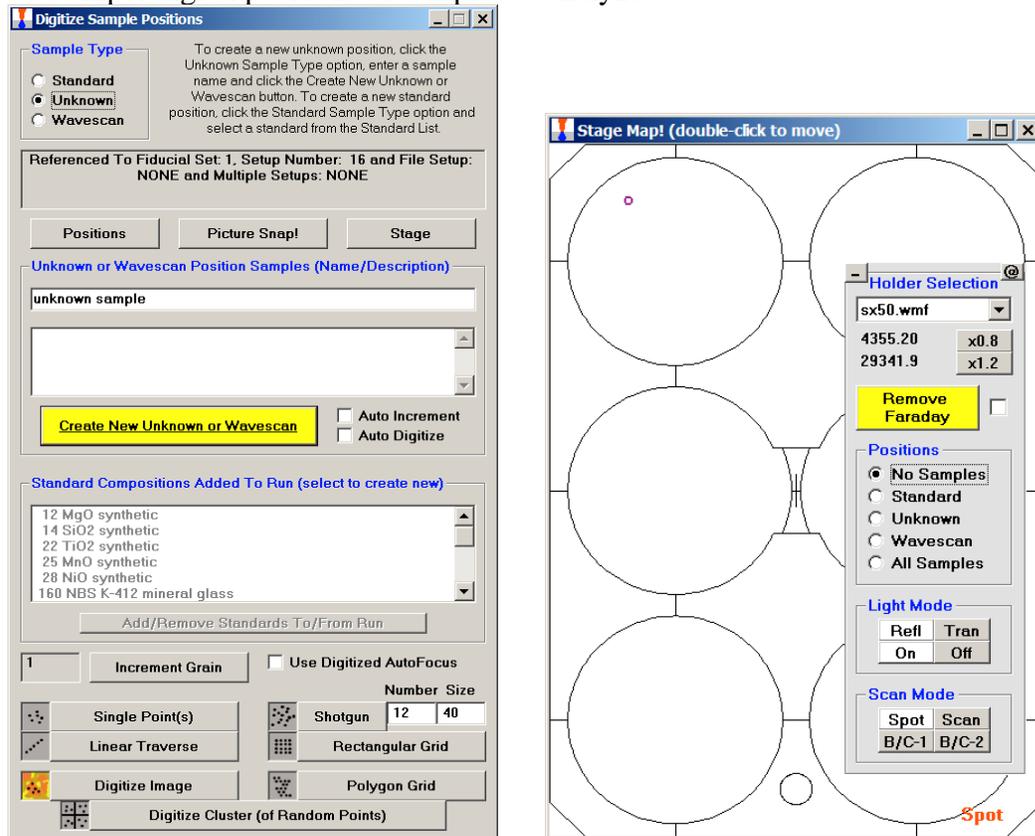
1. In the 'Automate!' window, click on Standards and Select Stds. You have previously confirmed standard positions during peaking but you can check that button again if you would like. Check Acquire Standard Samples and then click on Run Selected Samples.
2. Once standards have been run, you can check MAN assignments and adjust as necessary. Ask John if you need help.



3. It is useful to run some standards that contain none of the elements you are analyzing for in order (and samples that are not used as the standard for a particular element!) to check that you are getting accurate measurements.
4. Also Run a standard (like BIR-16 glass) or something else as an unknown and make sure that the measurements agree with what the true values are.
5. Again, ask John if you have any trouble.

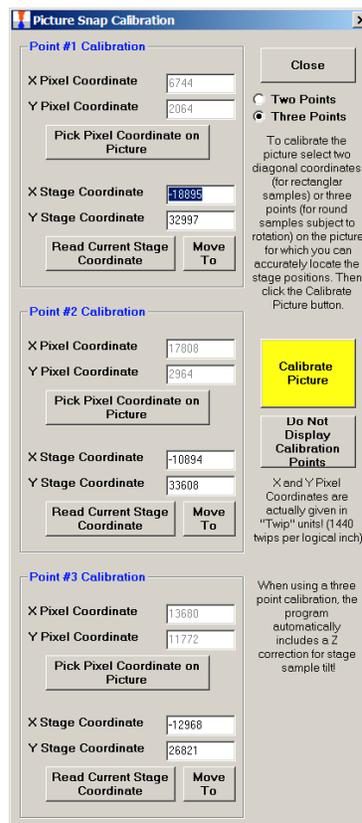
## Digitizing Samples & Running

1. Go back to the ‘Automate!’ window and click on Digitize. You will now be able to input digital positions of samples to analyze:



2. 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.
3. In the ‘Stage Map!’ window, double-click on where you think your sample is. Use the stage controls located to the right of the keyboard and locate a good spot. Once you find a good spot, focus on the sample surface (easiest in reflected light), using the Z adjustment knob.

