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U. S. GEOLOGICAL SURVEY

JEOL 8900 SUPERPROBE USER'S MANUAL

by

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SECTION 1: Introduction

Welcome to the United States Geological Survey's Electron Microprobe user's manual. The following will give the first-time operator the basic knowledge and procedures needed to perform analyses on the microprobe and provide the already familiar user with a quick reference guide. This manual will not go into the techniques and theory behind microanalysis, but will instead concentrate on the physical and practical operation of the JEOL 8900 electron microprobe. Each chapter is a detailed explanation of one particular aspect of the probe, complete with figures and references. A quick reference guide or list of items and procedures for the more experienced user is included for each section (where applicable) in Appendix B.

This manual was written for all three US Geological Survey microprobe laboratories and is intended to be an overall user's guide to the operation of a JEOL 8900 series Superprobe. To accommodate the differences in the labs, there are three separate layout descriptions in Section 3. Some local discrepancies of procedure will exist, and where appropriate the following designations have been made:

- (R) Applies to the Reston probe
- (D) Applies to the Denver probe
- (M) Applies to the Menlo Park probe

Check with the local lab personnel for site-specific procedures and consult the appropriate personnel if you have any questions regarding lab procedures not covered in this manual.

This manual does not attempt to include descriptions of every menu option found in the software and every button, knob, dial, or switch found on the hardware devices. Information about most of these items can be found in the JEOL reference manuals entitled JXA-8900 WDS/EDS BASIC SOFTWARE and JXA-8900S/M/L ELECTRON PROBE MICROANALYZERS and their supplements.

SECTION 2: Beginner's Guide to X-Windows

General introduction:

This section is mainly concerned with the operation of the computer software interface used to control the functions of the microprobe. Those who are familiar with UNIX's X-Windows or Microsoft's 'Windows' for DOS computers can skip this section as it deals with opening and closing windows and using the mouse. Apple/Macintosh computer users should note that there are differences between operating X-Windows and Macintosh windows systems, one major difference being that 'double-clicking' is usually not recommended, and is replaced with a more complex system of opening and placing windows. Other peculiarities in operation of the X-Windows environment are described below.

The operation of the microprobe is controlled by a Hewlett Packard Apollo workstation using an 'X-Windows' environment. X-Windows is a system of 'windows', 'menus', and 'icons' which has been programmed to control almost all of the functions of the microprobe.

A 'window' is essentially an extra terminal screen through which a procedure can be controlled. The use of windows allows more than one procedure to be running at a time. Generally the controlling software will create appropriately designed windows.

A 'menu' is list of options to choose from. To select one of the options given in the menu, you must use the 'mouse' which is located on a pad to the right of the keyboard.

Throughout this manual, the hierarchy of menu choices is indicated by the symbol "->", as in Main Menu->Sub-Menu->Sub-Menu.

The basic mouse operations are 'clicking', 'dragging' and 'double clicking'. A menu option may be selected by moving the mouse to point the cursor at the option, then 'clicking' the left button on the mouse once and releasing. Pressing the left button on the mouse and holding it down while moving the mouse is called 'dragging'. Pressing the left mouse button twice in rapid succession is called 'double-clicking' and is rarely used in the X-Windows environment.

An 'Icon' is a small picture that represents a menu item or window which is not open at the present time. Icons in the X-Windows environment are used to save space on the screen without closing windows completely. Further discussion of icons can be found below in the section describing windows.

Select menu options by moving the pointer over the box desired and clicking the left mouse button. A vertical list of options will be displayed. Move the pointer until the item you want is highlighted and click the left mouse button again. Once a menu option has been selected, an hourglass picture will be displayed indicating that the computer is busy loading software.

Important Note: Do not click the mouse buttons or type any characters while the software is loading, as this can lead to system lock-ups, loss of valuable operating time and damage to files.

After the software has loaded, a window outline will appear which can be moved about the screen by moving the mouse. Maneuver the outline to an appropriate position and click the left mouse button. A window will appear inside the outline. More than one window may be open at the same time, and they may overlap one another. In order to use a window when more than one is present on the screen, it must be 'active'. The active window is the window in which the

pointer is located. An active window usually has a green or blue border; an inactive window usually has a gray border. If the desired window is partially covered by another, it must be brought forward to the “top of the pile” by clicking on the window frame.

There are many control and display functions inside a window and along the border or frame around the window. Figure 2.1 is a basic window frame; the functions associated with it are described below:

1. **Title bar**...Displays the title of the window. Clicking on the title bar will bring a window to the top of the pile. The window can be moved by placing the pointer in the title bar, clicking and dragging to a new position.
2. **Minimize button (iconize/iconify)**...Placing the pointer over this button and clicking the left mouse button reduces the window to an icon and displays the icon at the right of the monitor screen. Iconizing a window does not halt any processes, but does unclutter the screen and speed up processing. To restore the window form, double-click on the icon.

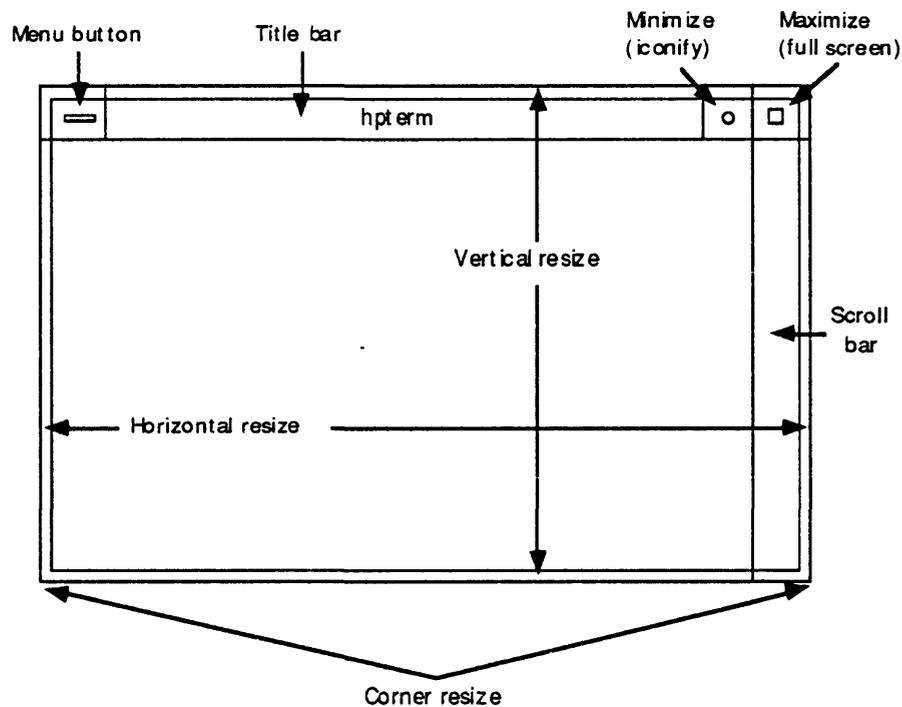


Figure 2.1. Controls of a basic X-window frame.

3. **Maximize button (full screen)**...Placing the pointer over this button and clicking the left mouse button enlarges the window to fill the entire monitor screen. In some windows, the displayed contents may not be enlarged. To return the window to its original size, click on the maximize button a second time.
4. **Resize border**...Changes the size of the window as desired, and is located on all sides of the window, and at all four corners. The resize borders at the top and bottom adjust the window vertically, the left and right borders adjust horizontally. The borders at the corners will adjust the window both directions simultaneously. Clicking and holding the mouse button on a resize border will cause an outline representing the size of the changing window to appear. Drag the border to the desired size and release the mouse button.

5. **Scroll bar**...Controls the position of the cursor in the lines available for viewing in the window and allows one to scroll through more information than can fit on one screen. When a window is new, the scroll bar is a uniform gray. When the first text scrolls out of view, the gray section leaves a clear area at the top of the scroll bar. The more text has scrolled out of view, the smaller the gray area becomes, until the maximum number of saved lines (default=256) is reached. Scrolling is accomplished by clicking on the scroll bar:

- | | |
|---------------------|------------------------------|
| Right mouse button | scroll toward end |
| Left mouse button | scroll toward beginning |
| Middle mouse button | jump to the location clicked |

With the pointer at the bottom of the scroll bar, scrolling is done one screen at a time. With the pointer at the top of the scroll bar, scrolling is done one line at a time.

6. **Window menu button**...Brings up a separate menu which can be used to close, enlarge, shrink, or 'iconize' the window. For window menu operation, refer to the operation manual supplied with the workstation.

Inside the window frames are the windows themselves, which may contain numerous function controls. A typical window is displayed in Figure 2.2, and the basic window controls are described below:

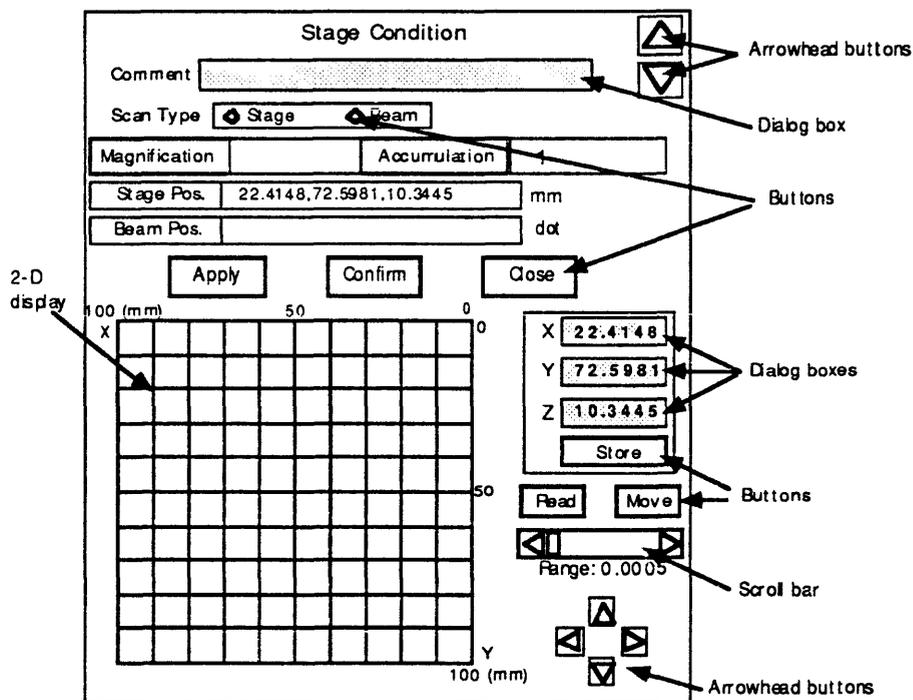


Figure 2.2. Basic window controls.

1. **Buttons**...Execute commands when the user positions the pointer over the button and clicking.
2. **Arrow head buttons**...Allow devices such as the specimen stage and spectrometers to be driven in the specified direction by clicking on the appropriate button. Also used to move through choices in a list.

3. **Scroll bar**...Scrolls the item row displayed in a window or increases /decreases the numerical value in the key entry area. The scroll bar contains a position indicator and two arrow heads. To use the indicator, position the pointer over it and click and drag in the desired direction, releasing the mouse button when the desired position is reached.
4. **Key entry area or dialog box**...An area where numerical values or alphabetic characters are entered directly from the keyboard. **Note: The pointer must be positioned inside the key entry area in order to type.**
5. **Two-dimensional display**...Changes the position of specified items, such as the specimen stage. Position the pointer at the desired location and double-click the mouse button, or click and drag the box to the desired position.

SECTION 3: Layout

It is important to familiarize yourself with the physical layout of the controls for the microprobe in order to operate it efficiently. An extended description of each button, dial, switch, and knob is available in JEOL's JXA-8900S/M/L ELECTRON PROBE MICROANALYZERS manual and its supplements, so only a brief overview will be given here. Each microprobe laboratory of the US Geological Survey contains basically the same hardware devices, but due to the physical constraints of each laboratory and the particular tastes of the lab supervisors, their arrangements are slightly different.

The rest of this section contains an illustration of the standard layout of the JEOL 8900, a description of hardware and controls, and brief descriptions of the variations in hardware of each USGS installation as of May 1994.

Column

Control and Display System

Workstation

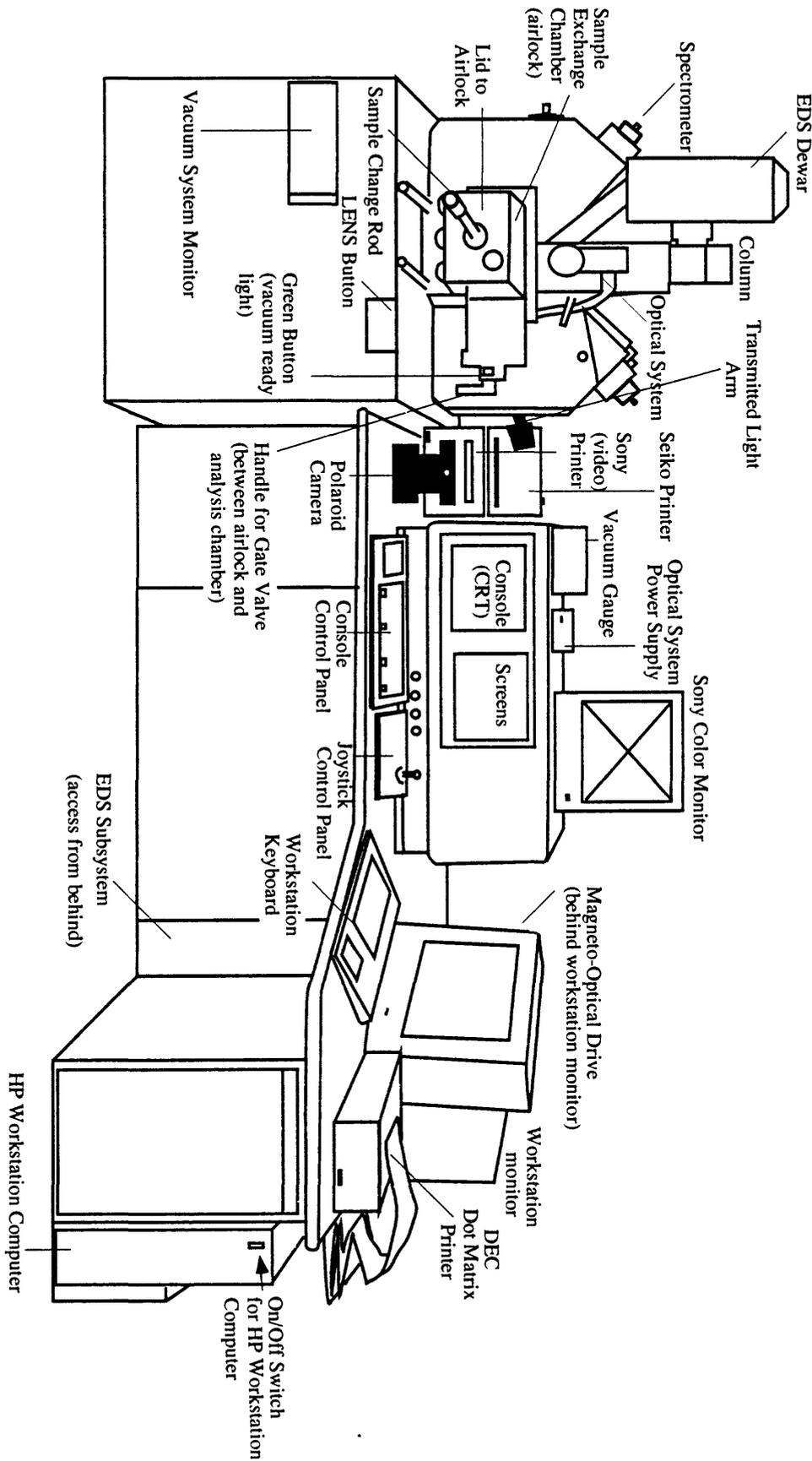


Figure 3.1. Hardware layout of USGS microprobe laboratories.

The three JEOL 8900 Superprobes owned by the USGS have five wavelength spectrometers, four with two crystals each and one with four crystals. They are also equipped with an EDS system. For optical focusing, it has an optical microscope equipped with a TV camera, which transmits an image to the SONY color monitor using either reflected or transmitted light. Almost all functions of the JEOL 8900 can be accessed from the software; however, some functions also have hardware access from the console for convenience. More information on the instrument itself can be found in the JEOL manuals or in the Configuration window (choose Initialize -> Configuration from the main menu.)

Monitors:

The two lower screens are electron image (CRT) screens. The left screen displays a live time image. The right screen displays a digitized average of the image on the left screen and may be frozen with the FREEZ button which toggles off and on. The scan speed for the screens can be set to FAST or SLOW buttons using buttons on the console control panel discussed below.

The VIEW button toggles the electron image screens through the possible image types: secondary electron (SEI), back-scattered electron topography (TOPO), back-scattered electron composition (atomic number contrast, COMP). The INST button will change the current image display conditions to SEI conditions; i.e., PCD out, probe diameter zero (minimum), PRBSCN (probe scan) on, scan mode PIC and speed SR.

The MODE button toggles through the display types: B_UP adds crosshairs to the display, LSP is a line scan profile, SPT is for spot positioning, and NOR is the normal display.

The SONY monitor displays an optical image of the sample stage, magnified to approximately 600X. You can also see this image by looking through the eyepiece on the instrument and pulling out the back rod. With the rod in place, the image is displayed on the screen. The OMTV button on the console toggles the optical microscope light on and off. The SONY monitor is also used to display images of the right SEM screen, for subsequent printing on the SONY printer. See section 6 for further information on the imaging and printing capabilities of the system.

SONY video monitor:

Magnification of the optical image on the video screen is about 600X.

The tick marks on the SONY screen are spaced approximately 10 microns apart.

The cross hairs on the SONY screen are not necessarily aligned with the beam position. Use a fluorescent sample to determine the position of the beam relative to the optical image. A small pen mark may be put on the screen for reference.

Cameras and printers:

The Polaroid camera is used to record images from the SEM screens. The photo LEFT and RIGHT buttons are used to choose which screen to photograph.

The SONY printer can print images displayed on the SONY monitor. Details for its operation are in section 7 of this manual.

Summary files generated at the HP workstation and analytical data are printed on the dot matrix printer.

The Seiko color printer will print an image of the computer screen (sometimes referred to as a screen dump) when either the remote green COPY button or the START-ENTER button on the printer is pressed.

Console/Control panel features:

The vacuum gauge displays the gun vacuum pressure. The display should typically read in the $(2-6) \times 10^{-6}$ torr range, although a rise into to the 10^{-5} torr range is normal during the sample exchange procedure. Ask your lab supervisor about acceptable and optimal vacuum readings.

The green high voltage (HV) indicator light below the right console screen will not light if the vacuum is not adequate. The high voltage will not go on if this indicator light is not lit.

The ACB button causes contrast and brightness for an SEI image to be adjusted automatically.

BRIGHTNESS and CONTRAST knobs on the console control panel are used to adjust the secondary electron image (SEI) display only. Back-scattered (COMP) and topographic (TOPO) image brightness and contrast knobs are located below the console screens.

The PCD (pneumatic cup device/probe current detector) button controls the insertion and removal of the Faraday cup--a device which blocks the electron beam and measures the probe current. When PCD button is lit, the cup is inserted into the line of the electron beam, blocking the beam from hitting the sample. With the PCD in, the beam current measured at the cup is displayed in the Present Values monitor window. With the cup removed from the beam path (button not lit), the beam hits the sample. The value of the sample current, that which is absorbed by the sample, is displayed.

The PRBSCN (probe scan) button toggles between scan mode and spot mode. When the button is lit, the beam rasters (moves very rapidly across an area) to provide scanned images. When PRBSCN is off (unlit), the beam is in spot mode.

The PROBE CURRENT knob adjusts the condenser lens current to increase or decrease probe current. The FINE button toggles between coarse and fine adjustment mode; if the FINE button is lit, movement of the current knob will cause very small adjustments in the current.

The MAGNIFICATION knob adjusts the electron image magnification between 40x and 300,000x, though the quality of the image resolution degrades when the magnification is above 1000x.

The FOCUS knob adjusts electron focus by changing the objective lens current. In scanning mode, this knob adjusts the electron image focus. In spot mode, this knob adjusts the electron beam spot size (visible on a fluorescent sample such as MgO). When the probe diameter is set to a value other than zero in the Electron

Optical System (EOS) conditions in the EOS window, the FOCUS knob is disabled.

Joystick control panel:

The joystick controls the x-y movement of the stage. The READY light must be blinking for the joystick controls to work. Press the REQ button to activate the joystick controls if the READY light is not blinking or the stage will not respond to the controls.

The Z-buttons control the z-direction movements of the stage (i.e., optical focus). Depressing the FAST button in conjunction with one of the Z-buttons will increase the speed of movement.

The TEST button jogs the stage to remove backlash and check x, y, z positioning.

The STORE button stores the stage position if the software is expecting a stage position to be input.

Optical microscope:

Images from the Optical (light) microscope are viewed primarily on the SONY monitor. These images may be transferred to the PC or recorded on the SONY video printer.

Workstation computers:

All USGS microprobe laboratories are controlled through a Hewlett-Packard 9000/745I computer. All laboratories also have Data General 300 series Aviion workstations which function as servers, and can be used to access the HP data processing software (see Data Management, Section 15).

