JEOL JXA-8200 SUPERPROBE OPERATOR'S MANUAL

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Revised edition including **Probe for EPMA**

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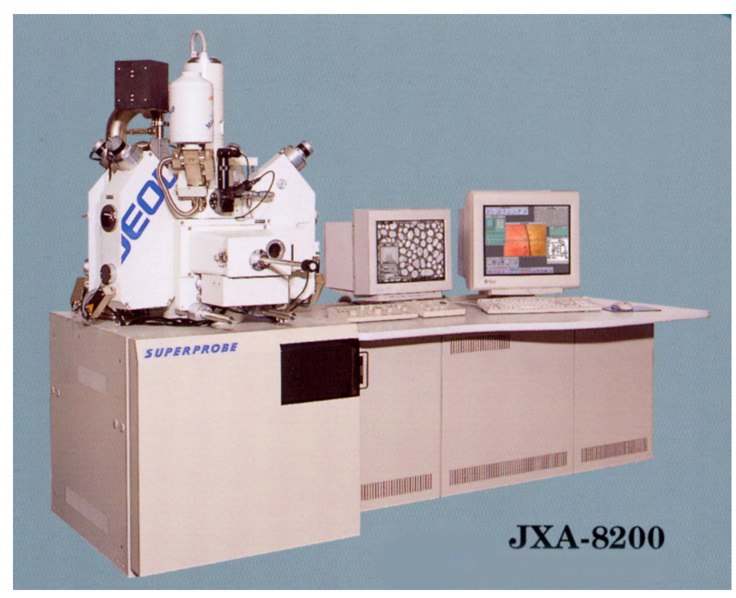

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Institute of Meteoritics

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Note: In the following text, **Bold Blue** indicates a software selection and **Bold Green** denotes a control on the instrument.

LOGIN TO JEOL SOFTWARE (SUN WORKSTATION)

Enter your account User Name and password on the Solaris system.

If the previous user has left the probe in an overnight run, you will need to enter the common password to activate the screen.

Under the **Initialize** menu in the **EPMA Main Menu**, select **Logout**.

Select **OK**, wait for the Login screen and then enter your User Name and the password.

Login proceeds automatically, displaying the EPMA Main Menu on the monitor when it is complete (Fig. I-1).

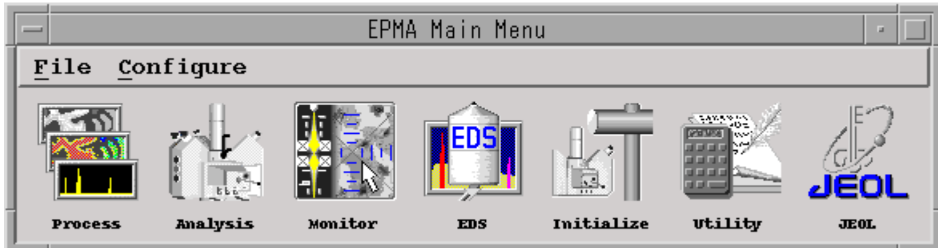


Figure I-1. All JEOL software is accessed from the EPMA Main Menu.

CHANGE OR INSERT SAMPLES

If the previous user has left a sample in the chamber, the bar on the **Specimen Exchange Rod** will be turned toward the right. You must remove the sample before inserting yours.

If there is no sample in the chamber, the **Specimen Exchange Rod** will be turned toward the left: continue to "Insert a sample" below.

REMOVE A SAMPLE

Make sure the **PCD** is "in" (button is lit).

Go to the **Monitor** menu in the **EPMA Main Menu** and select **Stage Monitor**.

From the **Stage Monitor** window, select **Sample Change**.

Press **OK** in the "Stage: Sample Change" menu.

Leave the next dialog window ("Stage") up until sample exchange is completed. DO NOT hit OK yet.

Evacuate the Exchange Chamber

1. Press the **Vacuum Operation Button** on the **Specimen Exchange Chamber**. The button should be lit green. After about 90 seconds, the light should go out.

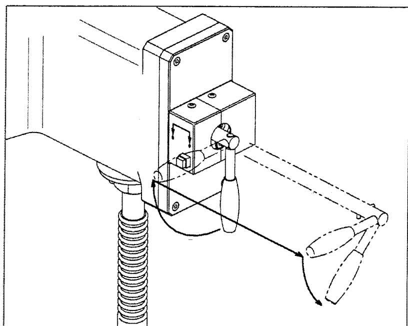


Figure I-2. Operation of the Sample Exchange Door

2. Turn the **Isolation Valve Control handle** 90° clockwise or outward toward you (See Fig. I-2).
3. Pull the **valve handle** all the way to the right.
4. Turn the **valve handle** about 45° down to lock.
5. Insert the **Specimen Exchange Rod** and turn the **knob** to the left. Then withdraw the **Specimen Exchange Rod** all the way out until it latches.
6. Close the **Isolation Valve** by reversing steps 1-4 above.
7. Unlatch the **Specimen Exchange Door** and swing open.
8. Remove the Sample Shuttle and Sample Holder by lifting shuttle straight up.

Remove the sample from the holder and place in the steel desiccator or in the person's lab desiccator. If you will not be using that holder, place it in the steel desiccator marked "Sample Holders".

Insert a sample

Make sure the **PCD** is "in" (button is lit).

Go to the **Monitor** menu in the **EPMA Main Menu** and select **Stage Monitor**.

From the **Stage Monitor** window, select **Sample Change**.

Press **OK** in the "Stage: Sample Change" window.

Leave the next dialog window ("Stage") up until sample exchange is completed. DO NOT hit OK yet.

Open the **Specimen Exchange Door,** remove the shuttle, and slide the holder onto the Sample Shuttle (Fig. I-3).

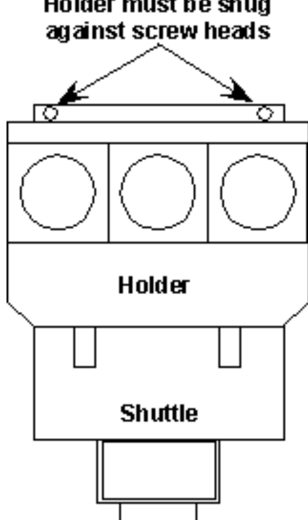


Figure I-3. Proper orientation of the sample holder on the shuttle.

Place the **Sample Shuttle** onto the **Sample Exchange** tray, making sure to engage the **Specimen Exchange Rod**. Close and latch the door.

Evacuate the Exchange Chamber

1. Press the **Vacuum Operation Button** on the **Specimen Exchange Chamber**. The button should be lit green. After about 90 seconds, the light should go out.
2. Turn the **Isolation Valve Control handle** 90° clockwise or outward (see Figure I-2).
3. Pull the **valve handle** all the way to the right.
4. Turn the **valve handle** about 45° down.
5. Push the **Specimen Exchange Rod** all the way in, turn the **knob** to the right, and then pull all the way back out.
6. Close the **Isolation Valve** by reversing steps 1-4 above.
7. Press **OK** in the "Stage" dialog window.

If you have changed the type of sample holder, from the **Stage Monitor** window, select **Holder**.

In the "Stage: Holder" window, select the appropriate holder.

Click **Apply** and **Close**.

START-UP

Hot Start (Normal)

Confirm that the HV READY lamp on the main panel is lit up or that the HT "button" in the upper left of the EOS (Electron Optical System) display is green.

Go to the **Monitor** menu in the **EPMA main menu** and select **EOS Monitor**.

1. In the **EOS Monitor** window select **Filament**.
2. In the **Filament** window click **Auto Saturation**.
3. Click **Start**. The software will heat the filament and select the saturation point.
4. Exit from the **Auto Saturation** window and the **Filament** window.

ALIGNMENT

Automatic alignment of the electron gun

In the **EOS Monitor** window select **Alignment**.

1. In the **Alignment** window, select **Auto Alignment**.
2. Make sure the "Auto Tilt/Shift" button is selected (pink) and that the "Fine" button is green.
3. Select **Start**. The alignment procedure will progress automatically through the tilt and shift operations.
4. When the alignment is done, you should see values for Tilt and Shift in the **Alignment** window. [If Auto Alignment returns an error ("Center is in corner…"), try selecting the "Middle" search button and run the alignment again.

Close the **Auto Alignment** window. Proceed to "**OL aperture alignment**" below.

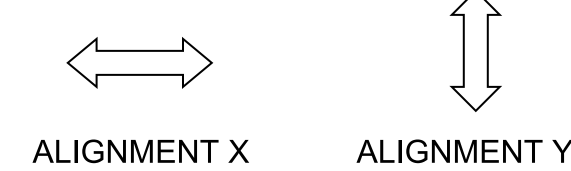
Note: If Auto Alignment fails after "Middle" search (there are no values for Tilt and Shift), see Manual Alignment in the Troubleshooting Section.

OL aperture alignment

Centering is necessary when the objective lens aperture or the accelerating voltage is changed. Select the appropriate **OL aperture** for the intended purpose: use aperture #4 for microanalysis and #1, 2, or 3 for high magnification imaging.

Verify CL Coarse number is 45-60 in the EOS monitor (adjust with the **PROBE CURRENT** knob).

1. Move the stage to the sample holder.
2. In the **Monitor** menu select **Optical Microscope.**
3. Turn on the light by either selecting the light bulb icon in the **OM Control** or click in the **OM** check box below that. Set the brightness slider to 100 or less.
4. Press the **PCD** out and switch **PRB scan** on.
5. Focus the optical microscope.
6. Find a small image feature in **SEI** mode (use the **VIEW** button to find the correct mode) at the center of the viewing display using image magnification of ~1,000x.
7. Press the **ACB** button or manually adjust the **CONTRAST** and **BRIGHTNESS** knobs on the operation panel for the SEI, and focus the image.
8. Press the **RDC IMAGE** button on the operation panel.
9. Press the **WOBB** button on the operation panel. If the feature shifts, use the **ALIGNMENT X-Y** knobs to minimize the image shift. The knobs control the motion along the directions shown below:



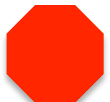
ALIGNMENT X　　ALIGNMENT Y

10. Press the **STIG** to release the wobble function.

Stigmation Correction

Making sure the **STIG** button is lit, adjust the **ALIGNMENT X-Y** knobs and the **FOCUS** to get the sharpest image.

Press the **RDC IMAGE** button to release its function.



Proceed to the appropriate section for imaging, mapping or quantitative analysis. When your work is complete, return to the Shut Down section below.

Shut Down

Standby (Normal)

Remove your samples (see **Remove Sample** section above).

Close all windows other than the EPMA Menu on the JEOL computer.

Go to the **Initialize Menu** in the EPMA Main Menu and select **Gun Startup.**

In the “Gun Automatic Startup” program window, use option #6 (Standby). Enter **6 [Rtn]**.

Input final “Filament code” of 90. **[Rtn]**.

Enter **Y [Rtn]**.

Once auto gun program is finished, press **[Rtn]** to exit the terminal window.

In the **Initialize Menu** in the EPMA Main Menu, select **Log out.**

Click on **OK.**

Enter filament hours in log book.

Full Gun Shutdown

Use this only if the filament is blown, if the sample falls off the sage during sample exchange or if there an accidental chamber venting.

Close all windows other than the EPMA Menu.

Go to the **Initialize Menu** in the **EPMA Main Menu** and select **Gun Startup.**

In the “**Gun Automatic Startup**” program window, use option #5 (Shutdown). Enter **5 [Rtn]**.

Enter **Y [Rtn]**.

Once auto gun program is finished, press **[Rtn]** to exit the terminal window.

Push the **ACCEL VOLTAGE** button on the console to your left to shut off the accelerating voltage.

Notify the lab manager.

QUANTITATIVE ANALYSIS USING PROBE FOR EPMA

Summary of the process:

Step 1: Launch Probe for EPMA (PFE) on the PC

Setup an .MDB file for the day's run.

Step 2: Add Standards to the Run

Select all the standards that will be calibrated including calibration test standards.

Step 3: Setup an Analysis File

Enter the elements and conditions or read from a previous file.

Step 4: Calibrate Standards

Set standard positions, conduct peaks searches, and measure standards.

Step 5: Acquire measurements on the unknowns

Set automated or manual analysis points and then measure the unknowns.

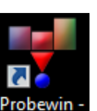
Step 6: Analyze the data

Set analysis parameters and quantify the unknown measurements.

Step 7: Export the data

Select and export the data to an Excel spreadsheet.

Step 1: Launch Probe for EPMA (PFE)



Start PFE by clicking on the **Probewin** icon.

Answer **Yes** in the dialog box: “Do you want to interface to microprobe hardware?”

Setup a database file

Users should have setup their own folder in D:/UserData/*UserName*

Select **File – New**

Name the run and save to your folder. Recommended file structure: *FileName_Date.mdb*

The "**File Information**" window will popup. This information is needed for billing:

Enter your *User Name, Department, Acct Number (or advisor),* and a brief *Description*

The **Log** window will now open (Fig. Q-1). Keep this open throughout your session.