4. In the 'Digitize Sample Positions' window, click on Single Point(s) (or whatever you want) and click OK once you have established the places you wish to analyze.
5. Repeat steps 14-16 until all of your unknown samples have been digitized.
6. In the 'Stage Map' window, choose to show locations of the unknowns to double check that you are not looking at the same sample twice or missing one.
7. Tip: Picture Snap! is a great tool included in the Probe for Windows software. It simply requires an image file (jpeg, bmp, or gif?) of your mount. Access Picture Snap! from the 'Probewin' window by selecting \_\_\_\_ >> Picture Snap! Load your image file and then choose "Calibrate". In the 'Picture Snap Calibration' window, select either Two Points (for square samples) or Three Points (for round samples). Use the stage controls and navigate to 2 or 3 points that you can recognize on the image and click  Read Current Stage Coordinate. When finished choosing reference points click  Calibrate Picture. Now you should be able to navigate anywhere on your image by double-clicking there, also your digitized positions show up.



8. 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.

- Now click the following:
  - Confirm Standard Positions,
  - Acquire Standard Samples (optional since you may have just done this),
  - Acquire Unknown Samples,
  - Acquire Standards Again, and
  - Use Filament Standby Mode Afterwards (IMPORTANT if you do not want to get charged for beam time after your samples are done!),
  - Use Confirm During Acquisition, and
  - Use Digitized Multiple Setups. See picture below for confirmation.

The screenshot shows the Automate! software interface. The main window is titled "Automate!" and contains several sections:

- Position List (multi-select) (double-click to see data):** A list of samples with radio buttons for selection. The "All Samples" radio button is selected. The list includes:
  - Standards: Un 3 Fid 0 7-10-07c
  - Unknowns: Un 4 Fid 0 7-10-07f
  - Wavescans: Un 5 Fid 0 7-10-07i
  - All Samples: 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 7-11-07q, Un 14 Fid 0 7-11-07r, Un 15 Fid 0 7-11-07t
  - Standards: Wa 514 Fid 1 Silicon metal, Wa 527 Fid 1 Cobalt metal, Wa 530 Fid 1 Zinc metal
- Automation Actions:** A section with checkboxes for:
  - Confirm Standard Positions (checked)
  - Confirm Unknown Positions (unchecked)
  - Confirm Wavescan Positions (unchecked)
  - Peak Spectrometers (unchecked)
  - Acquire Standard Samples (checked)
  - Acquire Unknown Samples (checked)
  - Acquire Wavescan Samples (unchecked)
  - Acquire Standard Samples (again) (checked)
- Automation Options:** A section with checkboxes for:
  - Peak on Assigned Standards (checked)
  - Use "Quick" Standards (unchecked)
  - Use Filament Standby Afterwards (checked)
  - Use Confirm During Acquisition (checked)
  - Use Beam Deflection For Position (unchecked)
  - Suppress ROM Based Backlash (unchecked)
  - Confirm All Positions In Sample (unchecked)
  - Combine Multiple Sample Setups (unchecked)
  - Use ROM Auto Focus (unchecked)
  - New Sample (radio button selected)
  - Every Point (radio button selected)
  - Digitized (radio button selected)
  - Interval (radio button selected)
- 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 (radio button selected)**
- Use Digitized Conditions (radio button selected)**
- Use Digitized Sample Setups (radio button selected)**
- Use Digitized File Setups (radio button selected)**
- Use Digitized Multiple Setups (radio button selected)**
- Run Selected Samples** (yellow button)

At the bottom, there is a table of sample positions:

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

Below the table, there is a status bar with the following information:

- 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

- Double-check everything and click  Run Selected Samples. This will bring up a time estimate. Click  YES and the run will start. The standard positions will be checked again. Make sure they are still in focus.

## Accessing your Data

1. Go through and make sure you like what you see....
2. In the 'Probewin' window, select Output >> (H.W.)... and then select Yes for unknowns. This should export your data to Excel.