Reston Laboratory Information:

The Eastern Region microprobe is located in room 3B-205a (phone: 703-648-6743) at the Reston National Center of the USGS. The laboratory in Reston can be contacted via electronic mail at the following address: jmcgee@lunatic.er.usgs.gov. Please address correspondence to James McGee.

Differences from the standard layout illustrated in Figure 3.1 for Reston include the following: the SONY thermal printer and the Polaroid camera are located to the left of the console, as is the vacuum gauge. The Seiko color printer is to the right of the console behind the monitor, with a remote control button located near the monitor. The dot matrix printer is to the right of the monitor, and the magneto-optical disk drive is on top of the console. To the right of the HP monitor is a 386 PC for use in data transfer and image processing.

In addition to the obvious components, there is a Data General Aviion 310 workstation in the outer lab (room 3B-205) which is connected to the probe via the network and can be used for analyzing data, image processing, etc. The Aviion has an associated HP Laserjet III printer as well as a 3 1/2" floppy drive and a cartridge tape drive.

Denver Laboratory Information;:

The Central Region JEOL 8900 Superprobe is located in room G1601 in building 20 of the Denver Federal Center (phone: 303-236-3188). The laboratory can be contacted via electronic mail at the following address: gmeeker@usgsprobe.cr.usgs.gov. Correspondence should be addressed to Greg Meeker.

In Denver the only differences in the set up illustrated in Figure 3.1 are in the location of peripheral devices.

The Denver microprobe lab also has a 486 DOS system PC and a Data General Workstation. The PC contains spreadsheet, image processing, and word processing software, with both 3 1/2" and 5 1/4" floppy drives.

The Data General 300 Workstation has a 3 1/2" floppy drive, a 32 track cartridge drive and a CD ROM drive. An HP Laserjet IV laser printer is connected to both the PC and the DG workstation via the USGS computer network.

Menlo Park Laboratory Information:

The Western Region microprobe is located in Building 11 on the Menlo Park campus (phone: 415-329-5089). The laboratory in Menlo Park can be reached via electronic mail at the following address: lcalk@condor.wr.usgs.gov. Please address correspondence to Lewis Calk.

There is a separate alarm system for building 11, and users must get instructions on arming and disarming the system before running the instrument alone or after hours.

The instrument, console and workstation are a standard JEOL layout (Figure 3.1).

The workstation and 386 PC, are hooked up to the HP printer between them. The 386 has two floppy drives, one 3.5 inch and one large format (5.25 inch).

The Data General Aviiion 300 Workstation has CD ROM and cartridge tape drives and is networked to the HP Workstation for use in data processing.

SECTION 4: Normal Start-up and Shutdown

The different centers have slightly different routines; steps below which do not apply to all centers are marked for the appropriate locations. (R=Reston, D=Denver, M=Menlo Park).

Start-Up:

1. Turn the HP and the SONY monitors on. If the previous user has shut down normally, the workstation computer screen should have the JEOL EPMA menu at the top and the EPMA and CONSOL icons in the lower right portion of the screen. The EOS Monitor, (R, D) Filament and Present Values windows (R, M) should also be displayed. If the EPMA menu is not displayed, click the mouse anywhere on the background, choose **EPMA Menu** from the list, and wait for the system to load the software.
2. Check the vacuum on the vacuum gauge located on top of or adjacent to the main console (see Figure 3.1 or laboratory description). Except during or immediately after a sample change, or while the vacuum is recovering for some other reason, the vacuum should remain stable in the 10^{-6} torr range. Consult your lab supervisor for the optimal and acceptable vacuum reading at each location.
3. Turn on any peripheral systems which may have been shut down (i.e., printers, the PC, the magneto-optical drive), and turn up the brightness and contrast controls using the knobs located underneath the two CRT screens.
4. Bring up the EOS Monitor window if it is not already open (Monitor -> EOS Monitor). This will display all the imaging control settings at a glance, and allow you to enter the commands using the software. (For most of these operations there are both hardware and software controls).
5. If the high voltage has been turned off, turn it on by pressing the ACCEL VOLTAGE button on the main control panel; a representation of the beam will appear in the EOS Monitor diagram. (The accelerating voltage cannot be controlled from the EOS Monitor window unless it has already been turned on using the button on the main control panel.) In the EOS Monitor window, click on Acc. V. and in the resulting window incrementally adjust the accelerating voltage to the desired setting (normally 15 kV).
6. (R only) If the Filament window is open, indicating that the filament was left under-saturated, the previous saturation level can be reached automatically by clicking on **Reset**. (This does not work if the Filament window has been closed since desaturating the filament.). If the filament needs to be resaturated, see the instructions at the end of this section (Section 4, Filament Saturation).
7. Start the beam rastering by pressing the PRBSCN button on the main control panel (lit = ON) or by clicking **Scan** in the EOS Monitor
8. Set the view to secondary electron image (SEI) using the VIEW button on the console control panel or the button in the **Image signal** section of the Scan Control window. Choosing **B UP** mode will cause cross-hairs to be superimposed on the image. The CRT screens will display the VIEW and the magnification.

9. To scan at TV speed, click on the FAST button on the main control panel or choose Scan speed **SR** in the Scan Control window of the EOS Monitor. Set the EOS scan mode to normal either by toggling through the options with the MODE button on the control panel or by choosing **PIC** in the Scan Control window.
10. Adjust the magnification to 40x using the **Magnification** knob on the main control panel or using the button in the EOS Monitor. This button is only functional if the beam is rastering (the PRBSCN button is lit).
11. Unblock the beam by toggling the PCD button (lit = beam blocked) or by clicking on the picture of the cup on the EOS Monitor. Pressing the INST button on the main control panel will set the instrument to the basic secondary electron image (**SEI**) configurations described above.
12. Adjust the brightness and contrast using the control knobs located on the lower right side of the main control panel until a satisfactory image is displayed.
13. Press the OMTV button to illuminate the sample and get a light optical image on the SONY monitor. Also make sure that the SONY monitor, the optical power supply and the SONY color monitor are turned on. If the SONY monitor is dark or blank and all the above settings are correct, the focus is probably out of adjustment. Adjust the focus with the joystick controller, using the FAST button in conjunction with one of the arrow buttons until the cross hairs appear on the SONY screen. If you still cannot get an optical image, the beam may not be on the top surface of a sample or holder and you should move the stage using the Stage Monitor window from the Monitor menu and/or the joystick.
14. (**R** always; **D**, **M** as necessary) Check the Electron beam focus and location in relation to the crosshairs by moving to a fluorescent sample such as benitoite, wollastonite or MgO--see stage map or ask lab attendant to locate an appropriate sample. With PRBSCN off decrease the illumination (knob on spectrometer panel beneath the sample airlock lever) to make the spot clearly visible on the SONY monitor. With the probe diameter set to zero (EOS Monitor window), use the FOCUS knob on the main control panel to minimize the spot size. The SEI image is in sharpest focus when the spot size is at a minimum. If the fluorescent spot is not well centered, the gun shift needs to be adjusted by a lab attendant. Also, if the fluorescent spot does not expand concentrically around the crosshairs as the focus knob is adjusted, the objective lens aperture must be centered by a lab supervisor.
15. If everything looks normal, change to your personal directory space by choosing Initialize -> Change Directory. A window will open stating the current home directory. Type the path to your login directory in the bar provided, then click **OK**. If you do not have your own separate directory space, the appropriate path name here is `/users/jx1`. The current home directory is displayed in the title bar of the EPMA menu.
16. At this point you can continue to the sample exchange procedure and begin your normal routine for an analysis session.

Filament Saturation and Beam Alignment:

This procedure is not usually necessary in Denver and Menlo Park, though the beam alignment and position should be checked no more than once per session to insure excellent results.

1. Open the EOS Monitor window. (Monitor -> EOS Monitor) A schematic diagram of the column will appear. Changes made in this window are sent to the system.
2. The ACCEL VOLTAGE button on console must be lit and the high voltage (HV) on. In the EOS Monitor window, click on **Acc V.** and incrementally set the desired voltage (usually 15 kV).
3. Open **Gun Bias** in the EOS Monitor window and select the setting button marked in green. If only pink buttons are visible, the setting is already correct.
4. Position the beam on a smooth, non-vulnerable spot (like brass holder).
5. Turn the PRBSCN on and take the PCD out.
6. Choose **SEI** mode, **FAST** scan mode, magnification ~ 1000x.
7. Click on **Filament** to open the Filament window. Click **Signal** to open the Scan Control window and set **EOS mode** to **EMP** (emission pattern) to display an image of the filament. The spot should be centered on the screen; if it is not, choose **Gun Tilt** and adjust until the spot is centered. The adjustment of **Gun Tilt** should not usually be necessary. (If starting from a very low filament value, use this **EMP** display for initial filament saturation.).
8. With **EOS mode** set to **EMP** in the Scan Control window, set **Scan mode** to **LSP** (line scan profile). Adjust brightness and contrast knobs on console to obtain a Gaussian profile visible on left screen, then click arrows in the Filament window to lower values until peak begins to shrink.
9. Cycle between these two steps until increasing the filament current causes little change in the Gaussian profile:
 - Increase current (click right arrow in Filament window)
 - If the profile flattens on top, lower baseline with SEI brightness control on the main control panel until the profile is Gaussian again.
10. Back the filament down 2 clicks from the max, and record the filament setting on your log sheet. Saturation often occurs near 115-125 for 15 kV. It may be higher (~125-135) for a new filament, and it usually decreases slightly as the filament ages.
11. Set **EOS mode** back to **NOR**, make sure **Scan mode** is set to **PIC**, and close the windows.
12. Check the gun shift adjustment by moving to a fluorescent sample such as benitoite, wollastonite or MgO--see stage map or ask lab attendant to locate an appropriate sample. If the fluorescing spot is not visible with PRBSCN off, either turn off the OMTV light or decrease the illumination (knob on spectrometer panel beneath the

sample airlock lever) to make the spot clearly visible on the SONY monitor. If the fluorescent spot is not well centered, the gun shift needs to be adjusted by a lab attendant.

13. After the filament is saturated and gun tilt and gun shift are properly aligned, adjust the beam current to the desired value (PCD in).
14. It is necessary to center the objective lens aperture so that the beam has the proper shape. Follow the instructions in step #12 above for checking the gun shift. Find a defect, pit, or any feature on the fluorescent mineral that you can center under the beam. With the beam diameter set at 0, turn the Focus knob on the control panel counter clockwise. Note if the beam "swings" off of the feature under the beam. If the objective lens aperture is properly centered, the fluorescent spot will simply expand concentrically around the point. If the point "swings" the aperture must be adjusted. Consult your lab supervisor for assistance if the aperture is out of adjustment.

Shut-Down :

Leaving the microprobe in the proper condition when you are through working is important for both the machine and the next user. In addition to following the shut-down procedure which follows, remember to take with you anything that you brought into the room, as items left behind may get misplaced by others. If you must have your samples immediately, remove them from the sample chamber before you start the shut-down procedure; otherwise the next user will remove them and you can retrieve your samples later. If you are shutting the instrument down for the day follow Procedure 1 below. If running the instrument overnight, use Procedure 2. If possible, standards, sample holders and samples should be stored in a dessicator.

Procedure 1: (normal shut-down)

1. Set the beam on brass or somewhere off of standards, samples or epoxy.
2. Retract the transmitted light lens if it was being used by pressing the LENS button located inside the sliding cover on the front of the main console, below the sample exchange lever. The LENS button is not illuminated when the transmitted light is in the retracted position.
3. Select the Secondary Electron Image (SEI) using the VIEW button on the main control panel or the **Signal** button in the EOS Monitor window. The image type being displayed is identified along the bottom of the left CRT screen.
4. Set the scan mode to the full picture setting either by pressing the PIC button on the main control panel, or by using the **Mode** button in the EOS Monitor window and choosing **FULL** under **Reduce Mode**.
5. Turn up the magnification to its highest level (300,000x) using the MAGNIFICATION knob on the main control panel, or use the **Mag** button in the EOS Monitor window (only functional if the PRBSCN is ON at the console). The magnification setting is indicated at the bottom of the left CRT screen.
6. Select the slowest speed setting by pressing the SLOW button on the main control panel until the slowest scan speed is attained, or use the **Speed** button in the EOS

Monitor window and choose **Scan speed S3**. Watch the left CRT screen to check the speed. You may have to turn up the brightness to see the scan line on the monitor.

7. Blank the beam by pressing the PCD button on the main control panel, or click on the picture of the cup in the EOS Monitor window.
8. Turn the optical system off by pressing the OMTV button on the main control panel so that it is no longer lit.
9. Turn the probe scan off by pressing the PRBSCN button so that it is no longer lit, or use the control button in the EOS Monitor window.
10. **(D, M)** Turn the acceleration voltage off by pressing the ACCEL VOLTAGE button on the main control panel so that it is no longer lit and no beam image appears in the EOS Monitor window.
11. Turn off the SONY color monitor (green button below screen).
12. Turn down the brightness and contrast of the left and right CRT monitors as far as they will go using the thumb knobs located immediately underneath the monitors. •
13. **(R)** Open the Filament window in the EOS Monitor. Decrease the filament saturation by clicking 10 times on the left-arrow button in this window. Leave these windows displayed on the screen in addition to the items described in step 14.
14. Close the open windows on the HP screen until only the main EPMA menu, the EPMA and CONSOL icons and the Present Values window remain.
15. **(M)** Turn the PC and its monitor off. **(D)** Turn off the DG Workstation.
16. **(D, M)** Turn off the SONY and Seiko color printers.
17. Turn off the HP color monitor. The power switch is located on the lower right front of the monitor.
18. **MAKE SURE THE GREEN VACUUM CONTROL BUTTON IS LIT!**
The green button is located on the right side of the vacuum chamber next to the specimen exchange chamber isolation valve lever (refer to Figure 3.1). The green button light indicates that the sample exchange airlock is at atmosphere. If this button is not lit, press the button to vent the airlock. In Reston this will happen automatically whenever the gate between the sample chamber and the airlock is closed.
19. Complete the log book entry for your session, noting problems and solutions and any other pertinent information. **(R)** Please tape a copy of your measurement conditions to the back of the page.
20. Remove your printed analyses and any floppy disks used for saving your data, turn out the lights in the room and lock the door behind you.

Procedure 2: (run overnight)

1. Retract the transmitted light lens if it was used and is still in the chamber. This is done by pressing the LENS button located inside the sliding cover on the front of the main console, below the sample exchange lever. The LENS button is not illuminated when the lens is in the retracted position. **FAILURE TO DO THIS CAN SEVERELY DAMAGE THE INSTRUMENT.**
2. Turn the optical system off by pressing the OMTV button on the main control panel so that it is no longer lit.
3. Turn off the SONY color monitor.
4. Turn off the HP color monitor. The power switch is located on the lower right front of the monitor.
5. (D, M) Turn the PC and its monitor off as well as the DG workstation if in use.
6. (D, M) Turn off the SONY and Seiko color printers.
7. (D, M) Turn down the brightness and contrast of the left and right CRT monitors as far as they will go using the thumb knobs located immediately underneath the monitors.
8. (D, M) **Make sure the green vacuum control light is lit before leaving.** It is located on the right side of the vacuum chamber next to the specimen exchange chamber isolation valve lever. If this is not lit, press the green button once so that it lights up.
9. (R, M) Make an entry in the log book.
10. Clean the instrument area of any material that was not present at the beginning of your session. **PLEASE KEEP THE LAB CLEAN.** Turn out the lights, close the blinds (M) and close/lock the door behind you.

SECTION 5: Sample Changing

This procedure assumes that your samples are ready to load into the microprobe, i.e., that they are in an appropriate holder, are polished, have been appropriately mapped or diagrammed for locating positions of interest, and have been carbon-coated to prevent charging, etc. These preliminary steps should have been taken prior to the beginning of the probe session. When the probe start-up routine has been completed, or whenever you need to exchange samples, follow the procedure below.

1. Check to make sure the LENS button on the control panel below and to the right of the sample exchange chamber (Airlock, see Figure 3.1) is not illuminated. If illuminated, press the LENS button (which is protected by a flip-down window) to remove the transmitted light lens. This will take about 30 seconds, and the LENS button light will go out when removal is complete.

WARNING: If the transmitted light lens is left inserted, serious damage will occur when you attempt to remove the sample holder assembly from the analysis chamber!!!

2. Block the electron beam by either pressing the PCD button (lit = blocked beam) on the main control panel, or by clicking on the picture of the Faraday cup in the EOS Monitor.
3. Select Monitor -> Stage Monitor. Clicking on the **Sample Change** button opens a small confirmation window. Click on **OK**, and the stage will move to the proper position for the exchange procedure.
4. After ensuring that the sample exchange chamber lid is in firm contact with the rubber gasket on the inside of the sample exchange chamber, press the lighted green vacuum control button located on the right side of the airlock next to the gate valve handle (see Figure 3.1).
5. After approximately 2 minutes the light in the green button will go out, indicating that the sample exchange chamber (or airlock) has been evacuated. (**D, M:** Wait an additional 2 minutes after the light goes out to allow the vacuum to stabilize before opening the isolation gate valve.) Open the gate valve slowly by rotating the handle forward and up 90° to horizontal.

Watch the vacuum gauge as you slowly begin to pull the handle out (to the right if standing in front of the chamber), pausing to keep the vacuum in the 10⁻⁵ torr range. Once the vacuum is stable in the proper range, pull the handle firmly all the way open, then turn the handle down 45° to lock it in position.

6. Retrieve the sample holder inside the analysis chamber by sliding the handle at the end of the sample exchange rod all the way to the base of the sample exchange chamber lid. With the handle as close to the lid as it goes, turn clockwise 90° to engage the sample holder, and gently pull outward to the end of the rod. You can monitor the retrieval of the samples from the analysis chamber by looking through the sample viewing window.
7. Once the sample holder is in the airlock, close the isolation valve by reversing step 5 above. Rotate the valve handle up 45° to a horizontal position, and push it in. When it

is completely inserted, rotate it down 90° until it locks in place. When the handle is in the proper position, press the green vacuum control button (not necessary in Reston); you should hear valves activating and a light hiss as the sample exchange chamber is vented. The green vacuum control button will light.

8. When the exchange chamber is at atmosphere (venting takes about 10 seconds), pull the entire exchange chamber lid outwards by lifting it lightly from the bottom edge and pulling gently on the sample exchange handle, in order to open the airlock for the sample exchange.
9. **ALWAYS WEAR GLOVES WHEN HANDLING THE SAMPLES AND SAMPLE HOLDERS.**
10. Remove the sample holder assembly by lifting it straight up off the T-shaped inner rod. The holder itself slides off the adapter base along two brass dovetail grooves if you need to change holders. Exchange samples and replace sample holder onto the adapter base, making sure the sample holder is pushed all the way to the front. Then replace the sample holder assembly on the T-shaped rod on the sample exchange platform.
11. Make sure the O-ring gasket is seated properly in the groove on the airlock lid. Close the sample exchange chamber lid and see that it is tightly pressed against the gasket.
12. Press the green vacuum control button to evacuate the sample exchange chamber. After approximately 2 minutes, the light will go off, indicating evacuation is complete. (**D, M**): Wait an additional 2 minutes before opening the isolation gate valve.)
13. Open the gate valve between the analysis chamber and the airlock (see Figure 3.1) by rotating the gate valve handle forward and up 90°. Watch the vacuum gauge as you slowly begin to pull the handle out (to the right if standing in front of the chamber), pausing to keep the vacuum in the 10⁻⁵ torr range. Once the vacuum is stable in the proper range, pull the handle firmly all the way open, then turn the handle down 45° to lock it in position. (see step 5, above).
14. Insert the shuttle and attached sample holder into the analysis chamber very slowly (15mm per second or slower to preserve the actual positions of stored stage coordinates) by sliding the handle at the end of the sample exchange rod all the way in to the base of the sample exchange chamber lid. With the handle as close to the lid as it goes, turn it counterclockwise 90° to disengage the sample holder, and pull it out to the end of the rod. You can monitor the insertion of your samples by looking through the sample viewing window in the lid.
15. Once the sample holder has been replaced, close the isolation gate valve by rotating the handle up 45° to a horizontal position and pushing it in. When the handle is in the proper position, **press the green vacuum control button (not necessary in Reston)**; you will hear valves activate and a low hiss will be heard as the sample exchange chamber vents. The green vacuum control button will be lit when the exchange chamber is again at atmosphere.
16. Click the **OK** button in the Sample Exchange confirmation window on the computer screen to complete the exchange procedure.

To Initialize Stage Position after Sample Exchange:

Often, after changing samples, the stage coordinates of the standards do not match the stored positions. This usually is the case when the user has pushed the sample holder into the analysis chamber too quickly (see step #14 above). Individual positions can be stored or their positions changed by choosing Monitor->Stage, clicking on **Memory**, highlighting position number and storing a position.

To check the integrity of previously stored positions, se the positioning button in the Stage Monitor window to drive the stage to the stored position for the periclase crystal (MgO). The resulting position should be close to the previously stored position for this mineral. If the initialization point has drifted considerably, leave the stage in this position (i.e., don't move it manually from the position stored with the MgO button).