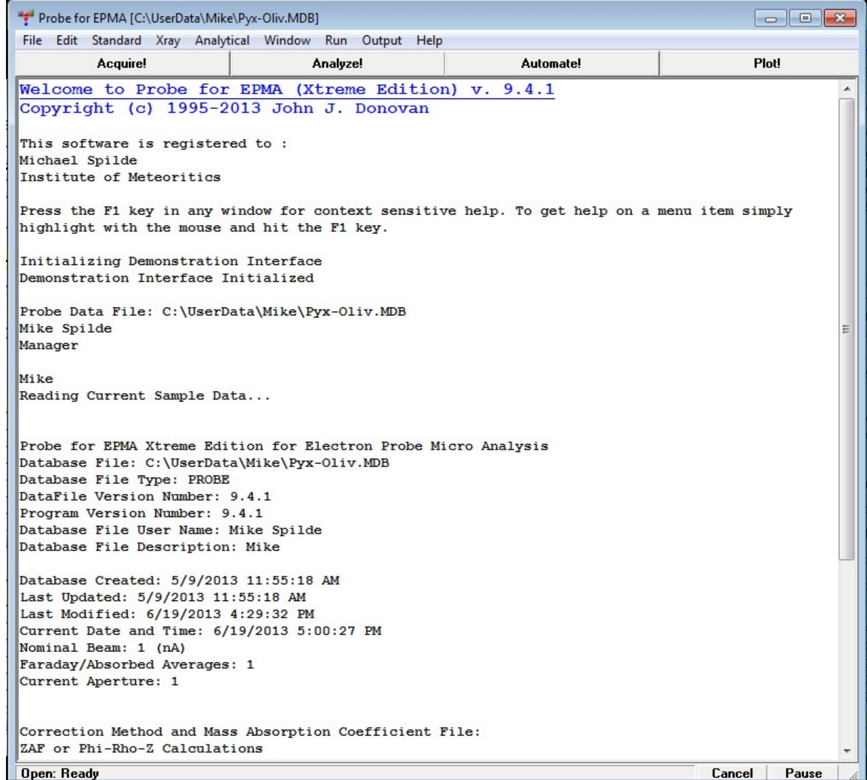


Figure Q-1. PFE Log window. Tabs across the top open additional windows, as do the Acquire, Analyze, Automate and Plot buttons, just below the tabs.

Open **Acquire**, **Analyze** and **Automation** windows from the buttons at the top of **Log** window (see Fig. Q-1 for locations).

Step 2: Add standards

Select **Standard** Tab from the **Log** window.

Select **Add/Remove Standards To/From Run**

Select standards that you will use. Include secondary standards to evaluate quality of calibration. Standards can be removed if you haven't measured them.

Click on **Add Standard to Run>>**

Click **OK**

Step 3: Setup analysis file

To setup an element file, you can either:

A) Read the setup from a previous file, including the calibration if desired,

or

B) Setup a new file from scratch

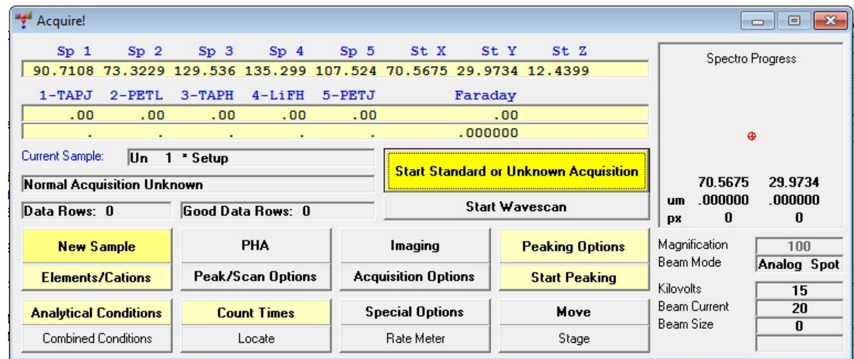


Figure Q-2. Acquire window.

A) Read from a previous file:

From the **Acquire** window (Fig. Q-2):

Select New Sample

Select type **Unknown** and enter a name, usually "Initial" or "Setup"

Click on **Load File Setup**

The "Load File Setup" window will open (Fig. Q-3). On the right side of the window, navigate to the folder containing the file to load (yours or an other user's).

Select the file on the left side of the window. The elements in that file are shown at the bottom right and unknowns at left.

Click **OK**.

A dialog box will ask if you want to load the standard intensity data. Click **Yes** if you want to use the previous calibration (then proceed to step 4). Otherwise, answer **No** to just load the element setups.

Click **OK** at the "New Sample" window

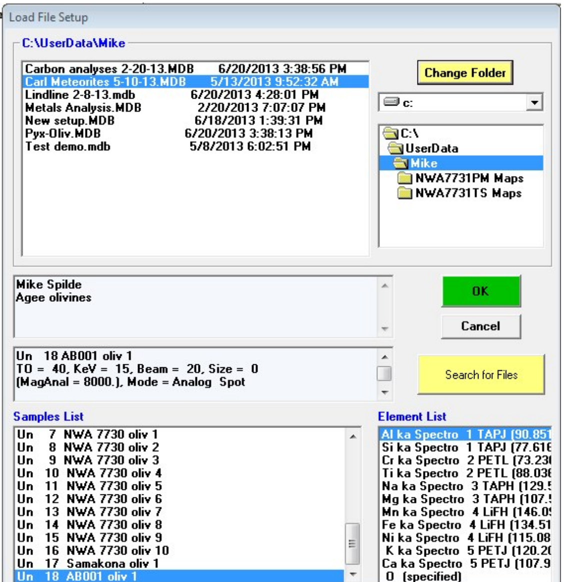


Figure Q-3. Load file setup window.

B) Set up a new file:

From the **Acquire** window (Fig. Q-2):

Select **New Sample**

Select type **Unknown** and enter a name, usually “Initial” or “Setup”

1) Select **Elements/Cations** button.

To set up a new element, click on a blank line

1. Enter **Element** name (Fig. Q-4).
2. Enter **X-Ray Line**, **Spectrometer**, and **Crystal**. Appropriate default values will be automatically entered for each element.
3. Enter Peak Offsets: **High Off-Peak** & **Low Off-Peak**. (Default values have 4 decimals. If you edit, use 0-2 decimals; this way you will know if you have set this).
4. Select **Off Peak** Background type (usually **Linear**)
5. Select an appropriate **Gain** value (look up plot if necessary),
6. Then click **Calculate Empirical PHA**. A green number in the **Bias** window indicates a legitimate value.

Repeat steps 1-6 to enter all the elements that you plan to analyze.

To add calculated oxygen, enter **O** in last position

Set **X-Ray Line** to blank (last position).

Add carbon the same way if carbonates are analyzed.

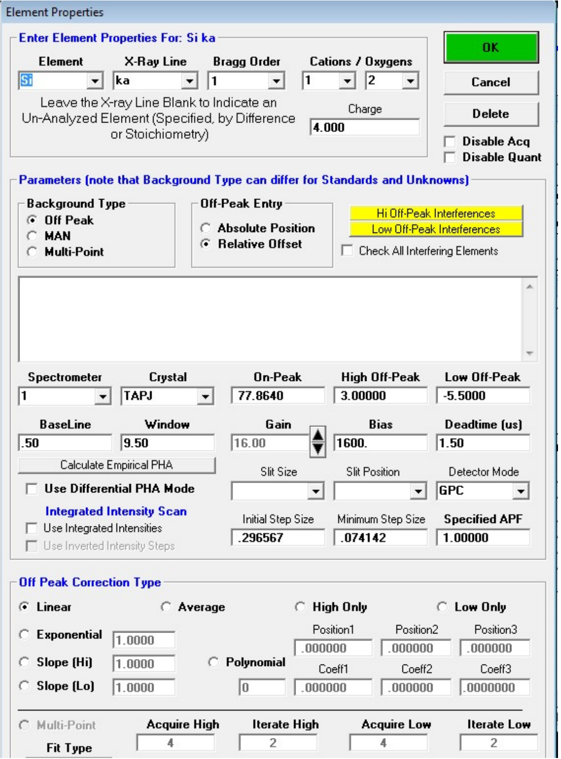


Figure Q-4. Element setup window.

2) Select **Analytical Conditions**

Check **Kilovolts** (usually 15 keV)

Enter **Beam Current** and **Spot Size**. For calibration, enter **10** μm spot size.

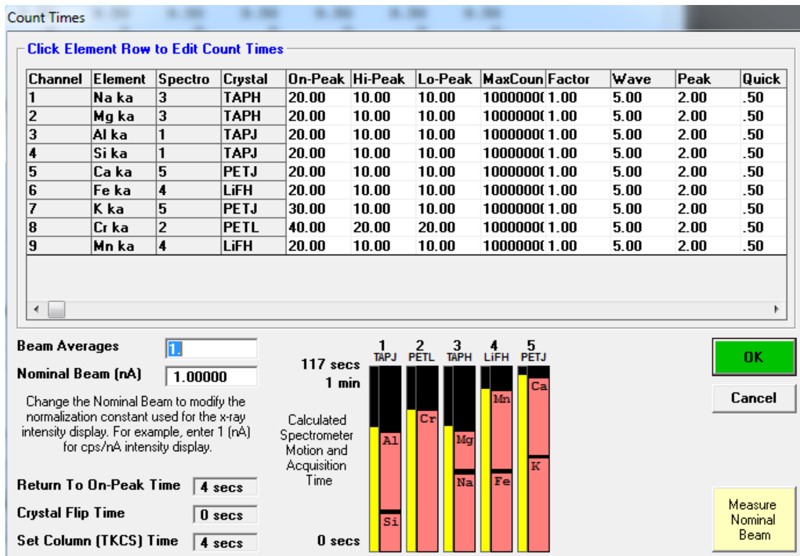
Click **OK**

When OK is hit, the instrument will be set to these conditions (window will be grayed-out for a short time while column conditions are set).

3) Select **Count Times**

Click on an element to set peak and background times for analysis. A map of the spectrometers shows the layout of elements on each spectrometer and the calculated time for analysis (Fig. Q-5).

Default values are usually sufficient for calibration. If longer counting times are desired for analysis, a ***New Sample*** with longer times may be setup from the **Acquire** window after calibration or new ***Conditions*** can be set from the **Automate** window.



Channel	Element	Spectro	Crystal	On-Peak	Hi-Peak	Lo-Peak	MaxCoun	Factor	Wave	Peak	Quick
1	Na ka	3	TAPH	20.00	10.00	10.00	1000000(	1.00	5.00	2.00	.50
2	Mg ka	3	TAPH	20.00	10.00	10.00	1000000(	1.00	5.00	2.00	.50
3	Al ka	1	TAPJ	20.00	10.00	10.00	1000000(	1.00	5.00	2.00	.50
4	Si ka	1	TAPJ	20.00	10.00	10.00	1000000(	1.00	5.00	2.00	.50
5	Ca ka	5	PETJ	20.00	10.00	10.00	1000000(	1.00	5.00	2.00	.50
6	Fe ka	4	LiFH	20.00	10.00	10.00	1000000(	1.00	5.00	2.00	.50
7	K ka	5	PETJ	30.00	10.00	10.00	1000000(	1.00	5.00	2.00	.50
8	Cr ka	2	PETL	40.00	20.00	20.00	1000000(	1.00	5.00	2.00	.50
9	Mn ka	4	LiFH	20.00	10.00	10.00	1000000(	1.00	5.00	2.00	.50

Figure Q-5. Count Time window. Spectrometer map provides an estimate of total analysis time.

4) Select other options as needed:

Acquistion Options

Change element order during analysis, e.g. Na first.

To run spectrometers in consistent direction (*best=saves time*), select **Ascending Angstroms**. This will move each spectrometer from highest atomic number (longest wavelength) to lower atomic number (short wavelength).

To provide a specific order on a spectrometer (e.g. Cl>S>P), select **User Defined Order Number**.

Select the 1st element on the spectrometer. Change **Acquisition Order Number** to 1. Click **OK**.

Select next element on the spectrometer and change to 2, etc.

To use to set one background measurement for a number of analyses (usually used for major elements), select the element in the upper window and check **Nth Point Off-Peak Acquistion** box for that element. Set number points between measurements.

Select **Use Nth Point Acquisition for Off-Peaks** on the Options window.

Combined Conditions

Different conditions can be set per element, e.g: different spot size for Na, different current for trace elements.

Select element in upper left window (**Element X-ray, Spectrometer, Crystal**)

To change settings, click **Apply Conditions to Selected Element**.

Best to group conditions together (e.g. all beam current changes).

Select element in lower window. Click up or down arrow to change order.

Peak/Scan Options

Use to define length and number of steps for “Peakscans” or “Wavescans”

Display changes depending on which button is selected: **On/Off Peaks, Wave Scan Limit, Peak Scan** or **Peaking Parameters.**

Special Options

Use this option to set up Time Dependent Intensity (TDI) acquisitions. This should be used for elements that change intensity during analysis, such as Na or F.

Also used to setup Quick Wavescans for full-length WD spectrometer scans.

