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Imaging and Qualitative Spectral Analysis using the Environmental Scanning Electron Microscope

- User Guide for the ESEM Facility at GSC Atlantic-

Frank C. Thomas and Marie-Claude Williamson



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1. Introduction

The ElectroScan E3 Environmental Scanning Electron Microscope (ESEM) housed in GSC Atlantic's MicroAnalysis Facility (MAF) is, in most operational respects, a typical scanning electron microscope (SEM).

SEMs are sophisticated instruments capable of imaging (and in some cases analysing) microscopic samples or microscopic parts of larger samples. Instead of light, an SEM employs a finely controlled beam of electrons. The interaction of the sample and beam can yield a variety of analytical information. Surfaces can be minutely examined and characterized and, with the proper detector, the elemental composition of samples can be determined.

A great deal of literature is available on this subject, but an excellent general reference is Scanning Electron Microscopy and X-Ray Microanalysis 2nd edition by J.L. Goldstein, D.E. Newbury, P. Echlin et al., published by Plenum Press, New York, in 1992. A more concise summary can be found in Principles of Instrumental Analysis 4th edition by D.A. Skoog and J.J. Leary, Saunders College Publishing, New York, 1992.



Figure 1.1 Photograph of the entire ESEM Voyager Unit.

The Electron Probe Microanalyser (EPMA) is a sort of first cousin to an SEM, differing in that it is designed for optimal x-ray microanalytical capabilities, with less emphasis on imaging capabilities.

The "gun" at the top of the column (Fig. 1.1) contains an electron source, in this case a single lanthanum hexaboride (LaB_6) crystal. An ion pump and a diffusion pump, both running constantly, keep the gun area in a high and stable vacuum state – typically around 10^{-6} torr. When a high voltage current is passed through this cathode, large numbers of free electrons are generated. A positively charged ring (anode) placed just below the gun, draws these electrons down into the column in a stream, where electromagnets (condensers) further form this stream into a controlled beam of electrons.

The beam travels through several apertures which further reduce its size, and help to maintain different levels of vacuum in the various parts of the column. In a typical SEM, a similar high vacuum state exists all through the column and in the specimen chamber at its base, which contains the stage and various detectors. In an ESEM, on the other hand, the chamber is kept at a fairly low vacuum state, and is typically operated at about 2 torr.

In a standard SEM, non-metallic samples to be examined must be coated with some substance such as carbon, gold, palladium, or some other metal in order to provide a source of secondary electrons for imaging and to provide electrical conductivity to the stage. In our ESEM, secondary electrons are provided by the ionization of water vapour which makes up the 2 torr partial vapour pressure of the chamber during operation. As a result, even non-metallic samples can be examined with no special preparation.

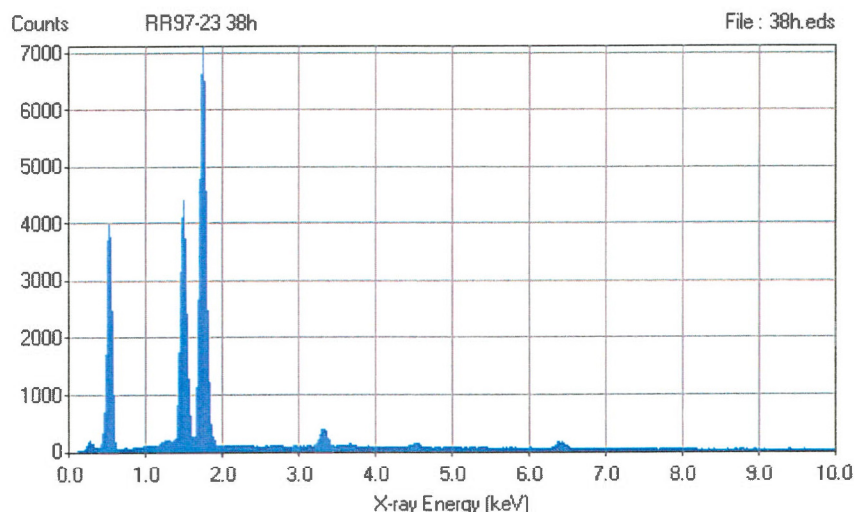
The energy dispersive spectrometer (EDS) attached to our ESEM (Fig 1.1) is a NORAN Voyager Version 3.3 unit. The large tank mounted on the left-hand side of the ESEM's column contains liquid nitrogen to keep the detector itself (which resides on the end of a long steel tube which protrudes through the ESEM's chamber wall) cold and stable. The computer which controls the EDS is a Sun Solaris V UNIX machine which we simply called Voyager. It contains the software necessary to control the spectrum acquisition process, as well as to acquire digital images from the ESEM. Although the software is UNIX-based, the graphic interface is MS Windows-like in appearance and usage.

