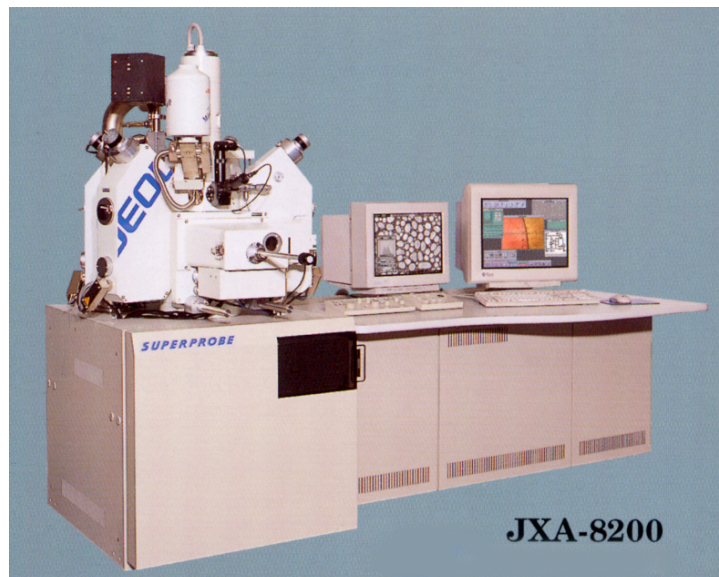


JEOL JXA-8200 SUPERPROBE OPERATOR'S MANUAL

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

LOGIN

- A. Enter your account login code and password.
 1. If the previous user has left the probe in an overnight run, you will need to enter your password to activate the screen.
 - a. Under the **Initialize** menu in the **EPMA Main Menu**, select **Logout**.
 - b. Select **OK**, wait for the Login screen and then enter your Code and Password.
- B. Login proceeds automatically, displaying the EPMA Main Menu on the monitor when it is complete.

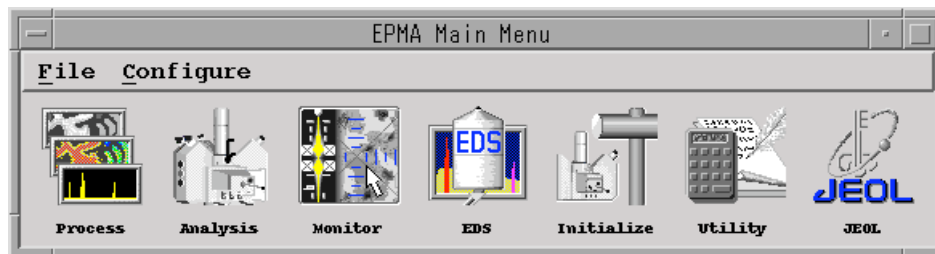


Figure 1. All microprobe software is accessed from the EPMA Main Menu.

CHANGE OR INSERT SAMPLES

If the previous user has left a sample in the chamber, for example on an over-night map, 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

- A. Make sure the **PCD** is “in” (button is lit).
- B. Go to the **Monitor** menu in the **EPMA Main Menu** and select **Stage Monitor**.
 1. From the **Stage Monitor** window, select **Sample Change**.
 2. Press **OK** in the “Stage: Sample Change” menu.
 3. Leave the next dialog window (“Stage”) up until sample exchange is completed. **DO NOT hit OK yet.**
- C. Press the **Vacuum Operation Button** on the **Specimen Exchange Chamber**. The button should be lit green.
 1. After about 90 seconds, the light should go out.

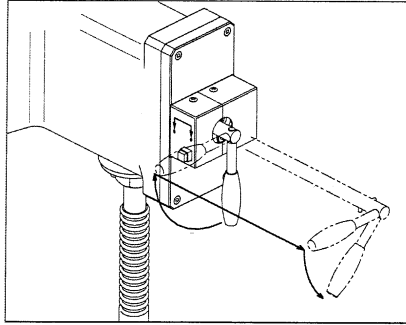


Figure 2. Operation of the Sample Exchange Door

2. Turn the **Isolation Valve Control handle** 90° clockwise or outward toward you (See Figure 2).
 3. Pull the **valve handle** all the way to the right.
 4. Turn the **valve handle** about 45° down to lock.
- D. Insert the **Specimen Exchange Rod** and turn the **knob** to the left.
- E. Withdraw the **Specimen Exchange Rod** all the way out.
- F. Close the **Isolation Valve** by reversing steps 1-4 above.
- G. Unlatch the **Specimen Exchange Door** and swing open.
- H. Remove the Sample Shuttle and Sample Holder by lifting shuttle straight up.
1. Remove the holder from the shuttle.
 2. If you are through with the sample exchange, return the shuttle to the Specimen Exchange Door and close and latch the door.
 3. 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

If you have just removed a sample, skip to section C.

- A. Make sure the **PCD** is "in" (button is lit).
- B. Go to the **Monitor** menu in the **EPMA Main Menu** and select **Stage Monitor**.
 1. From the **Stage Monitor** window, select **Sample Change**.
 2. Press **OK** in the "Stage: Sample Change" window.
 3. Leave the next dialog window ("Stage") up until sample exchange is completed. DO NOT hit OK yet.
- C. Secure your sample into the appropriate Sample Holder.
- D. Open the **Specimen Exchange Door**, remove the shuttle, and slide the holder onto the Sample Shuttle (see Figure 3).

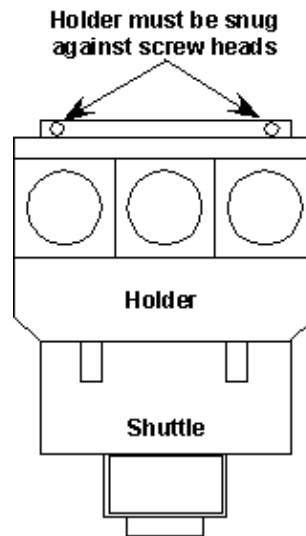


Figure 3. Proper orientation of the sample holder on the shuttle.