Step 4: Calibrate standards

This is a 3-step process that you will usually want to do as separate steps: 1) confirm standard positions, 2) peak the spectrometers, and 3) acquire standard intensities. You can do them sequentially together, but there will not be the opportunity to inspect peaks before acquisition.

From the **Automate** window (Fig. Q-6):

1) Load standard positions

Click **Standards** radio button

Genereally standards are kept loaded in the database.

If no standards are listed or if you want to add standards, click **Import from ASCII.**

Answer **Yes** to the dialog window that asks if you want to delete all positions.

A window will open to select a .POS file. If a recent "working standards" file is available, select and load that. Otherwise, backup one level and open "StandardPOSData" folder.

Select the first standard set, usually "Taylor Std Block.POS"

Answer **No** to transforming the positions using fiducials. **Cancel** the next window.

If another set of standards is needed, select **Import from ASCII**.

Answer **No** to the dialog window that asks if you want to delete all positions.

Repeat the above steps to load the additional standard sets.

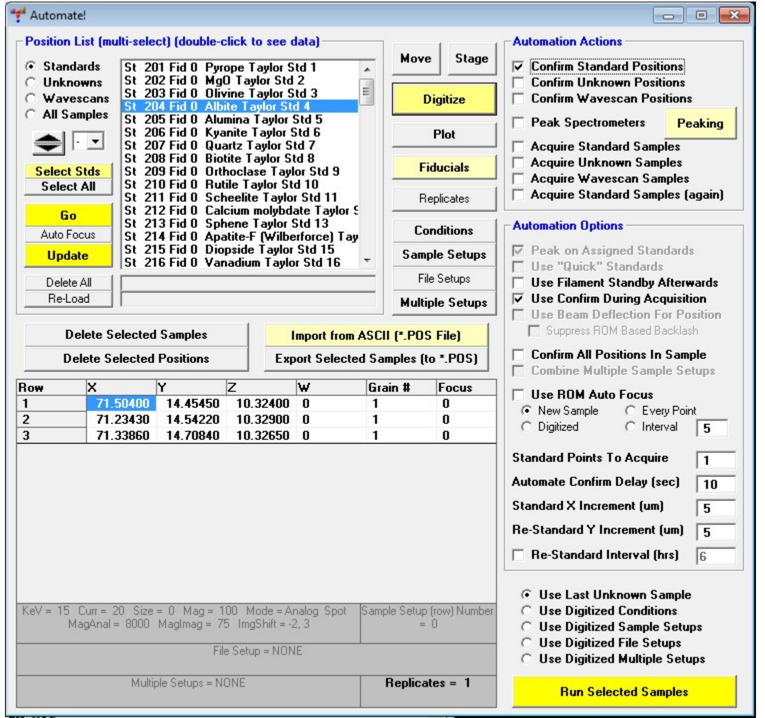


Figure Q-6. Automate window with sample type "Standards" selected.

2) Update standard positions

Click on **Select Stds**. Standards that were loaded into the run will be selected.

Check **Confirm Standard Positions**

Check **Use Confirm During Acquisition**

If multiple points on each standard are to be acquired, check **Confirm All Positions in Sample**. If the points are not in the database, use **Digitize** from the **Automate** window to set additional points.

Click **Run Selected Samples**. This will move to each the standards loaded in the run to allow you focus and adjust the position(s). A 10 second countdown is used; you can **Pause** for more time.

3) Peak the spectrometers

Uncheck **Confirm Standard Positions** and **Use Confirm During Acquisition**.

Check **Peak Spectrometers**

Click on **Peaking**

Confirm that the elements are selected that you want to do peak searches on (usually all of them).

Make sure that **ROM based** peak search is checked.

Check **Skip P/B Check Before Peaking Spectrometer**

Click **OK**

Click **Run Selected Samples**.

4) Inspect peak scans for centroid fit

In the **Log** window, select **Run—Display Fit and Export Spectrometer Peaking and PHA Scans**

Make sure **Display Fit** is checked with **Highest** selected (Fig. Q-7).

Check **Smooth** box.

Confirm that the selected centroid is at or very near the top of the scanned peak for the "Fine" scan.

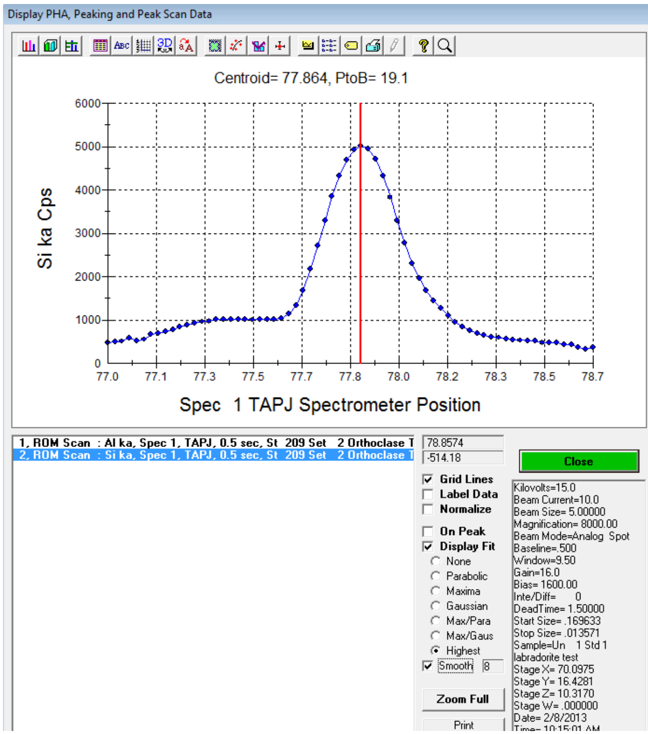


Figure Q-7. Display Spectrometer Peak Fit window.

5) Acquire standard measurements

Uncheck **Peak Spectrometers**

Check **Acquire Standard Samples**

If multiple points are to be acquired, change **Standard Points to Acquire** to **3** (or how many you want). If the points are not in the database, the stage will be incremented by the **Standard X Increment** for each additional point during acquisition.

If you are using specific positions or have mixed numbers of standard points (e.g. 3 on some, 1 on others), you should use only the "digitized positions." From the **Acquire** window, select **Acquisition Options—Use Only Digitized Standard Positions**.

6) Analyze and check standard data

Open the **Analyze** window (Fig. Q-8).

Click **Standards** radio button if not already selected.

Click on **Select Standards** to select all the calibrated standards.

Select analytical standards for each element that will be appropriate for your analysis by clicking on **Standard Assignments**.

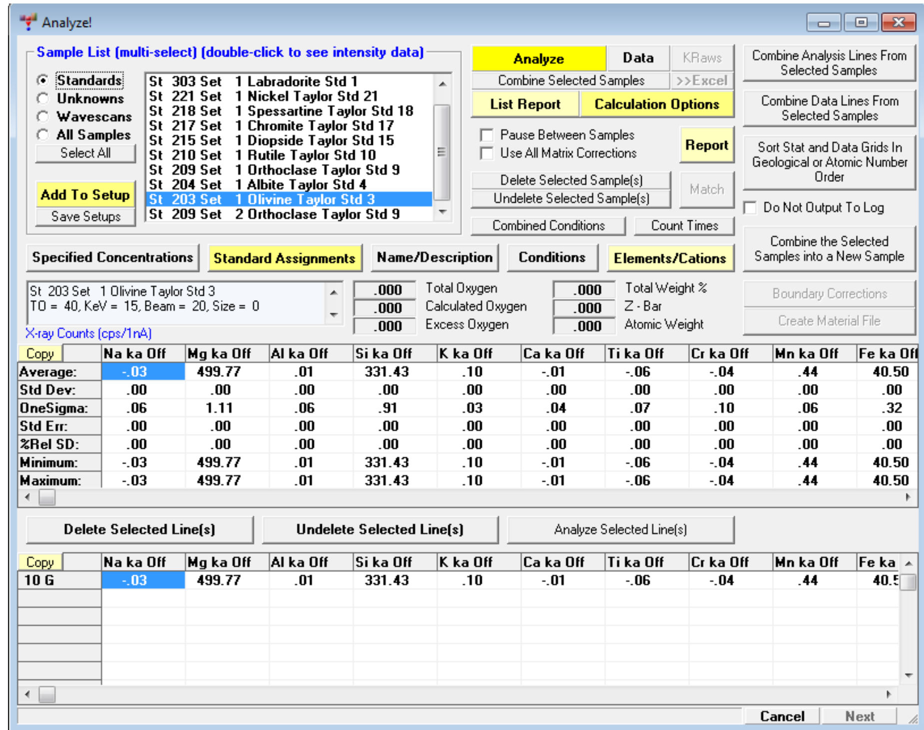
Click on the element, and then select the correct standard from the pull-down list (highest concentration of each element will be selected by default).

If analyzed standards are oxides, go to **Calculation Options** and check **Display Results as Oxides.**

Click the **Analyze** button. Results will print to the **Log** window. Verify that calibration is good for each element on each standard.

If an analysis point needs to be removed to improve standard deviation, double-click on that standard in the **Analyze** window.

In the bottom subwindow, select the bad line. Click on **Delete Selected Lines(s)**.



Copy	Na ka Off	Mg ka Off	Al ka Off	Si ka Off	K ka Off	Ca ka Off	Ti ka Off	Cr ka Off	Mn ka Off	Fe ka Off
Average:	-.03	499.77	.01	331.43	.10	-.01	-.06	-.04	.44	40.50
Std Dev:	.00	.00	.00	.00	.00	.00	.00	.00	.00	.00
OneSigma:	.06	1.11	.06	.91	.03	.04	.07	.10	.06	.32
Std Err:	.00	.00	.00	.00	.00	.00	.00	.00	.00	.00
%Rel SD:	.00	.00	.00	.00	.00	.00	.00	.00	.00	.00
Minimum:	-.03	499.77	.01	331.43	.10	-.01	-.06	-.04	.44	40.50
Maximum:	-.03	499.77	.01	331.43	.10	-.01	-.06	-.04	.44	40.50

Copy	Na ka Off	Mg ka Off	Al ka Off	Si ka Off	K ka Off	Ca ka Off	Ti ka Off	Cr ka Off	Mn ka Off	Fe ka
10 G	-.03	499.77	.01	331.43	.10	-.01	-.06	-.04	.44	40.5

Figure Q-8. Analyze window with an olivine standard selected.

Step 5: Acquire measurements on the unknowns

Sample data can be acquired by two methods:

A) As individual points acquired in real-time,

or

B) As a group of digitized points set before acquisition begins.

A) Individual points:

From the **Acquire** window:

Select **New Sample**

Select type **Unknown** and enter a name, e.g. NWA7731 Pyx1

Click **OK**.

Make any adjustments to spot size, beam current, etc. by clicking the **Analytical Conditions** button. The PFE software will use the previous conditions until they are changed.

Click **Start Standard or Unknown Acquisition** in the **Acquire** window

B) Set digitized points:

From the **Automate** window:

Click **Unknowns** radio button.

Click **Digitize**

Select type **Unknown** and enter a name, e.g. NWA7731 Pyx1

Click on **Add New Unknown to Position List**

Several options are available to set points:

Single Points(s): set multiple points under the same sample name. They will be treated as the same mineral type but will have separate line numbers in the database.

Linear Traverse: define a line traverse by setting starting and ending points. Define the traverse by either the ***Number of points*** or ***Distance between points***.

Retangular Grid: define a grid by setting starting and ending points. Define the distance between points in X and Y and the software calculates and sets the array of points.

Check that positions are being added to the **Automate** lower subwindow.

When all points are set, close the **Digitize** window.

Uncheck **Acquire Standard Samples**

Check **Acquire Unknown Samples**

If you want to make any adjustments to spot size, beam current, etc., select all the samples that you want to run at a given condition. Click the **Analytical Conditions** button in the **Automate** window. The PFE software will use the previous conditions until they are changed.

Select all the samples that you want to run.

If you have changed Analytical Conditions, check that "**Use Digitized Conditions**" is selected instead of "**Use Last Unknown Sample**."

Click **Run Selected Samples**

Step 6: Analyze the data

After the data have been acquired, it needs to be processed for matrix corrections. Standards can be changed and different corrections and calculation options applied.

From the **Analyze** window:

Select Sample Type **Unknown**

Specify the appropriate analytical standards for each mineral type:

Select specific unknowns or click on **Select All** to select all the unknowns.

Select the standard for each element that will be appropriate for your analysis by clicking on **Standard Assignments**.

Click on the element, and then select the standard from the pull-down list (highest concentration of each element will be selected by default).

Click **Calculation Options** (Fig. Q-9).

If samples are analyzed as oxides, check **Display Results as Oxides**

If stoichiometric minerals were analyzed, click the **Calculate with Stoichiometric Oxygen** radio button.

Check the box **Calculate Formula Based on** and enter the number of Oxygens (e.g. 6 for pyroxene) and select **O** (oxygen) from the pull-down menu.

You can have end-members calculated for certain minerals by unselecting the **No Mineral End-member Calculation** radio button and selecting the appropriate mineral family.

For carbonates, instead of the above, check **Stoichiometry to Calculated Oxygen** and enter **0.33** Atoms of **C** to 1 Oxygen (carbon must in the Elements/Cations file as an unanalyzed element).