1. Choose Initialize -> Present Positions -> Stage. While this window is open, reposition the stage, with the cross-hairs on top of a distinctive feature (this will be unique to each lab, so ask lab personnel where to position the window), and click on the Position button to store this initialization point. Close this window.
2. Now if you drive the stage away from the periclase crystal and click on the MgO button in the Stage Monitor window, the stage should be driven to the feature. This procedure makes it possible to reposition the stage accurately even after a sample exchange has taken place.

This procedure will fail (i.e., the new initialization point will not be stored) if the adjustment of the initialization point exceeds 0.5 mm, but a message to that effect will appear on the screen. If that occurs, see a lab attendant for a more complete initialization of the stage positioning.

The only consequence of the failure of this procedure (or of omitting it) is that driving the stage to positions stored prior to the most recent sample exchange will not position the beam precisely at the previous point on the sample. If the off-set from the initialization point becomes large enough, the markers indicating position which are displayed in the Stage Monitor may be noticeably offset relative to the proper position on the sample holder diagram in the Stage Monitor window..

SECTION 6: Imaging

This section is designed to help the inexperienced user obtain the various types of images possible on the JEOL microprobe. It will not go into detail on high-resolution, high-magnification imaging as that subject is lengthy and beyond what the typical user requires. In addition to optical (light microscope) images using either reflected or transmitted light, there are three different types of electron images available to the user on the JEOL 8900: secondary electron images (SEI), composition back-scattered electron images (COMP), and topographical back-scattered electron images (TOPO). The default on system start-up and the easiest image to work with is the secondary electron image.

Optical Imaging

Reflected light imaging:

Reflected light is the usual method of viewing samples optically. The OMTV button on the console toggles the light source on and off. The illumination level can be adjusted using the OM/OMT ILLUMINATION knob on the control panel at the front of the column.

Focus the image by using the Z-arrows on the joystick console. If the image brightens, you are headed in the right direction. Holding down the FAST button in conjunction with the Z-arrows causes faster motion. The image is considered to be in focus when the crosshairs are in sharp focus. Poorly polished samples and samples which are not level will be difficult to focus properly.

Transmitted light imaging:

You **MUST**** be using the transmitted light (thin section) stage (platform has rectangular openings with space beneath them) or the instrument will be damaged.**

The light source must be inserted into the column. If the LENS button on the control panel below and to the right of the sample exchange chamber is not lit, the transmitted light source is out of the column; to insert it, press the LENS button. (Flip down the little door which protects the button from being pushed accidentally.) The light source (black tube on the right side of the column) takes about 30 seconds to move into place. Open the EOS Monitor (Monitor -> EOS Monitor).

Click on the button marked OMTV until it is pink and displays "Transmit ON". This button is a toggle which cycles among transmitted light, reflected light ("OMTV On") and light off ("OMTV off").

The toggle for the polarizer is next to the LENS button on the control panel.

Always remove the transmitted light arm from the column before changing sample holders. To remove the transmitted light arm, simply press the LENS button on the control panel. If the transmitted light arm is not removed before samples are exchanged, serious damage will result to the microprobe.

Electron Imaging:

Images are viewed on the two CRT monitors of the control unit. The left CRT displays a live image of the sample, while the right CRT is a continuous average of scans (up to 64). Also displayed on the CRT monitors are the image type, accelerating voltage, magnification, and a micrometer bar showing the scale of the image. Directly underneath the CRT monitors are brightness and contrast dials for each screen; on the left screen, these should be turned to their maximum for imaging. Adjust brightness of the right screen to match the left screen.

Image preparation

In order to view an image, the instrument must be configured properly. Most of the basic settings are set as defaults upon start-up of the machine, so the user may only need to adjust a few settings.

First, the accelerating voltage and probe current must be set and the electron beam must be focused. For basic imaging the user should use the same settings he or she will be using during analysis, typically 15-20 kV and 2.0×10^{-8} amp beam current. With the probe diameter set to zero (EOS Monitor window), use the FOCUS knob on the main control panel to focus the electron image. (See Section 4, step 14 under Start-Up Procedure, for complete instructions if there is a problem achieving good focus.)

Make sure you have a sample under the beam. To do this, turn on the microscope light (OMTV button) and use the joystick controller to find and optically focus on a suitable position. Remember to optically focus with reflected, not transmitted light. Turn the probe scan on (PRBSCN button lit), set the magnification to 40x and unblock the beam (PCD button not lit). Select the image type to be displayed using the VIEW button on the main control panel; this button cycles through the imaging options. Begin by selecting the SEI setting.

SEI (secondary electron imaging)

Once the probe scan is on and the beam is unblocked, an image should be visible on the CRT screens. For a basic SEI image configure the instrument as described below. If the image is entirely bright or dark after the beam is unblocked and the probe scan is on, the contrast and brightness are probably far out of adjustment. You can manually set the contrast and brightness by adjusting the knobs in both directions and "feeling" your way to the proper setting until an image appears, or you can let the instrument guide you.

Setting the contrast and brightness: Press the WFM (wave-form mode) button on the main control panel. A series of six horizontal bright lines will appear on the left CRT screen. Adjust the brightness and contrast knobs until a separate video wave-form line appears on the screen, and continue adjusting the knobs until the line is contained between the six bright lines and produces an irregular wave-form. Press the WFM button again and an image should appear on the CRT screens.

Refining the image:

1. Set the scan speed to super rapid (SR) by pressing the FAST button on the main control panel or clicking on the Speed button in the EOS Monitor window. A Scan Control sub-window will appear and the Scan speed can be set.
2. Set the Scan mode to Picture (PIC) and the Reduce mode to Full in the Scan Control sub-window as above, or press the PIC button on the main control panel.
3. The magnification should initially be set to 40x to view the image, but should be increased to sharply focus the beam.
4. Set the probe diameter to 0 in the EOS Monitor window. The beam cannot be manually focused without doing so.
5. Focus the electron image using the focus knob located on the main control panel; toggle between coarse and fine using the COARSE/FINE button. The AFD button, located above the focus knob, will automatically focus the image in about 15 seconds, but most users will want to adjust it manually. For best results, increase the magnification of the image and then re-focus. Repeat this at higher and higher magnification settings until turning the focus knob when it is on the fine setting no longer alters the focus.
6. Adjust the brightness and contrast of the SEI image using the control knobs on the main control panel until the quality of the image is satisfactory.

COMP (back-scattered electron imaging)

The compositional (COMP) or back-scattered electron image is used to show differences in mean atomic number. The image varies in brightness depending on the mean atomic number of the phases in the sample. The higher the average atomic number in the phase, the brighter it will appear in the image.

There are several differences between setting up a SEI image and setting up a COMP image. First and most important is that they have different contrast and brightness adjustment knobs. The COMP image knobs are located underneath the two CRT monitors on the control unit and consist of separate coarse and fine contrast and brightness knobs. A second difference is that the sample illumination (OMTV button) should be turned off while viewing a back-scattered electron image because the detector is light sensitive. If you are using the COMP image to locate your analysis points you can leave the OMTV on, but you will have to continuously adjust the brightness and contrast settings in response to the varying reflectivity across your sample.

Select the COMP image using the VIEW button on the main control panel. Once the beam is unblocked and the probe scan is on, an image should be visible. For a basic COMP image configure the instrument as described in steps 1-6 under SEI imaging above, but use the contrast and brightness knobs for COMP images located below the console screens. If the image is dark or bright, the contrast and brightness are probably far out of adjustment. See the section on adjusting contrast and brightness controls in the SEI instructions above.

TOPO (topographic imaging)

Setting up a topographical image is almost identical to that of the COMP image, but the pseudo-topographic effect is used to distinguish fine differences in height of the sample surface. The steps and controls are exactly the same except the TOPO fine brightness knob is separate from the COMP fine brightness knob. Follow all the procedures listed under the COMP image above to obtain a TOPO image.

Single-element survey imaging

It is possible to map the distribution of single elements and display the result in real time on the console screens as a pattern of dots. The size of the image is limited to approximately 0.75 mm or less, depending on magnification. At magnifications below about 2000x, x-ray intensity will be diminished by at least 10% at the edges of the image. This effect becomes visibly noticeable between 100 and 500x. **NOTE:** This technique is a much faster way to see chemical variations over a small area than making a preset digital map. However, it is totally qualitative and generally less sensitive; you can only view one element at a time; and it can only be documented on the SONY printer or with a Polaroid image (see Section 7.1). In short, this is a survey technique intended for locating phases and for choosing elements for preset mapping or quantitative analysis.

1. First, position a spectrometer on a desired element peak:

Peak-search positioning method: Choose Monitor-> Peak Search. Make sure that the beam is on a material that contains a sufficiently high concentration (greater than 10 wt %) of the element of interest for the peak search to succeed. Click on **Element**, and with the arrow in the appropriate box, type in the element abbreviation. The software will automatically choose a channel and crystal for that element, but you may change it by clicking on the appropriate buttons.

Manual positioning method: Choose Monitor -> Rate Meter. Click on the button for the channel you wish to use. In the window for the chosen spectrometer, click on **Element** to open a periodic table. Choose the element from those available to you on that spectrometer, then click on the **Set** button at the bottom of the window and close the window. The chosen spectrometer should move automatically to the proper position. This method should only be used if you are sure that the correct peak position is stored.

2. On the console control panel, press the XRAY button once for channel 1, twice for channel 2, etc., until you have the desired channel displayed on the console screen. One can tune multiple spectrometers and toggle among five different elements (one for each spectrometer).
3. Set the scan speed to the slow so that white dots (not lines) are displayed on the console screens. Each dot represents an x-ray pulse counted by the detector from the tuned spectrometer. By zooming in and out one can see chemical zoning of single elements within a crystal or material.
4. To compare the single element image to the back-scattered (COMP) or SEI images, simply click on **VIEW**. When XRAY is chosen again, the console screen will show the last element viewed.

SECTION 7: Photography and Printing

Using the Polaroid Camera:

The Polaroid camera can capture images from either the right or the left console screen. In general, if the image is acceptable on the screen, the picture will turn out. If the picture is too dark, increase the contrast and brightness and try again, or use the waveform monitor as described in Section 6 under "Setting the contrast and brightness".

1. To adjust the image, the brightness knobs under the CRT screens should be set to their maximum. Follow the instructions under SEI imaging in Section 6 to get a suitable image on the screen. Although most users will want to set the contrast and brightness themselves, an automatic contrast and brightness adjustment can be performed by choosing the ACB button on the main control panel. For optimum results, set the monitor to the slowest scan speed before using this feature.
2. To load the camera, insert the film with the lever on the camera turned to **L**. With the proper side down (see film sheet), push the film in until it stops against the back and makes a clicking sound. Pull the film sleeve out gently until it catches.
3. To make a photograph choose the photo button located at the right side of the main control panel: **LEFT** to print an image of left screen, **RIGHT** for the right screen image. A slow scan will occur. When the scan is complete, the photo is finished and ready for developing.
4. To develop the photograph push the film sleeve back in until it stops. Turn the lever on the camera to **P** and pull the film firmly and evenly out of the camera. Follow the developing instructions on the film package. Then return the lever to **L**.

Using the SONY Video Printer:

The SONY video printer is used to capture images from the SONY monitor. The same functions are available on both the remote controller and the front panel of the printer.

Pictures to be printed must first be stored in the memory of the printer. Under some conditions, two full-screen pictures may be stored. A letter **M** in the lower right of the SONY screen indicates that you are seeing the image stored in memory, a letter **S** indicates that the picture is from the source.

To produce a basic picture:

1. (**D**, **M**) Select the image type to be photographed using the **A** and **B** source buttons on the front panel of the SONY monitor. These buttons toggle between the optical (normal operation) image and the image displayed on the right CRT screen (the "averaged" image). (**R**) The image must be selected using the printer's MENU functions: **S-Video** selects the optical image, **Video** selects the SEM image. Current selections are shown in green. Arrow buttons allow you to move around the menu. Choose MENU again to exit.
2. Set the scan speed to **SLOW** (or **S3** in the EOS Monitor window). **OMTV** should be **ON** for optical microscope images and **OFF** for SEM images, though this will only affect the contrast settings. Set brightness and contrast either manually or by pushing the ACB button.

3. To store this image for printing, choose **MEMORY-IN** on the remote control or on the front panel of the **SONY** printer. If you are satisfied with the picture, choose **PRINT** and the image will be printed. If you want to make further adjustments, select the **MEMORY/SOURCE** button to toggle back to the source image and make the adjustments, then press **MEMORY-IN** again and press **PRINT**. If you try to print from the source image, the image in memory will be displayed, and printing will not occur until **PRINT** is chosen again.

To customize a picture:

1. Choose **MENU** on the printer or remote control to display an image of the menu choices on the **SONY** monitor. Current selections are highlighted in green, and the arrows are used to move around the screen.
2. (**M, D**) Select the image type to be photographed by using the **A** and **B** source buttons on the front panel of the **SONY** monitor as described above. Scan speed must be set to slow, and the **OMTV** should be **OFF** for **SEM** images, **ON** for optical images, although this affects only the contrast settings. Alternatively, (**R**) the image type can be chosen from the menu on the screen (the on-screen menu is displayed by pushing the **MENU** button on the remote control or on the front of the printer). Choose **VIDEO** from the on-screen menu for **SEM** images, i.e. whatever image is being viewed on the right **CRT** screen. Choose **S** (**S-Video**) for optical images.
3. Continue to Menu-2 if you wish to add a caption to appear outside the picture area in the lower left corner. To produce a caption, use the arrow to highlight the letter or symbol desired, then press **EXEC** to add that letter to the caption. If the caption screen displays **OFF** in the upper right corner, highlight the **ON** button and press **EXEC** to activate captions. After exiting the menu, a **C** will be displayed in the upper left of the screen if captions have been activated. This caption appears only on the **SONY** print.
4. If you wish to annotate your image (put words or symbols on the image), open the Monitor menu and choose **EOS Keyboard** (Monitor-> **EOS keyboard**). When the window opens, put the cursor arrow on the keyboard. Pressing the middle mouse button locks the arrows/cursor onto the **EOS** keyboard so that you do not lose control of this function by moving the mouse. Push "insert character" on the **HP** workstation keyboard and begin typing your comments or symbols. This will temporarily remove the information at the bottom of the screen, but this information can be re-displayed by pressing "insert character" again. Arrow symbols (<, >) will put arrows on the screen. You can move the cursor with the space bar or the directional keys to the right of the alphanumeric key pad on the key board. Annotated comments will stay on the screen for the next image unless they are removed using the "delete line" or "delete character" keys. Press the middle mouse button to unlock the cursor and exit from the **EOS Keyboard**. The annotations appear directly on the right **CRT** screen and are captured along with the image when the print is made. These annotations will remain and will print on the next image if they are not removed.
5. Press **MENU** to exit from the menu display and the source image will be displayed on the screen. Choose **MEMORY-IN** to store the image for

printing and **PRINT** to produce the picture. To adjust the picture further, select the **MEMORY/SOURCE** button to return to the source image, make the necessary adjustments, then choose **MEMORY-IN** and **PRINT**.

To produce multiple small pictures on one page:

1. Choose **MENU** on the printer or remote control to display an image of the menu choices on the SONY monitor. Choose the number of pictures desired on the page (1, 4 or 9). Set the **SEPARATOR** option to **ON** if you want a border to separate the multiple pictures. If you are using separators, you cannot have a caption.
2. On exiting the menu, stars will appear on the SONY screen to indicate the positions of the pictures. The green star indicates the location of the next image to be stored, and can be changed using the arrow keys.
3. Proceed as with single pictures, toggling to source mode to set up a frame, then placing it in memory, and using the arrow keys to move, until all the frames are set as desired. Choose **PRINT** as before.

Using the Seiko Color Printer:

The Seiko color printer prints the screen of the HP workstation monitor. On the HP monitor, bring up the item you wish to print (most users take pictures of area analyses, or element condition windows). Press the **START** button on the top of the Seiko printer or the green button on the Seiko remote control. The printing process takes about a minute. Wait for printing to start before altering the monitor images.

When re-printing plots from a phase analysis it is best to reverse the image to get a white background rather than black. This can be done using the arrow keys next to the **START** button on the printer. First press **ONLINE**; the display on the Seiko will read "#1 PAPER NOR 1", with the final "1" blinking. Press the left arrow and NOR will blink. Press the down arrow to change NOR to REV, then press **ONLINE** and dump the image as above.

SECTION 8: Standard Analysis

Standard analysis calibration data is shared among all users. Only the most recently acquired count data for a standard is stored on the disk.

Menu: Analysis -> Standard Analysis

After a few seconds the menu bar outline will appear and must be placed with a click of the left mouse button.

Sample Button:

This displays a menu of previously analyzed standards available, the last date they were measured, and whether data are available for WDS or EDS analysis or both. Be patient, as this window is somewhat slow to come up. Choose **Name** to sort the standards alphabetically or **Date** to sort them by most recent analysis date. Be aware that the element of interest may not have been standardized on the spectrometer and crystal you have selected on the date shown in this window... this is the last date the standard was used for any element. Use the pointer to highlight the standard being selected.

Choose **Search** and enter the element of interest in the dialog box to examine all the previous standardizations for that element. A list of possible standards is displayed, and an appropriate one may be selected at this point.

If a previously unmeasured standard is needed, it is added by clicking the **New** button. This will prompt you only for the name of the new standard; enter the composition under Measurement -> Element Condition as described below.

Other options include renaming samples, deleting samples, and printing the standard data for the highlighted sample. **PLEASE do not rename or delete standards without consulting a lab attendant.**

Select OK when you have completed standard selection.

Measurement Button:

Condition Load:

If you previously created and saved analytical conditions for this standard in the Condition Store window, choosing **Condition Load** will allow you to recall them. Locate the stored conditions on the list presented, highlight it, and click the **Load** button. You still must update the beam current in the EOS Condition window, and check the stage positions in the Stage Condition window. It is wise to check other conditions to make sure that all is as you expect. **NOTE: this Condition Load routine will only retrieve the conditions for one standard, not an entire suite of standards. See the discussion of default analysis conditions in Section 16.**

Standard Type:

You must decide whether you want to analyze your unknowns as metals or oxides. If you are analyzing silicates, generally, you will want to analyze them as oxides. However, if you are analyzing for oxygen, or non-silicate materials, you must create new standardization files and standardize for all elements in your quantitative analysis setup as "metals". Mixing standards analyzed as oxides and metals in Quantitative Analysis routines leads to miscalculations by the workstation software. Previously created oxide and metal files should be distinguishable from among the standardization listings (metals may have the designation "m" or "metal" in the file name. Once a standardization is created as an oxide or a metal it should not be changed. Before closing this window, make sure **Calibration Curve** is turned OFF. (Its function is described in the Map Analysis section, p. 11-6).

Element Condition:

Standard Composition:

This item should not generally be needed by users. However, when a new standard is being entered, choosing this opens a window which allows you to enter the known composition of the standard in atomic, mole or weight percent. The element symbols, valences and percentages are typed in a column format. For an existing standard, this window will display the composition data which has been entered for it. If you need to copy a standard composition from an existing standard file to a new one (as you might if the same standard is on two different standard blocks) see the directions at the end of this section.

WDS Elements:

This window allows you to select the elements for measurement with WDS (wavelength dispersive spectrometry) on this standard by clicking on the desired elements in the periodic table. The available elements are those entered in the standard composition. You can choose to measure an element more than once, allowing the same element to be measured under different conditions (e.g., different spectrometers). Do not try to standardize on an element of low concentration; use major elements only.

WDS Condition:

Choosing this menu option will display the conditions that will be used for measuring the selected elements for this standard. Clicking on the element box at the top of any element condition description will open a screen which displays the master list of conditions for analyzing this element. (There should be a routine set for each possible crystal on the spectrometers on which this element can be measured).

You may choose to use or modify an existing set-up. To make a modification, click in the box of the item you wish to change and make your selection from the list presented or by typing in desired values. When you are finished, highlight the box above the set of conditions you wish to apply and exit the screen by clicking **OK** at the bottom.

Please do not change the PHA settings (Peak Height Analysis gain and window voltages) for any routine without consulting a lab attendant first.

EDS (Electron Dispersive Spectrometry) Elements:

Choose elements for EDS analysis in this window. This window functions in the same manner as the WDS Elements window (above). If the same element is specified twice for EDS analysis, you must specify two different x-ray lines.

EDS Condition:

Set the x-ray and ROI (Region of Interest) for analysis for EDS elements. If no data is set here, the most recently set conditions will be used, and by default the ROI will be set to include both α and β lines.

EOS Condition:

This window displays the Electron Optical System conditions to be used for analyzing this standard. Be aware that each standard has its own EOS condition file. On the console, turn the PRBSCN (probe scan) off to stop the beam from rastering. With the PCD in, set an appropriate beam current (typically on the order of 2×10^{-8} amps = 20 nanoamps). Choose **Read** to enter the present instrument conditions into this window.

The EOS condition window should display the present probe current and accelerating voltage (usually 15 kV. This setting must be the same for both the standard and unknown when doing quantitative analysis). Do not alter the settings for the Condenser Lens or Objective Lens currents as these are set in conjunction with the probe current. Make sure the beam diameter is larger than 10 microns (adjustable in the EOS Monitor window).