- E. Place the **Sample Shuttle** onto the **Sample Exchange** tray, making sure to engage the **Specimen Exchange Rod**. Close and latch the door.
- F. Press the **Vacuum Operation Button** on the **Specimen Exchange Chamber**. The button will light up green.
 1. After about 90 seconds, the light should go out.
 2. Turn the **Isolation Valve Control handle** 90° clockwise or outward (see Figure 2).
 3. Pull the **valve handle** all the way to the right.
 4. Turn the **valve handle** about 45° down.
- D. Push the **Specimen Exchange Rod** all the way in, turn the **knob** to the right, and then pull all the way back out.
- E. Close the **Isolation Valve** by reversing steps 1-4 above.
- F. Press **OK** in the “Stage” dialog window.
- G. If you have changed the type of sample holder, from the **Stage Monitor** window, select **Holder**.
 1. In the “Stage: Holder” window, select the appropriate holder. Type 3M150 holds 3 metallurgical samples, type 6M100 holds 6 round or 4 rectangular thin section, and the JEOL holder is a single brass holder.
 2. Click **Apply** and **Close**.

START-UP

HOT START (Normal)

- A. Confirm that the HV READY lamp on the main panel lights up or that the HT “button” in the upper left of the EOS (Electron Optical System) display is green.
- B. Go to the **Initialize Menu** in the **EPMA Main Menu** and select **LaB6 Gun Startup**.
 1. In the “Gun Automatic Startup” program window, use option #1 (Hot Start). Enter **1 [Rtn]**. Remember that the block cursor must be flashing for the program to accept entries: move the mouse cursor into the terminal window.
 2. Use the default “Filament code” (should be less than 150). **[Rtn]**.
 3. Select the program run time that is 7-10 minutes long. Enter **2 [Rtn]**.
 4. Confirm for the program that the HV is enabled. Enter **Y [Rtn]**.
 6. Once auto gun startup is finished, press **[Rtn]** to exit the terminal window.
- E. Go to the **Monitor** menu in the **EPMA main menu** and select **EOS Monitor**.







ALIGNMENT

Automatic alignment of the electron gun axis

- A. 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. If Auto Alignment fails after “Middle” search, use the **Manual Alignment Section** below.]
 5. Select “Auto Tilt” button and select **Start** again. This will refine the **Tilt alignment** and help that the beam current becomes more stable.
- B. Close the **Auto Alignment** window. Proceed to “**OL aperture alignment**” below.

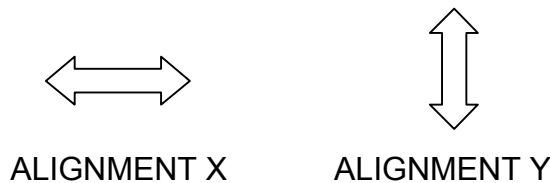
Manual alignment of the electron gun

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.

The OL aperture alignment of the electron column

- C. The mechanical aperture centering is necessary when the objective lens apertures are changed. When the accelerating voltage is changed, electrical centering of the aperture may be used.
1. Go to a spot on the sample holder.
 2. Press the **PCD** out and switch **PRB scan** on.
 3. Focus the optical microscope.
 4. Find a small image feature in SEI mode at the center of the viewing display using image magnification of ~1,000x.
 5. Press the **RDC IMAGE** button on the operation panel.
- D. Select the appropriate **OL aperture** for the intended purpose: use aperture #4 for microanalysis and #1, 2, or 3 for high magnification imaging.
- E. Verify **CL** Coarse number is 40-50 in the **EOS monitor** (adjust with the **PROBE CURRENT** knob).
- F. Adjust the **CONTRAST** and **BRIGHTNESS** knobs on the operation panel for the SEI, and focus the image.
- G. Press the **WOBB** button on the operation panel and the focus of the image changes periodically.
- 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:



- H. Press the **STIG** to release the wobble function.
- I. Making sure the **STIG** button is lit, adjust the **ALIGNMENT X-Y** knobs and the **FOCUS** to get the sharpest image.
- J. Press the **RDC IMAGE** button to release its function.

In case you just brought the filament up, wait at least 15 minutes before doing any Quantitative analyses.

COLLECTING AN IMAGE

Electron imaging

There are several ways to get an image on EOS system.

- A. Manually (buttons will be green when active).
 1. Activate the beam scan by pressing the **PRB scan** button on the EOS operation panel.
 2. Press the **PCD** button out.
 3. Toggle between **SEI**, **BSE Compo**, and **BSE Topo** by using the **VIEW** button.
 4. Adjust the image using the **CONTRAST** and **BRIGHTNESS** knobs (or press the **ACB** button to automatically set contrast/brightness).
 5. Adjust **MAGNIFICATION** and **FOCUS** knobs to get a clear image.
- B. Use the INSTANT button (starts image in SEI mode).
 1. Press **INST** button on the operation panel (if it was previously set, it may be necessary to hit it twice).
 2. Toggle to **BSE Compo** or **BSE Topo** (or toggle back to **SEI**) by using the **VIEW** button.
 3. Adjust the image using the **CONTRAST** and **BRIGHTNESS** knobs (or press the **ACB** button to automatically set contrast/brightness).
 4. Adjust **MAGNIFICATION** and **FOCUS** knobs to get a clear image.

EOS image storage

There are 3 ways to collect and store an image on the probe, through the EOS system.