## Tips & Common Problems

### 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. This is especially important for trace elements. Note that by acquiring intensity data for all the elements in each standard you can examine the complete analysis of each and see that you should be able to get close to zero concentrations on standards that do not contain the element of interest. If the concentration is significantly negative then it is likely that one or more off-peak positions is being interfered by a secondary x-ray peak.
3. Do a simulation of the samples you will be running in Stratagem to pick the best operating voltages. Low voltages will give small excitation volumes but poor sensitivity.
4. Establish which spectrometers should be used for which standards. You will want to avoid crystal flipping within a single sample. Put in proper count times for what you are doing. Check that the count times are approximately balanced for all spectrometers to avoid a spectrometer sitting idle (you might as well be counting photons).
5. 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. This is most common when the probe has been idle for a long period of time with the filament off.

SX50 Only:

- 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.
- 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.

**Element Setup Database**

**Current Sample:**  
Un 20 \* test

si ka Spectro 2 LPET (81480.0)  
 mg ka Spectro 1 TAP (38490.0)  
 fe ka Spectro 5 LIF (48077.0)  
 ca ka Spectro 2 LPET (38431.0)  
 ni ka Spectro 3 LLIF (41175.0)  
 mn ka Spectro 4 PET (24068.0)  
 o (specified)  
 h (specified)

Double click Analyzed Element List to see Element Setups

**<< Add to Sample**

Delete from Sample

Add To Database >>

Close

SETUP.MDB  
 SETUP2.MDB (MAN)  
 SETUP3.MDB (Interf.)

Import Export

**Element Setup Data From SETUP.MDB Database**

Enter Search Element >>  Total Records = 613

Element/Xray/Cat      
 Spec/Crystal/2d

User Name   
 Sample Name   
 Date - Time   
 Probe Data File

On/Hi/Lo Pos     
 BgdType/Offset      
 S-Hi/Lo/Exponen     
 Base/Win/Gain/B      
 KeV/TO/DT/DIFF

**Standard Intensity Data**

Std/PeakToBgd    
 On/Hi/Lo sec     
 On/Hi/Lo cps     
 Beam/Abs current    
 Stage Position

**Wavescan and Peaking Parameters**

Wavescan Hi/Lo/Points/Time      
 Peakscan Hi/Lo/Points/Time      
 Start/Stop/PB/Count/Attempts

Analyze!

**Analyze!**

Sample List (multi-select) (double-click to see intensity data)

- Standards
- Unknowns: Un 11 2-MS-14-04-c.ol, **Un 12 2-MS-14-04-d.ol**, Un 13 2-S17-3-a.ol
- Wavescans: Un 14 2-S17-3-c.ol
- All Samples: Un 15 2-S17-3-d.ol, Un 16 2-S17-3-d2.ol, Un 17 2-S17-3-e.ol, Un 18 2-S17-3-f.ol, Un 19 2-S17-3-g.ol, Un 20 \*test

Buttons: Select All, Add To Setup, Save Setups

Buttons: Analyze, Data, KRows, Combine Selected Samples >>Excel, List Report, Calculation Options

Buttons:  Pause Between Samples,  Use All Matrix Corrections, Report, Delete Selected Sample(s), Undelete Selected Sample(s), Match

Specified Concentrations | Standard Assignments | Name/Description | Conditions | Elements/Cations

Un 19 2-S17-3-g.ol  
TO = 40, KeV = 15, Beam = 50, Size = 10  
(MagAnal = 8000.), Mode = Analog Spot  
Results in Oxide Weight Percent

42.666	Total Oxygen	99.394	Total Weight %
42.666	Calculated Oxygen	12.033	Z - Bar
.000	Excess Oxygen	21.281	Atomic Weight

Copy	SiO2	MgO	FeO	CaO	NiO	MnO	O	H2O	Total
Average:	39.821	46.959	12.110	.147	.195	.163	.000	.000	99.394
Std Dev:	.112	.119	.128	.002	.011	.015	.000	.000	.199
ZAF Corr:	1.3939	1.4531	1.1820	1.0895	1.1825	1.2008			
Std Err:	.065	.069	.074	.001	.006	.008	.000	.000	.115
%Rel SD:	.3	.3	1.1	1.4	5.6	9.0	.0	.0	.2
Minimum:	39.700	46.825	11.962	.145	.188	.147	.000	.000	99.185
Maximum:	39.922	47.052	12.187	.148	.207	.175	.000	.000	99.582