A proper beam diameter should be set based on the types of phases you are analyzing. Some phases are less robust than others and can be damaged by a focused beam (especially at high beam currents), and damaged standards are often difficult to replace. Although a small beam diameter and higher probe current will give better counting statistics and more precise measurements, and the size of the samples may limit the beam size in some cases, for some phases the beam size will need to be expanded to protect against standard/sample damage and alkali loss (see Troubleshooting, section 16). It is advisable to keep the beam size used on the standard as close as possible to the beam size used on the unknown. Large differences in probe diameters can affect the measurement. If the probe diameter needs to be reset in your analysis file, click on the button labeled **Probe Diameter** (μm) and adjust the setting in the sub-window.

The settings for **Magnification**, **Scan Mode** and **Scan Speed** are unimportant since in normal standardization the beam will not be rastering during analysis. Click **OK** to save the conditions for automatic use during measurement. Choosing **Set** will set the instrument to the conditions currently displayed in this window.

EDS Condition:

This window displays the operating conditions for EDS. The options in this window should read as follows for Standard Analysis and Quantitative Analysis;

Energy Full Scale: 10
Spectrum Data Points: 1024 (default)
Measurement Mode: LIVE
Measurement Time: (has no effect)
Aperture: 1

The aperture setting in the software bears no relation to the actual aperture setting of the detector. The aperture must be adjusted manually to a setting that will keep the dead time of the EDS detector between 20 and 40%. If the dead time is too low, not enough counts are reaching the detector, but if the dead time exceeds 40%, the detector is being swamped and missing too many counts. If both EDS and WDS analyses are being done, the beam current should be adjusted for WDS (e.g. 20 nA) and then the aperture should be adjusted to yield 20-40% dead time. It is strongly advised that you discuss the EDS aperture setting with the laboratory supervisor before doing EDS quantitative analysis.

Stage Condition:

This window is used to set the stage positions for the points to be measured. Only the last position entered will be displayed. Click on **Position Input**, and a window with a diagram of the stage will appear.

To enter new positions:

1. To move the stage into position, click to indicate the approximate position on the stage with the pointer and select **Move** or use the joystick to move to the point of interest. If this is a standard that has been analyzed recently, the coordinates which appear in the Position Input window should be fairly accurate. In this case, click the **Move** button and check to see that it has located the proper sample.
2. Focus the image optically using the Z-axis controls on the joystick control pad. **Always focus the cross-hair by using the up arrow last so that the backlash is taken out of the gears and the computer will return to that position more precisely.** Press the **TEST** button on the joystick control pad to check the location and focus of your position. If it is off, correct the position and repeat the test until it is satisfactory.

If for some reason, you wish to only use one standardization point (not recommended) proceed to steps 3 and 4. If you wish to accumulate and average multiple analysis points for your standardization, proceed to step 5.

3. Click the **Read** button and then the **Store** button in the Position Input window, or press the STORE button on the joystick control pad to enter this as the stage position for measurement on this standard. If you use the STORE button on the joystick console, you will then have to press REQ to reactivate the joystick.
4. If necessary, edit the comment line by placing the cursor in the dialog box and typing. If no comment is typed in, the comment from the previous point will automatically be used.
5. Set the number of accumulations desired for the standard analysis by clicking on the **Accumulations** button. Typically you will want to accumulate and average at least three points per standardization. Type in the number of accumulation points desired, or slide the bar to the desired number, and then click **OK**.

To set coordinates for each of the accumulations click **Confirm** in the Position Input window. A tone will sound to alert you that the computer is ready to begin storing points, though no position data has yet been stored. A small window will open and remain open until positions are stored for each accumulation.

On the joystick control pad, choose a point to be analyzed, focus, press TEST to remove stage motor backlash, then re-focus if necessary. When satisfied with the focus and the position, press the STORE button on the joystick control pad to store that position as an accumulation point. A tone will sound, this time to alert you that the point has been stored, and you can move to the next position and repeat the procedure until all the accumulations are set, at which point the accumulations window will close automatically.

6. Click **Apply** to store the information in permanent memory, then click **Close** to close the stage window.

To modify existing positions:

1. Choose **Input** from the Stage Condition window. When the screen with the image of the stage appears, choose **Move** to drive the stage to the last previously set position for this standard.
2. Select **Confirm**. The stage will position itself at the point set for the first accumulation, and a tone will sound. You may move to a different position or adjust the focus of this position or make no change at all. Press STORE. Once the points have been set, they can be checked by clicking on **Confirm**. The stage will move to the next analysis point and beep when the STORE button is pressed. If you begin checking the analysis points using the **Confirm** function, you must go to all of the accumulation points you wish to analyze.
3. The sequence described in (2) will continue until all the stored positions have been checked. When you are satisfied with the positions chosen, click on **Apply**, then **Close**.

After the Position Input window has been closed, you may choose **One-by-One** if you only want to analyze one standard at this time. Press **Acquire** in the window that appears and the analysis will be performed only for the standard listed in the **Standard Analysis** dialog box.

If you prefer to set up several standards to run consecutively, return to the **Sample Button** and select the next standard to be analyzed. Follow the steps above until you have set up all the standards you wish to measure, then proceed to the section entitled **Preset Measurement** below.

Condition Store:

If you are happy with the conditions as they are set up, and might want to use the same conditions again, save them here with a recognizable name. If this is done, you can retrieve them later using **Condition Load**.

Print-out Condition:

Use this to choose a full or brief output. Use full if you wish to have a hard copy of your count data. If you do not want a print out, turn off the printer or the print function. As stated below, only averaged counts are printed unless the X-Text window itself is printed by clicking on the **Print** button.

Measurement Mode:

If both EDS and WDS standard analyses are being executed, choose whether to do them at the same time (parallel) or separately (serial). If they are to be done separately choose the order in which they should be accomplished. If using parallel mode, be advised that EDS peaks may broaden due to noise from the WDS spectrometers.

Check Data:

If you need to eliminate a point from the calculation of the average for the standardization, or alter any other standard data after it has been collected, the data files can be altered via this window. There is a long delay in opening the Check Data window with no indication that anything is happening...be patient.

To examine an accumulation point, click the column heading designated **Accm**, and the number of accumulations for each standardization will become a new button. Click on the accumulations for the desired standardization, and a window will open showing the value for each accumulation in counts per second. Turn off the analyses you wish to omit by clicking on the buttons to the left of the numbers, and they will be left out of the standard calculation.

Choose **Save** to keep the adjusted values for use in calculations of unknowns. Even if **Preset Measurement** was used, only the standard named in the dialog box of the **Standard Analysis** window will be available to change. To examine to data from other standards you must select each standard, one at a time, using the **Sample** button on the left side of the **Standard Analysis** window as described in the beginning of this section.

Survey Measurement:

Choose this process to take a quick measurement at the point currently under the beam to check the measurement conditions you have set up. These results are for checking conditions only, and cannot be used for quantitative or standard analyses.

Pre-set Measurement:

When all points on all standards have been chosen and entered as above, choose the standards to be measured by clicking on their buttons (ON=blue). Make sure the number of standards you expect to analyze matches the number at the bottom of the window. Click on **Acquire** and the chosen standards will be measured automatically.

- While the analyses are occurring, a window will be open showing a moving pump and keeping you informed about how many accumulations have been completed, etc. This window will close when measurement is complete, but to stop the analyses at any time click the **Accum Stop** or the **Stop** button in this window and the measurement will end. Note that **Accum Stop** will not stop the analysis immediately. The microprobe will finish the analysis it is working on before quitting and then ask you if you wish to stop the analysis. Be patient.

*Aborting the current analysis by pressing the **Stop** button is not recommended.* Doing this often leads to software failures. If it is necessary and software problems do occur, follow the troubleshooting procedures (Chapter 16) to reestablish the software communications. Using the Utility -> Reset Analysis function immediately following use of the **Stop** button seems to prevent software failures.

Count data appears in a window labeled "X-Text" as the measurement is occurring. When the analysis is complete, a message to that effect is displayed in the window and the instrument beeps.

A summary of the standard data will be printed automatically; this summary does not contain the individual counts for each accumulation point.

If you wish to print the raw counts for each accumulation, choose **Print** in the X-Text window before you close this window. Although accumulation data may be viewed using **Check Data** after the X-Text window has been closed, the only convenient way to print out the individual count data is to print the X-Text window. Once X-Text is closed, this information is not readily available for printing.

To copy an existing standard composition to a new file:

Standard compositions will be copied to new files when the same standard is present on more than one standard block; or the same standard analyzed under significantly different conditions.

Menu: Utility ->File Utility ->Data Arrange -> Standard Data

Select an existing file to copy, assign a name for the new file and click on **Copy**. In order for this new file to show up on the list of possible standards, you must close and then re-open the standard analysis window if it has been open during this procedure.

SECTION 9: Quantitative Analysis

Menu: Analysis -> Quantitative Analysis

After a few seconds, a menu bar will come up and must be placed by clicking the left mouse button.

Sample:

Choose a group by pressing the **Group** button and highlighting your choice on the list. If you are a new user, press **New** and set up a new group name. Choose **OK**. If the list of group names which appears is unfamiliar to you, you may be in the wrong directory. Reset the initial directory to use your own. (Initialize -> Change Directory. Type the path to your login directory: /users/*your directory*. If you are using the common-use directory, the appropriate path name here is /users/jx1.)

A menu of samples previously set up in this group (if any exist), the last date they were measured or updated, and whether data was measured using WDS or EDS analysis or both, will be displayed. Use the pointer to highlight the sample being chosen. To add a new sample, choose **New**, and you will be prompted for a new sample name. Click on **OK** when you are finished.

Measurement: *If you are setting up a new analysis routine, skip to Correction Method.*

Condition Load:

If you already have an analytical routine set up and wish to repeat those previously designed and saved conditions for this analysis, choosing **Condition Load** will allow you to recall those conditions. See discussion of default analysis conditions in Section 16. A list of your previously saved condition files will be displayed; highlight the one you wish to use and click **Load**.

Check to make sure that the conditions set are those you want for this measurement, paying special attention to element conditions to make sure the most current peak position is in use. You must update the **EOS Condition** because only the condenser and objective lens settings are saved directly. The instrument will not adjust these settings to get the "correct" reading at the cup. Therefore, if filament conditions have changed since the analytical conditions were saved, those same lens settings may not produce the desired beam current. Therefore adjust the instrument conditions to those desired for analysis and choose **Read** in the **EOS Condition** window.

Correction Method: *If you are using a preset condition and you have checked analysis conditions, you can skip to Stage Condition.*

Quantitative Analysis -> Measurement -> Correction

Choose the correction method you wish to use for your analyses.

The ZAF correction was developed by JEOL (reference nos. 4-7, 9) "BandA" is the correction method of Bence and Albee (2), the PRZ method selects the following:

- a) if **Oxide** is chosen, "PRZ" performs CITZAF matrix corrections of Armstrong (1).
- b) if **Metal** is chosen, "PRZ" performs the XPP correction of Pouchou and Pichoir (8).

Consult your lab supervisor if you have any questions about the available correction routines.

Element Condition:

WDS Elements:

Select elements to be measured in this sample using Wavelength Dispersive Spectrometry by clicking their symbols in the periodic table. Clicking an element more than once results in multiple analyses of that element. To clear existing elements, highlight them in the box below the table, then choose **Clear**. The order in which you choose the elements is the order in which they will be analyzed unless you change it in the WDS Condition window. Volatile elements (Na, K, F, etc.) should be measured on the first pass to minimize loss due to beam exposure.

WDS Condition:

Choosing this will display the current spectrometer settings for measuring the elements selected for WDS (wavelength dispersive spectrometry) analysis. Clicking on the element box at the top of any element condition description will open a screen which displays alternative existing spectrometer analysis conditions for this element. (There should be a set of analytical conditions for each possible crystal on each spectrometer on which this element can be measured). You may choose to use one as it stands or to modify an existing set-up by changing counting times, peak search conditions, backgrounds, etc.

To make modifications, place the cursor over the area you wish to change and press the left mouse button; a window will appear in which you can alter the chosen value. Please **do not alter the PHA (voltage, baseline and window) conditions** without consulting a lab supervisor first. PHA settings for all elements must match the settings used during standardization for an accurate quantitative analysis.

When the conditions are satisfactory, highlight the box above the set of conditions you wish to apply and exit the screen by clicking **OK**. Do this for each element to be analyzed.

NOTE: In order to assure that the most up-to-date instrument settings are used for your analysis, it is wise to open this window for each element and click on **OK**, because parameters such as peak positions are automatically updated here but not in each user's condition files.

EDS Elements:

Choose elements for EDS analysis in this window, which functions the same way the WDS Elements window (above) are selected.

EDS Condition:

Only the x-ray line can be chosen. The other conditions will be the same as those set for the standards and cannot be changed here.

Calculation:

Elements chosen here will be calculated stoichiometrically instead of analyzed directly. Typically the dominant anion in your samples is calculated. If you have chosen the oxide correction method, oxygen is calculated by default.

Calculation Condition:

Select from six modes of element calculation.

Valence: If the sample is to be analyzed as an oxide, click on **Valence** to open a window displaying default valences. Make any necessary changes and click **OK**.

EOS Condition:

This window summarizes the operating conditions to be used for analysis. Make sure the beam diameter is set to an appropriate size for the sample in question (adjustable in the EOS Monitor window). On the console control panel, turn the PRBSCN off and, with the PCD in, set an appropriate beam current (typically on the order of 2×10^{-8} amps = 20 nanoamps). Choose **Read** to enter the current instrument conditions into this window, where they may then be modified if necessary. Click **OK** to save the conditions for automatic use during measurement. Choosing **Set** will set the instrument to the conditions currently displayed in this window.

EDS Condition:

This window displays the operating conditions for EDS. The options in this window should read as follows for Standard Analysis and Quantitative Analysis;

Energy Full Scale:	10
Spectrum Data Points:	1024 (default)
Measurement Mode:	LIVE
Measurement Time:	(has no effect)
Aperture:	1

The aperture setting in the software bears no relation to the actual aperture setting of the detector. The aperture must be adjusted manually to a setting that will keep the dead time of the EDS detector between 20 and 40%. If the dead time is too low, not enough counts are reaching the detector, but if the dead time exceeds 40%, the detector is being swamped and missing too many counts. If both EDS and WDS analyses are being done, the beam current should be adjusted for WDS (e.g. 20nA) and then the aperture should be adjusted to yield 20-40% dead time. It is strongly advised that you discuss the EDS aperture setting with the laboratory supervisor before doing EDS quantitative analysis.

Standard Conditions:

Check to make sure that the standards you want are the ones selected for this analysis. If you wish to change a standard, click the button above the element and select the desired standard from the list presented. If no standard has been measured under your measurement conditions, the only available choice will be "calculated".

The button in this window which says "set standards meas. ON" will turn on the standards chosen here for use in standard analysis, preset measurement. If the standard you want to use is not on the list, then probably it was standardized on a different spectrometer or crystal than what you have selected.

Either change your element conditions to that of your standard or go back to Analysis -> Standard and re-standardize using the conditions you have set up for this analysis. Note that the standardizations can be changed after the analysis using Process -> Quantitative -> Off-Line Correction as described later in this section.

Stage Condition:

This window is used to set stage position coordinates for the points to be measured. If positions have already been set for this sample, they will be displayed. **Point Table Edit** can be used to edit existing points; this can be especially helpful if you need to edit comments. Highlight an existing position to confirm or modify it, or highlight the next available empty position to add a new point. Select **Pos. Input** and a window with an image of the stage will appear.

To enter new positions:

1. To move the stage into position, click to indicate the approximate position on the stage with the pointer and select **Move** or use the joystick to move to the point of interest.
2. Focus the image optically using the Z-axis controls on the joystick. Always focus the cross-hair by using the up arrow last so that the backlash is taken out of the gears and the computer will return to that position more precisely. Press **TEST** on the joystick to check the focus and X and Y position. If any coordinate is off, correct and test again until satisfactory. Good optical focus is critical for an accurate analysis.
3. Click **Read** and then **Store** in the window, or press the **STORE** button on the joystick to enter this as a stage position for measurement on this sample. If you use the **STORE** button on the joystick console, you will then have to press **REQ** to reactivate the joystick.
4. For more than one accumulation, click the **Accumulation** button and type in the number or slide the bar to the desired number, then click **OK**. The accumulation function will average all the points selected and the individual point analyses will be lost. *This is not recommended for quantitative analysis, since data can be averaged later using the off-line summary function.*

To set positions for the accumulations click **Confirm**; a small window will open and remain open until a position is stored for each accumulation. On the joystick console choose a point to be measured, focus, choose **TEST** to remove stage motor backlash, then re-focus if necessary.

When satisfied with the focus and the position, press **STORE** to store that position as an accumulation point to be measured. A tone will sound and you can move to the next position and repeat the procedure until all the accumulations are set. After the last point is set, the instrument does not beep, instead the accumulations window disappears and the joystick is deactivated.

5. Edit the comment line if necessary by placing the cursor in the box and typing.
6. Click on **Apply** to store the information for this point unless you have used the **STORE** function on the joystick control (see step #3).
7. Use the arrows at the upper right of the stage window to highlight the next blank line or move to the next point you wish to modify. The information on the currently highlighted line will be overwritten the next time you choose **Apply**, so it is important to remember to use the arrows to advance to the next line.

Repeat steps 1-5 until all points have been entered or modified. Click on **Close** to close the stage window. The data in this window is saved automatically when the window is closed. Until a **Save** button is added to the stage position window in quantitative analysis, the user should close the stage window periodically so that the data will be saved to the hard disk. Otherwise your position data is at risk if the software fails. Once a **Save** button is added to the window, simply click on **Save** periodically for the same reason.

To modify existing positions:

1. Choose **Pos. Input** from the Stage Condition window.
2. Select **Confirm**. The stage will automatically be positioned at the point set for the first accumulation. You may move to a different position or adjust the focus of this position or make no change at all. Press **STORE**. The stage will automatically drive to the position previously set for the next accumulation.
3. The sequence described in (2) will continue until all the stored positions have been checked. When you are satisfied with the positions chosen, click on **Apply**, then on **Close**.

After the **Position Input** window has been closed, you may choose **One-by-One** if you only want the highlighted position to be analyzed at this time. Press **Acquire** in the window that appears and an analysis will be performed for the highlighted point only. If you have set up a number of measurements to be done automatically, choose all the points you wish to analyze by clicking the buttons to the left of their numbers in the **Stage Condition** window (blue=ON). Close this window when all the points have been selected. Select **Preset Measurement** in the measurement menu, and click **Acquire**. All the points selected will now be analyzed in turn.

Print-out Condition:

This window allows you to choose the order in which the elements are printed, full or brief print-out, and other features of the output. Full output is preferable. You also set the number of oxygens used in the mineral formula calculation (cations) mode from this window.

Condition Store:

If you are happy with the conditions as they are set up, and might want to use the same conditions again, save them here with a recognizable name. If this is done, you can retrieve them later using **Condition Load**.

Measurement Mode:

If both EDS and WDS analyses are being done, choose whether to do them at the same time (parallel) or separately (serial). If they are to be done separately choose the order in which they should be accomplished. If using parallel mode, be advised that EDS peaks will broaden due to noise from the WDS spectrometer motors.

Survey Measurement:

Choose this process to take a quick measurement of the point under the beam to check the conditions you have set up. Data measured here will be stored as point #9999

under the present Group/Sample, and will be overwritten the next time a survey measurement is done. Thus only the last survey analysis will be saved to your data files.

Additional Functions:

In this window you can choose whether to do background measurements and peak searches on all measurements, not at all, or every n^{th} measurement. Peak seeking is recommended to overcome any spectrometer or crystal-flipping backlash, except for volatile or minor elements. If minor elements are being measured (i.e., low count rate and unsuccessful "peak-peek"), peak-searching is automatically disabled and the default peak position is used. If it is possible to mistakenly pick up a nearby peak, peak searches are not recommended. Background measurements are recommended if phases are heterogeneous or zoned and if minor element measurements are important.

Pre-set Measurement:

As described in the section on setting stage positions, when all the points have been entered, selecting this menu item and choosing **Acquire** will cause the designated points to be analyzed automatically.

If you are doing EDS measurements, the EDS window must either be open or present on the screen as an icon while measurement is occurring or an error will cause measurement to stop. Hopefully this problem will be solved with a software upgrade.

As with standard analysis, while an analysis is occurring, a small window will be open showing a moving pump, and keeping you informed about how many accumulations have been completed, etc. This window will close when the analyses are complete. To stop the analyses at any time click the **Accum Stop** or the **Stop** button in this window and the measurement will end abruptly. By clicking on **Stop** one runs the risk of temporarily disabling the software or losing communication between the instrument and the workstation. If this happens, please refer to the appropriate Troubleshooting sections of this manual and/or consult a lab attendant.

Data appears in a window labeled "X-Text" as measurements are taken. When an analysis is complete, a message to that effect shows in the window, and the instrument beeps. At the end of a preset group of analyses, the instrument will beep three times.

SECTION 10: Line Analysis

Menu: Analysis -> Line Analysis

This function is useful for qualitative "traverses" of element compositions in samples, or for setting up points for a quantitative line traverse. Many options for line analysis are similar to items in the quantitative analysis menu, so are described here only briefly. Refer to Section 9, Quantitative Analysis for more complete information.

Sample:

Choose a group by pressing the **Group** button and highlighting your choice on the list. If you are a new user, consult Section 9, Quantitative Analysis, or laboratory personnel. To add a new sample, choose **New**, and you will be prompted for a new sample name.

Select **OK** when you are finished.