- A. You must first set the file path to the folder where you would like to save the images. This is important so that your images are not saved in someone else's folder.
 1. Select **Setup** on the EOS display (left-most monitor).
 2. Click on **Network Set**.
 3. Select your "**host name**" from host list at left side of window.
 4. Click on **Regist** button and the host should change to your login name.
 5. Click **OK**.
- B. Method 1 (slower but higher resolution): Use the PHOTO button.
 1. Before starting, set the file name.
 - a. Choose **Image** on the EOS display.
 - b. Click on **Network Save**.
 - c. Enter the filename, WITHOUT SPACES (**make sure to hit enter**)
 - d. Set the picture number (it will be incremented up by 1 with each photo).
 - e. Select **CLOSE**.

2. Adjust the image on the EOS display.
 3. 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.
- C. Method 2 (faster but lower resolution): Use the FREEZE button and “Network Save”.
1. Produce an image on the EOS display
 2. Select **FINE** view on the operation panel (you may have to hit the button twice to get the slowest possible scan).
 4. Press the **FREEZE** button on the operation panel. It should start to flash.
 5. When the image is fully frozen, the **FREEZE** button will stop flashing and go solid green.
 6. Choose **Image** on the EOS display.
 7. Click on **Network Save**.
 8. Enter the filename, WITHOUT SPACES (**make sure to hit enter**)
 9. Set the picture number (it will be incremented up by 1 with each photo).
 10. Hit **Save**.
 11. When finished, release the **FREEZE** button to return to a live image.
- C. Other methods: Network Clip and Clipboard
1. Images may be saved indefinitely on the clipboards and can be loaded back to the EOS at a later time and transferred to the network.
 - a. Produce an image on the EOS display using SEI, BEI or Xray at the appropriate scan speed set from **EOS Monitor** window.
 - b. Choose **Image** on the EOS display.
 - c. Click on **Network Clip** or **Clipboard**.
 - d. Select an image box and click **Save**.

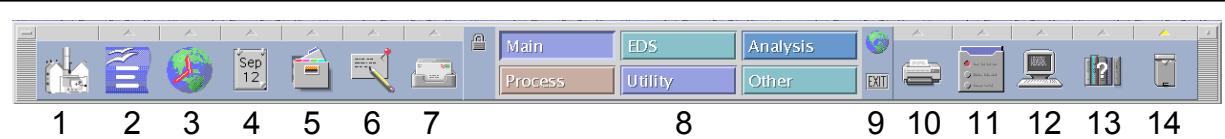
Other image collecting methods

You can also use the EPMA mapping program to take images. This can be useful for making montages, or taking large amounts of automated images. Please see the “MAPPING” section.

How to view and print a picture

Images are generally stored in the **Image** folder.

- A. To access your images, select the **File Manager** (see below) from the Solaris menu bar.
 1. Open the **Images** directory.
 2. Double-clicking on the image icon will open it in **ImageMagick**, a simple image manipulation program, with limited print control.
 3. To print an open image, left-click anywhere in the image.
 - a. Select **File** and **Print**.
 - b. Select **Letter**. Hit **Select** to print.
- B. For image editing and better print control, use **Gimp** (Openware equivalent of Photoshop).
 1. In the **EPMA Main Menu** go to **Utilities** and **Graphics**.
 2. Select **GIMP**.
 3. The GIMP menu will appear. Select **File**.
 - a. Select the **Images** directory from the left window and hit the **return** key.
 - b. Select the file name from the right window and hit **OK**.
 - c. To print or perform any function on the image, right-click anywhere in the image.
 - d. Select **File** and **Print**. Set **Scaling** and **Brightness**. Hit **Print**.