Energy dispersive spectrometry operates on the principle that when an electron beam interacts with a sample, a certain number of electrons from the beam do not simply produce secondary electrons which can be gathered to create an image. A few will penetrate the sample and strike an atom in such a way as to produce x-ray fluorescence, which then exit the sample and fly off in random directions. These x-rays can then be picked up and counted by an EDS detector, and their energy levels (expressed in electron volts – eV's) can be measured. The theory and practice of EDS analysis is well described in Goldstein et al. (1992), p.341-356.

As part of the beam/sample interaction, a certain number of beam electrons striking the sample bounce back towards the beam (at least in samples where the surface is relatively flat, such as in a thin or polished section). These are termed "backscattered electrons" and can be collected and turned into a useable image in the same way that secondary electrons are, providing a backscatter detector is present, as it is in our ESEM. Backscattered electrons are produced in greater numbers by materials of greater density (higher atomic number), and so can cause such materials to appear "brighter" in the field of view than less dense materials. Goldstein et al (1992) provide a complete discussion of backscattered electrons, their behaviour and uses (p.91-106).

A spectrum is produced by arranging all the “counts” gathered during a specific period of time (e.g. 100 seconds) in graphic form, with increasing eV along the x-axis and numbers of counts on the Y-axis (Fig. 1.2).

Figure 1.2 A typical EDS spectrum with peaks of varying intensities.



Since the eV level of a given particle is dependent upon the atomic species and electron shell from which it was derived, the elemental composition of a sample can be determined from the positions of peaks along the x-axis. The areas of peaks are proportional to elemental concentrations.

The Voyager software can acquire spectra from samples of unknown composition and identify its component elements, and assign “semi-quantitative” atomic percentages to the various elements. These are usually accurate to within a few percentage points. However, with the right sample configuration, beam control, and standards of similar materials but known composition for comparison, full quantitative analysis can be performed.

Another microanalysis technique used in some systems is wavelength dispersive spectrometry (WDS). In these analysers, the x-ray fluorescence from the beam/sample interaction is directed through a crystal (or one in an array of crystals) and the wavelength of the diffracted energy is then measured. An advantage of WDS compared to EDS is the lack of spectral artifacts (spurious peaks) produced: a disadvantage is the greater amount of time required for a single analysis (often 10 times that required for a single EDS run). The accuracy of both systems is more or less comparable, though both are sample-dependent.

Our EDS system can detect all elements except the four lightest - hydrogen (H), helium (He), lithium (Li) and beryllium (Be). The lower detection limits (i.e. the lowest concentration at which a given element can be detected and identified) vary with the element, sample, and length of time used for the acquisition. Concentrations of less than 1% (by atomic weight) can usually be picked up, espe-

cially if acquisition times are longer than normal. The default setting on our system is 100 seconds of live-time acquisition, which is usually sufficient for the kind of reconnaissance analysis most clients need.

The next few chapters are intended to lead the operator through all the steps necessary to achieve sample installation and the acquisition of images and spectra using GSC Atlantic's Microanalysis Facility instrumentation.