Measurement:

Condition Load:

If you wish to duplicate a previous run for which you stored the conditions, select the stored condition file in this window and click on **Load**. There is more information about loading stored conditions in Section 9: Quantitative Analysis; a discussion of default analysis conditions in Section 16 is also applicable.

Element Condition:

Element:

Select elements for analysis by clicking their symbols in the periodic table. Pressing an element more than once results in multiple analyses of that element. To clear existing elements, highlight them in the box below the table, then choose **Clear**. The order in which you choose the elements is the order in which they will be analyzed.

Condition:

This menu option will display the current spectrometer settings for measuring the elements selected for this sample. Clicking on the element box at the top of any element condition description will open a list of alternative measurement conditions for this element. (There should be a column of data for each possible crystal on each spectrometer on which this element can be measured).

You may choose to use or modify an existing set-up, or create a new one. To make a modification, click in the box of the item you wish to change and make your selection from the list presented. To create a new set-up, click **New** and then modify the default conditions as above.. The eight items in the list from **Back(+)** to **Bac Count** are ignored in line analysis. In particular, counting times set here do not apply to line scans, since the dwell time per pixel is set under **Stage Conditions**. Highlight the box above the set of conditions you wish to apply and exit the screen by clicking **OK**. Do this for each element to be analyzed.

IMS Signal:

Open this box to choose the signal type for measurement: **SEI** (secondary electron), **TOPO**, or **COMP** (back-scattered electron). **AUX** has no meaning.

EOS Condition:

This window summarizes the operating conditions to be used for analysis. Make sure the beam diameter is set to a proper size (this is related to the measurement interval, which is discussed under **Stage Condition**).

Turn the PRBSCN off, and with the PCD in, set an appropriate beam current (typically on the order of 2×10^{-8} amps = 20 nanoamps). Choose **Read** to enter the current instrument conditions into this window, where they may then be modified if necessary. Click **OK** to save the conditions for automatic use during measurement. Choosing **Set** will set the instrument to the conditions currently displayed in this window.

EDS Condition:

Refer to Section 9, Quantitative Analysis for an explanation of proper EDS settings.

Stage Condition:

This function is used to assign stage position coordinates for the line analysis. If a line has already been set, its end points are displayed in this window. Highlight an existing line to confirm or modify it, or highlight the next available empty position to add a new line. Choose **Pos Input** and the stage window will appear.

To enter lines:

1. To move the stage to the proper position, click on **Move** if the previously stored position is appropriate. Otherwise, either click on the approximate position and choose **Move**, or use the joystick to move to the sample of interest. Focus the image optically.
2. Edit the comment line if necessary.
3. Choose to move either the stage or the beam during the scan. A stage scan allows a larger area to be covered than a beam scan.
4. Choose normal or micro step size. Normal is appropriate unless a very small area is under study.
5. Use **Accumulations** to set the number of times the scan is to be repeated. Note that the accumulations will be averaged together to give one analysis.
6. Choose an appropriate dwell time for each point analyzed. This parameter, in combination with the number of pixels to be measured and the accumulations will determine how long the analysis will take. The time estimate given is approximate.

Method 1:

- Set a position, direction, number of pixels (points), analysis interval
- After choosing a location, select **Read**, then **Store** to enter this as the initial stage position for measurements on this sample. Choose whether this point is to be the beginning, the mid-point, or the end of the line.
- Choose the number of pixels up to a maximum of 1024.
- Highlight the appropriate arrow on the display to choose the direction you want the line to go. The arrows correspond to the electron image, which means that **x** is the reverse of the optical image but **y** is the same.
- Set **XY Size**. (the measurement interval in microns)

Method 2:

Set the beginning, end, and distance between points

- After choosing a location select **Read**, then **Store** as the beginning of the line.
 - Move to the position where you want the line to end and focus. Select **Read**, then **Store** as the end of the line.
 - Set **XY Size** (the measurement interval in microns).
7. To check the line to be mapped, choose **Confirm**. The sample will be positioned at one end of the line. Focus, choose **TEST** to remove stage motor backlash, then re-focus if necessary. When satisfied with the focus and the position press **STOR** on the joystick controller, The sample will then be positioned automatically at the other end of the line; repeat the focusing procedure.
 8. Choose **Apply** to store the information. A new blank line will open in the parent window; use the arrow at the upper right of the stage window to highlight the new line and repeat the above steps to define another line.
 9. If you want to do quantitative analysis at each point of a line you have defined, you can choose **Convert to Point table**. This will convert your line points (up to a max of 9999) to points in the quantitative analysis table (Analysis -> Quantitative -> Measurement -> Stage Conditions) under the same sample name you have chosen for the line, creating it as a new sample name for quantitative analysis if necessary.

The conditions for the analysis **must** be set in the Analysis -> Quantitative menu; only the point coordinates are transferred from line analysis to quantitative analysis.
 10. Choose **Close** when you have finished defining lines.

After the stage window has been closed, you may choose **One-by-One**, then **Acquire**, to analyze only the highlighted line at this time. If you are setting up a number of measurements to be done automatically, define all the lines, select the ones to be analyzed by clicking the button at the left of the desired lines

(blue=ON), then close the stage window and choose **Preset Measurement** from the Measurement menu. Click on **Acquire** to start your analyses.

Print-out Condition:

Choose to print out measurement conditions and results.

Condition Store:

If you might want to use the same conditions again at a later date, save them here. You can then retrieve them at any time with the **Condition Load** command.

Survey Measurement:

This will execute a single line scan measurement. Unfortunately, data is written in position #9999 of the current Group/Sample, which means that each successive survey measurement will overwrite the previous analysis data, and there is no way to recover the data once that happens. The print-out is the only record you will have for any survey analyses except your final survey analysis.

Pre-set Measurement:

When all lines on all samples have been chosen and entered as above, choose **Acquire** to start the analyses. A small window will be open showing a moving pump and keeping you informed about how many accumulations have been completed, etc. This window will close when the analysis is complete. To interrupt the analyses, choose **Accum Stop** or **Stop** in this window.

Viewing Line Scans:

To view the analysis on the screen as it is happening select **Analysis -> Line Real-time**. Select the proper sample using the **Sample** button, then click on **Real-Time** to start the display.

To recall or reprocess line analyses after they have been acquired, select **Process -> Line Analysis**. Choose the appropriate group name, highlight the analysis of interest and a list of stage positions for that line analysis will appear. Highlight the desired stage position, then choose which elements (up to 5) you wish to display on the screen. Clicking the **Max Spectra** button opens a window where the number of spectra to be displayed is chosen. When you are ready, choose **OK** and be patient, as the loading process takes a while, but eventually the appropriate scans will appear on the screen. Clicking the mouse or pressing the keyboard while the files are loading may lead to system lock-up.

When the spectra are displayed, the **Spectra Specify** window is also opened. Click on the element to be processed, then click on the **Operation** button to open a menu of available processing operations.

The display can be manipulated using the options found under **Spectra Display**. Choosing **Spectra Analysis** allows examination of x-ray intensity or concentration for points on the line. The **Spectra Calculation** menu accesses arithmetic operations which can be performed on the spectra.

Calibration Factor allows specification of factors for use in converting intensity to concentration (instructions for evaluating the calibration factors are given in the description of map analysis).

Reset Display releases all the processing and restores the display to the original data. There is more information about these options in the JEOL software manual..

SECTION 11: Map Analysis

Menu: Analysis -> Map Analysis

Many options for map analysis are similar to items in the quantitative analysis menu, so are described here only briefly. Refer to Section 9, Quantitative Analysis, for more complete information.

Sample:

Choose a group by pressing the **Group** button and highlighting your choice on the list. If you are a new user, consult a laboratory supervisor before proceeding. See Section 9 for information on setting up a new group directory. To add a new sample, choose **New**, and you will be prompted for a new sample name. Select **OK** when you are finished.

Measurement:

Condition Load:

If you created and saved analytical conditions for a previous map and wish to use those conditions again, select the stored condition file and choose **Load**. There is more information about loading stored conditions in Section 9: Quantitative Analysis; a discussion of default analysis conditions in Section 16 is also applicable.

Element Condition:

Elements:

Select elements to be measured by clicking their symbols in the periodic table. Selecting an element more than once results in multiple analyses of that element. To clear existing elements, highlight them in the box below the table, then choose **Clear**. The order in which you choose the elements is the order in which they will be analyzed.

Condition:

Choosing this will cause the current spectrometer settings to be displayed for measuring the elements selected. Clicking on the element box at the top of any element condition description will open a list of alternative measurement conditions for this element. (There should be a set of analysis conditions for each possible crystal on each spectrometer on which this element can be measured.) You may choose to use or modify an existing set-up, or create a new one. To make a modification click on the item you wish to change and make your selection.

To create a new setup, choose **New** and modify the default conditions which appear. The eight items on the list from **Back(+)** through **Bac Count** are used only for quantitative and standard analysis and have no meaning here. Highlight the box above the set of conditions you wish to apply and exit the screen by clicking **OK**. Try to minimize the number of elements per spectrometer (sequence number ideally should be 1) as the total analysis time for making a map is a function of this sequence number.

IMS Signal:

Open this box to choose the signal type for measurement: SEI (secondary electron), TOPO, or COMP (back-scattered electron). AUX has no meaning. You may choose more than one signal type, but be aware that two signals will double your analysis time...three will triple the time.

EOS Condition:

This window displays the Electron Optical System conditions to be used for analysis. Make sure the beam diameter is set to an appropriate size. For maximum resolution, the probe diameter should be less than the pixel size. For pixel sizes less than 5 microns, the probe diameter should be 0 (see **Stage Condition**, step 7). Turn the PRBSCN off and, with the PCD in, set an appropriate beam current (typically 5×10^{-8} amps, or 50 nanoamps are used for map analysis in order to increase the signal).

Choose **Read** to enter the current instrument conditions into this window, where they may then be modified if necessary. Click **OK** to save the conditions for automatic use during measurement. Choosing **Set** will set the instrument to the conditions currently displayed in this window.

EDS Condition:

See Section 9, Quantitative Analysis, EDS Condition, for a discussion of proper EDS quantitative analysis conditions. Consult lab personnel before using EDS for map analysis or quantitative analysis.

Stage Condition:

This item is used to assign stage position coordinates for the area to be measured. If areas have already been set, their limits are displayed. Highlight an existing area to confirm or modify it, or highlight the next available empty position to add a new area. Choose **Area Input** and the stage window will appear.

To enter areas:

1. To move the stage to the proper position, click on **Move** if the previously stored position is appropriate. Otherwise, click on the approximate position on the stage and choose **Move**, or use the joystick to move to the area of interest. Focus the image optically.
2. Select **Read**, then **Store** to enter this as the initial stage position for measurement of this area. Choose whether you want this position to be the center of the desired area, the beginning (upper right corner) on the CRT image, or the end (lower left corner) of the area.
3. Edit the comment line if necessary.
4. Choose to move either the stage or the beam during the scan. A stage scan is usually appropriate and allows a larger area to be measured. Beam scan should be used for magnifications >2000X or with EDS only. A unidirectional stage scan (**Stage(uni)**) minimizes the influence of backlash and stage alignment problems without increasing measurement time considerably.

5. Use **Accumulations** to set the number of times the scan is to be repeated. The accumulations will be averaged together to give one analysis; therefore one is the normal value.
6. Choose an appropriate dwell time. This parameter, in combination with the number of pixels to be measured and the number of accumulations chosen, will determine how long the analysis will take. The time estimate given is approximate.
7. Choose the number of pixels in each direction and the pixel size desired. There is a maximum of 1024 pixels in either direction. The pixel size (microns) should be larger than the probe diameter (see EOS Condition above). These two parameters determine the size of the area to be mapped.
8. To check the area to be mapped, choose **Confirm**. The stage will be driven to each corner of the area in turn. At each corner the focus must be set using the joystick console z-axis controls. Choose **TEST** to remove stage motor backlash, then re-focus if necessary. When satisfied with the focus and the position press **STOR** button on the joystick control. The stage will automatically move to the next corner.

If the area is not satisfactory, reset the reference point (center or beginning or ending corner) or adjust the number of pixels and their size, then repeat this step.

9. Choose **Apply** to store the information. A new blank line will open in the Stage Condition window; use the arrow at the upper right of the stage window to highlight the new line and repeat the above steps to define another area.
10. If you want to do quantitative analysis at each point of an area you have defined, choose **Convert to Point Table**. This will convert your map points (up to a maximum of 9999) to points in the quantitative analysis table (Analysis -> Quantitative -> Measurement -> Stage Conditions) under the same sample name you have chosen for the area, creating a new sample name for quantitative analysis if necessary. The conditions for the analysis **must** be set in the Analysis -> Quantitative menu; only the point coordinates are transferred from map analysis to quantitative analysis. If you are tempted to use this technique, remember that a 100x100 pixel area is 10000 points, and with typical analysis times greater than one minute, it would take several days to quantitatively analyze the area, even if the Stage Condition files could store that many positions.
11. Choose **Close** when you have finished defining areas.

After the stage window has been closed, you may choose **One-by-One** to analyze only the highlighted area. If you are setting up a number of maps to be done automatically, choose the maps to be measured by clicking their button (blue=ON), then close the stage window and choose **Preset Measurement** from the Measurement menu. Choose **Acquire** to start your analyses.

Print-out Condition:

Choose to print out conditions for measurement and results.

Condition Store:

If you wish to save your measurement conditions for possible later use, do so in this window. Your conditions can then be retrieved at any time using **Condition Load**.

Survey Measurement:

If you wish to get rough results, you only need to open the **Stage Conditions** window and set the scan type, dwell time, pixels and pixel size. The current instrument conditions will be used to carry out a rough measurement, which will be stored as #9999 in the current Group/Sample. If you wish to preserve the data in a more accessible form, use the **Pre-set Measurement** option.

Pre-set Measurement:

When all areas on all samples have been chosen and entered as above, choose **Acquire** in this window to start the analyses. While measurement is occurring, a small window will be open showing an active pump moving, and keeping you informed about how many accumulations have been completed. Choose **Stop** or **Accum Stop** in this small window to interrupt the analysis.

Viewing Maps:

To view the analysis on the screen as it occurs select **Analysis -> Map Real-time**. To enter calibration factors to relate the map colors to composition rather than counts, choose **Operation -> Map Display -> Level Modify** within the real-time map display window. See below for information on how to obtain calibration factors.

To recall or re-process area analyses select **Process -> Map Analysis** from the EPMA Menu. Choose the appropriate group name, highlight the analysis of interest, and the window will display the stage position and the elements for which the analysis was done. Choosing **Max Maps** in this window allows you to set the display for the number of maps you wish to see on the screen and the number of colors. The more maps you display, the fewer colors you may use. If you choose too many colors, you will get an error message to that effect when you try to close this window.

Highlight the elements for which you want maps displayed in the order desired, press **OK**, and be patient because the loading process takes a while. Eventually the appropriate scans will appear on the screen. Choosing **Operation -> Calibration Factor** here will allow you to enter factors to relate the map colors to element concentrations rather than counts. See below for information about how to obtain calibration factors. For a description of other processing options, see Section 10: Line Analysis or refer to the chapter on Map Analysis in JEOL Instructions.

To Get Calibration Factors A and B for Data Conversion from Intensity to Concentration:

Menu: Analysis -> Standard -> Measurement -> Standard Type

Choose **Calibration Curve**, then click **OK**.

Menu: Analysis -> Standard -> Measurement -> Check Data

A calibration line will be displayed, with values for A and B. According to the JEOL Instructions manual,

A = X-ray intensity per 1% concentration, in units of
counts/((microamps)(microseconds)%);

B = background, in units of counts/((microamps)(microseconds)).

Choose **Element** to display a window with A and B values for all the elements analyzed on this standard. In the Element window, choosing A or B will allow you to adjust the value manually.

Choosing **Name** will make each standard name into an active button; choosing one of these buttons will bring up a display showing cps/nanoamp for this analysis. Choosing **Search** in this window brings up yet another window where an element name can be entered, and a list of the standard names which have analyses for that element will appear, along with information about the analysis such as kV, channel, crystal, cps/nanoamp. This window is informational only... choosing an analysis here by clicking its button has no effect on anything else.

Menu: Analysis -> Standard -> Measurement -> Standard Type

Turn off the **Calibration Curve** toggle when you are through, as it interferes with some of the other functions in standard analysis.

SECTION 12: Serial Auto Analysis

Menu: Analysis -> Serial Auto Analysis

Serial Auto Analysis enables you to schedule a number of analyses requiring different analytical conditions to be run in one unattended session. The analysis order, along with some information about each analysis already chosen for inclusion, is displayed. Below is a brief recommended procedure followed by a more complete description of each menu option.

Recommended Procedure:

1. Before coming to Serial Auto Analysis, set up each individual analysis package in the usual way (under Quant, Line or Map) and save a condition file for each package you set up using Condition Store.
2. In the Serial Auto Analysis window, choose File -> Open Serial Directory. Either choose one of your previous series set-ups to modify or enter a new name for the series you are about to set up.
3. Choose Condition -> Input Condition or click on the **Input** button to open the Input Condition window.
4. For each analysis you wish to place in the series:
 - Choose the appropriate analysis type: map, quant, line, etc.
 - Click on **Group/Sample** and choose the group and sample name for the analysis as you set it up in step 1.
 - Choose **Condition Load**. In the resulting window, choose to load from stored condition files, then highlight the name of the condition file you set up in step 1 for this analysis and click **OK**.
 - Choose **Apply**, then use the arrows at the top of the Input Condition window to move down to a blank line. Repeat the sequence in step 4 for each analysis you wish to place in the series.
5. Measurement -> Preset.

Serial analysis creates one file for each analysis in the series in a special directory under your user name. Into each file is copied the set of conditions which you indicated (by doing a **Condition Load**) should be used for this analysis.

Changes to conditions made in the Serial Analysis window do NOT modify the original condition file, only the copy of that file which will be used to direct the serial analysis. By the same token, changes you make to the original condition file by going back to the original analysis menu (Quant, Map, Line) will not be transferred to your serial analysis set-up unless you specifically re-load those conditions to the serial analysis after making the changes.

File Menu:

Open Serial Directory:

Give your serial analysis file a name in this window or choose a previous series to modify.

Printout:

Print out the serial setups to be used and the analysis order.

Condition Menu:

Input Condition:

The **Input** button in the Serial Analysis window is equivalent to choosing Condition -> Input Condition. A window will open where one chooses the analysis type (Quantitative Analysis, Map Analysis, etc.) and the analysis conditions (EOS conditions, elements, etc.) for whichever analysis in the sequence is currently highlighted in the parent Serial Analysis window.

Measurement type:

Click the type of measurement this analysis in the sequence will be.

Condition load:

Choose either to accept the present configuration of the instrument (i.e., the most recent conditions used for this type of measurement, which will not be the most recent conditions for this type of measurement for this sample if that was not what you were doing last) or to load a set of stored conditions. If you choose to load stored conditions, a list of your stored condition files for this sort of measurement will be displayed for you to choose from.

Condition change:

A window will open allowing you to choose which conditions to examine and/or change. You can only examine **Element conditions**. Choosing **EOS Conditions**, **EDS Conditions** or **Standard Conditions** will open windows similar to those presented when you set up the analysis as an individual analysis, and most of those items can be modified here. Be aware, however, that you are modifying them for the serial analysis only, and changes you make here will not carry back to your condition files stored under the individual analyses.

Group/Sample :

This button presents you with a display of the groups and sample names you have set up for this kind of measurement. Click on the analysis you are choosing for this position in the series of analyses.

Stage Condition:

Clicking this button will open the familiar stage condition window where you can choose which points to include in your analysis and you can re-position or re-focus existing points as well as add new ones.

Check:

This option allows you to review all relevant analysis conditions. They cannot be changed in this window, but can be printed out.

Apply:

Clicking here will save the conditions you have just set up for use with the analysis which is highlighted in the sequence window. If you try to exit the Input window without applying, or if you use the arrows in the upper right corner of this window to move to set up another analysis without having chosen **Apply** first, you will be prompted to save the conditions you just chose.

Copy, Move, Delete, etc.:

These are basic operations which can be used to modify the order of analysis. They are fairly self-explanatory.

Measurement:

One-by-one runs the highlighted analysis routine only.

Preset runs all the analysis routines which have been selected (marked with a blue box).

To get a summary of the data gathered using Serial Auto Analysis, use the appropriate Process menu for each individual analysis type used in the series. There is no separate compiled summary file for Serial Auto Analysis.

SECTION 13: EDS Analysis

Menu: EDS -> EDS

The following commands are found in the main EDS window:

Two boxes w/an arrow between them:

This function clears previous spectra and should be used before every spectrum acquisition.

Acq. Spectrum:

Clicking this button toggles EDS acquisition on and off. The analysis will stop automatically and the beam will be blocked after the counting time set under **Preset.Preset:** This function allows the user to choose a set counting time, analysis time and regions-of-interest (ROI). If the time is set to zero, counting will continue until it is turned off by clicking again on the **Acq. Spectrum** toggle.

ROI:

(Region Of Interest) This allows the user to highlight a region of interest and determine integrated counts between samples in that energy range. To set, click on **ROI** and move the red lines on the spectrum with the arrow keys in the ROI sub-window. Multiple ROIs can be set, and the ROI Integral window will display data for all of them. It is necessary to set regions of interest when using EDS for quantitative analysis.