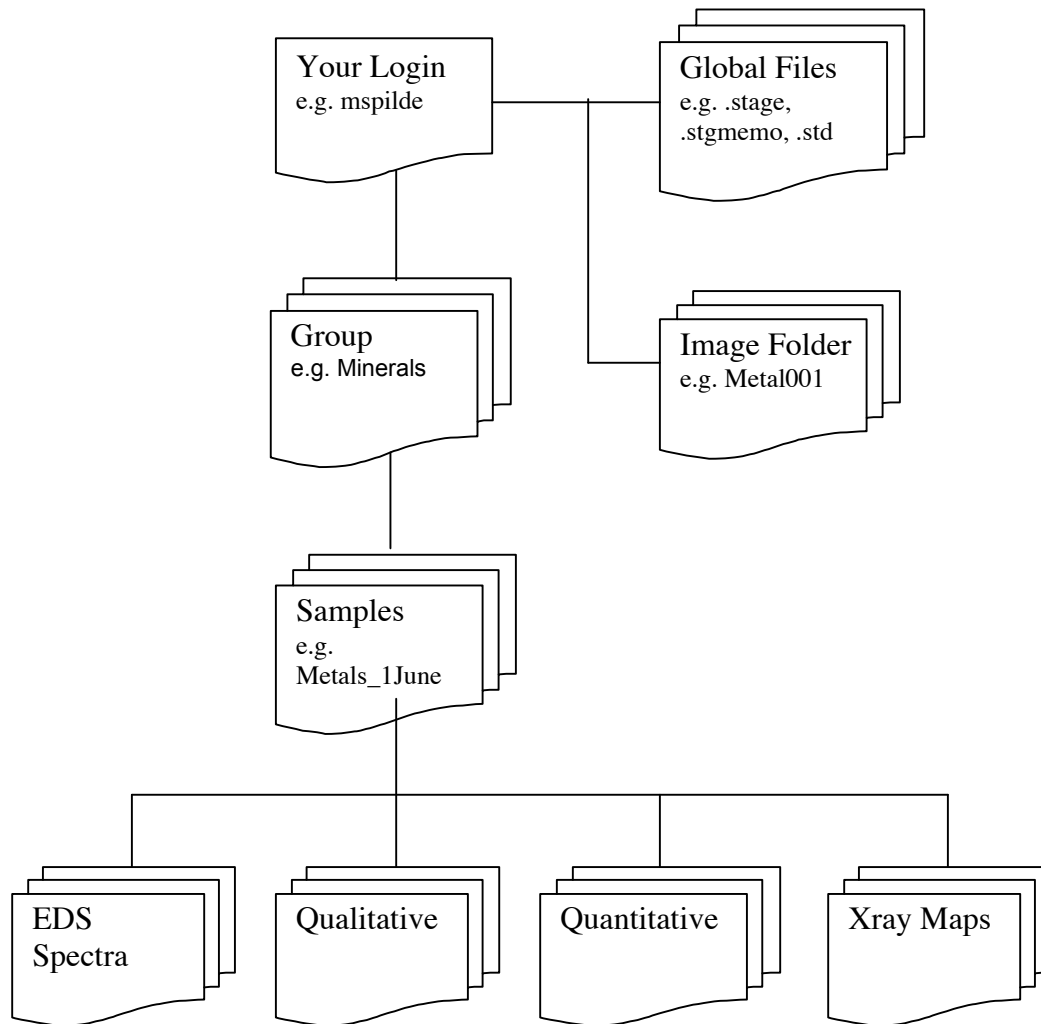


The Solaris Menu Bar:

- 1) Start the EPMA Menu (if it is closed or put away)
- 2) OpenOffice Suite (similar to Microsoft Office)
- 3) Firefox web browser
- 4) Calendar
- 5) File Manager
- 6) Text Editor
- 7) Mail
- 8) Desktop levels (like having 6 separate desktops)
- 9) Logout (use only after setting desktop preferences; use Logout from EPMA menu otherwise)
- 10) Print manager (we only have one printer)
- 11) Desktop controls
- 12) Terminal program (shell)
- 13) Help manager
- 14) Trash

File Structure on the Microprobe

Files stored in your login directory include standards, standard positions, job files (Groups), sample files and images.



Important Note About File Names

In the UNIX system, a space indicates that something different will come next, as in following command:

ls -al Minerals/Metals/ | more

(tells the computer to list the directory Metals in the text viewing utility “more”)

Therefore, a space in a filename tells the computer that a command or options will follow. It does not know that you meant for the space and following text to be part of the filename. Therefore, if you want to avoid shooting yourself in the foot:

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

QUANTITATIVE ANALYSIS

List of steps:

1. Set up **Quantitative Analysis** file (refer to [section A](#)). Decide which elements you want to measure and fill out a worksheet, which you can find in the back of this binder. Even if you use an old file, make sure that all element conditions are appropriate.
2. Using **Standard Analysis**, set points on the standards for the calibration (refer to [section B](#)).
3. Let the calibration run while having some green or red Chili.
4. Check if the calibration (refer to [section C](#)).
5. Write down the new peak positions (from **Standard Analysis**) and check if they are updated in the **Quantitative Analysis** file (under [Element Condition](#)).
6. Make sure that the calibrated standards are selected in the **Quantitative Analysis** file (under [Standard condition](#)).
7. Measure appropriate secondary standards and do analyses on your sample ([section D](#)).
8. Summarize the data ([section E](#))

There are **FOOTNOTES** in the following text for which you will find the explanations after [section E](#). We did this to shorten the text, but still have additional information available for you in case you need it.

A. Set up a “Quantitative Analysis” file

In order to work out problems before setting up standards, do the following Quantitative Analysis setup first, because then your Master file is available from the Standard - Element conditions files!

1. In the **EPMA Main Menu** select **Analysis** and go to **Quantitative Analysis**.
2. Click on **Sample**. Select or create a group. Within the group select or create a sample name.
3. Click on **Measurement** and go through the following points of the menu:
 - a. **Correction method**: Make sure that “**oxide**” is selected when you want to measure silicates or oxides. If you want to measure metals or sulfides select “**metal**”. Select “**metal**” also if you want to measure oxygen directly.
 - b. **Element Condition**:

Under **WDS**:

- ✓ Click on **Elements** and select the elements that you want to measure. Click **OK**.
- ✓ Click on **Meas. order** and ensure that every element is measured on the right crystal and in the right order (increasing atomic number from top to bottom or volatile elements first). You can change the order by dragging and the crystal with a right click on the element.
- ✓ Click on **Condition** and make sure that every element is measured in the way you want it; compare SCA data with information sheet in the back of this binder. Set appropriate times for measuring peak/background. Don't worry about the peak positions as they will be updated during the calibration. Choose **Diff** for elements measured on H-type spectrometers.
- ✓ Select **Peak Overlap** to look for possible overlaps (red are bad overlaps, yellow are possible overlaps). Set the backgrounds accordingly!

Under **CAL*1**:

- ✓ Click on the element that should be calculated. If you are calculating oxygen by stoichiometry, you should choose oxygen here.
 - ✓ Set the appropriate number of anions (oxygens) under “**Condition**”.
- c. **EOS Condition**: Set the instrument to the conditions (accelerating voltage, beam current, scan off, etc.) that you will use for measurement. Click on **Read** in the “**EOS Condition**” window. It is important that you do calibration and analyses at the same conditions!