Buttons: Delete Selected Line(s), Undelete Selected Line(s), Analyze Selected Line(s)

Copy	SiO2	MgO	FeO	CaO	NiO	MnO	O	H2O	Total
141 G	39.700	47.001	11.962	.147	.207	.167	.000	.000	99.185
142 G	39.922	46.825	12.187	.148	.188	.147	.000	.000	99.417
143 G	39.840	47.052	12.181	.145	.189	.175	.000	.000	99.582

Buttons: Cancel, Next

**Calculation Options**

Selected Samples: Un 12 2-MS-14-04-d.ol

Buttons: OK, Cancel

**EDS Calculation Data Options**

- Do Not Use EDS Element Data
- Use EDS Weight % Element Data
- Use EDS Spectral Element Data

Assign EDS Spectral Elements

**Integrated Intensity Data Options**

- Do Not Use Integrated Intensities
- Use Integrated Intensities

**Sample Conductive Coating**

Element: c, Density: 2.1, Thickness (A): 200

Use Conductive Coating

**Calculations Options**

- Display Results As Oxides
- Calculate Atomic Percents
- Calculate Detection Limits and Sensitivity
- Calculate Homogeneity Ranges
- Calculate Alternate Homogeneity Ranges
- Calculate Pearson's Linear Correlation Coefficients
- Element By Difference: h
- Stoichiometry To Calculated Oxygen: Atoms Of: To 1 Oxygen
- Stoichiometry To Another Element: Atoms Of: To
- Hydrogen Stoichiometry To Excess Oxygen: H:O Ratio: .00, OH = 1, H2O = 2

Use Particle/Film Calculations

**Formula and Mineral Calculations**

- Calculate Formula Based On: Atoms Of: Sum
- No Mineral End-Member Calculation
- Olivine
- Feldspar
- Pyroxene
- Garnet (Ca,Mg,Fe,Mn)
- Garnet (Al,Fe,Cr)
- Amphibole (Ague, Auto Normalization)
- Biotite (Brimhall and Ague, Halog Code)

**Standard and Interference Assignments**

Selected Samples  
Un 12 2-MS-14-04-d\_ol

OK Cancel

Save Element Setup  
Save Sample Setup

Add/Remove Standards  
Reload Standard Assignments  
Remove Volatile Correction

1 2 3 4 5 6

Click Element Row to Edit Standard/Interference/Time Dependent Intensity (TDI) Assignments

Channel	Element	X-Ray	Analyzed	Standard	Interf-Ele	Interf-Std
1	si	ka	Yes	273	....	0,0,0,0,0
2	mg	ka	Yes	273	....	0,0,0,0,0
3	fe	ka	Yes	395	mn....	25,0,0,0,0
4	ca	ka	Yes	358	....	0,0,0,0,0
5	ni	ka	Yes	28	....	0,0,0,0,0
6	mn	ka	Yes	25	....	0,0,0,0,0
7	o		No	0	....	0,0,0,0,0
8	h		No	0	....	0,0,0,0,0

Acquire! (example run & off)

**Acquire!**

SP1	SP2	SP3	SP4	SP5	X	Y	Z
38490.0	38431.0	23951.0	32472.0	48077.0	-6052.0	27468.0	-40.000
Mg-TAP	Ca-LPET	Mn-LPET	Al-TAP	Fe-LIF	Faraday		
27.65	74.29	13.73	6.67	15.00	1.00		
13395.	104533.	911.	6797.	1059.	9.99170		

Current Sample: Un 11 \* 2-MS-13-04-c

Combined Conditions Sample

Data Rows: 0 Good Data Rows: 0

Start Standard or Unknown Acquisition

Start Wavescan

Magnification: 8000  
Beam Mode: Analog Spot  
Kilovolts: 15  
Beam Current: 50  
Beam Size: 10  
Col. Condition:

Progress/Beam Deflection  
TAP LPET LPET TAP LIF  
Ti  
P  
Cl  
S  
F  
Mn  
Ca  
Mg  
Na  
Si  
K  
Al  
Fe  
-6052.0 27468.0  
um .000000 .000000  
px 0 0

New Sample PHA Move Stage  
Elements/Cations Peak/Scan Options Acquisition Options Imaging  
Analytical Conditions Count Times Special Options Peaking Options  
Combined Conditions Locate Rate Meter Start Peaking