Analysis:

Peak ID:

This gives possible peak identifications for the current cursor position.

Auto. Ident.:

This function identifies peaks in the EDS spectrum display.

Quantitative Analysis:

This window displays analysis conditions for quantitative analysis.

F (Spectra Calc.):

This window displays options for mathematical manipulation of EDS data.

Periodic Table:

Peaks can be identified by guessing with the Periodic Table keys. Clicking on an element when **Peak Label** is ON causes lines coinciding with the expected peaks and the element name to appear at the appropriate positions. **Always Analyze** keeps the peak labels up as other elements are selected so that multiple peak positions can be seen at once. To clear the spectra of unwanted peak labels, click on **Analyze if Found** and then on the elements in colors other than blue. The element button will turn blue, and the peak label will disappear from the spectrum. Turning the **Peak Label OFF** will prevent the element name from appearing on the spectrum when the element is clicked on in the table.

The **Auto Ident** key is non-functional and misleading in the **Periodic Table** window. To identify an unknown peak, put the cursor on the peak and click, click on the **Analysis** button in the main EDS window, and then choose **Peak ID** or **Auto Ident**.

Print:

This is intended to print the spectra on the dot matrix printer.

1234:

This window displays conditions and comments for EDS measurements. It also allows for temporary storage of up to four EDS spectra and their conditions. The highlighted number indicates the set-up in use and spectrum currently displayed.

Parameter List:

The parameters listed here are the analysis conditions for scans 1, 2, 3 and 4.

Data Management:

This allows for storage and saving of conditions and spectra.

Vertical/Horizontal scale:

These controls let the user zoom in and out on an energy range by clicking on the appropriate arrow buttons.

Cursor:

This function moves the cursor in the home window. However, it is simpler to move the cursor by clicking directly at the desired spot.

Net/Gross:

This is a toggle between spectra types. The spectra options are Net (background corrected) and Gross (the same as in the small window).

Display Mode:

This lets the user to manipulate the display (colors, etc.).

Analyzer:

This window displays the detector conditions of the EDS system and allows the user to manipulate them. Most of these conditions should only be changed by someone who knows what they're doing (like a JEOL engineer). The only thing routinely done by users in this window is to toggle between **Bias on** and **Bias off** in response to a common error message saying that the gun bias is off.

Aperture #:

The aperture should always be left on #1. See Section 9, Quantitative Analysis for the proper setup of EDS analysis conditions..

Using EDS for Qualitative Analysis on Preset Points :

It is possible to preset multiple points and analytical conditions for qualitative EDS analyses by choosing Analysis->EDS Qualitative Analysis.

Sample:

Choose group and sample names as in quantitative analysis (Section 9).

Measurement:

EDS Conditions:

Set the following conditions for EDS analysis:

Els keV: Set the energy range that you are scanning.

Mode: Choose whether to measure for a fixed real time or live time (detector live time as inferred by the dead time bar at the side of the spectrum--approximately 70% of real time).

Seconds: Set the acquisition time.

Aperture: Use #1. Refer to Section 9, Quantitative Analysis, EDS Conditions for explanation.

EOS conditions:

This is the same window as in quantitative analysis, and it summarizes the Electron Optical System conditions to be used for the analysis. Refer to Section 9: Quantitative Analysis for details.

Stage position:

As in quantitative analysis, this window allows the user to set the stage for the points to be measured. Proceed as described in Section 9: Quantitative Analysis.

Preset Measurement:

This window also works the same way as in Quantitative Analysis. Make sure the positions you want to measure have all been selected (buttons in **Stage Condition** window are blue) and click on **Acquire** to begin analyzing.

SECTION 14: Qualitative WDS Analysis

Menu: Analysis -> Qualitative

This function is used for obtaining WDS spectra, similar to EDS spectra, of elements present in a sample.

Sample Button:

Choose your group and sample as described in Quantitative Analysis (Section 9).

Measurement:

Condition Load:

If the spectrometer conditions from a previous run have been saved, they can be recalled by choosing **Condition Load** and highlighting the desired qualitative analysis condition file, then clicking on **Load**,

Spectrometer Condition:

This window displays the measurement conditions for the spectrometers. To alter the conditions, click directly on the desired setting value. Values for the measurement starting and ending positions can either be set directly or by element designation.

To use element designation, click on the **Elem-** box at the bottom of the condition list for the spectrometer of interest and a window containing a representation of the periodic table will appear. Click on the **Order** button in this window if it is necessary to set the order of the lines. Use the **Line** button to specify which line type. Click on the desired elements, and the measurement range will be set to include all x-ray and background analysis positions for these elements.

All elements can be chosen simultaneously by clicking on the **All Elem** button. Clicking on the **Back (+)** or **Back (-)** button allows you to change the measurement range for the peak position. Click **OK** when everything is set properly.

EOS Condition:

This is the same window as is used in Quantitative Analysis (see Section 9), and is used to set the electron optical system conditions for use during measurement.

Stage Position:

This functions in the same way as it does in Quantitative Analysis. Refer to Section 9 for details about how to set points for measurement.

Condition Store:

This option allows you to save the conditions used for this analysis (especially spectrometer conditions) for use again later. Give the file an appropriate name and choose **Store**.

Print-out Condition:

Choose items to be included in your printed output.

Survey Measurement:

A measurement will be performed at the current instrument settings and the results stored at position #9999 for the current Group/Sample.

Preset Measurement:

Measurements will be performed using the measurement conditions set in the **EOS Condition** window for all the points chosen in the **Stage Position** window.

Real-time Display of Spectra:

Choosing Analysis -> Qualitative Realtime will allow you to watch the analysis as it is occurring.

Realtime:

This will open a window where you can choose conditions for the live display, such as the number of spectra to display and the order in which to display them. Choose **Start** to begin the realtime display, **Stop** to stop it.

Sample:

This can only be used after the realtime display has been stopped, and will allow you to display spectra previously collected for a different sample. Enter the sample name for which you wish the spectra to be displayed.

Operations:

These operations can also be used only after the realtime display has been stopped:

Change Spectrum Pattern:

Choosing this will allow you to change the scale, color and format (vector, dot, bar) of the spectra.

Display Parameter:

Change the display items and the abscissa units for the parameters at the right of the realtime display window.

KLM Marker:

Labeled markers will appear on the spectra at the positions of the characteristic x-rays for the chosen elements.

Peak ID:

In this window, choose the spectrum of interest, then click with the cursor positioned on peaks in that spectrum, and the names of the characteristic x-rays in the vicinity of the pointer will be displayed.

Spectra Calculation:

A variety of calculations such as smoothing, background subtraction, etc., are available through this menu choice.

Write Text:

Characters may be written in the spectrum display area.

Reset Spectra:

Restores spectrum to the state in which it was originally displayed.

Print-Out:

Choose items to print.

Processing Qualitative WDS Spectra:

Menu: Process -> Qualitative

Sample:

Choosing this will open a window listing the samples available in the current group. The group name can be changed by choosing **Group** in this window. Highlight the sample name of interest, and the available stage positions will be displayed. Highlight the stage position, and a list of available spectra will be shown. The number of spectra to be displayed is changed with the **Max Spectra** button; highlight the spectra of interest and click on **OK**, and the spectral data will be displayed in the data display window.

Operations:

This button accesses a number of possible processing operations which are described more fully in the JEOL Basic Software notebook in the Qualitative Analysis chapter.

Spectra Display:

Set zooming windows, normalization factors, horizontal and vertical scales. Change spectrum color, type (vector, dot, bar) and parameters on display at the right of the spectrum. Add text, or arrange to display multiple spectra to illustrate points of similarity or difference.

KLM Marker:

Add markers to the display for characteristic x-rays for chosen elements.

Peak ID:

Display names of characteristic x-rays for chosen peak positions.

Off-Line ID:

Display names of existing elements at the corresponding peak positions on the spectra. Choose **Apply** to save these.

Spectra Calculation:

A variety of mathematical operations are available for application to the spectra (smoothing, background subtraction, etc.).

Reset Spectra:

Release processing executed in the **Operations** menu and restore the settings to the original state.

Print-Out:

Choose data for printing.

SECTION 15: Data Management

Phase Analysis: Process-> Phase Analysis

Phase analysis creates useful plots of data from microprobe files. However, this software is not compatible with most IBM or Macintosh graphics programs and the results can be printed only as a screen dump via the Seiko color printer.

Click on **Sample** and choose your group and sample name in the usual way, then choose **Operation**. In the Operation menu, choose Analysis to indicate the type of data to be processed (quant, map, etc.), the type of output desired (weight %, atom or intensity) and whether or not to apply the least-squares fit line. An intensity display using quantitative data displays net intensities. If map or line data is displayed, the raw intensity is used.

Choose **Element**, to select a plot type (histogram, ternary plot, 2-dimensional scatter plot (x-y)) and assign appropriate axes and display levels. **Color** allows you to choose the marker symbol, color and size for the plot. **Save Result** gives you an opportunity to save the plot file. **Plot** actually initiates the plotting, either as an overlay to a plot already displayed, or as a new plot. Because conditions for phase analysis don't default to the previous analysis conditions, you must choose the elements again for each plot you create.

Summarizing Data: Process -> Quantitative -> Summary

Choose your group by clicking on **Group**, highlighting your choice in the resulting list, then clicking on **OK**. Choose the samples you want to include in the summary by highlighting them in the list. Click on **OK**. Then select which analysis points you wish to summarize by clicking on the appropriate buttons or selecting them in groups using the **Some** or **All** buttons. Choose the type of summary you wish to create.

Normal:	output as produced on screen during measurement
Calc:	choose ratios, sums from a list which appears
Atom:	cations based on # of cations chosen in printout condition
100%:	analyses normalized to 100%.
Wt %:	weight %
Net:	net counts
Current:	beam current
Total:	totals only, weight %
L-Value:	peak positions
K-ratio:	uncorrected weight % analyses
Stage:	total weight %, stage position, date and time, comment
Standard:	standard data
Spreadsheet:	weight % summary suitable for export to spreadsheet

Depending on the type of summary you have chosen, options may be available which affect the appearance of the output (e.g. choosing to use row or column format).

When you have chosen your summary options, click on **Type Out** and the summary will be displayed in a new window on the screen. To print a copy of your summary, choose **Print** in the data display window.

To save the summary for transfer to a floppy or to another computer over ethernet, choose **Save** in the display window. Another small window will open to allow you to choose a path and file name, though a default is provided. Please use the default path name to prevent the proliferation of summary files on the disk.

In the display window, choose **Clear** to clear the current data. If you just **Close**, other summaries you create before exiting the Process Menu will be appended to the data already on display, so if you then choose to do a **Save** you will be saving multiple summaries in a single file.

Off-line Data Correction:

Menu: Process -> Quantitative -> Off-line Correction

This will open a window similar to the window for Analysis -> Quantitative.

Within this window, choosing **Sample** will give you the opportunity to choose a group name and the sample of interest.

Conditions will offer a series of choices similar to those under Analysis -> Quantitative -> Measurement.

Standard Data will open a window allowing you to edit your standard data... this window is very slow to come up, so be patient.

K-Ratio Input & Correction will open a window which allows you to manually adjust K-ratios and re-correct your data.

Intensity Input & Correction (for WDS analyses only) will open a window where you can manually adjust net intensities and beam currents and re-correct your data.

File-Read & Correction (for WDS analyses only) will open a window where you enter the old file name under **Read**, the new file name under **Save**, and choose the samples you wish to correct. *If you do not rename the new file the original data will be overwritten.*

Read Intensity and Apply will perform and print the correction.

Recommended Use and Warnings about Off-line Corrections:

The most common use of off-line correction is to check the quality of various possible standards available. There is generally no problem with substituting standards and recalculating your analysis as long as the new standard was analyzed under the same conditions as the unknown (check PHA settings, peak and background positions and EOS conditions to make certain they are identical between standard and unknown). Note that if a re-calibration of a standard has been done between the original analysis and the off-line correction, the most recent standard data will be used in the correction procedure. Off-line correction may also be used to recalculate analyses using different oxidation states (e.g. Fe₂O₃ and FeO) without any problems.

Other uses of Off-Line Correction software are up to the users' discretion, but manually changing counts of k-ratios of either standards or unknowns will tend to be subjective and will generally degrade the scientific integrity of the analyses. Adjusted data should not be published as truly quantitative results unless thorough explanation of one's methods and reasoning for such corrections are also presented. It is essential that users understand what they are doing when using the Off-line Correction software. If there is any question of the quality of either the standard or unknown data, it is generally considered better to repeat the analyses rather than correct them off-line.

To insure that the original analysis files will not be overwritten by the off-line correction, enter another file name in which to save the new data. The user should also make note of the original conditions and what is being changed.

Transferring data to a floppy or over ethernet (for IBM/PC users):

After creating summary files, you may transfer files over ethernet by following the transfer instructions below (omitting references to floppies).

To transfer the files to a floppy, turn on the PC and load your floppy in disk drive A or B. The floppy must be IBM formatted, and therefore must be converted later to Macintosh format to be read by Apple computers.

At the DOS prompt, change to the appropriate floppy drive by typing A: or B:. Then type

```
ftp workstation
```

where *workstation* = name or IP address of the workstation. Check with lab personnel.

In Menlo Park, you may open the connection by simply typing "f" at the first prompt, thus avoiding typing in the IP address.

You should receive a message saying you are connected to the workstation and asking for a login. Log in the same way you would log onto the HP workstation when running the microprobe.

If you want your spreadsheet file transferred so that it can be opened in Excel, you must type

```
binary  
at the next prompt.
```

To transfer the files to the floppy, type

```
get pathname/filename new file
```

If you omit "new file", the file will be copied as "file name".

Be aware that although UNIX allows long file names and extensions, the resulting DOS names will be truncated to eight characters and extensions to three.

The "get" command doesn't recognize the * convention for file naming. If you have a number of files to transfer and need to use the * convention, the command is "mget" instead of "get".

When the transfer is complete, remove your summary file from the probe workstation by typing

```
rm pathname/filename
```

Type "quit" to exit from the file transfer program and return to the DOS prompt.

You can check the contents of your floppy by typing

```
dir a: (or b: if that is where your floppy is)  
on the PC.
```

View your transferred data on the PC a screen-full at a time by typing

```
type filename | more
```

Transferring Data Files to Your Mac Using Fetch

Fetch is free-ware. It is a Macintosh-style version of FTP (file transfer protocol), which allows a user to log onto another computer remotely and copy files to their own system through a network. Therefore, in order to do this you must have a copy of Fetch loaded onto your Mac, you must be on the network, and the network must be functioning. You cannot create files using Fetch. Summary files must be created on the workstation.

1. Open Fetch. If the HP Workstation you wish to contact is already nicknamed in your shortcuts menu, go to step 2. If you have not already loaded the appropriate workstation information in the Shortcuts directory, click on Fetch in the command bar at the top of the screen and choose Edit Shortcuts from the pull-down menu. Enter in the following information highlighted in bold below into the Edit Shortcuts window:
 - Shortcut (this is the official "nickname" of the HP workstation): *see lab personnel for info.*
 - Host (this is the IP address of the workstation computer): *see lab personnel for info.*
 - User ID (this is the sub-directory that holds all user files): *see lab personnel for info.*
 - Password: *see your lab personnel for info.*
 - Click on OK or hit the return key.
2. In the main window, click on the arrow next to the word Shortcuts, and choose the nickname of the HP Workstation from the pull down menu.
3. Choose the Binary option if you are transferring a spreadsheet file.
4. From the Fetch window, click on "open connection". A new window will open.
5. If the workstation information is not already loaded in the new window, click on the arrowhead pull-down menu next to the word "Shortcuts" and choose the HP Workstation name from the Shortcuts list of aliases, or load in the required information manually. Click on OK or hit return.
6. When the little dog stops running, find the file you want to transfer in the directory on the left side of the Fetch window. Highlight the file you want to transfer and click on "get file". A new window will open.
7. At the bottom of the new window there will be two boxes filled with question marks, indicating that the file is unformatted. You will need to change them to indicate spreadsheet formatting. To format the transferred file as an Excel file, click on the arrow button in the lower right corner and choose Microsoft Excel. Choose the directory on your machine to which you want the file transferred. Click on OK or hit return. You should now find the file you transferred in the appropriate directory and be able to open it directly by double clicking on its icon, or by opening it through Excell.

Backing Up Data

It is very important that at the end of each probe session you back up the data you have just collected. Each person has been given a portion of a magneto-optical (MO) disk for this purpose, and is responsible for his or her own data. Load your MO disk into the MO drive with your label facing up.

Menu: Utility -> File Utility

Choose **Media Access** from the resulting display, and then **Backup** from the options which are presented. A new window will open. Choose **MO Disk** as the media type, and make choices about what you wish to back up. Because of the time involved, you will probably only want to back up your measurement conditions and your new data each time. Standard data is in common use and will be backed up on a regular basis as part of the lab management schedule.

To restore data that has been backed up on MO disk, choose **Restore** under **Media Access**. A new window will open in which you choose **MO Disk** as the Media type. A window will inform you that current data will be destroyed. It is advisable to transfer your data sample by sample to avoid re-configuring your directory and wiping out data that has not been backed up. Data files with the same names will be overwritten. Be patient--this is a very slow process. It is possible to access other functions of the microprobe (set up and analysis, etc.) while the **MO Disk** is being searched.

Using the Data General Workstation to access Microprobe Software:

It is possible to access microprobe software via the Data General workstation. However you ****MUST**** limit your activities to items which will not affect the operation of the microprobe, such as creating summary files, operating Phase Analysis software, etc. If you have any questions or concerns about possibly interfering with the HP or microprobe functions, consult with lab personnel before proceeding.

To access the HP workstation, log on to the DG, then at the prompt, type

```
xhost HP workstation name
```

```
telnet HP workstation name
```

Log on as you usually would on the microprobe.

```
setenv DISPLAY DG workstation name :0  
off_menu
```

At this point you will be in the EPMA menu on the microprobe. Again, do not interfere with microprobe analyses by removing or moving files, running Off-Line corrections or trying to set up or run analyses from the DG.

To exit:

```
Initialize-> End Menu
```

This will exit the EPMA menu on this terminal (the DG) only. At the prompt, type "logout" or "lo", and you will be returned to the DG workstation environment.

SECTION 16: Tips and Troubleshooting

Alarms:

There are four alarms you may encounter while running the microprobe: a stage limit alarm (loud fast beeps which occur after a stage movement); a vacuum alarm, which is rather faint and sounds like it is coming from behind the instrument (you should have heard valves closing before this); EDS loss of communication alarm (two alternating tones which are rather loud); and a faint alarm signaling that the liquid nitrogen in the EDS liquid nitrogen tank (EDS Dewar in Figure 3.1) is low. Follow the instructions below if you hear an alarm.

Stage Limit:

press the REQ button and move away from the stage limit with the joystick. If the joystick is disabled, open the Stage Monitor window and move the stage position (by clicking on the appropriate buttons). Notify laboratory personnel immediately whenever this happens.

Vacuum loss or decay:

First check the vacuum gauge to confirm that the vacuum has degraded (greater than approximately 1×10^{-5} torr and rising). If you cannot tell whether or not the vacuum is degrading, or if the vacuum looks fine, consult lab personnel. Once you confirm that the vacuum has degraded, turn the High Voltage off by pushing the HV button on the console. Check to see that the green button to the right of the airlock is lit (see lab layout Figure 3.1). If the button is dark, push it once. It should light up and the airlock should vent. Then call for help from a lab attendant. A loss of vacuum may destroy the filament (which will cost you several hours of down probe time), and it may also damage delicate and costly parts within the column.

EDS communication loss:

See instructions below about EDS loss of communication.

Liquid Nitrogen tank low:

See Figure 3.1 for location on the instrument. Do not use the EDS until this is refilled. Notify the appropriate personnel immediately.

Default Analysis Conditions:

Sometimes a user will set up an analysis and discover that the analysis was performed using conditions other than those they thought they chose, causing frustration and loss of time. To prevent this unfortunate occurrence, it is essential for users to understand the nature of system defaults on the microprobe in order to be confident that the analysis conditions which they intended are the conditions which will actually be used.

Difficulties stem from the fact that the defaults are, in most instances, dynamic, i.e. they are determined by "last use" rather than returning to values set by the controlling software. Therefore, while on one occasion the defaults that you find may be precisely what you want, you cannot depend on the same defaults being in effect the next time you set up an analysis. Default conditions do not automatically accompany the sample; that is, the defaults are based on the last use of the analysis package in question (Quant,

Map, etc.), not the last use of the analysis package for this sample. If a user sets up Sample 1 with a set of conditions Cond 1, then sets up Sample 2 with conditions Cond 2, then returns to Sample 1 to measure it, unless the user resets the conditions to Cond 1 values, the conditions in Cond 2 will be used for the measurement of Sample 1.

The user can gain control of the defaults by taking advantage of the Condition Load and Condition Store features in the different analysis packages. When all the conditions for analyzing a certain sample have been defined, the user should use Condition Store to store those conditions to a file. Then that set of conditions can be recalled using Condition Load at any time to become the current defaults for that type of analysis. Storing standard conditions thus simplifies returning to your previously used conditions through Condition Load, though the data in the standardization file will be from "last use".