B. Calibration (“Standard Analysis”)

1. In the **EPMA Main Menu** select **Analysis** and go to **Standard Analysis**.
2. Click on **Sample** and choose the standard material that you want to calibrate specific elements on^{*2}.
3. Move the standard of interest under the beam.
4. Click on **Measurement** and go through the following points of the menu:
 - a. **Standard Type:** Make sure that “**Oxide**” is selected if the standard mineral is a silicate or an oxide. If the standard mineral is a metal or sulfide select “**Metal**”.
 - b. **Element Condition:**
Under **WDS**:
 - ✓ Click on “**Element**” and select or deselect the elements that you want to calibrate on this standard.
 - ✓ Click on “**Condition**” and make sure that the element parameters are identical with the parameters that you just set up in the “Quantitative Analysis” file.
 - c. **EOS Condition:** Check in the EOS monitor that the high voltage, beam current and probe diameter have the values that you want to measure with and that the probe scan is off. Then click on “**Read**” in this window. The calibration and analyses should be done with the same values (except for Na and K → choose a larger beam diameter in calibration. 10 μm spot size is preferred on albite standard)!
 - d. **Stage Condition:** Now it’s time to select points on the standard for the calibration. Click on “**Pos. input**” and enter the name of the standard. Under “**Accumulation**” choose at least 3. Focus the beam in the optical microscope^{*3} and click “**Read**”, “**Store**”, “**Confirm**”. Use the **Store** button on the joystick to confirm the point, select more points and confirm every point with the **Store** button on the joystick. Be sure to focus with **up** button last. Click on “**Apply**” and close the window.
 - e. **Additional functions^{*4}:** Peak Search – “**Always**” for standards.
5. Do the same procedure for all other elements on other standards that you will measure (just select another standard under **Sample** and go through the same steps again).
6. To watch peak searches during calibration, select **Analysis – Peak search** before starting calibration. IMPORTANT: Do not open the Peak Search window after calibration has started. Opening the window after the calibration has started will result in errors in the search.
7. In the **Measurement** window of your last standard go to **Preset Measurement**, select all the standards you want to calibrate and click **Acquire**.

C. Check calibration

After the calibration is done, check each element in the calibration listing that is on the monitor screen. Make sure that the **s.v.** is below 0.5 and that the 3 values for the **Net cps** are close. If the standard was calibrated previously, see that “previous” values for peak position, counts, etc. are not significantly different.

Write down the new peak positions!

If you want to remove a bad data point, go to **Measurement** in the “**Standard Analysis**” window and select **Check Data**. Select **Acum** and check the standard data by clicking on the number of accumulations (turns into a button when “**Acum**” is selected). If a data point is bad, it can be removed by selecting it. Don’t forget to save! (Data is not deleted, it can be added back in by reselecting it.)

D. Measurements (“Quantitative Analysis”)

Before you do the analyses on your sample, you can use the same procedure described below to do analyses on a few standards to find out if your calibration is ok!

1. In “**Quantitative Analysis**” window, click on **Measurement** and go now through the following points of the menu:
 - a. **Element Condition:**
Under **WDS**:
Click on **Condition** and make sure that the peak position of every element has been updated (compare to peak positions that you wrote down after the calibration from the “**Standard Analysis**” listing). If a position is not updated in the front window, click on the element header, then click on the correct element header in the master element list window that opens. Click **OK** and it should be saved in the front window, too.
Select **Peak Overlap** again to see if the shifted peak positions cause any new overlaps. If so, set backgrounds accordingly.
 - b. **EOS conditions:** Click on **Read** to update the EOS conditions to make sure that the lens conditions are the same in the EOS monitor and the EOS conditions window. Also, before you “**Read**”, make sure that the beam current has still the value that you want to measure with and that beam scan is off.
 - c. **Standard Condition:** Make sure that the standards are those that you just calibrated (easiest way is to look at the dates!). You can change the standard by clicking on the element and choosing the correct one.
 - d. **Stage Condition:**^{*5} Click on **Pos. input**, give your analysis a name and focus the spot on your sample/standard that you want to analyze^{*3}. Click on **Read & apply**. Click on the down button ▾ to add a new point (if your sample name ends with a number, this will increment the number as a new point is added).

- e. **Additional function:** Peak search: usually “None”

Background measurement:

Set to “Always” for background measurement at each point. To measure background at 1st point and then some interval, set to “Every” and enter number such as 3 or 5 to measure every 3rd or 5th background.

Synchronous Measurement: “Asynchronous”

- f. **Preset Measurement:** Click on **Acquire** and the machine will start the measurements.

E. Summarize data

In order to transfer the data to a spreadsheet or word processing program, use must first summarize the quantitative analysis data.

1. In the **EPMA Main Menu** click on **Process** and select **Quantitative Analysis**.
2. Select **Summary**.
3. In the “**Summary**” window, click **Sample** button.
 - a. Verify the **Group** file. If it is incorrect, click on **Group** button.
 - b. Select group file and hit **OK**.
 - c. Select desired sample file from list and hit **OK**.
4. Click on **Summary** button.
 - a. By default, all box are checked and will be listed.
 - b. If you only want some analyses, uncheck the individual boxes or click the **Clear all** button, then the **Some** button and enter the range of desired analyses in the “Select some...” window, and click the **Select** button.
5. Click **Mass%** button (**Mass%** is the element wt%; **100%** is the element wt% normalized to 100%; **Atom** is the cation %; **Stage** provides the X, Y and Z stage coordinates).
6. Click the **Spreadsheet** button (with **Row** selected, each analysis is printed out across and elements down).
7. To change the order of elements listed in the printout, click the **List order** button in the upper left corner of the “**Summary**” window.
 - a. Click **Clear order** button.
 - b. Click in the check boxes for each element in the desired order. Leave box unchecked if you want to omit it.
 - c. Hit the **Type Out** button. Don’t close “**List order of elements**” window if you want to add cations or stage coordinates to the list.
 - d. To add cations, select **Atom** from the “**Summary**” window.
8. To save, click **Save** button on the “**Listing**” window.
 - a. By default the file will go to the sample file. You can define another directory in the top line of the window (hit **return** after) and a new file name in the bottom line of the window. Files can be .txt for Excel or .csv for other programs.