2. Sample Holder

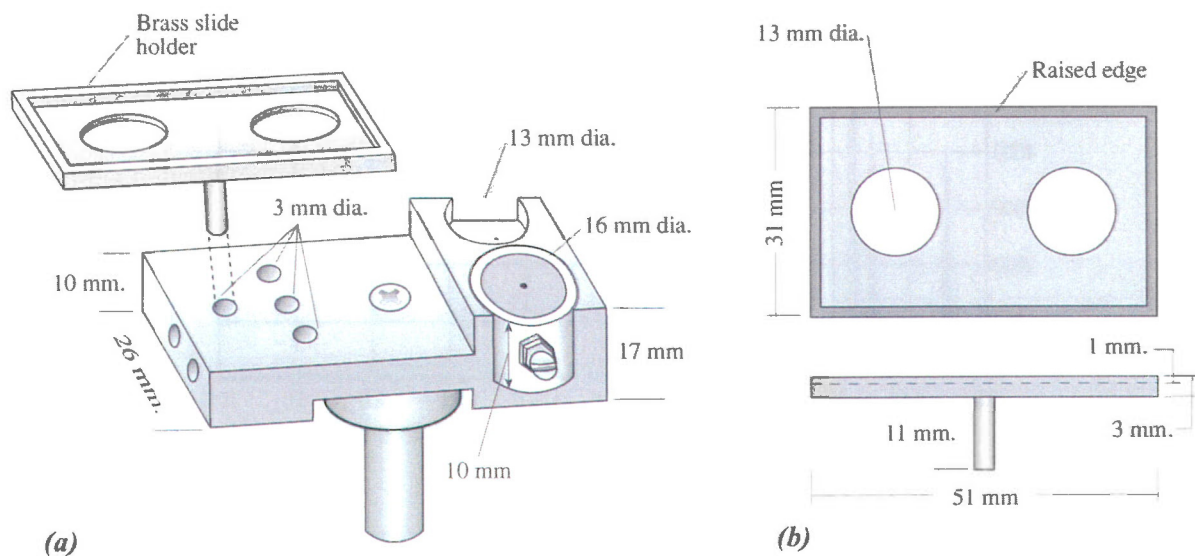


Figure 2.1 (a). Aluminum thin section holder (TSH) showing mounting position of the brass slide holder. (b). Plan and dimensions of the brass slide holder.

To perform quantitative EDS of mineral samples in our ESEM, whether mounted on a standard SEM stub (e.g. grain mounts) or on a thin section, you must use the aluminum thin section holder (TSH). A drawing of the TSH is shown in Figure 2.1.

The mount permanently contains a Faraday cup, and holds a thin section and a 13 mm aluminum stub. The stub usually contains mineral standards for the purpose of quantitative analysis.

- If your sample is mounted on an ESEM stub, place it in one of two positions illustrated on Figure 2.1. Secure with set screws.
- If your sample is a standard glass-mounted thin section, place it on the brass thin section carriage as shown in Figure 2.1.
- Use carbon tape to secure the edges of the thin section to the brass holder.

3. Start Up Procedure

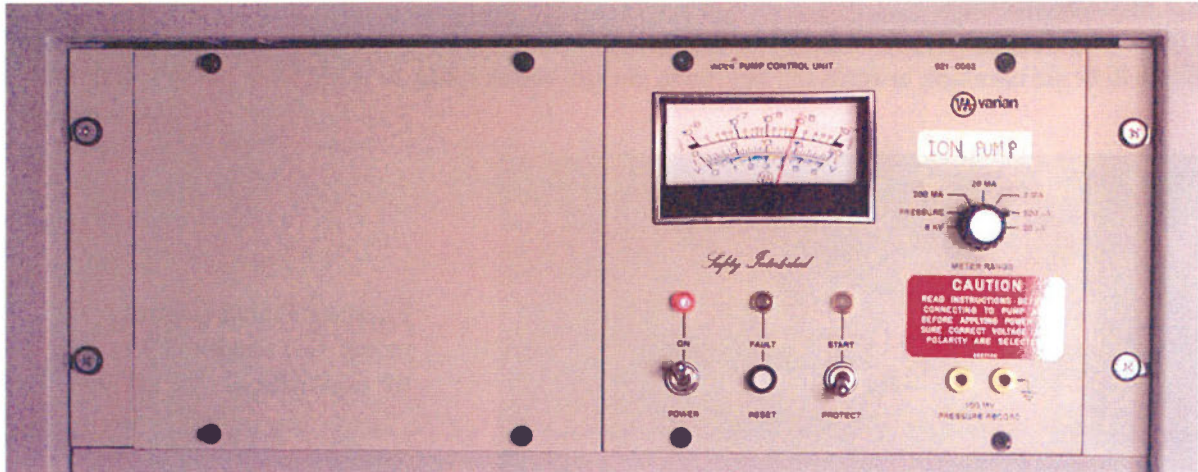


Figure 3.1 Photograph of the ESEM Ion Pump Control Unit.

3.1 Vacuum Check

Under the console, on the left-hand side, check the readings on the pump control unit (Fig. 3.1). The red power light should be on. The knob on the Ion Pump should be set on the 200 mA scale, at the value shown in Figure 3.1. You can also check the vacuum pressure in the gun by switching the knob to the *pressure* setting. The value should read about 10^{-7} torr.

3.2 The ESEM Console

Set the console to **ON** by pressing the green CONSOLE ON button (extreme right of console). When the video displays are on the RH video display should look like Figure 3.2..