**Acquire!**

SP1	SP2	SP3	SP4	SP5	X	Y	Z
38490.0	81480.0	41175.0	24068.0	48077.0	-15663.	-9204.0	-28.000
1-TAP	2-LPET	3-LLIF	4-PET	5-LIF	Faraday		
30.00	20.00	5.00	5.00	45.00	1.00		
1533.	2059.	498.	611.	31092.	49.9282		

Current Sample: Un 20 \* test

Self Time Dependent Intensity (TDI) Correction Sample

Data Rows: 0 Good Data Rows: 0

Start Standard or Unknown Acquisition

Start Wavescan

Magnification: 1252.648  
Beam Mode: Analog Spot  
Kilovolts: 15  
Beam Current: 50  
Beam Size: 10  
Col. Condition:

Progress/Beam Deflection  
-15663. -9204.0  
um .000000 .000000  
px 0 0

New Sample PHA Move Stage  
Elements/Cations Peak/Scan Options Acquisition Options Imaging  
Analytical Conditions Count Times Special Options Peaking Options  
Combined Conditions Locate Rate Meter Start Peaking



## Element Setup Database

### Current Sample:

Un 20 \* test

si ka Spectro 2 LPET (81480.0)  
 mg ka Spectro 1 TAP (38490.0)  
 fe ka Spectro 5 LIF (48077.0)  
 ca ka Spectro 2 LPET (38431.0)  
 ni ka Spectro 3 LLIF (41175.0)  
 mn ka Spectro 4 PET (24068.0)  
 o (specified)  
 h (specified)

Double click Analyzed Element List  
 to see Element Setups

<< Add to Sample

Delete from Sample

Add To Database >>

Close

- SETUP.MDB
- SETUP2.MDB (MAN)
- SETUP3.MDB (Interf.)

Import

Export

### Element Setup Data From SETUP.MDB Database

◀◀ Data Cursor ▶▶ Delete from Database

Enter Search  
 Element >>

Total Records = 613

Element/Xray/Cat ti ka 1 2

Spec/Crystal/2d 5 LIF 4.0267 0.000058

User Name Beth Erlund

Sample Name TiO2 synthetic

Date - Time 7/12/2006 11:50:16 AM

Probe Data File C:\UserData\Erlund\7-2006\P-04-06\_07-

On/Hi/Lo Pos 68258 68749.5 67833.3

BgdType/Offset OFF LIN 491.5 -424.69999

S-Hi/Lo/Exponen 1 1 1

Base/Win/Gain/B 0.56 4 700 1850

KeV/TO/DT/DIFF 15 40 2.97E-06 0

#### Standard Intensity Data

Std/PeakToBgd 22 90.294

On/Hi/Lo sec 10 5 5

On/Hi/Lo cps 1090.3 3.9 20.25

Beam/Abs current 19.75995 0

Stage Position -16243 -5075 -3 0

### Wavescan and Peaking Parameters

Wavescan Hi/Lo/Points/Time 69117.23 67398.77 100 6

Peaksan Hi/Lo/Points/Time 69117.23 67398.77 30 8

Start/Stop/PB/Count/Attempts 22.1735 2.21735 5 10 30

### Count Times

Click Element Row to Edit Count Times

Channel	Element	Spectro	Crystal	On-Peak	Hi-Peak	Lo-Peak	MaxCount	Factor	Wave	Peak	Quick
1	si ka	2	LPET	20.00	5.00	5.00	10000000	2.00	6.00	8.00	2.00
2	mg ka	1	TAP	30.00	5.00	5.00	10000000	3.00	6.00	8.00	2.00
3	fe ka	5	LIF	45.00	5.00	5.00	10000000	3.00	6.00	8.00	2.00
4	ca ka	2	LPET	20.00	5.00	5.00	10000000	4.00	2.00	8.00	2.00
5	ni ka	3	LLIF	30.00	5.00	5.00	10000000	3.00	4.00	8.00	2.00
6	mn ka	4	PET	30.00	5.00	5.00	10000000	3.00	2.00	8.00	2.00

#### Beam Averages

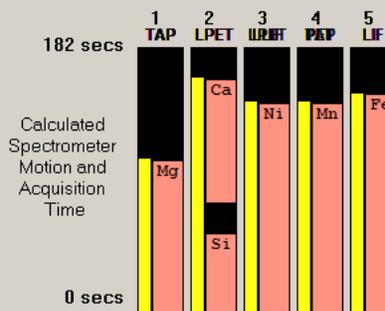
#### Nominal Beam (nA)

Change the Nominal Beam to modify the normalization constant used for the x-ray intensity display. For example, enter 1 (nA) for cps/nA intensity display.