Click **OK**

Click the **Analyze** button.

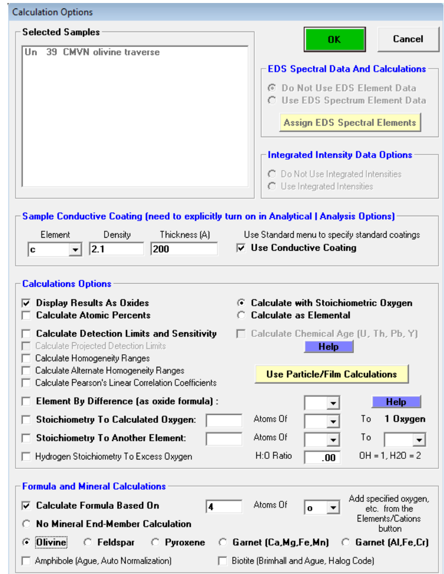


Figure Q-9. Calculation Options window with olivine calculation conditions.

Step 7: Export the data

After completion, data is exported directly into an Excel spreadsheet.

Make certain that **Analyze —Calculation Options** are properly set or that groups of unknowns have been previously analyzed with appropriate Calculation Options.

From the **Log** window:

From the **Output** tab, select **User Specified Format Output.**

In the dialog window, check boxes for desired data output, such as Sample Number, Line Number, Totals, Oxide Wt %, etc.

Click **OK**

Answer **Yes** to: "Do you want data output to spreadsheet?"

Corrections will be written to log window and then to Excel sheet

“Do you want to save changes to Excel”

Yes will save and close Excel

No will close Excel sheet

Cancel leaves sheet open

OPTIONAL PROCEDURES FOR PROBE FOR EPMA

Navigating with Picture Snap in Probe for EPMA

The input image can be an acquired digital image, e.g. BSE, or imported digital image, such as a scan or photomicrograph. Picture snap can also open maps to use for digitized analyses of map objects.

Must be in BMP, JPEG or JIF format.

2k x 2k is a good size. Use *Irfanview* to resize and to change format.

Images should be in current folder for user sample

Start from or from **Automate—Digitize.**

Select **File—Open JPEG** (or BMP or JIF)

Select **Window – Calibrate Image to Stage Coordinates**. Use 2 pt (diagonal) calibration unless image is rotated, then use 3 pt (equilateral triangle).

Move stage to identifiable point on sample. Click **Read Current Stage Coordinate.**

Then click **Pick Pixel Coordinate on Picture** and click on the same point on image that the stage is at.

Move to second point and repeat (and 3rd if doing 3 pt calibration.

Click on **Calibrate Picture. **Close window.

You can now move the stage around by double-clicking on image.

Set sample positions from Picture Snap

After calibrating the stage to the image:

Enter a new sample of type **Unknown** and click **Add New Unknown to Position List**.

Double click on Picture Snap image to locate point

Click **Single Point** to set point, adding as many points to the job as needed. "Linear Traverse" or other point types can also be added.

Collect & digitize BSE image (provides stage-calibrated image)

From the **Automate** widow, select **Digitize**

Enter a new sample of type **Unknown** and click **Add New Unknown to Position List**.

Click **Digitize Image**

Set **Image Type** (BSE) and **Image Size**

Initially, you may want to do a small test image first (e.g. 128x128) as a quick quality check.

Then do a high-resolution image (e.g. 1024x1024).

Select **Save As** and save to your folder

Note: "Save to Database" puts image in the database file rather than your image folder.

You can set points from this window, but is better to Save, Close, and then use **Digitize – Picture Snap** to set points.

To display analysis points on Picture Snap

To show data points that have been previously set or analyzed:

Select **Run – Display Picture Snap**.

Note: sample positions must be loaded into the Automate window if they are not already there.

Select **Display – Digitized Unknown Position Samples** to show points and **Display – Digitized Position Long (or Short) Labels** to show point labels.

To save image with positions and labels, select **File – Save as BMP (with graphics objects)**.

To save to another image program (e.g. *Photoshop*) use **File – Copy to Clipboard (method 2).**

Paste into the program and save

Correcting X-ray line interferences

Check for interferences after all elements are entered into your analysis file. Interferences such as Ti Kβ on V Kα can be corrected after calibration. The Standard Database can also be used to calculate interferences.

In **Analyze** window:

Select **Standard Assignments**

Click the element line of interest.

In the Properties window, select **Calculate Interferences**

The program calculates the percentage of interference based on 1 Wt% of the interfered element and 100 % of the interfering element. A "Maximum Order" of 3 (III) is usually sufficient.

Declare and correct an interference:

In order to correct for interfering X-ray lines, you must calibrate on standards that do not contain the interfering elements within the same standard.

For example, to correct Cr Kβ interference on Mn Kα:
You will need a Cr standard without Mn, such as Cr metal
and a Mn standard without Cr, such as Tephroite.

After calibration, go to the **Analyze** window

Click **Standards** radio button.

Click **Select All** to select all the standards.

Select **Standard Assignments**

Select element to be corrected (e.g. Mn)

In the section "Interference Standard Assignments for Interferred Element"

Select **1st Intrf Elem** (Interfering Element) from pull down box (Cr).

Select **Intrf Order** if necessary (usually I).

Select **Interference Standard** (in this example, Cr metal)

(Remember: the interfering correction standard must not contain any of the interfered element).

Add other corrections if needed.

Reanalyze and check that interferences are removed.

Important: Interferences on the unknowns will have to be declared separately (this is in case a correction isn't necessary on the unknown. Large negative corrections will raise a red flag with the program).

Click **Unknowns** radio button.

Select the appropriate unknowns or **Select All** to select all the unknowns.

Proceed as above.

Corrections can be turned off globally: go to menu **Analytical—Analysis Options**. Unselect “Use assigned interference corrections.”

Specifying Unanalyzed Elements

All elements should be including in the file, even if they are not analyzed, in order for the matrix correction to work properly. For example: the analysis of trace elements in a sample in which you knew the concentration of the major elements.

In the **Acquire** window:

Start a **New Sample** of type **Unknown.**

Select **Element/Cations**

Enter all the elements that you plan to analyze.

To add an unanalyzed element, enter the element.

Set **X-Ray Line** to blank (last position).

Add any remaining unanalyzed elements.

To add calculated oxygen, enter **O** in last position and set **X-Ray Line** to blank.

In the **Analyze** window:

Select **Specified Concentrations**

Scroll down to the unanalyzed elements and click on the element(s).

In the next window, enter the specified concentration in elemental or oxide weight percent.

To do an element by difference:

To do an anion (e.g. PO_4) by difference, enter the element as unanalyzed in **Elements/Cations** (e.g. P).

In the **Analyze** window, select **Calculation Options**.

Click the radio button for **Calculate with Stoichiometric Oxygen**.

Check the box for **Stoichiometry to Calculated Oxygen:**

Enter "**0.25** Atoms of **P** to 1 Oxygen"

Analyze the same element on multiple spectrometers

Set up the file with the same element on several spectrometers, e.g. Ti on Ch 2 PET and Ch 4 LiFH.

Calibrate and run as usual.

Before analyzing, select **Analytical–Analysis Options** in Log window

In the "**Analysis Calculation Options**" window, under "**Calculation Options**" (right side):

Select **Use aggregate intensities for Duplicate Quantitative Elements**.

You can check the individual results of each spectrometer by selecting **Elements/Cations** from the **Analyze** window.

Select one of the multiple element entries:

Check **Disable Quantification**

Click **Analyze**

Wavescans

An element file should be set up with appropriate elements in Elements/Cations window.

In the **Acquire** window:

Start a **New Sample** and select file type **Wavescan**

Select **Peak/Scan Options**

In the "Peak and Scan" dialog window, check **Wavescan Limits**

Click on each element and the "Peak Scan Properties" dialog window will open.

Wavescan Hi and Low limits should be set beyond the background offset positions

Wavescan Points should be 100 or 200.

ROM-based spectrometer scanning should be checked.

Select **Count Times**

Click on each element. From the "Count Time Properties" dialog window:

Set **Wave Scan Time** to **5** seconds or less. If 3 seconds/point are used, each 200 pt scan will take 10 minutes.

After running scan, open **Plot!** window

Click **Wavescan** radio button and highlight your sample (Fig. Q-10).

In the X-Axis column, highlight "**Element-line-spectrometer**"

In the Y-Axis column, highlight the corresponding "**Element-line-Wavescan counts**." Multiple elements can be selected to overlay spectra.

Set **Graph Type = line**.

Click **Output**.

Move between element plots by clicking the up and down arrows on the right side.

To change background positions from the plot window:

Select **Low** button, then click on graph to set the low background to that position.

Do the same for the **High** background. This edits Element/Conditions File.

To do wavescan on only one element per spectrometer:

Setup a new sample of type **Wavescan**

Select **Elements/Cations**

For each element not wanted in a wavescan, click on "**Disable Acq**" e.g. Si only scanned on TAP containing both Al & Si, Al is turned off.

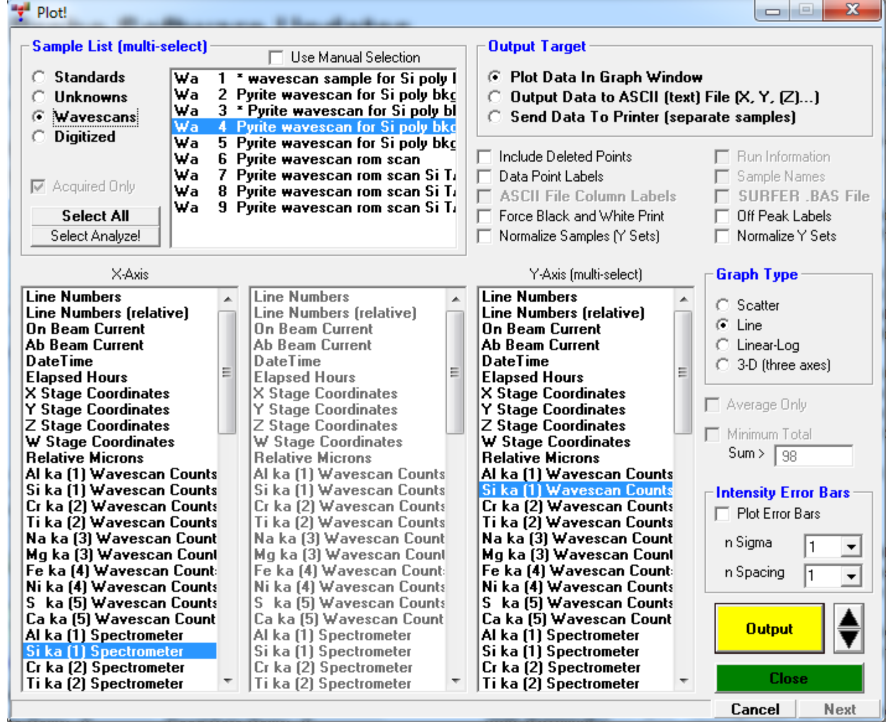


Figure Q-10. Plot! window with wave scan data for Si selected.

PHA Scans

This method is used to setup manual PHA scans.

From the **Acquire** window:

Set up a new sample of type **Unknown**.

Click **PHA** button.

Select the element of interest.

If necessary, do a Bias scan first to determine proper bias voltage:

Set **Gain** at top of window

Select **Aqcuire and Graph Bias Scan Distribution**.

To do PHA scan:

Set **Gain** and **Bias** at top of PHA window

Set Intervals at 80 and select **Aqcuire and Graph PHA Distribution**.

After acquisition, plots can be viewed by selecting **Run—Display, Fit and Export Spectrometer Peaking and PHA Scans** from **Log** menu.

Polynomial Background Correction

Setup a new sample of type **Wavescan**.

Example, checking Si background in low Si materials such as pyrite

Turn off acquisition of other elements except Si

Select **Elements/Cations**

Select each element to turn off. Check **Disable Acq**

Select **Peak/Scan Options**

Scan perimeters are set from **Peak and Wavescan** window

With ROM-scan box checked, scan is continuous.

Without, it is a stepped scan (recommended).

Click **Wavescan** button and select element.

Set **Hi** and **Low Limits** far enough out to cover background

Set number of **Wavescan Points** (usually want fine steps for this, 201 steps).

Select **Count Times** and click on element to be scanned.

Enter number of seconds per step in **Wave Scan Time** box.

For this example, 201 steps were setup at 1.5 sec/step (16 minutes total)

After running scan, open **Plot!** window (Fig. Q10).

Select **Wavescans** radio button and highlight your sample.

In the X-Axis column, highlight "Element-spectrometer"

(e.g. Si Ka (1) Spectrometer)

In the Y-Axis column, highlight the corresponding "Element-Wavescan counts" (e.g. Si ka (1) Wavescan Counts)

Click **Graph Type: Line** and click **Output** button.

If necessary, check box for **Smooth** (= 4 or 6).

Click **Model Background** button

Select **Polynomial** button (Fig. Q-11).