Quantitative analysis footnotes

- *1 - How to let the probe calculate compounds like CO₂ or OH⁻ when measuring carbonates or hydrated minerals, respectively. Setup for magnetite.
 - *2 - How to add a standard mineral to the list of samples.
 - *3 - Using Auto Focus during measurements.
 - *4 - Using separate standards for peak search and calibration.
 - *5 - Setting a line or grid of points.
-
- *1 - How to let the probe calculate compounds like CO₂ or OH⁻ when measuring carbonates or hydrated minerals, respectively. Setup for magnetite.
-

In the **Measurement** menu of your quantitative analysis file, select **Element Condition** and then **CAL**. Under **Elements** select the element(s) that should be calculated. Under **Condition** setup the following by clicking on the element:

Analyzing oxygen by stoichiometry:

Use O as anion (set “**Fraction**” to appropriate number of anions).

Analyzing carbonates:

Use O as anion (set to 3 anions).

CO₂ – Do as Atomic Ratio

No Elem	Fraction
1 *	3.00

Analyzing biotite:

Use O as anion (set to 11 anions).

Add OH by difference.

Analyzing magnetite:

Use O as anion (set to 4 anions).

Use XO to make up difference.

Analyzing hydrous mineral:

Use H calculated by Difference.

***2** - How to add a standard mineral to the list of samples.

1. In the “**Standard Analysis**” window under **Sample** click on **New** and enter the name of the standard mineral.
2. Under **Measurement** go to **Standard type**. Make sure that “**Oxide**” is selected if the standard mineral is a silicate or an oxide. If the standard mineral is a metal or sulfide select “**Metal**”.
3. Under **Measurement** go to **Element condition** and then **Standard composition**. Enter the composition of the standard mineral (you have to have the cursor inside the window where you want to write):

Standard type: oxide

(click on “**Mass**” and “**Horizontal**”)

Enter the oxide wt% value for every element in the standard

Example: Taylor spessartine

No.	Elem.	Val.	Value
1	Si	4	36.1700
2	Al	3	20.5000
3	Fe	2	2.2600
4	Mn	2	40.5000
5	Ca	2	0.0800
6	Sn	4	0.0700
7	Ti	4	0.0100
8			
9			
10			
TTL		7	99.5900

Buttons: OK, Cancel, Delete

Standard type: metal

(click on “**Atom**” and “**Horizontal**”)

Enter the atom% value for every element in the standard

Example: Taylor nickel

No.	Elem.	Value
1	Ni	1.0000
2		
3		
4		
5		
6		
7		
8		
9		
10		
TTL		1 1.0000

Buttons: OK, Cancel, Delete

***3** - Using Auto Focus during measurements.

You can use Auto Focus instead of having to focus every single point you save:

1. In the **EPMA Main Menu** under **Monitor** click **Auto Focus**.
 - a. Under **Mode** select **Auto**.
2. Select **Auto Focus** in the “**Stage Pos. Input**” window under **Standard Analysis** or **Quantitative Analysis**.
3. Be sure to leave the optical microscope camera on for your analyses! Be aware that adding the Auto Focus feature adds time to your analyses. So, if you have a lot of analyses running overnight, we do not recommend using it.

***4** - Using separate standards for peak search and calibration.

To use a separate standard for peak search and another for calibration, select **Synchronous** in **Additional Functions** menu. Under **Peak Search Position** select **Stage position**. Move to a strong standard to perform the peak search. Click **Read** button. When calibration is performed, stage will move to the peak search standard to locate the peak and then move to the calibration standard to take the measurements.

***5** - Setting a line or grid of points.

1. Press the **Line Set** button in upper left corner of the “**Stage Condition**” window.
2. To set line of points, select **Line** button.
 - a. Set start point. Be sure to focus carefully. Press **Read** button. Move to end point and focus. Press **Read**.
 - b. Set number of points or distance (width) between points. The computer will calculate the other parameter.
 - c. Set “**Segments**” if line is not in focus throughout. Can also use “**Autofocus**” but this will add time to each analysis.
 - d. Click **Apply**. Software will add points to stage file conditions file.
3. To set grid of points, select **Grid** button.
 - a. Set stage to upper right corner. Press **Read** at “**A.**”
 - b. Set stage to lower left corner. Press **Read** at “**B.**”
 - c. Set steps and/or width. Program calculates the other parameter.
 - d. Save the points. The grid will be added to the stage conditions file.

Map analysis

Set HV 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.

A. Start setting up your map analysis file:

1. In the **EPMA Main Menu** select **Analysis** and go to **Map Analysis**.
2. Click on **Sample**. Select or create a group. Within the group select or create a sample name.
3. Click on **Measurement** and go to **Element Condition**. Select the elements to be measured with WDS and EDS (for major elements), check if conditions and measurement order are ok.

B. Do peak searches for your selected elements:

1. In the **EPMA Main Menu** select **Monitor** and go to **Peak search**.
2. Click on the **Element** button, select **Element** and then **Map**. This will show you the elements you selected for the map.
3. Select one element at a time, go to the standard that you want to use for this element and do the peak 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.
4. 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).

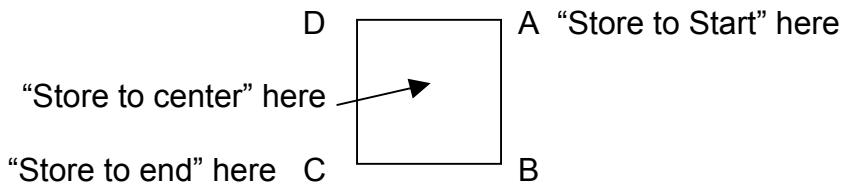
C. If you selected elements to be measured with EDS:

1. In the **EPMA Main Menu** select **EDS**, go to **EDS** and collect spectrum.
2. Check that deadtime is between 20 and 30%. Change aperture or process time (usually T2 or T3, T4 only for light elements) accordingly. With lower aperture the deadtime will be higher.
3. Determine high and low energy values for each element (ROI).
4. Compare these values with your **Element condition** setup under **EDS Conditions**. Change if necessary - default energy windows are usually pretty broad.