Return To On-Peak Time

Crystal Flip Time

Set Column (TKCS) Time



OK

Cancel

Measure Nominal Beam

### Peak and Scan

Click Element Row to Edit Peak and Scan Parameters

Channel	Element	Spectro	Crystal	On-Peak	Hi-Peak	Lo-Peak	Offset	Hi-Off	Lo-Off
1	si ka	2	LPET	81480.0	82022.5	80937.4	-25.523	542.500	-542.60
2	mg ka	1	TAP	38490.0	39634.0	37358.3	9.15234	1144.00	-1131.7
3	fe ka	5	LIF	48077.0	48660.2	47568.6	38.3867	583.199	-508.40
4	ca ka	2	LPET	38431.0	39737.3	37165.4	-31.414	1306.30	-1265.6
5	ni ka	3	LLIF	41175.0	41758.7	40591.3	32.0977	583.699	-583.70
6	mn ka	4	PET	24068.0	25065.3	23070.6	-28.648	997.299	-997.40

#### Display:

- On/Off Peaks
- Wave Scan Limits
- Peak Scan Limits
- Peaking Parameters

Spectrometer "Offsets" are the difference between the theoretical or predicted peak position (from x-ray tables) and the actual or measured peak position. If the spectrometer peak position has not had a peak center procedure performed then the "Offsets" value will usually be close to zero. The calculation is "Offset = Predicted - Actual"

OK

Cancel

Use ROM Based Spectrometer Scanning

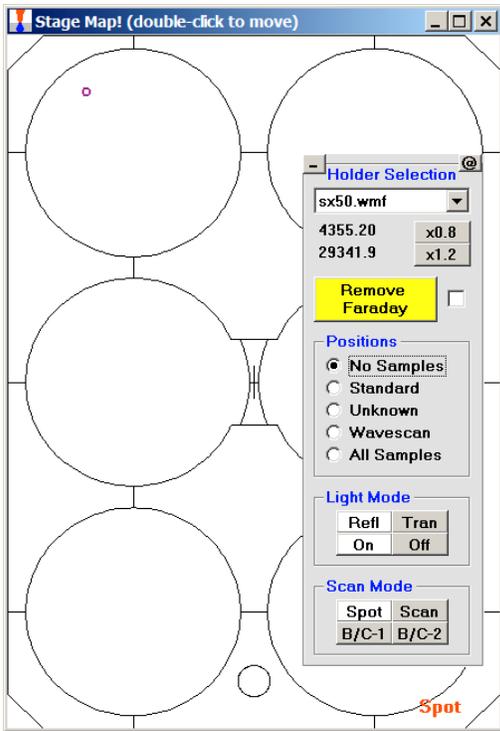
#### Increment Stage During Peakscan/Wavescan or Peaking (X and Y Axis)

- Use Increment During Scanning
- Use Increment During Peaking

Increment Size (microns)  
X  Y

Increment Interval (seconds)

Stage Map!



Picture Snap! Calibration Window

Picture Snap Calibration
✕

**Point #1 Calibration**

X Pixel Coordinate

Y Pixel Coordinate

Pick Pixel Coordinate on Picture

X Stage Coordinate

Y Stage Coordinate

Read Current Stage Coordinate
Move To

**Point #2 Calibration**

X Pixel Coordinate

Y Pixel Coordinate

Pick Pixel Coordinate on Picture

X Stage Coordinate

Y Stage Coordinate

Read Current Stage Coordinate
Move To

**Point #3 Calibration**

X Pixel Coordinate

Y Pixel Coordinate

Pick Pixel Coordinate on Picture

X Stage Coordinate

Y Stage Coordinate

Read Current Stage Coordinate
Move To

Two Points

Three Points

To calibrate the picture select two diagonal coordinates (for rectangular samples) or three points (for round samples subject to rotation) on the picture for which you can accurately locate the stage positions. Then click the Calibrate Picture button.

**Calibrate Picture**

**Do Not Display Calibration Points**

X and Y Pixel Coordinates are actually given in "Twip" units! (1440 twips per logical inch)

When using a three point calibration, the program automatically includes a Z correction for stage sample tilt!

Close