If the probe software is configured so that each user has his or her own directory, the dynamic defaults will be set by that user's own "last use". If any of the analyses are shared across the system (e.g., standard data is shared by creating a link to a common standard file structure) then the "last use" defaults will be those conditions set by the last user of that type of analysis. Defaults for standard analyses are determined by the last use for the standard in question, not the last use of standard analysis in general. Storing standard conditions thus simplifies returning to your previously used conditions through Condition Load.

Common Errors

Impatient user:

If the workstation software fails to execute a command instantaneously, some users become impatient and click on the same command button or buttons several times. This can cause the system to crash because the redundant commands go into a buffer and the system tries to execute the same process over and over. The remedy is simple: **click the mouse button once and be patient.**

Some software operations may take several seconds before you see results, and often there is no indication on the screen that anything is happening. One common result of over-clicking is overloading the print buffer--jobs will be printed as many times as you click PRINT. To clear the print buffer, see the instructions below.

Caps-Lock key:

When the caps lock key is depressed, the EPMA menu is effectively disabled, since an upper-case mouse click does not have the same effect as a lower-case mouse click. If the menus will not open, the caps-lock key is the first thing you should check.

Airlock: (automatic in Reston)

After loading a sample into the analysis chamber and closing the gate valve, it is easy to forget to vent the air lock. The green button next to the gate valve handle is lit when the air lock is vented and dark when the air lock is evacuated. If the air lock is not vented by pressing the green button, the roughing pump will continue to pump on the air lock rather than backing the diffusion pumps, causing the vacuum to degrade. The accuracy of your analyses will be severely affected; you may lose the filament and if the vacuum becomes too badly degraded, the entire system will shut down. **Always vent the airlock after changing samples.**

Useful Operational Information

Updating window displays:

Not all window displays are updated automatically when the information on display is changed. Before becoming concerned that a change that you have made wasn't entered into the system, back out of the window sequence where you are currently operating. Close the windows, perhaps as far back as to the main EPMA menu, then re-open the windows and see if the expected change has occurred.

Setting spectrometers to default conditions:

Occasionally the spectrometer conditions are changed to other than the default values. To assure that the peak position, gain, etc. are set to the hardware default values for this crystal on this spectrometer, you may choose **Channel** and **Crystal** in the WDS Conditions window (Measurement -> Element Conditions -> WDS Conditions) in each of the analyses packages (Standard, Quant, Line, Map) even though you don't want to change them.

Clearing the print buffer:

- Turn off the printer (switch on back right side of the printer).
- Open an HP terminal window by clicking on the gray screen background, then dragging to NEW WINDOW and clicking, or by opening an existing HP Term icon.
- Type **lpstat** to show the active printer jobs. Lp1 is the system name for the dot matrix printer in Menlo Park and Denver. The line printer is called lp132 in Reston.
- The command **cancel lp1 -a** clears all jobs in the print queue for the printer named **lp1**. To cancel a specific job, substitute the job number (use lpstat to determine the number) for the "a" in the previous command (e.g., **cancel lp1 -1874**).
- When you turn the printer back on it should print "canceled" and then quit.
- To re-create print jobs that you deleted from the print queue, you must repeat the procedure you used to create them the first time. There is no way to retrieve jobs deleted from the print buffer.

Freeing a frozen joystick:

Users should understand that the joystick works most effectively when it is treated as a sensitive piece of high precision equipment. Do not treat the joystick controls roughly or use sudden movements and then expect the stage to respond immediately.

Occasionally the joystick on the console locks for no apparent reason. It can sometimes be freed by pressing the TEST key.

If the TEST key doesn't free the joystick, try pressing the STORE and REQ keys on the joystick console. Be aware that by doing this you may be storing a position

you really don't want, so you would be wise to confirm your positions after doing this.

If the above methods fail to free the joystick, try opening the Stage Monitor window from the main menu (Monitor -> Stage) and clicking on the directional arrows or **Test**, or clicking **Read** and then **Move** in the Pos. Input window.

Initialize -> Reset Analysis will sometimes free the joystick.

As a last resort, do an **End of Menu**. This procedure is described in the section of this chapter about regaining control if the system hangs up. **Close any open windows before doing this.**

Regaining control if processing hangs up: (arranged in order of increasing desperation)

Reset Analysis: (Initialize -> Reset Analysis)

This resets the windows for whatever analysis routine is open. Reset Analysis is quick and relatively harmless, but it is wise to check for lost information after using it.

End of Menu: (Initialize -> End of Menu)

Close any open windows (if possible) before doing this, as information in open windows may be lost, and temporary files created by the system will be deleted.

Choosing this command will shut down the EPMA software. A window asking you to confirm that you wish to end the menu will appear; click on OK. The screen will clear and the EPMA icon will disappear from the screen.

Wait for all disk activity to cease (a few minutes), then click in the gray background field and drag and click to select "EPMA MENU" from the blue menu box. As EPMA is reloading, the background screen will gradually become a lighter gray, and eventually the EPMA main menu will reappear. Be patient. This will also take a few minutes.

Even after the EPMA menu is functional again, the system will be slow for about 20 minutes as the hard drive goes through what JEOL calls "garbage collection", searching the entire hard drive for files which must be deleted. The restart process can be monitored from the EPMA window whose icon is at the bottom right of the workstation screen.

System Shutdown: (Initialize -> System Shutdown) (Notify lab personnel before doing this)

This will shut down processing in an orderly way, and will destroy all temporary files created by the program. Close any open windows (if possible) before doing this, as information in open windows may be lost.

- A window asking you to confirm that you wish to shutdown will appear; click on OK. The screen will clear, and the message "halted..." will appear when shutdown is complete.

- Turn off the computer, using the rectangular ON/OFF button on the computer. Wait at least 15 seconds, then push this button again to restart the computer.

The computer will reboot automatically, and you will be prompted for a login name and a password. The EPMA software will automatically be restarted, so be patient at this point; the screen will gradually become light gray and eventually the EPMA menu will appear, and the system will become functional again, although it may remain sluggish for 15-20 minutes while it continues its "garbage collection".

Soft reboot: Press the SHIFT-CONTROL-RESET keys simultaneously.
(Notify lab personnel before doing this)

A soft reboot will dump everything and put you back at a login command. This is a very messy way to end things, so only resort to this if mouse control is totally locked up and none of the above options can be used.

After login, you will have to start the EPMA menu manually by clicking on the background to get the root menu, then dragging and clicking to choose EPMA MENU. Be patient as the screen gradually becomes lighter gray and eventually the menu appears.

Hard reboot: ON/OFF switch (white button on the HP workstation computer. See Fig. 3.1):
(Notify lab personnel before doing this)

This is truly a desperation move. If you have not been able to do an End of Menu or System Shutdown, rebooting will take 15-20 minutes before the login prompt appears, as the system must do a major clean-up. The procedure for restarting is the same as described under System Shutdown.

Re-establishing software-to-hardware communication:

If the EDS alarm is sounding (it is two alternating tones, like a European police car), if the EDS system is not working, or if the Present Values or EPMA window lacks information on one or more spectrometer, or for some other reason you believe that communication is broken between the workstation and the instrument, **first notify a lab attendant.**

Do an End of Menu operation as described above. But before restarting the EPMA menus press the appropriate restart button or buttons as described below.

EDS reset button:

This is located on the back of the EDS control unit, below the desk level and behind the main console. Buried beneath wires coming out of the unit is a button clearly marked RESET. Push this button once and a series of tones will be heard which sound like a major scale or a vocal exercise, indicating that communication has been re-established. Restart the EPMA windows and continue normal operation.

WDS reset buttons:

These are located on the back left side of the visual consoles, next to the instrument. They have rounded caps and stick out of the console about an inch. To re-establish communication between the workstation and the instrument, push both buttons

simultaneously. Restart the EPMA menus and check the Present Values and EPMA windows to make sure that communication has been re-established. If the channel and crystal information are still incomplete in the Present Values window after this operation, notify a lab attendant.

Visual console reset button:

This is a recessed orange button located near the front of the visual console on the right side of the unit next to the workstation monitor. Pushing this button once resets the console screens to SEI images and resets the focus of the images. It is not necessary to do an End of Menu before resetting the console screens.

Dealing with a power outage:

- Turn off the system by turning the key located to the left of the HP workstation monitor to the OFF position.
- Press HV button, to turn off the high voltage switch. The button should be raised, not depressed.
- Turn off the HP workstation computer by pushing the white rectangular button on the computer (see Fig. 3.1) and turn off the workstation monitor.
- Turn off other computer, monitors and printers.

Restarting after a power outage:

- Wait several minutes after power is restored to avoid subjecting the equipment to large power surges and fluctuations which often occur at such times.
- Turn on the room lights and see that they are fully bright. If they are not and any dimmers are turned all the way up, don't turn on the probe.
- Turn the key on the console to START. It should automatically spring back to ON and stay there.
- Check the vacuum monitor, and turn it on if necessary (ON/OFF switch is on the back). When the vacuum is stable within the acceptable range, you should be able to run. The acceleration voltage must remain off until the vacuum has recovered.
- While waiting for the vacuum to recover, turn on the workstation computer, monitor, and any other auxiliary items. The computer will probably take a total 15-20 minutes to reboot, so be patient. You will eventually be prompted to log in and give a password, after which the EPMA software will be loaded automatically.
- Choose Monitor -> Present Positions, and leave this window open. Choose Monitor -> EOS Conditions, and make sure the PCD (Faraday cup) is blocking the beam. If the vacuum is in the proper range and the workstation is communicating with the instrument, turn on ACCEL VOLTAGE on the main console. The Present Values window should display the beam current. If the current is reasonable (about 20 nanoamps) and stable, you should be able to run, and can continue with the normal start-up procedure. If the current is abnormally high, low or non-existent, notify a lab attendant. The filament may have blown, or the beam may need to be realigned.

Useful Analytical Information

Poor totals during Quantitative Analysis (mechanical causes):

For samples with no H, HO, H₂O, Li or other elements or species not analyzed, totals should be between 98.5 and 101.0 weight %. There are many possible causes for poor totals, including poor standardization. Some possible causes and solutions are described below.

You may be on the wrong sample or standard. Consult images of the standards or unknowns, or use EDS to determine the elements present.

The beam may be rastering. If you see an image on the console screens while you are analyzing your sample or your standard, open the EOS conditions window and change the probe scan setting to OFF for the analysis or standardization. Rastering during standardization can give very high totals on the unknowns. Rastering during quantitative analysis will generally give low or high totals (depending on what the beam is scanning).

The vacuum may be degraded. Make sure the green button next to the gate valve handle on the airlock is lit. If it is dark, push the button once to vent the airlock. The vacuum in the sample chamber should slowly recover (see the section above entitled "Venting the Sample Chamber Airlock" for more information).

You may have missed an element. Acquire an EDS spectrum on the sample to see if you missed any major or minor elements.

The beam may be mis-aligned. Ask a lab attendant for assistance if you think this might be the source of your problem.

The carbon coat may be too thick or too thin on either the standard or the unknown, the standard may be damaged, or the sample may not be properly grounded to the holder. Consult a lab attendant if you suspect carbon coat problems or damage to the standards.

You may be using the wrong standard. There are preferred standards for each element depending on the type of phase being analyzed. If you analyze a glass with an olivine standard, you may find the totals to be low because the peaks have different shapes or slightly different peak positions. Ask a lab attendant which standards are preferred for the phase(s) you are analyzing.

Loss of alkali metals, F and Cl may bring totals below 100%. Always analyze for sodium and/or potassium first. In general, do not use a peak search for analyses of alkali metals. Also, use a defocused beam, short counting times and low beam currents whenever possible. High silica glasses are notorious for Na and K loss. If significant loss is still occurring even under these conditions, consider cutting down analysis times and/or sample current, or increasing the beam diameter. Alkali loss can be monitored using Monitor -> Chart Recorder. The chart recorder should be used to determine analysis times for alkalis. Roaming around on the sample with the joystick during the analysis may be a last resort if the loss of alkalis during analysis is so rapid that shortening counting times and lowering the beam current will degrade the counting statistics appreciably.

Loss of volatiles may bring totals below 100%, but nothing can be done because it is not possible to analyze quantitatively for water with an electron microprobe. The instrument is capable of analyzing for carbon and oxygen. Consult with laboratory personnel if you are interested in analyzing for these elements. If you wish to analyze for volatiles, try using SIMS or IR spectroscopy. Otherwise, using standard microprobe techniques and not analyzing for oxygen or carbon directly, expect totals lower than 100% for hydrous phases and glasses when using the microprobe. Consult Deer, Howie and Zussman (3) or another appropriate reference for expected cation ad weight % totals for the mineral or phase of interest.

Different oxidation states of elements (e.g. Fe^{+3} and Fe^{+2}) in your standard or unknown (other than those set-up in analysis conditions) could cause low or high totals.

Inclusions or holes may be present below the surface, leading to high or low totals.

Low totals from Quantitative Analysis (software-related causes):

Stoichiometry may not be correct. Check that the data is being calculated correctly for all elements (e.g. SiO_2 not Si_2O_3).

The default peak position for an element may not have been updated to the current best position, so data was not collected at the optimum peak position. This is most likely to be a problem only for minor elements when not enough counts are generated to have a peak search performed at the time of analysis, or if the peak search has specifically been turned off. To check this, choose Element Conditions in the Measurement menu of the appropriate analysis type and compare the peak positions there with the peak position in the window "behind" it which contains all the possible configuration choices for the element in question. Peak positions are automatically updated in the window containing the configuration choices, but not in Element Condition files.

Not all of the analytical information may have been reported. If all elements being analyzed are not included in the correction, check the element order in the printout conditions window. Include any missing elements by clicking on the appropriate element names. The element order shown at the bottom of the window is the order in which the analytical results will be reported. Remember, this is independent of the order in which they will be analyzed.

Analysis conditions may not be the same as the standardization conditions. Check the voltage settings: PHA gain, high voltage (HV), baseline (Base L.), voltage, window, and differential (D) versus integral(I) settings--in the standard analysis and quantitative analysis condition columns that you are using. Note that if the integral mode is chosen, the window setting is ignored. If your analytical condition columns for standard and unknown do not have the same settings, consult a lab attendant. **Please do not change PHA settings or voltage settings without consulting a lab attendant first.**

The beam current for your analysis may differ from the beam current for your standardization. (Check the EOS conditions window or the printout). If the standardization was done at a significantly different beam current than the analysis for one or more elements, you may have to re-standardize. Raw data are stored as counts/sec/nanoamp, so beam current differences should only lead to problems if the counts are low and the difference in beam current is great.

Spectrometer choice:

Even though there is approximately 1% reproducibility on flipping the crystal, it is best to avoid flipping crystals during quantitative analysis whenever possible. If you do flip crystals, be sure to do a peak seek after flipping.

Spectrometer 2 has a smaller Roland circle radius and uses larger crystals, thus it gives a higher count-rate for better sensitivity to minor elements. The lower limit for crystal positioning is higher than the other spectrometers, which means that some elements accessible on LIF or PET on other spectrometers will not be accessible on spectrometer 2. Also the peak to background ratios are not as good as other spectrometers. Therefore, your best sensitivity may be on another spectrometer. When choosing a spectrometer and crystal, first choice is to use the $K\alpha$ line. If more than one crystal possibility exists, it is sometimes better to choose the one with the lower peak position. Doing this will give a higher count rate but poorer resolution. It may in fact be better sometimes to use the higher position to resolve interferences such as S-Pb, V-Ti, S-Mo, etc.

Peak search:

If the x-ray intensity for a minor or trace element is too low for a peak search to be successful, the last measured position for that element is used. (See, however, the notes about default analysis conditions and software-related causes of poor totals earlier in this section.) Peak search thresholds are:

15 kV	100 cps
20 kV	200 cps
25 kV	300 cps

When setting up measurement conditions for an unknown, a normal peak seek choice would be 1. Peak seek = 0 means no peak search is ever done. For standards, the peak seek parameter is automatically set to 2 unless it is set to 0, meaning two iterations are done on the peak search over successively narrower regions even if the peak seek number set in Analysis->Standard->Measurement->Element Conditions is 1.

In Analysis -> Quantitative -> Measurement -> Additional Functions, it is possible to choose to skip peak searches during analysis or only to do them once every n points, so there is seldom a reason to set the peak seek parameter to a value other than 1.

EOS conditions:

Possible kV range is 0-40, but 15-20 is usually suitable for most geological applications. Check with lab personnel before running the instrument at a higher voltage.

Normal probe current (PCD in) range is 10-50 nanoamps ($1-5 \times 10^{-8}$); usually 10-25 nanoamps is appropriate for silicates. If possible, use a higher current for mapping.

References:

1. Armstrong, J.T. (1991) Quantitative elemental analysis of individual microparticles with electron beam instruments. *In* Electron Probe Quantitation, K.F.J. Heinrich and D.E. Newbury, Eds., Plenum Press p. 261-315.
2. Bence, A.E., and Albee, A.L. (1968) Empirical correction factors for the electron microanalysis of silicates and oxides: *Journal of Geology*, v. 76, p. 382-403.
3. Deer, W.A., Howie, R.A., and Zussman, J. (1992) An introduction to the rock forming minerals. New York: Wiley.
4. Henke, B.L. et al. (1982) Low-energy s-ray interaction coefficients: photo absorption, scattering and reflection: *Atomic Data and Nuclear Data Tables*, v. 27, no. 1, p. 1-144.
5. Heinrich, K.F.J. (1966) X-ray absorption uncertainty *in* The Electron Probe, T.D. McKinley, K.F.J. Heinrich and D.B. Wittry Eds. New York: Wiley. p296-377.
6. Philibert, J. (1963) A method for calculating the absorption correction in electron probe microanalysis: *Proceedings of the 3rd International Conference on X-ray Optics and Microanalysis*. New York: Academic Press p. 379.
7. _____ and Tixier, R. (1968) Electron penetration and the atomic number correction in electron-probe microanalysis: *Journal of Physics*, v. D1, p. 685.
8. Pouchou, J.L., and Pichoir, F. (1991) Quantitative analysis of homogeneous or stratified microvolumes applying the model "PAP". *In* Electron Probe Quantitation, K.F.J. Heinrich and D.E. Newbury, Eds., Plenum Press p. 31-75.
9. Reed, S.J. B. (1965) Characteristic fluorescence corrections in electron-probe microanalysis: *British Journal of Applied Physics*, v. 16, p. 913-926.

Appendix A: Some Useful UNIX Commands

Please do not use UNIX commands before consulting lab personnel .

Create a UNIX window:

Move the pointer to anywhere on the gray background. Push and hold the mouse button, and drag the pointer down the menu that appears until the option "New Window" is selected. Release the mouse button. An outline of the window will appear, with a small box open which gives the current coordinates on the screen. Move the outline with the mouse to a convenient position for the window you are opening and click once to create the window.

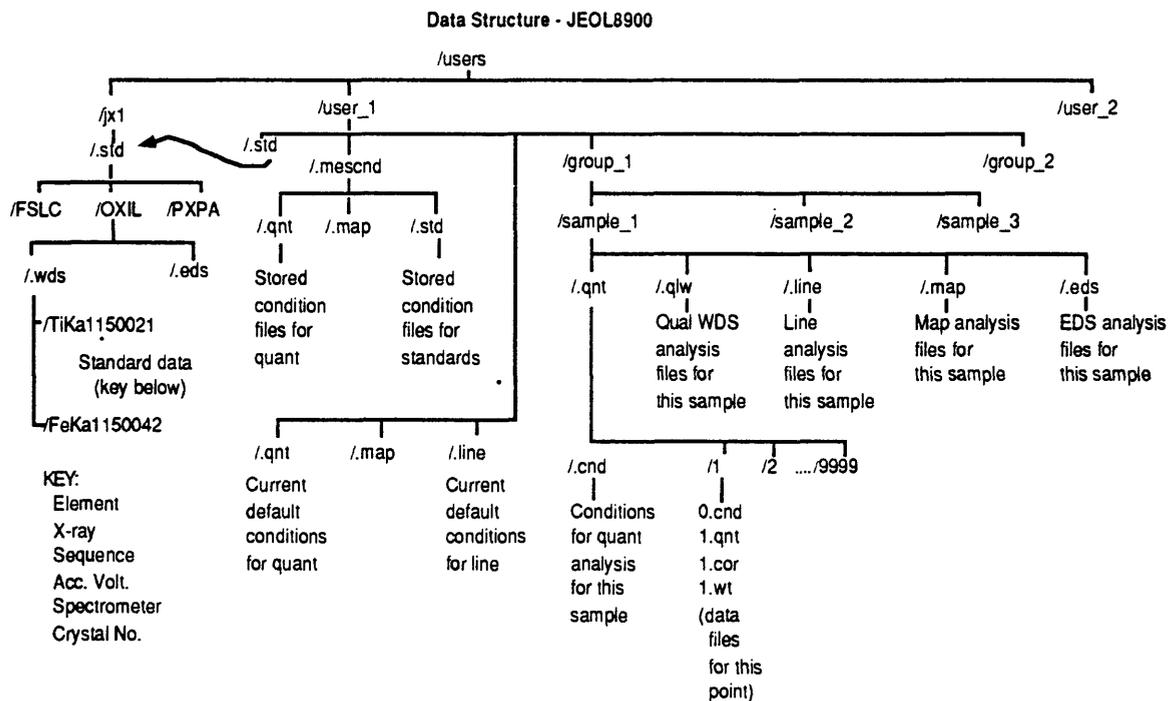
Destroy a UNIX window:

Type "exit" or CTRL-D in the window.