In the 3 boxes for **Poly Fit Positions**:

Adjust high and low boxes to be coincident with linear background points. As the values are adjusted, the blue lines in the plot will move according to the box operated on. Set the high and low blue lines on top of the high and low green lines representing the two backgrounds.

Adjust center box to give a curve matching the background. Move the center blue line until the modeled background line (light blue) fits the measured background (Fig. Q-11).

Select the unknowns in the lower window and click **Assign Background Model to Selected Samples**

This should be done after calibration before starting measurements (start new sample – it will be assigned to following samples)

However, it could be done later if you discover that there was a problem with the background.

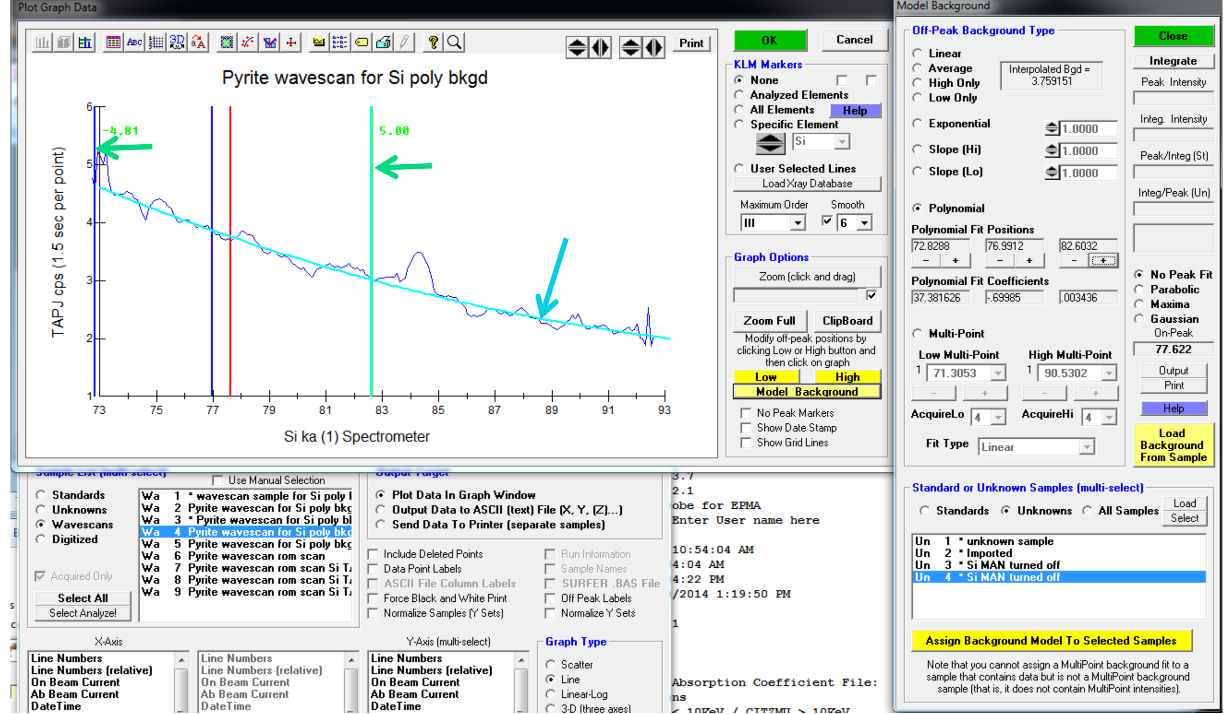


Figure Q-11. Polynomial background setup. Calculated background plot at left (light blue) is derived from settings in Model Background Window at right. Green arrows are linear background positions.

Using the Mean Atomic Number (MAN) Background Correction

MAN Background Correction is an empirical calibration curve for background subtraction. The MAN Method is used especially for mapping, but is also good for complex analyses situations, e.g. REE analysis.

To set up, a range of standards will be needed, with Z-numbers above and below the anticipated Z of the samples analyzed. One or two of them must not contain any of the measured element (e.g. Al2O3 and Fe2O3 might be included for Si backgrounds).

Select **Element/Cations**

Select each element.

In the **Element Properties window**, check the **MAN** radio button under **Background Type.**

Calibrate as usual.

After calibration, from the Main window select **Analytical – Assign Man Fits**

Select each element one at a time, e.g. Na, in the table "Click Channel Row to Plot MAN Fit."

If one or more standard points are significantly off the general trend, especially above the trend, **Ctrl-click** on the deviant point in the **Standards (multi-select)** window to remove (see point 519 in Fig. Q-12).

Click on **Update Fit**

Remove more points if necessary to get a good fit. "Force Straight Line Fit" should be used for 2 points.

After all elements are done, click **OK**

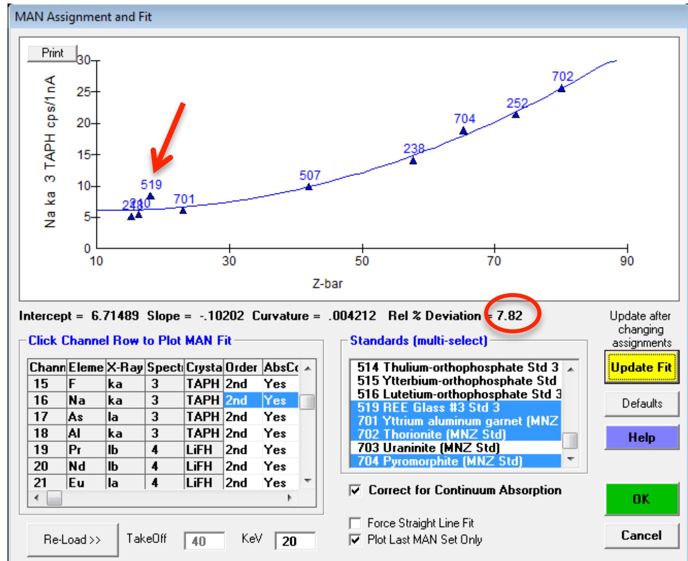


Figure Q-12. MAN background fit for Na. Standard 519 contains a small amount of Na not listed in the standard database. Removing the point improves the background fit to 3.52 Rel % Deviation.

Time Dependent Intensity Correction (TDI)

Volatile elements such as Na and S decrease intensity over the time of a measurement. TDI correction should be applied to correct for intensity drift.

The correction will be done on first element on each spectrometer. Set up the element file so that major elements, including the volatiles are first.

Ch1	Ch2	Ch3	Ch5	
Si	Ti	Na	K	←TDI on these only
Al	Cr	Mg	Ca	

Set up new sample, then select **Acquire – Special Options.**

Select the **Self-Calibration Time Dependent Intensity (TDI) Acquisitio**n option.

Enter the number of **Count time intervals.** Example: if Na is counted for 40 sec on peak, entering 10 intervals will result in counting ten 4-second intervals (10 data points).

Click on **Start Standard or Unknown Acquisition** to start measurement.

After acquiring the unknown sample, display the correction from the **Analyze** window.

Select **Standard Assignments** button

Select an element to view the TDI calibration (e.g. Na)

Check **Use TDI "Self" Calibration Correction**

Click **View TDI Plot**

Select either "**Use Log-linear (exponential) Fit**" or "**Use Log-Quadratic (hyper-exponential) Fit**," depending on shape of the displayed curve.

Volatile elements (e.g. Na) will show a decrease in counts with a subsequent increase for major elements (e.g. Si). May need to turn off TDI on minor elements.

If samples are particularly sensitive to the beam, measurements should be done in "synchronous mode" to reduce the time delay between beam unblanking and counting.

From the **Acquire** window:

Select **Acquisition Options**

Click the **Synchronous** radio button under "**Spectrometer Motion**"

Turn on/off globally: from the **Log** window, select **Analytical – Analysis Options**

QUANTITATIVE X-RAY MAPS WITH PROBE FOR EPMA

Step 1: Set up Quantitative Analysis in Probe for EPMA

1) Create a PFE analysis file for all required elements.

You should include all major elements in the map in order to do quantitative analysis, but trace elements of interest can also be added. Major elements not specifically mapped should be added by difference or fixed concentration (e.g. carbonate or sulfide) for proper matrix corrections.

Set the file to measure backgrounds using the Mean Atomic Number (MAN) method (see the section above on *Miscellaneous Procedures*). This will save time during mapping in that background maps will not have to be acquired.

2) Do a full calibration for all elements using MAN background measurements.

3) Setup at least one unknown with appropriate conditions, and run an analysis on a mineral in the map or on a standard as an unknown.

Under **Acquisition Options**, make sure "**Return to On Peaks After Acquisition**" is checked.

If elements are analyzed as oxides, oxygen must be specified in the element file as unanalyzed.

In the **Analyze** window, select "**Oxygen by Stoichiometry**"

If carbonates are done, C should be added as unanalyzed and "**Stoichiometry to Calculated Oxygen**" selected in the **Analyze** window. Use "0.33 Atoms of C to 1 Oxygen."

4) Save the map position by setting map center from **Automate – Digitize**

Step 2: Set up and acquire maps in Probe Image

Start **Probe Image** by clicking on the icon.

Select **Setup – Acquisition**

1) Enter sample name for first map pass.

Click on the first box under **Sample Name** (Fig. QM-1)

Enter name of first map in **Sample Name** box.

2) Setup Sample Parameters in the boxes to the right.

Use **Beam** for small-scale maps (greater than 600x).

Use **Stage** for large-scale (stage raster) maps and select either:

Stage Ctr to start at center of map area

Stage 2Pt to set 2 corners of map

Enter a value in **Pixel Time**. For trace elements, this should be on the order of 80-100 msec.

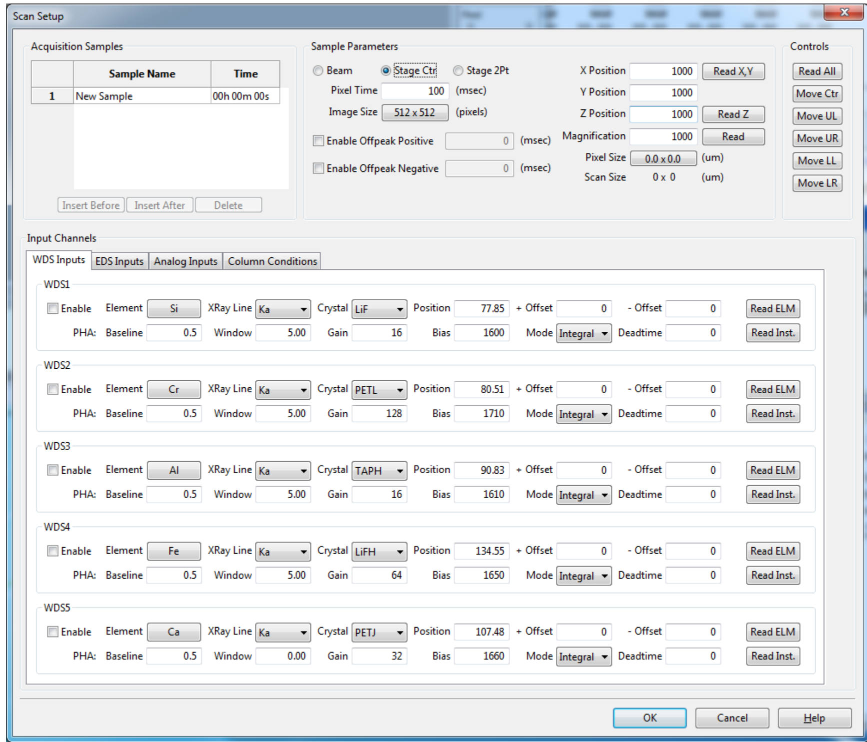


Figure QM-1. Probe Image setup window.

Select **Image Size**

Maps should be done as square images.

Image size of 512 x 512 at 100 msec will require about 7 hr 20 min per pass.

If MAN background correction *is not* used, select **Enable Offpeak Pos or Neg**. to acquire background maps.

Move to the map area and focus the optical microscope. Hit the **Test** button on the JEOL joystick to jog the stage. Refocus if necessary.

Click **Read X,Y**

Click **Read Z**

Move to the corners of the map by selecting **Move UL** (upper left), **Move UR**, **Move LR**, and **Move LL** to check focus.

If the stage is not focused through the 4 corners, record the Z reading at each corner and calculate the average Z of the corners.

Click **Move Ctr** and manually set the JEOL stage to the calculated Z position (remember to hit the **Test** button)

Click **Read Z**

Move through the map corners again to double check.

Enter a large value like 50,000x in the **Magnification** box.

Enter an appropriate value in **Pixel Size** box.

Pixel Size (μm) x Number of X-axis Pixels = Map Width (μm)

3) Setup Input Channels

For the first mapping pass, click on **Read Elem** for WDS1 through WDS5.

This will read the MDB file set up in Step 1. Check that **Peak** position, **Gain** and **Bias** are correct for each of the elements to be mapped in this pass.

Check that each spectrometer is enabled.

Click on the **Analog Input** tab and enable the **BSE** input.

4) Setup parameters for the second map pass.

Note: If more than 5 elements are used, map will make separate 2 passes.

Under the **Sample Name** box, click on **Insert After** to add new name

Enter the name of second map pass in the lower **Sample Name** box. It must be a different name from the first (e.g. A & B).