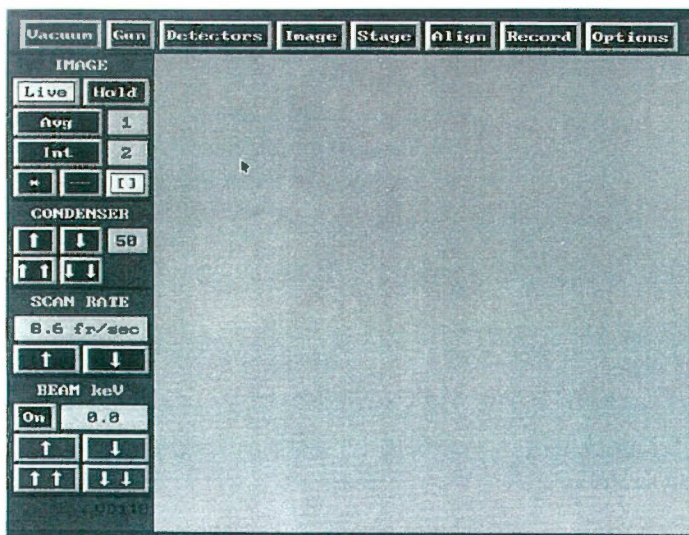


Figure 3.2 Starting menu on the RH video display. See section 3.2 for explanation.

3.3 Heating the Gun

Under the [Gun] menu, select the [Heat Filament] button (Fig. 3.3). The value shown in the Filament box will begin climbing towards the Set Point, usually about 1.88A.

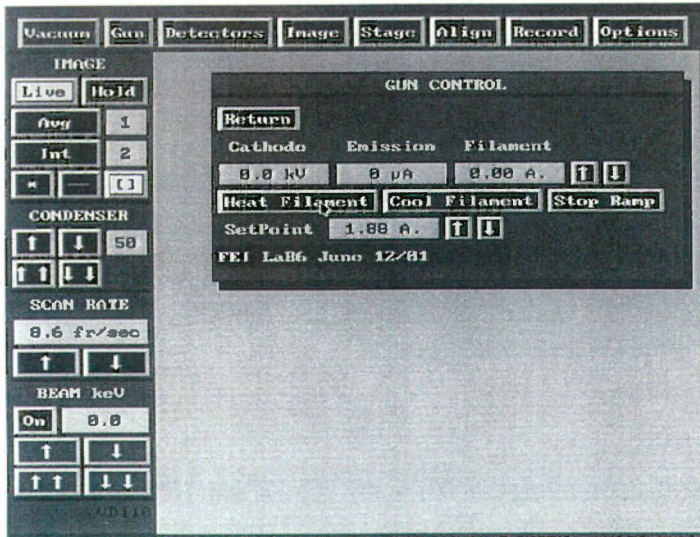


Figure 3.3 Gun Control menu. See section 3.3 for explanation.

3.4 Vacuum Control

This procedure can be done while the filament is heating up. Before you go to the [Vacuum] menu, please check that the EDS detector (mounted on the left side of the ESEM column; Fig. 1.1) is fully

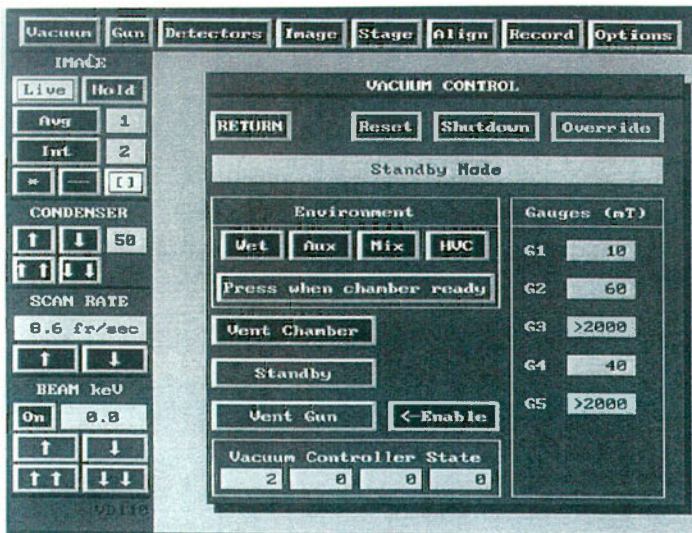


Figure 3.4 Vacuum Control menu; See section 3.4 for explanation.

retracted (pointer on 17 cm). If it is, you can go ahead and press the [Vent Chamber] button (Fig. 3.4). If not, manually retract it to that position using the little wheel on the slider beneath the LN dewar. Wait until the “hissing” sound has stopped before attempting to open the chamber door. Ensure that the door is opened gently, to avoid bumping the stage against the door frame. If necessary to avoid a collision, use the joystick to adjust the position of the stage as you open the door.