Alternative method:

Use the window menu option "Close" accessed by clicking on the upper left corner of the window.

File Structure of Software for JEOL 8900 Superprobe



UNIX is case sensitive!!

UNIX Command

Definition

cancel *job #*
cd *dir-path*
cp *file1 file2*

cancel a print job
change directory; default to home directory
copy *file1* to *file2*

exit

exit the current process, quit a window

find *dir -name file -print*

find *file*, starting search at *dir*

head *-n file*

list first *n* lines in *file*

id

list one's user id and group

lp *file*

print *file*

ls *-aF*

list contents of current directory

more *file*
mkdir *dir*
mv *file1 file2*

list *file* one screen-full at a time
create a new directory
move or rename *file1* to *file2*

pwd

print current working directory

rm *file*
rmdir *dir*

delete (remove) *file*
delete an empty directory

tail *-n file*

list last *n* lines of *file*

who am i

show own user id

Appendix B: Quick Reference Summaries

SECTION 4: Start-Up (Reston)

- 1) Turn the SONY and HP monitors on.
- 2) Check vacuum monitor for stability and acceptable vacuum reading.
- 3) Reset filament, check saturation level and beam focus (spot size).
- 4) Choose Initialize -> Change Directory to set your personal directory
- 5) Proceed with sample exchange, standard analysis, etc.

SECTION 4: Shut-Down (Reston)

Normal shut-down

- 1) Put the beam on brass or somewhere off the samples, transmitted light lens retracted.
- 2) Select full picture mode and SEI image.
- 3) Set magnification to 300,000X and scan speed to SLOW (S3).
- 4) Block the beam (PCD button lit) and turn OMTV and PRBSCN off.
- 5) Lower the filament saturation by ten clicks (Filament window in EOS Monitor). Leave these windows open.
- 6) Close any other open windows, leaving the EPMA menu, Present Values window and the EPMA and CONSOL icons.
- 7) Turn the SONY and HP monitors off,
- 8) Make sure a log book entry is completed, noting problems, solutions and a summary of your analysis conditions. Leave the lab area tidy.

SECTION 4: Start-Up (Denver, Menlo)

- 1) Turn the SONY and HP monitors on.
- 2) Check vacuum monitor for stability and acceptable vacuum reading.
- 3) Turn other peripherals on (color printers, PC, standard printer, optical drive)
- 4) Turn up the contrast and brightness located under CRT screens .
- 5) Turn the accelerating voltage on if it is not already on.
- 6) Set console screens to SEI image, FAST scan speed, PRBSCN on, magnification 40X.
- 7) Take the Faraday cup (PCD) out.
- 8) Turn on OMTV and focus light optical image.
- 9) Choose Initialize -> Change Directory to set your personal directory.
- 10) Proceed with sample exchange, standard analysis, etc.

SECTION 4: Shut-Down (Denver, Menlo)
Normal shut-down

- 1) Put the beam on brass or somewhere off the samples and retract the transmitted light arm.
- 2) Select full picture mode and SEI image.
- 3) Set the magnification to 300,000X and scan speed to SLOW (S3).
- 4) Put Faraday cup in (PCD button lit)
- 5) Turn OMTV and PRBSCN off.
- 6) Turn high voltage off (ACC VOLTAGE button not lit).
- 7) Close open windows, leaving only the EPMA menu and the EPMA and CONSOL icons on the screen.
- 8) Turn the SONY and HP monitors off,
- 9) Turn the DG monitor off. Turn the brightness and contrast knobs on the CRTs all the way down. Turn both color printers off.
- 10) Make sure that the **GREEN BUTTON IS LIT !!!** If it is not lit, press it to vent the sample exchange airlock to atmosphere.
- 11) Write an entry in the log book, noting type(s) of analyses, problems and solutions, and remove your belongings from the area. Turn off the lights, and (M) close the blinds before you leave.
- 12) Turn off the PC.

SECTION 5: Sample Exchange

- 1) **Remove the transmitted light arm from the chamber! (LENS button light off). Severe damage will occur if this is not done before inserting samples.**
- 2) Select **Sample Change** from the Stage Monitor menu, and click **OK**.
- 3) Blank the electron beam. (PCD button lit)
- 4) Seal the airlock lid and press the green button to evacuate the airlock.
- 5) When evacuation is complete (green light goes off wait 2-3 minutes), slowly open the isolation valve between the column and the airlock, keeping the vacuum in the 10^{-5} torr range.
- 6) Retrieve the sample holder using the sample exchange rod.
- 7) Close the isolation valve. In Reston, the airlock vents automatically. In Menlo and Denver, one must press the green button to vent the airlock.
- 8) Open the airlock by lifting and pulling on the lid, remove and replace samples.
Remember to wear gloves, and be sure you have oriented the sample holder properly!
- 9) Seal the airlock lid, and press the green button to evacuate the airlock.
- 10) When the airlock is evacuated (when the green light has gone out wait 2-3 minutes), slowly open the isolation valve, keeping vacuum in 10^{-5} torr range.
- 11) Replace the sample holder slowly, so as not to lose y-axis calibration.
- 12) Close the isolation valve. The airlock vents automatically in Reston; in Menlo and Denver, one must press the green button .
- 13) Click on the **OK** button in the Sample Exchange confirmation window.
- 14) **Make sure the green button is lit before proceeding.**

SECTION 6: Imaging

- 1) Set the accelerating voltage and adjust probe current.
- 2) Turn the probe scan (PRBSCN) on, set the magnification to 40X and unblock the beam (PCD out).
- 3) Use the VIEW button to select the image type (SEI, COMP, TOPO). If you are using COMP, turn OMTV off for best results.
- 4) Set the contrast and brightness. For SEI the control knobs are on the main panel; COMP and TOPO knobs are below the CRT screens.
- 5) Set the desired scan speed with the FAST and SLOW buttons.
- 6) Set the scan and reduce modes to PIC and FULL for the best image.
- 7) Find the desired location.
- 8) Set probe diameter to zero and focus the image at a high magnification.
- 9) The INST button can be used to set the instrument to the SEI conditions if you are having trouble getting an image.

SECTION 7: Polaroid Photography

- 1) Set the CRT contrast and brightness knobs to their maximums.
- 2) Set the brightness and contrast for the SEM image. The ACB button will automatically set these to the default if the slowest scan speed is on.
- 3) Load the film (lever in the **L** position), making sure film sheet is inserted with the proper side down, and pull the film sleeve back. Press the **L** or **R PHOTO** button. A slow scan occurs while the photograph is being taken.
- 4) Push the film sleeve back in, slide the lever to **P**, and pull out the film. Follow the developing instructions on the film.

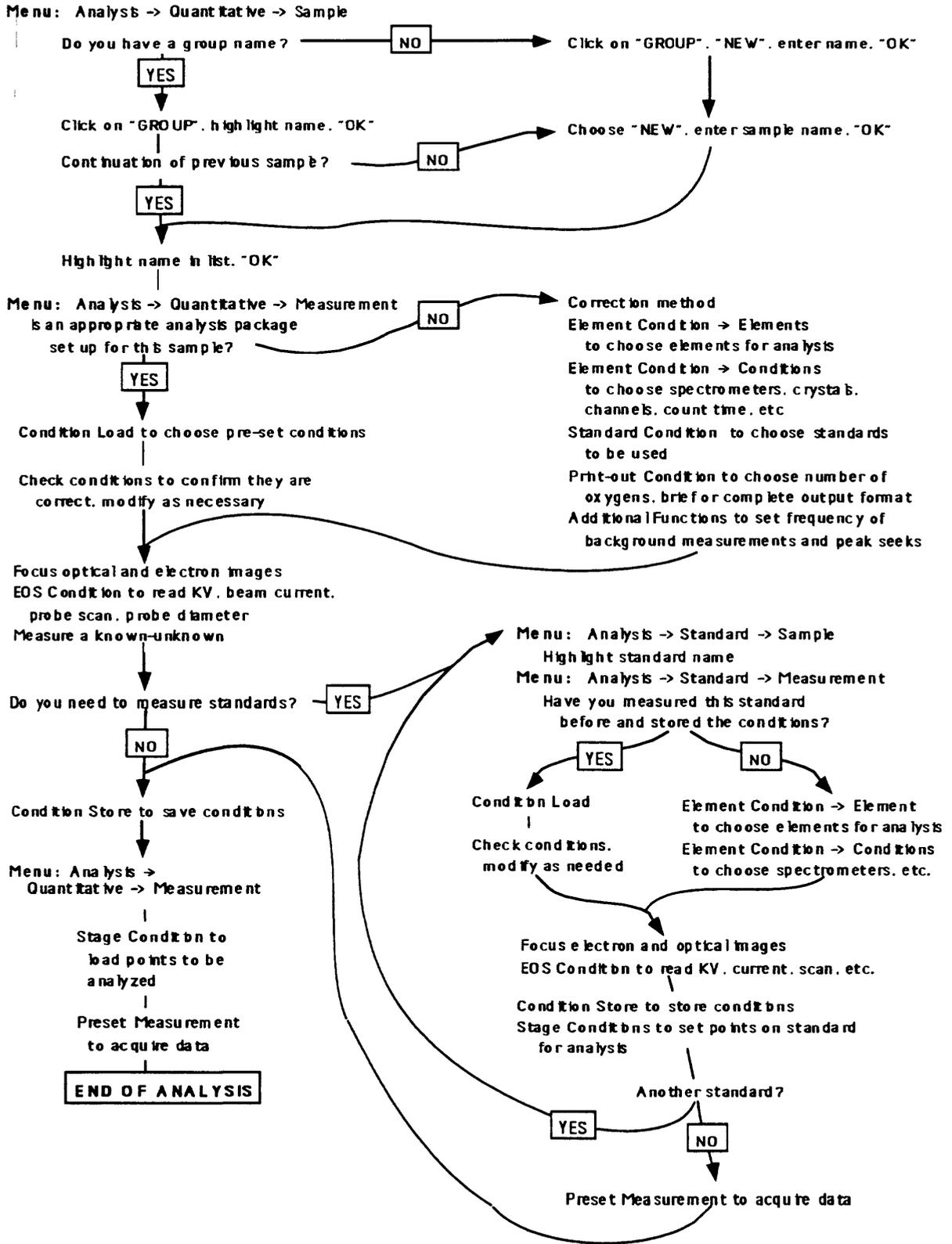
SECTION 7: SONY Printer

- 1) Select **MENU** on the remote or the printer to set multiple photos, separators, and comments. Choose **S** for optical image or **VIDEO** for SEM image. Or (**M, D** only) choose the image type by using the buttons on the lower front of the SONY monitor: **A**-source button for optical, **B**-source for SEI image.
- 2) For an SEM image, adjust the contrast and brightness on the SONY monitor using the knobs on the main control panel.
- 3) On the remote or the printer press **MEMORY-IN** to put the image in memory, then press **PRINT** to print the image.

SECTION 7: SEIKO Printer

- 1) Bring up the image to print on the HP monitor.
- 2) On the Seiko printer, select the number of copies and press **START**.
- 3) Colors can be reversed (meaning that the black background becomes white) using the arrow keys next to the start button.

**QUANTITATIVE ANALYSIS
FLOWCHART**



SECTION 8: Standard Analysis

- 1) Use the **Sample** Menu to select the standard sample you wish to analyze.
- 2) Go to the **Measurement** Menu; if you have previously set measurement conditions for this standard, recall them using **Condition Load**.
Load conditions for Quantitative Analysis before standardizing and re-store the conditions in order to update standard data.
- 3) Check the **Standard type**, metal or oxide, and make sure the calibration curve is OFF.
- 4) Under **Element Condition** choose **Element** and select the elements to be analyzed on WDS and/or EDS. Choose **Condition** to set-up the conditions for each element to be analyzed.
- 5) Set up the instrument for an analysis, make sure the probe scan is OFF, the beam diameter is correct, and the current is appropriate and choose **Read** in the **EOS Conditions** window.
- 6) If you wish to use EDS for the analysis, open **EDS Condition**. Set up the instrument for an analysis (10kV, aperture=1, 1024 spectrum data points) and **Read** in the current conditions.
- 7) Under **Stage Condition** choose **Position Input** to enter or modify/confirm the accumulation points for analysis. Click on **Confirm** and then **STORE** the accumulation points from the joystick control pad, after using the **TEST** button to check the focus of each point. Edit the comment line, press **Apply** to store all the information for that standard, and click on **Close** to close **Position Input**.
- 8) Select **One-by-One** followed by **Acquire** to analyze a single standard sample.
- 9) Choose **Condition Store** to save or update these conditions for future use.
- 10) To analyze multiple standards automatically, close the Stage Condition window and return to step (1). Choose the next standard sample and continue through step (9) for every standard you wish to measure.
- 11) In **Preset Measurement** select all the standards to be analyzed by clicking on their buttons, and press **Acquire** to start the analysis.
- 12) If any of the standard data needs to be altered after the analyses, select **Check Data** from the Measurement Menu. The data for the standard listed in the Standard Analysis dialog box will be displayed. **Note:** this menu item takes time to load, so be patient. Change the data as needed and click **Save**, then **Close** to exit the window.

SECTION 9: Quantitative Analysis

- 1) Click on the **Sample Button**. Choose or create group and sample names for this analysis.
- 2) If you wish to use analysis conditions previously stored, load them from **Condition Load** in the **Measurement** menu. Check that the settings are correct, paying special attention that the peak positions for minor elements are up-to-date, and skip to setting the **EOS Conditions** (step 7).
- 3) Choose the desired **Correction Method**.
- 4) In **Element Condition** choose **Element** to select the elements to be analyzed on WDS and/or EDS and those to be calculated. Choose **Condition** to set-up the conditions for each element to be analyzed.
- 5) In **Standard Conditions** select the standards that you have planned for your quantitative analysis. Be sure that the standardization dates match your standardization if you just did one.
- 6) Check output format and number of oxygens for calculation in **Print-Out Conditions**. In **Additional Functions**, set the frequency of background measurements and peak-searches.
- 7) Focus the electron and optical images. Set up the instrument for an analysis: make sure the probe scan is OFF, the beam diameter is correct, and the current is appropriate. Choose **Read** in the **EOS Conditions**.
- 8) In **Stage Condition** choose **Position Input** to enter new positions or to modify/confirm the accumulation points (generally, do not use accumulated data for Quantitative Analysis). Click on **Confirm** and then STORE the points on the joystick control pad. Be sure to use the TEST button to check the focus of each point. Edit the comment line and press **Apply** to store all the information for that point. Click on the down arrow in the upper right to move to the next line to enter another point location, and repeat until as many points as desired have been entered. Click on **Close** to close **Position Input**.
- 9) For an analysis of only the position which is currently highlighted in the list, choose **One-By-One**, then **Acquire**. To perform multiple analyses automatically, select all points to be analyzed (blue=ON). **Close** to exit this window and go to **Preset Measurement**.
- 10) If you are using analysis conditions which you expect to use again in the future, use **Condition Store** to save or update them.
- 11) In **Preset Measurement** press **Acquire** to start the analysis.

SECTION 10: Line Analysis

- 1) Click on the **Sample Button**. Choose or create group and sample names for this analysis.
- 2) If you wish to use analysis conditions previously stored, load them from **Condition Load** in the **Measurement** menu. Check that the settings are correct and skip to setting the **EOS Conditions** (step 6).
- 3) If you are setting up to do a qualitative scan, go to **Element Condition** and choose **Element** to select the elements to be analyzed on WDS and/or EDS and those to be calculated. Choose **Condition** to set-up the conditions for each element to be analyzed.
- 5) Focus the beam and light optical image. Set up the instrument for an analysis and choose **Read** in the **EOS Conditions**. Make sure the probe scan is OFF, the beam diameter is correct, and the current is appropriate.
- 6) In **Stage Condition** choose **Position Input** to enter a new line or to modify/confirm the line for analysis. Click on **Confirm** and focus each end of the line. Edit the comment line and press **Apply** to store the information. If you want to do quantitative analysis at each point on a line you have defined, choose **Convert-to-Point-Table** to store your points in the quantitative analysis point table, then proceed as for Quantitative Analysis (Section 9). To define another line, click on the down arrow in the upper right to move to the next blank line in the **Stage Condition** window and repeat until all lines have been entered. Click on **Close** to exit **Position Input**. For a single analysis of the line currently highlighted, choose **One-by-One**, then **Acquire**. To perform multiple analyses automatically, select the desired lines, choose **Close** to exit the Stage Condition window, and go to **Preset Measurement**.
- 7) If you are using analysis conditions which you may want to use again in the future, use **Condition Store** to save or update the analysis conditions.
- 8) In **Preset Measurement** press **Acquire** to start the analysis.
- 9) Choose **Analysis -> Line Real-Time** from the EPMA menu to view the analysis on the screen as it is being done.

SECTION 11: Map Analysis

- 1) Click on the **Sample Button**. Choose or create group and sample names for this analysis.
- 2) If you wish to use analysis conditions previously stored, load them from **Condition Load** in the **Measurement** menu. Check that the settings are correct and skip to setting the **EOS Conditions** (step 4).
- 3) In **Element Condition** choose **Element** to select the elements to be analyzed on WDS and/or EDS and those to be calculated. Choose **Condition** to set-up the conditions for each element to be analyzed.
- 4) Focus the beam and light optical image. Set up the instrument for an analysis and choose **Read** in the **EOS Conditions**. Make sure the probe scan is OFF, the beam diameter is correct, and the current is appropriate.
- 5) In **Stage Condition** choose **Position Input** to enter a new area or to modify/confirm the area for analysis. Click on **Confirm** and focus each corner of the area. Edit the comment line and press **Apply** to store all the information for that area. If you want to do quantitative analysis at each point in an area you have defined, choose **Convert-to-Point-Table** to store your points in the quantitative analysis point table. Be aware of the total number of points you have set before doing quantitative measurements of all of them, then proceed as for Quantitative Analysis (Section 9). Click on the down arrow in the upper right to move to the next line to enter another area, and repeat until as many areas as desired have been entered. Click on **Close** to close **Position Input**. For a single analysis of the area currently highlighted, choose **One-By-One**, then **Acquire**. To perform multiple map analyses automatically, select the maps desired, choose **Close** and go to **Preset Measurement**.
- 6) If you are using analysis conditions which you may wish to use again, choose **Condition Store** to save or update them.
- 7) In **Preset Measurement** press **Acquire** to start the analysis.
- 8) To obtain a calibration factor relating the map colors to composition, go to Analysis -> Standard -> Measurement -> Standard Type in the EPMA menu and choose **Calibration Curve**. Then go to Analysis -> Standard -> Measurement -> Check Data and choose **Element** to display a window listing A and B calibration values for all the elements analyzed on this standard.
- 9) Choose Analysis -> Line Real-Time from the EPMA menu to view the analysis on the screen as it occurs. To apply calibration factors relating map colors to composition instead of absolute counts, choose **Operation -> Map Display -> Level Modify** and enter appropriate A and B values.

SECTION 12: Serial Auto Analysis

- 1) Set up the **individual** analyses you will be doing in the appropriate analysis window: **Quant, Map, Line, etc.**. Save a condition file for each using **Condition Save** in each process.
- 2) Choose **File -> Open Serial Directory** to name your analysis sequence.
- 3) Choose **Input** to create an analysis "schedule". Choose the analysis type and use **Group/Sample** to name the desired analysis routine. **Stage Condition** may be used to modify stage positions or focus or to add new positions.
- 4) **Condition Load** must be used to load appropriate condition files.
- 5) Choose **Check** to review the analysis conditions. If they need to be modified, close **Serial Auto Analysis** and return to the proper analysis menu to make appropriate changes, then return to **Serial Auto Analysis** and re-load the conditions.
- 6) Though it is not recommended that you make adjustments in the Serial Auto Analysis window, it is possible to do so. **Condition Change** may be used to adjust or select standards or EOS or EDS conditions.
- 7) When conditions for this analysis are correct, choose **Apply**. Use the arrows in the upper right corner of the Input window to move to another analysis in the series, or close to exit this window.
- 8) Run a single analysis routine by choosing **One-By-One**, or choose **Preset** to select multiple analysis routines to run.

SECTION 13: EDS "Quick" Qualitative Analysis

- 1) Turn off the OMTV light (the EDS detector is light sensitive).
- 2) Choose EDS -> EDS from the EPMA menu. Wait for EDS to load and place window, then wait for the windows to be drawn (approximately 30 seconds total time).
- 3) To clear an existing spectrum, click the small button with the empty square on it in the EDS command window. A small window will pop up asking if you want to delete the acquired spectrum. Click **OK**.
- 4) Turn PRBSCN off to go to spot mode, or if it must be on, make sure the magnification is high enough so that you are not scanning epoxy or some other material that you wish to avoid.
- 5) Click on **Acq Spectrum**. This is a toggle switch. If EDS is not turned off by clicking again once the identification is made, the system will acquire for a set amount of time. The set analysis time can be adjusted from the EDS command window under **Preset**. If an error message appears saying the bias or voltage is zero, click on **Analyzer** and click bias to **OFF**, then **ON** to reset, and try **Acq Spectrum** again.
- 6) Identify peaks using the Periodic Table function.
- 7) Iconizing the EDS window (rather than closing it) when you have identified the phase in question will save time if EDS is to be used repeatedly, because the window will not have to be re-drawn.

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