For the second pass, spectrometer parameters will have to be hand entered.

All **Sample Parameters** should be the same.

For each WDS#, click on Elm to get a periodic table to select an element

Enter peak postion, bias and gain from the PFE file calibrated ealier.

5) Make sure all parameters are correct

Switch back and forth between the 2 sample sets.

6) Save setup

7) Set probe for mapping

Set probe to **Spot**; turn JEOL optical microscope light **Off**

Set **Probe Size** to equal pixel size of map

Set **Magnification** to 50,000x (gives fine control of beam)

Select **Acquire – Start**

Select **Window – Tile Horiz** to look at all windows

8) After completion, save the maps.

ProbeImage will save the files automatically, but just in case save again.

Select **File – Save All**. This may take a while and does not indicate what the PC is doing.

Images are by default in User Image File; move to your folder

Important! If map needs to be stopped, **Acquire—Halt/Reset** may be used. However, this may cause the JEOL to hang. The JEOL will continue to map but PFE is not collecting data.

If the computers hang, log out or quit PFE. Reset OPE Power on the JEOL and start PFE again.

Step 3: Calculate quantitative maps in CalcImage

Start **CalcImage** by clicking on the icon.

CalcImage utilizes *.probimg files from the *ProbeImage* program

Note: CalcImage will devise rather complex file names so it may useful to change the name of the MDB file that contains the map elements to something like a sample number (DON'T DO WHILE MDB FILE IS OPEN).

Set up a project file

1) Select **Project —Create (new) Project Wizard**

The wizard will walk you through the file selection process.

Select **PFE database file** (.mdb) that includes the MAN values and standard calibration.

Select **Conditions Sample** (unknown that was setup after calibration)

There must be at least one unknown sample in the *.mdb* file. If oxides are mapped, Oxygen should be specified as "Unanalyzed" in the element file, and "Calculate with Stoichiometric Oxygen" should be set in the **Analyze** window.

Select **.prbimg files** (map)

Open an **image file** from the 1st map pass. Only one map from each set need be selected (e.g. MAP1_*00013*_WDS1_Si_TAP.prbimg).

The **Open** window will come back to open another set (i.e. next set of maps from same run if there were more than 5 elements mapped)

Open an **image file** from the 2nd set (e.g. MAP1_*00014*_WDS1_ Al_TAP.prbimg)

Press **Cancel** when done. All maps will now load.

A dialog box will open to save the CIP (Calc Image Project) file

2) Select **Project —Specify Quantitative Parameters**

Check **Calculate Totals Image** and **Calculate Stoichiometric Oxygen Image** to check quality of fit.

If oxide maps are maps are desired in addition to the elemental maps, check **Output Oxide Percents**.

Select each **Element** mapped, and **Read** the appropriate map

(e.g. Na — MAP1_00013_WDS3_Na_TAPH.grd)

If MAN backgrounds were used, no off-peak maps will be needed.

Click **OK**. Save the *.CIP file again.

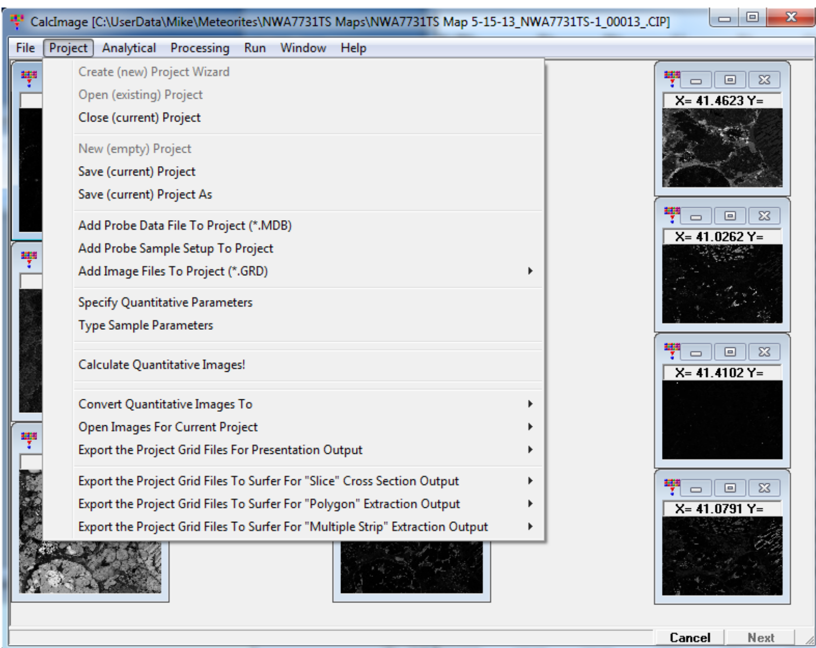


Figure QM-2. CalcImage Project Menu. Quantitative map processing is done from this menu.

3) The quantitative setup can be checked by selecting **Project —Type Sample Parameters**

Check that everything looks correct.

4) Select **Project —Calculate Quantitative Images!**

Calculations may take awhile (20-30 minutes). Check bottom status bar.

When complete, dialog window will ask to save the data into an ASCII file "_Quant_Image_Classify.dat"

Step 4: Process and Display images

You can use the current map or open a previous project file for processing.

Select **Project —Open (Existing) Project**

Navigate to the folder containing your maps and select the *.CIP file.

Process elemental maps for display:

Select **Project —Export Project Grid Files for Presentation—Output Quant Maps to Surfer**

A file *_Quant.bas" will be created and saved. The *.bas file is a Surfer Scripter™ file that will format and plot the maps. By default, it will print 4 maps to a page.

The software will ask if you want to run the script now. If the default 4 maps per page is fine, hit **Yes** to run the script.

Otherwise, hit **No** and edit the script before running (see Frequently Asked Questions for how to edit the Surfer script).

Process *oxide* maps for display:

Note: if oxides were not calculated, "Calculate Quantitative Images" will need to be repeated with oxides set (see" Select Project —Specify Quantitative Parameters" above).

Select **Project —Export Project Grid Files for Presentation—Output Oxide Percent Maps to Surfer**

To change intensity scale on quantitative maps

To stretch the contrast on a single map display or on an individual map on 4/page displays:

From the folder containing the maps, open the *_quant.srf file

Click on the map area to select it.

On left side, under **Property Manager—General—Colors**, click the button:

..

Change the scale at the bottom of the dialog box; e.g., to stretch the contrast, enter a smaller number in the "Maximum" box.

Click **OK**

To change to 1 quantitative map per page instead of 4

To change from the default 4 maps/page display to a single map display, it is necessary to edit the Surfer script.

From the folder containing the maps, open the *.bas file.

To change pages from 4 maps/page to 1 map/page:

Scroll down to around line #75 and look for "'Specify number of plots per page (must be 1, 4, or 9 images per page)"

Change “PlotsPerPage% = 4” to 1

Save the script.

Press the play button ▶ to run again after editing script.

(Note that scripts are path specific. You will need to modify directory path if the files are moved to another computer for processing.)

Process maps for polygonal area calculations:

Select **Project —Export Project Grid Files for Presentation—Output Quant (Elemental) Polygon Maps to Surfer**

A Scripter file will be created and you will be asked if you want to run the script. By default, the first map in the file will be used to draw the polygon. If this is a trace element map, don't run the script yet. Edit the script to use a map with higher elemental concentrations (see below).

Run the *_Polygon.bas script.

A dialog window will open to manually digitize an area on the map (if the area has been previously digitized, click "Finished Digitizing").

1) Select the image map
2) Select **Digitize** from the Map Menu
3) Left click on the image where you want the corners of the polygon to appear (note that it will automatically connect start and stop points).
4) In the "Digitize" popup box that appears with your data points, select **File—Save as**
5) Save the blanking file (*.bln) just created as "Digitized.bln" and save into current map data folder.
6) Click "Finished digitizing."

Scripter will calculate average values of all the elements in the ploygon and draw image files, saved as JPEG files.

To change the default map used for polygon extraction

To change from default map for polygon extraction or slicing, the Surfer script must be edited:

From the folder containing the maps, open the *_polygon.bas file.

Scroll down to around line #66 and look for "'DigitizeGRDNum%=1"

Change the number to correspond to a major element map such as Si, Fe, Mg, etc.

Save Script

Run the *_Polygon.bas script.

Step 5: (Optional) Perform phase analysis from CalcImage

You can use the current map or open a previous quantitative project for phase analysis. Note: the quant setup (*.BMP) must be in the same folder as the quant maps.

Select Project—Open (Existing) Project

Navigate to the folder containing your maps and select the *.CIP file.

Classify map image using Cluster Analysis

Select **Image Processing—Classify Image (from CalcImage quantification)**

Click the "Browse for Classify DAT File."

Look for "*_Quant_Image_Classify.DAT" file and Open it.

Set **Number of Clusters to Classify** to between 2 & 32. The number of clusters = number of phases. If you have some idea of how many phases are present, start with that. Too few clusters will cause phases to be lumped together so more is better. I usually start with something like 8 or 9.

Set **Iteration Tolerance** between 10 and 0.00001. This is a fractional value, i.e. 0.01 = 1% precision.

Select the "**Custom**" color radio button (loads the JEOL color table). One can also use other color tables such as "Rainbow," which tends to have better phase color separation.

Click the **Classify Clusters** button. Note that tighter tolerance and more clusters will run slower.

When complete, one needs to determine what phases the colors represent and whether the algorithm did a good job.

Some tools to help explore the clusters:

Load QuantMeter to load an interactive tool that shows composition at each pixel.

Load Analog Image to read in the BSE image or a map image.

One can extract the better data, if for example, there are a lot of really high or low pixels.

Select **Image Processing—Classify Points (from Probe for EPMA quantification)**

Browse for Classify input data file (*_Image_Classify.DAT). Load this file.

Set the Min and Max range, e.g. 85-115%. Click on "Extract Range Based" on Total and note file name.

Now open **Image Processing—Classify Image** again and load this file saved above. Pixels that fall out of Min-Max range are black.

Add clusters and tighten tolerance. Click **Classify Clusters** to redo analysis.

Deselect elements if they don't have much data (e.g. minor elements) by unchecking the box under "Select Data Channels to Include" at the left side of the window. Click **Classify Clusters** again.

Once you have a good set of clusters, proceed to **Modal Analysis** below.

Calculate Modal Abundances

Select **Image Processing—Calculate Modal Abundances (from CalcImage classification)**

Click the "Browse for Input TXT File."

Look for "*_Quant_Image_Classify.TXT" file and Open it.

This will load the clusters and their compositions from the classification process.

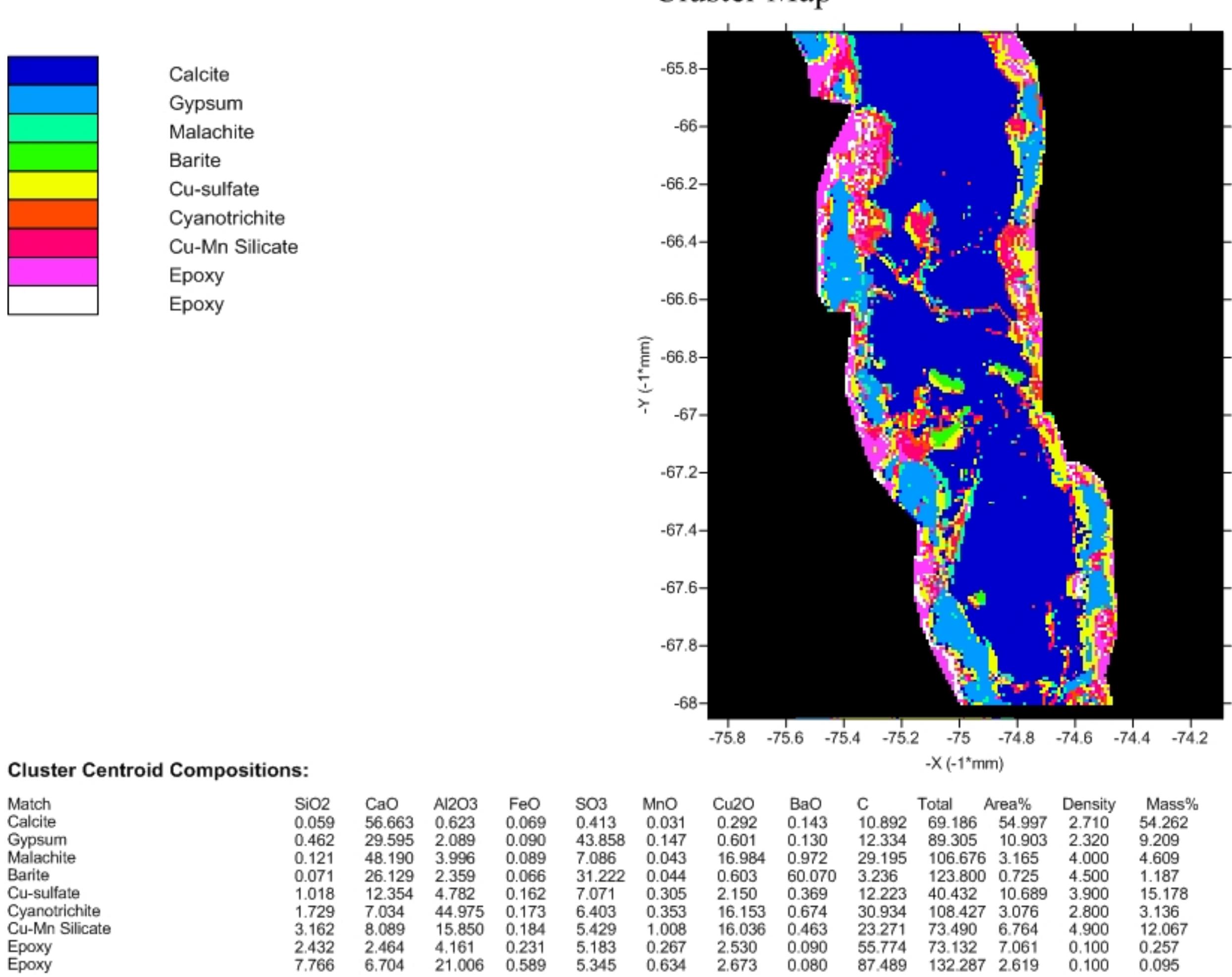
You can try match to a database (AMCSD or DHZ included in PFE) but most likely you will need to define phases yourself based on exploration in the clustering process.