D. Continue setting up the Map Analysis file:

1. Go to your sample and select a region for your map. Adjust contrast and brightness.
2. Under **Measurement** go to **Element condition**, under **IMS** select **Signal**. Get a good BSE image, select **Compo** and press **Read** to set to current values. If Read and Set are grayed out, uncheck the box, click **Read**.

3. Select **EOS conditions** and **Read** current conditions.
 - 3.1 Make sure that the **Probe scan** is “off” in the EOS monitor before you read the conditions! (Use Probe Scan “On” only for beam maps!).
 - 3.2 For long maps, set beam stabilizer. Under **Scan Conditions** within the EOS condition window, click **Stabilizer** and set to “CL & Tilt”. Click **ok** to close EOS condition window.
4. Select **EDS conditions** and **Read** current conditions.
5. Go to **Stage Conditions** and select **Position Input**. Enter a comment to name the map.
 - 5.1 Select **Scan Type**: usually “uni” (stage is moving only in 1 direction, “bi” = stage is moving back and forth; doesn’t save time except for very large maps). Use “beam” maps only above 1000x.
 - 5.2 Select **Stage Drive**. Select **Micro** at 1 μm steps (pixel size) or smaller for more precise control.
6. Get image of map area, go to ruler on EOS panel (turn off crossbar to see ruler button) and measure area. Calculate number of pixels and step necessary to cover area.
 - 6.1 Enter **Pixels** and **Pixel Size** to give the appropriate size for the map as shown on the EOS screen.
 - 6.2 Select **Dwell Time** considering the total measurement time.
7. Move to center or corner of map.



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

- 7.1 Press **Read**, then press **Store**. Select **Store to center** or **Store to end/start** depending on where you are.
- 7.2 Press **Confirm**. Stage will move to starting point. Check focus and press store button on joystick box. Stage will move to each of the four corners to check focus. If focus changed appreciably between points, repeat Confirm.
8. **Close** the **Stage Condition** window.
9. Check that **Print-out conditions** are off.
10. Start map from **Preset Measurement**.
11. 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.

TRANSFER DATA

There are several ways to move data from the probe to your computer, including using the on-board Windows computer to drag and drop files to the LAN “Common” directory or using ftp to transfer files. The easiest way is simply to “mount” the probe volume on your computer as a “read only” directory.

A. From your Windows computer:

1. Select **My Computer** and then **My network Places**
2. Select **Entire Network** icon
 - a. **Search** for the probe computer (“epsprobe.unm.edu” or 129.24.36.60)
OR
 - b. Select **View Entire Contents** and follow the path:
Microsoft Windows Network
Workgroup
epsprobe
3. Select the “**homes**” folder.
4. 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.

B. From your Mac computer:

1. From **Go** on the **Finder** menu bar, select **Network**.
2. Select **WORKGROUP** icon
 - a. Select “epsprobe” icon
 - b. Click **OK** in the “SMB Mount” window (“homes” is the only choice)
3. 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.

REMOVE SAMPLES

- A. Make sure the **PCD** is “in” (button is lit).
- B. Go to the **Monitor** menu in the **EPMA Main Menu** and select **Stage Monitor**.
 1. From the **Stage Monitor** window, select **Sample Change**.
 2. Press **OK** in the “Stage: Sample Change” menu.
 3. Leave the next dialog window (“Stage”) up until sample exchange is completed. **DO NOT hit OK yet**.
- C. Press the **Vacuum Operation Button** on the **Specimen Exchange Chamber**. The button should be lit green.
 1. After about 90 seconds, the light should go out.

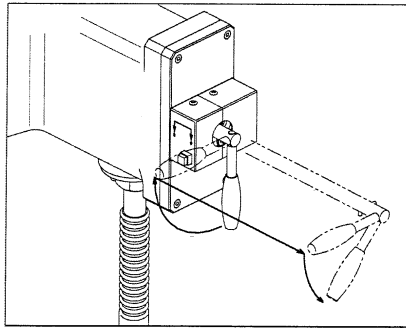


Figure 2. Operation of the Sample Exchange Door

2. Turn the **Isolation Valve Control handle** 90° clockwise or outward toward you (See Figure 2).
 3. Pull the **handle** all the way to the right.
 4. Turn the **handle** about 45° down to lock.
- D. Insert the **Specimen Exchange Rod** and turn the **handle** to the left.
 - E. Withdraw the **Specimen Exchange Rod** all the way out.
 - F. Close the **Isolation Valve** by reversing steps 1-4 above.
 - G. Unlatch the **Specimen Exchange Door** and swing open.
 - H. Remove the Sample Shuttle and Sample Holder by lifting shuttle straight up.
 1. Remove the holder from the shuttle.
 2. If you are through with the sample exchange, return the shuttle to the Specimen Exchange Door and close and latch the door.
 3. 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”.

SHUT DOWN

Standby (Normal)

- A. Close all windows other than the EPMA Menu.
 1. If Windows is running, click on the **Exit** button in the SunPC window.
 2. Select “Close Windows and Exit SunPC”
- B. Go to the **Initialize Menu** in the EPMA Main Menu and select **LaB6 Gun Startup**.
 1. In the “Gun Automatic Startup” program window, use option #6 (Standby). Enter **6 [Rtn]**.
 2. Input final “Filament code” of 85 or 90. **[Rtn]**.
 3. Enter **Y [Rtn]**.
 4. Once auto gun program is finished, press **[Rtn]** to exit the terminal window.
- C. In the **Initialize Menu** in the EPMA Main Menu, select **Log out**.
 1. Click on **OK**.
- D. Enter filament hours in log book.