Once you have decided on each cluster, enter a name in the "Match" column and press **Return**. Likewise, look up a viable Density and enter it followed by a **Return**.

Click **Calculate Modal Parameters.**

This will calculate Mass% of each phase and allow you to produce a nicely formatted output file by running the Surfer BAS file. This produces both a JPEG file and a Surfer file, shown in Fig. QM-3 below. The file can be edited by opening the “*IMAGE_CLASSIFY.SRF” in Surfer and editing the various parts.

Save the edited file, then export to a JPEG (or other format) file.



Cluster Centroid Compositions:

Match	SiO2	CaO	Al2O3	FeO	SO3	MnO	Cu2O	BaO	C	Total	Area%	Density	Mass%
Calcite	0.059	56.663	0.623	0.069	0.413	0.031	0.292	0.143	10.892	69.186	54.997	2.710	54.262
Gypsum	0.462	29.595	2.089	0.090	43.858	0.147	0.601	0.130	12.334	89.305	10.903	2.320	9.209
Malachite	0.121	48.190	3.996	0.089	7.086	0.043	16.984	0.972	29.195	106.676	3.165	4.000	4.609
Barite	0.071	26.129	2.359	0.066	31.222	0.044	0.603	60.070	3.236	123.800	0.725	4.500	1.187
Cu-sulfate	1.018	12.354	4.782	0.162	7.071	0.305	2.150	0.369	12.223	40.432	10.689	3.900	15.178
Cyanotrichite	1.729	7.034	44.975	0.173	6.403	0.353	16.153	0.674	30.934	108.427	3.076	2.800	3.136
Cu-Mn Silicate	3.162	8.089	15.850	0.184	5.429	1.008	16.036	0.463	23.271	73.490	6.764	4.900	12.067
Epoxy	2.432	2.464	4.161	0.231	5.183	0.267	2.530	0.090	55.774	73.132	7.061	0.100	0.257
Epoxy	7.766	6.704	21.006	0.589	5.345	0.634	2.673	0.080	87.489	132.287	2.619	0.100	0.095

Fig. QM-3. CalcImage Cluster Analysis of a secondary copper sample using 9 phases with 1% tolerance.

BASIC X-RAY MAPS WITH JEOL SOFTWARE

Step 1: Set initial column conditions

Set accelerating voltage and beam current to appropriate values before starting. You would generally want to use a higher beam current to increase X-ray counts for your map. 30-50 nA is suggested.

Step 2: Start setting up your map analysis file

In the **EPMA Main Menu** select **Analysis** and go to **Map Analysis**.

Click on **Sample**. Select or create a group. Within the group select or create a new **Sample**.

Click on **Measurement** and go to **Element Condition**.

Click the WDS **Element** button, and select the elements to be measured with WDS.

Click **Measurement Order** and arrange the elements, usually 1 per channel. Try to assign trace elements to Ch 2, 3, or 4. Click **OK**.

Click the **Condition** button. Check the **PHA Gain** and **High V.** against the plot in the back of this manual. Change if necessary and click **OK**.

If major elements are to be defined on the EDS, click EDS **Element** button, and select the elements to be measured with EDS.

Close the **Element Conditions** window.

Step 3: Do peak searches for your selected elements

In the **EPMA Main Menu** select **Monitor** and go to **Peak Search**.

Click on the **Element** button; select **Element** and then the **Map** radio button. This will show you the elements you have assigned to the map.

Select one element at a time, go to the standard that you want to use for this element and click **Search** (select **"Pksk no."** of 2 for a more accurate peak position and make sure peak is measured as **"Max"**). Save and write down the new peak position.

Click the **Save** button when the search is complete for each element.

Update the peak positions in your **Element Condition** setup in the **Map Analysis** file (click on each element, the new peak positions are usually in the back and you can update them by just clicking OK).

If you selected elements to be measured with EDS:

In the **EPMA Main Menu** click **EDS**, select **EDS** and collect spectrum (either on your sample or on standard minerals similar to your sample).

Check that the dead time is between 30 and 50%. Change aperture or process time (usually T2 or T3, T4 only for light elements) accordingly. With a smaller number aperture, the dead time will be higher.

Determine high and low energy values for each element Region of Interest (ROI).

Step 4: Continue setting up the Map Analysis file

Go to your sample and select a region for your map. Adjust **Contrast** and **Brightness**.

Define your map area

Get an image of map area. Go to ruler on **EOS panel** (turn off crossbar to see ruler button) and measure area. Calculate the number of pixels and step size necessary to cover area.

Setup the map

From the **Map Analysis window**, select **Measurement** and go to **Stage Condition**.

Click **Position Input**. Enter a comment to name the map.

Select **Scan Type**: usually **"uni"** (stage is moving only in 1 direction, **"bi"** = stage is moving back and forth; use only for very large maps).

Use "**beam**" maps only above 1000x.

Select **Stage Drive**. Use "**Micro**" at 1 μm steps (pixel size) or smaller for precise control.

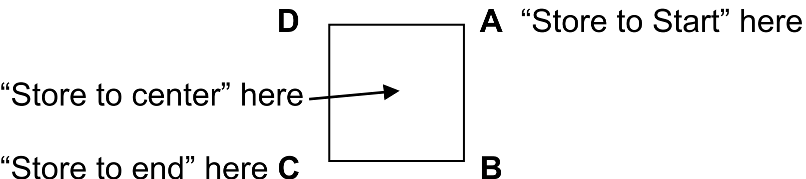
Enter **Pixels** and **Pixel Size** to give the appropriate size for the map as defined above.

Select **Dwell Time** considering the total measurement time.

For WDS maps, at least 50-60 msec dwell time is needed. Modify number of pixels (and size) to adjust total map time. Use only 1 **Accumulation** for WDS maps.

Move to center or corner of map.

Press **Read**, then press **Store**. Select **Store to center** or **Store to end/start** depending on where you are.



Note: Map starts at A, records each line toward B and advances toward D.

Press **Confirm**. Stage will move to starting point. Check focus and press **Test** button on joystick box. If focus is Ok, press **Store button.**

Stage will move to each of the four corners to check focus. If focus changed appreciably between points, repeat Confirm.

Press **Apply** and then **Close** to complete the map area setup.

Step 5: Setup the remaining conditions

Click on **Measurement** and select **Element Condition**.

Under **IMS**, click on **Signal**.

Adjust **Contrast** and **Brightness** on your sample to get a pleasing image.

Click on **Compo** button and then press **Read** to set to current values. If Read and Set are grayed out, uncheck the box at the right of **Compo**, then click **Read**.

Click **OK**

Select **EDS Condition**.

Check the **Start** and **End** voltage for the ROI for each element. Adjust voltage to avoid overlaps with adjacent peaks.

Click **OK** and **Close** to close the **Map Analysis** window.

Select **EOS conditions**

Make sure that the **Probe scan** is **"off."** (Use Probe Scan “On” only for beam maps!).

Set **Probe Diameter** to desired size. Use a size similar to your pixel size set in **Map Analysis** window above

Read current conditions.

For long maps, set beam stabilizer. Under **Scan Conditions**, click **Stabilizer** and set to **“CL & Tilt”**.

Click **OK** to close EOS condition window.

Select **EDS Conditions** and **Read** current conditions.

Check that **Print-out conditions** are off.

Step 6: Start map

Click on **Measurement** and select **Preset Measurement**.

Check that conditions are correct. Click **Acquire**.

Step 7: Check that map has started acquisition

Go to **Process Menu** and select **Map Analysis** to check map progress.

Select **Real Time** and **Start**.

Note: as maps are collected the stage drives the Y-axis for each line and increments each line on the X-axis; however each line is displayed on the X-axis so images are rotated by 90°. The rotation can be compensated in **Map Analysis**.

Step 8: Transfer element maps from the JEOL to the PC

Maps can be quickly exported as gray-scale images using a plugin in ImageJ, moved to the PC, and color added using a macro.

To do rapid gray-scale conversion:

Note: transfer can be facilitated by creating an empty folder named "Data."

From the **Utility** menu, select **Graphics** and then **ImageJ**.

Under the **Plugin** menu, select **JEOL Raw File Opener**

Select **Bulk Raw File Conversion**.

Don't add a scale bar yet.

Click **OK**

Select Map Directory window will appear. Navigate to the folder containing the map files. Highlight the subfolder containing maps but don't open. Click **Select** button.

Next, the **Select Output Directory** window will open. Navigate to the "Data" folder created. Click **Save**.

The **Select Start and End Maps** window will appear. Drag across or click on maps and press **OK**.

File names will be automatically created.

Add scale bar to BSE map:

It is best to add the scale bar only to the BSE image rather than a scale bar to each map, in case further processing is needed.

From the **ImageJ** menu, select **JEOL Raw File Opener** again.

Select **Open Files in ImageJ**

In the **Options Box**, add **Scale**, **Scale Color** and **Location**.

Click **OK**

Select Map Directory window will appear. Navigate to the folder containing the map files. Highlight the subfolder containing maps but don't open. Click **Select** button.

Select a map (only works on one at a time). Click **OK**. Maps will open in ImageJ.

Find the map "...CP_#" and bring to the foreground (title bar will be blue).

Select **File — Save as — TIFF**

Navigate to your "Data" folder and enter a name (e.g. BSE"). Click **OK**.

Transfer to the PC and add color:

Open the "Homes (EPSPROBE)" shortcut. Copy files from the probe to the appropriate folder on the PC.

Open **ImageJ** on the PC (double click shortcut in upper left of monitor). There are two ways to run a macro to colorize the maps:

1) Drag and drop the folder containing the maps onto the ImageJ toolbar to open all the files in **ImageJ**.

    Select **Plugins — Open to JEOL**

        All open maps will be colored with the JEOL color table, including the BSE.

    Select **File — Save as — TIFF** and save each map (except the BSE--you don't want to color it).

2) Use a batch macro to color maps in a folder. To avoid coloring the BSE, leave it out of the folder for now.

    Select **Plugins — Batch JEOL**

        Navigate to your map folder and select it. Click **OK**.

        Select a destination folder.

        The macro will ask for a file name for each file and save it in the destination folder.

        Copy your BSE map with scale bar into the folder.

MISCELLANEOUS PROCEDURES

Collecting an image

Select and compose the image.

Activate the beam scan by pressing the **PRB scan** button on the EOS operation panel.

Press the **PCD** button out. If cross-hairs are present, switch them off.

Toggle between **SEI**, **BSE Compo**, and **BSE Topo** by using the **VIEW** button.

Adjust the image using the **CONTRAST** and **BRIGHTNESS** knobs (or press the **ACB** button to automatically set contrast/brightness).

Adjust **MAGNIFICATION** to a higher value than you intend to take the picture.

Adjust the **FOCUS** and **STIG** knobs to get a clear image.

Turn magnification back to the desired level. Don't change the focus.

EOS image storage

Images can be sent to the user's folder on the JEOL or to the User Images folder on the PC.

Set the File Path

You must first set a file path from the JEOL EOS display. This is important so that your images are not saved in someone else's folder.

1. Select the **Setup** tab on the EOS display.
2. Click on **Network Set.**
3. To send the images to the ***Solaris***, select your "**host name**" from host list at left side of window. The images will appear in the "Images" folder in your directory.

   To send the images to the ***PC***, select "jxa1 PFE" from the list. The images will be sent to "D:/UserData/UserImages/Incoming" on the PC.
4. Click on **Regist** button and the host should change to your selected host.
5. Click **OK**.

Collect the image.

1. Before starting, set the file name.

   Choose **Image** on the EOS display.

   Click on **Network Save**.

   Enter the filename, **and make sure to hit enter**.

DO NOT PUT A SPACE IN A SOLARIS FILE OR DIRECTORY NAME!!!