Full Shutdown (Emergency)

- A. Close all windows other than the EPMA Menu.
 1. If Windows is running, see A. above.
- B. Go to the **Initialize Menu** in the EPMA Main Menu and select **LaB6 Gun Startup**.
 1. In the “Gun Automatic Startup” program window, use option #5 (Shutdown). Enter **5 [Rtn]**.
 3. Enter **Y [Rtn]**.
 4. Once auto gun program is finished, press **[Rtn]** to exit the terminal window.
- C. Push the **ACCEL VOLTAGE** button on the main panel to shut off the accelerating voltage.
- D. From the **Initialize Menu** in the EPMA Main Menu, select **SYSTEM SHUT DOWN**.
 1. Click on **OK**.
- E. After the computer shuts off, turn OFF the **POWER** key switch on the main panel

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.

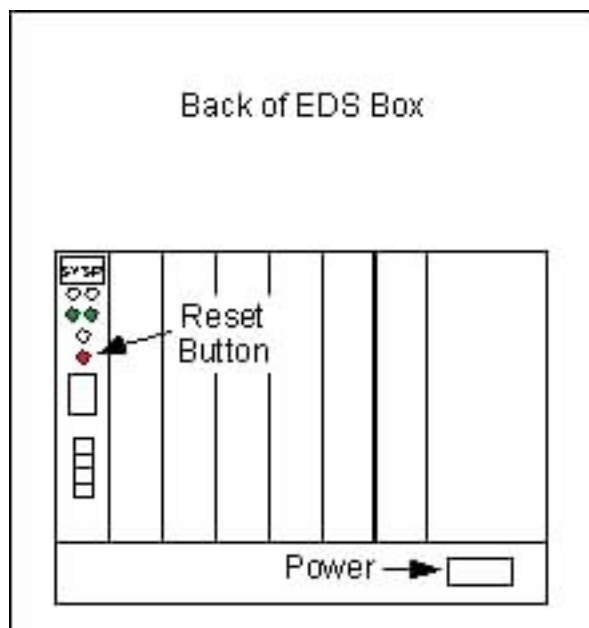
EDS Problems

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.



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**, go to **Utilities** and select **Connect EPMA system**. Hit the “**Yes reconnect**” button. Click **OK** in the Network Card Connection box.
- 4) 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.

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 ($>7.5 \times 10^{-8}$).

Stage File Error

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 menu bar):

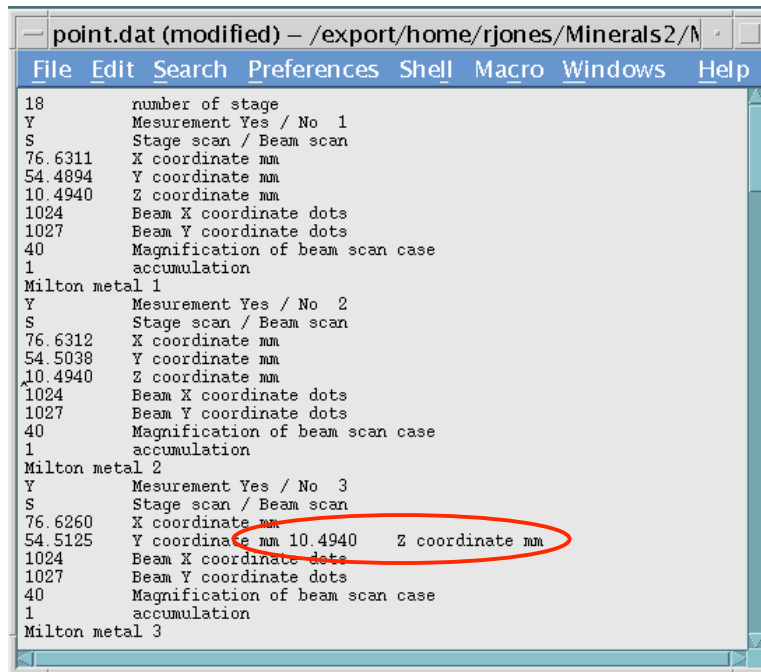
Open the group file (e.g. Minerals)

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.



The screenshot shows a text editor window titled "point.dat (modified) - /export/home/rjones/Minerals2/". The window contains a list of stage parameters for three different metals. The parameters are listed in a structured format with labels like "number of stage", "Measurement Yes / No", "Stage scan / Beam scan", "X coordinate nm", "Y coordinate nm", "Z coordinate nm", "Beam X coordinate dots", "Beam Y coordinate dots", "Magnification of beam scan case", and "accumulation". The parameters are grouped under "Milton metal 1", "Milton metal 2", and "Milton metal 3". A red circle highlights a line in the "Milton metal 2" section where the Z coordinate value "10.4940" is wrapped onto the same line as the Y coordinate value "54.5125".

```
18      number of stage
Y      Measurement Yes / No 1
S      Stage scan / Beam scan
76.6311 X coordinate nm
54.4894 Y coordinate nm
10.4940 Z coordinate nm
1024   Beam X coordinate dots
1027   Beam Y coordinate dots
40     Magnification of beam scan case
1      accumulation
Milton metal 1
Y      Measurement Yes / No 2
S      Stage scan / Beam scan
76.6312 X coordinate nm
54.5038 Y coordinate nm
10.4940 Z coordinate nm
1024   Beam X coordinate dots
1027   Beam Y coordinate dots
40     Magnification of beam scan case
1      accumulation
Milton metal 2
Y      Measurement Yes / No 3
S      Stage scan / Beam scan
76.6260 X coordinate nm
54.5125 Y coordinate nm 10.4940 Z coordinate nm
1024   Beam X coordinate dots
1027   Beam Y coordinate dots
40     Magnification of beam scan case
1      accumulation
Milton metal 3
```

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.