   Set the picture number (it will be incremented up by 1 with each photo).

   Select **CLOSE**.

2. Press the **PHOTO** button on the operation panel. The button will flash while the photo is being stored.
4. When finished, release the **FREEZE** button to return to a live image.
5. Subsequent images are saved by simply pressing the **PHOTO** button. The same file name will be used and the number incremented automatically.

FREQUENTLY ASKED QUESTIONS

How do I transfer data from the JEOL Solaris system?

There are several ways to move data from the probe to your computer. The easiest way is simply to “mount” the probe volume on your computer as a “read only” directory.

To a Windows (XP) computer:

Select **My Computer** and then **My network Places**

Select **Add Network Place**

Select the “Choose another network location” icon

Enter the network address for the probe computer and volume:

\\epsprobe.unm.edu\homes

Provide a name for the mount point, e.g. “JEOL Microprobe”

The “/export/home/” directory on the probe is now mounted on your desktop. Go to your directory within the “home” directory and drag and drop data files (text), maps (tiff), and images (bmp) directly to your computer.

To a Mac computer:

From **Go** on the **Finder** menu bar, select **Connect to Server.**

Enter the address:

smb://epsprobe.unm.edu

Click **OK** in the “SMB Mount” window (“homes” is the only choice)

The “/export/home/” directory on the probe is now mounted on your desktop. Go to your directory within the “home” directory and drag and drop data files (text), maps (tiff), and images (bmp) directly to your computer.

How do I transfer data from the Probe for EPMA PC?

The D drive on the Probe for EPMA (PFE) system is a shared drive.

To a Windows 7 computer:

The shared drive is:

\\epsb03-d2ynyvv1\UserData

At the prompt, enter:

UserID: "colleges\your unm id"

Password: "your unm password" (don't include the quotes)

To a Mac computer:

From a Mac, in the Finder, select Go--Connect to Server

enter: cifs://epsb03-d2ynyvv1/UserData

TROUBLESHOOTING

Login

Problem: After logging in, instead of the Solaris Toolbar at the bottom and the JEOL Main Menu at the top of the screen, you find that you have a blank screen with maybe a File Manager window present.

Solution: Logout of this session by clicking with the right mouse button and selecting "**Exit**". At the login screen "Welcome to epsprobe", select the "**Options**" button at the bottom of the window. Then select "**Session**" and "**Common Desktop Environment**" from the list. Continue logging in normally.

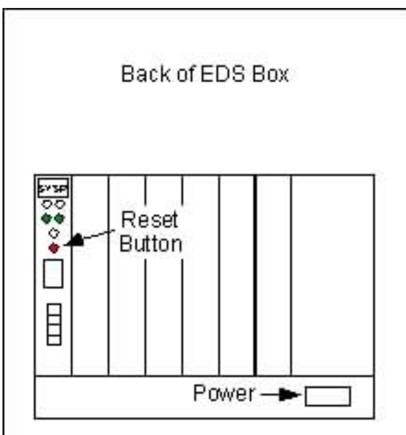
EDS Problems

Problem: The EDS exhibits strange behavior (e.g. very high dead time or very low acquisition rate).

Solution: **Exit** the EDS system. First try **Reset EDS** from the **EDS** menu in the **EPMA Main Menu** (below the option where you started the EDS). If the problem reappears, **Exit** the EDS system again, and reset the electronics in the EDS box at the left of the console. In the back, turn off the power to the EDS (see below), wait a few seconds and turn it back on.

Problem: After starting the EDS, a "Subsystem forbidden" error window appears.

Solution: First, **Exit** the EDS system. Turn off the power to the EDS (see below), wait a few seconds and turn it back on. Reopen the EDS application.



Hard reset of the EDS system is accomplished by switching the power off.

Problem: Sudden loss of EDS quality (poor spectrum resolution, high noise).

Solution: Check that a spectrometer light, the OM or OML light is off. To check spectrometer lights, select **Ratemeter** from **Monitor** menu. Click on each Channel button to see that "Spc Lamp" is off.

Problems with the stage

Problem: Stage control is lost.

Solution: First, check the "**STOR**" and "**TEST**" buttons on joystick. If they are lit (green), there is another problem (see "Lost Communications" or "Erratic Behavior" below). If the buttons are not lit, follow this procedure:

1) Open the black plastic door on the front of the probe console. Flip **OPE Pwr** switch off, count to 10 and flip back on.
2) Wait until menu returns on the left monitor. "**STOR**" and "**TEST**" buttons should now be green.
3) From the **EPMA Main Menu**, select the **JEOL** menu and **Connect EPMA system**. Hit the "**Yes reconnect**" button. Click **OK** in the Network Card Connection box.
4) Turn filament power back on by clicking in the far upper left button of the EOS monitor screen. Check that beam current is stable and continue with your work.

Lost Communications

Problem: Lost communication to Sun workstation.

Solution: First, try to "Reconnect the network." Under the **EPMA Main Menu**, pull down the **JEOL** menu and select **Connect EPMA System** near the bottom. Hit the "Yes reconnect" button. Shortly another window will come up. Answer "OK." System should now respond as normal.

If the problem includes monitor windows, such as the EOS Monitor or Stage Monitor window, taking an exceptionally long time to fill in values, restart the Sun computer.

Under the **EPMA Main Menu**, select **Initialize** and **System Shut Down**. After white screens stop and monitor turns black, press **ON** button on the front of the Sun computer, just to the left of the "SunBlade 2000". It will take a few minutes to start up (ON button and light on Plexwriter should be green. Log in as usual.

Erratic Behavior

Problem: Stage behaves strangely; jumping to unexpected places, or when using the joystick, movement can be seen on the SEM or OM image but axis positions are not changing in the Stage or "Monihome" windows. Test by adjusting coarse beam current with knob while watching beam current reading in EOS Monitor to see if it changes.

Solution: There is a zombie program hanging the system. Under the **EPMA Main Menu**, select **Initialize** and **System Shut Down**. After white screens stop and monitor turns black, press **ON** button on the front of the Sun computer, just to

the left of the “SunBlade 2000”. It will take a few minutes to start up (ON button and light on Plexwriter should be green. Log in as usual.

If the problem is still present, restart both systems:

1) Under the **EPMA Main Menu**, select **Initialize** and **System Shut Down**.
2) Open the black plastic door on the front of the probe console. Flip **OPE Pwr** switch off, count to 10 and flip back on.
3) After white screens stop and monitor turns black, press **ON** button on the front of the Sun computer, just to the left of the “SunBlade 2000”
4) Log in as usual.
5) Turn filament power back on by clicking in the far upper left button of the EOS monitor screen. Check that beam current is stable and continue with your work.

Cannot Use Beam Stabilizer

Problem: The BST returns an error message and cannot be used.

Solution: Make sure aperture #4 is in position. The BST cannot be used on any other aperture.

Note: BST does not function well at beam currents of 75 nA or higher (>$7.5x10^{-8}$).

Stage File Error in JEOL

Problem: After setting stage points, you receive an error message, or when trying to open an existing Stage Conditions file. Message says that the Stage Condition is broken and wants to delete the file.

Solution: Click cancel—don’t delete your file (unless you want to set new points). Open a **File Manager** window (select the file drawer icon on bottom Solaris menu bar):

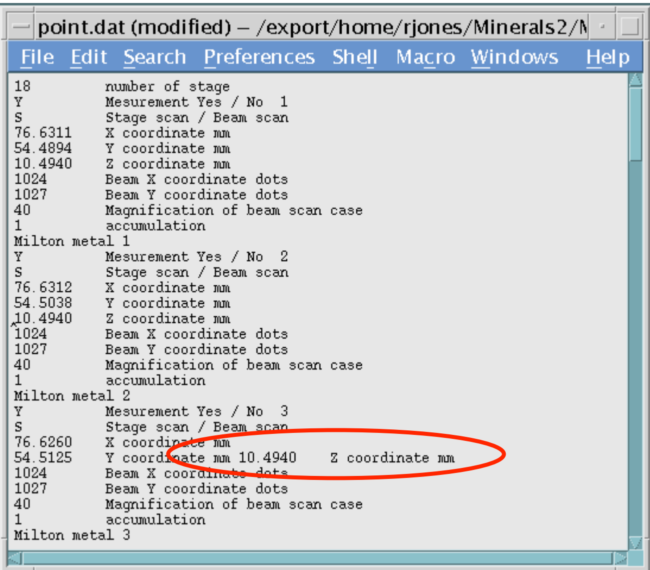
Open the group file (e.g. Minerals)

Open the sample file (e.g. “Pyx_June1” in the “Minerals” group)

Double-click on “**point.dat”** to open the file in **nedit**, the text editor. Scan through the file and look for anything unusual, such as an extra long line. (See figure below).

Edit the file so that there are values for X, Y and Z (and rotation if present).

Save the file (select **File—Save**) and exit **nedit**. You should be able to open the Stage Conditions now.



Example of text from **point.dat** file. Note that the Z coordinates have become wrapped with the Y coordinate line, causing a file error.

Images Disappear

Problem: After taking a photo, the image cannot be found.

Solution: First, check that you have registered the **Network Set** to your host name (See earlier section on **Collecting an Image**). You may be saving files to another user's directory.

As you take a photo, a gray window will appear over the image indicating that it is photographing. Immediately following the "Photographing..." window, another window will appear briefly, saying "Transferring file." If the window is so brief that you cannot read "Transferring File," then your image has not been saved. Open **Network Set** and type in your password again, being sure to hit **return** after you finish typing.

BSE Image Lost

Problem: Cannot get an image with the BSE detector (completely dark or light)

Solution: First, make sure you are on something on the stage. The metal web between standards or the metal sample holder works well. Turn off the optical microscope light. Also make sure that you scanning in **Fine View** mode.

First try the automatic contrast & brightness setting. Press the **ACB** button on the **EOS operation** panel.

If this fails, manually set the brightness and contrast to initial starting values. In the **EOS Monitor** window select **Contrast/Bright.** With the beam current around 20 nA, enter 3100 into the **Contrast** box and 2300 into the **Brightness** box. This should bring the contrast and brightness to a level that you can now adjust the contrast up or down with the brightness to get the desired contrast level.

Manual alignment of the electron gun

Use this method when the Automatic alignment fails with an error.

1. Verify that the CL Coarse number is 40-50 in the EOS monitor. Use **PROBE CURRENT** knob to adjust. Beam current should be in the 10^{-9} A range.
2. Go to a spot on the sample holder.
3. In the **EOS Monitor**, select **Filament** and click on **EMP** in the **Filament Window**. If you don't see the emission pattern on the left screen, adjust contrast and brightness.
   a. Press the **ALIGN** button on the operation panel to display the **Alignment** menu on the EOS viewing display, and select **Gun Alignment Tilt**.
   b. Select the scanning speed **S1**, and move the EMP pattern to the center of the viewing CRT using **ALIGNMENT X-Y** knobs.
   c. Select the speed **S3** and set the center of the EMP to the cross point of the cursors using **ALIGNMENT X-Y** knobs.
4. Click on the **EMP** button in the **Filament Window** to release EMP mode.
5. Press the **PCD** button **IN** (green).
6. Go to the **Monitor** menu in the **EPMA main menu** and select **Chart Recorder**.
   a. Deselect all buttons in the lower left of the window EXCEPT "**P. Current**".
   b. Set **Speed** to 1 minute.
   c. Click on ▶ to start recorder.
   d. Maximize the probe current using **ALIGNMENT X-Y** knobs.
   e. Click on ■ to stop recorder, then press **Clear** to clear the screen.
7. Adjust the probe current to around 10^{-7} A with the **PROBE CURRENT** knob.
   a. In the **Alignment** menu on the EOS viewing display, and select **Gun Alignment Shift**.
   b. Click on ▶ to start recorder.
   c. Maximize the probe current using **ALIGNMENT X-Y** knobs.
   d. Click on ■ to stop recorder, then press **Clear** to clear the screen.
8. Adjust the probe current back down to around 10^{-9} A with the **PROBE CURRENT** knob.
   a. In the **Alignment** menu on the EOS viewing display, and select **Gun Alignment Tilt**.

b. Click on ■ to start recorder.
c. Maximize the probe current using **ALIGNMENT X-Y** knobs.
d. Click on ■ to stop recorder.

9. Press the **STIG** button to close the **Alignment** menu.

Full Instrument Shutdown (Emergency)

If case of an emergency such as a power outage, sudden loud noises from the vacuum pump, fire, etc. do a full shutdown.

Close all windows other than the EPMA Menu.

Go to the **Initialize Menu** in the **EPMA Main Menu** and select **Gun Startup.**

In the "**Gun Automatic Startup**" program window, use option #5 (Shutdown). Enter **5 [Rtn]**.

Enter **Y [Rtn]**.

Once auto gun program is finished, press **[Rtn]** to exit the terminal window.

Push the **ACCEL VOLTAGE** button on the console to your left to shut off the accelerating voltage.

From the **Initialize Menu** in the EPMA Main Menu, select **SYSTEM SHUT DOWN.**

Click on **OK.**

After the computer shuts off, turn OFF the **POWER** key switch on the main panel.

Notify the lab manager.