3.5 Sample Installation

For simple qualitative analysis and/or imaging of samples, simply insert the pin of the stub into the hole at the top of the stage and tighten the set screw. For quantitative analysis, the TSH must be installed. **Note:** This is a delicate operation usually performed by MAF staff to avoid damage to the ESEM stage. Remove the existing 10 mm stub receptacle. This is the shiny steel cylinder with a central hole in which the post of an SEM stub fits, in the top of the stub carriage (Fig. 3.5 a). This can be done by opening the

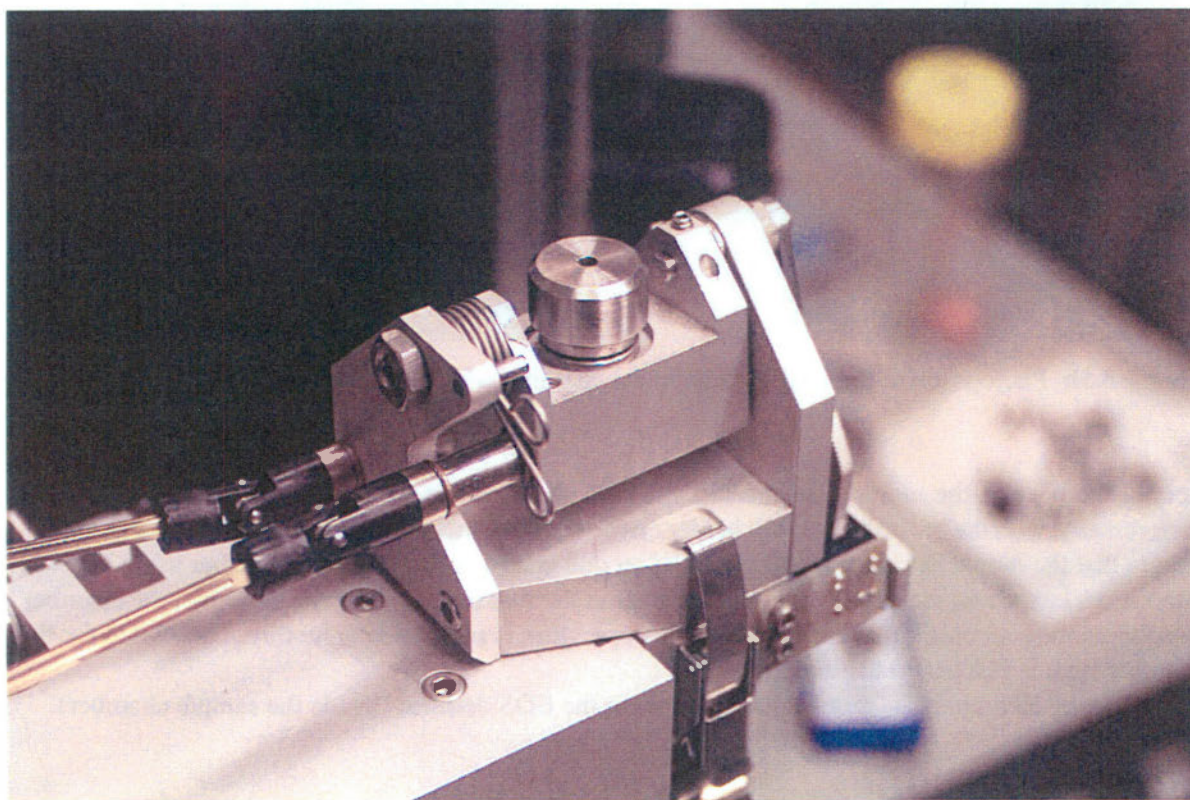


Figure 3.5a Photograph of the ESEM stage in the chamber, without the TSH.

chamber door and tilting the stage all the way to the left. This will expose the retaining screw at the bottom of the stub carriage. If performing EDS analysis, remove the screw with a 9/64 Allen (hex) key. Be careful not to lose the washers and bearing race associated with this screw. Once the screw is removed, the stub receptacle should just pull out from the top - please set aside carefully.

To mount the TSH (Section 2) on the ESEM stage, start by placing a bearing race on the white plastic

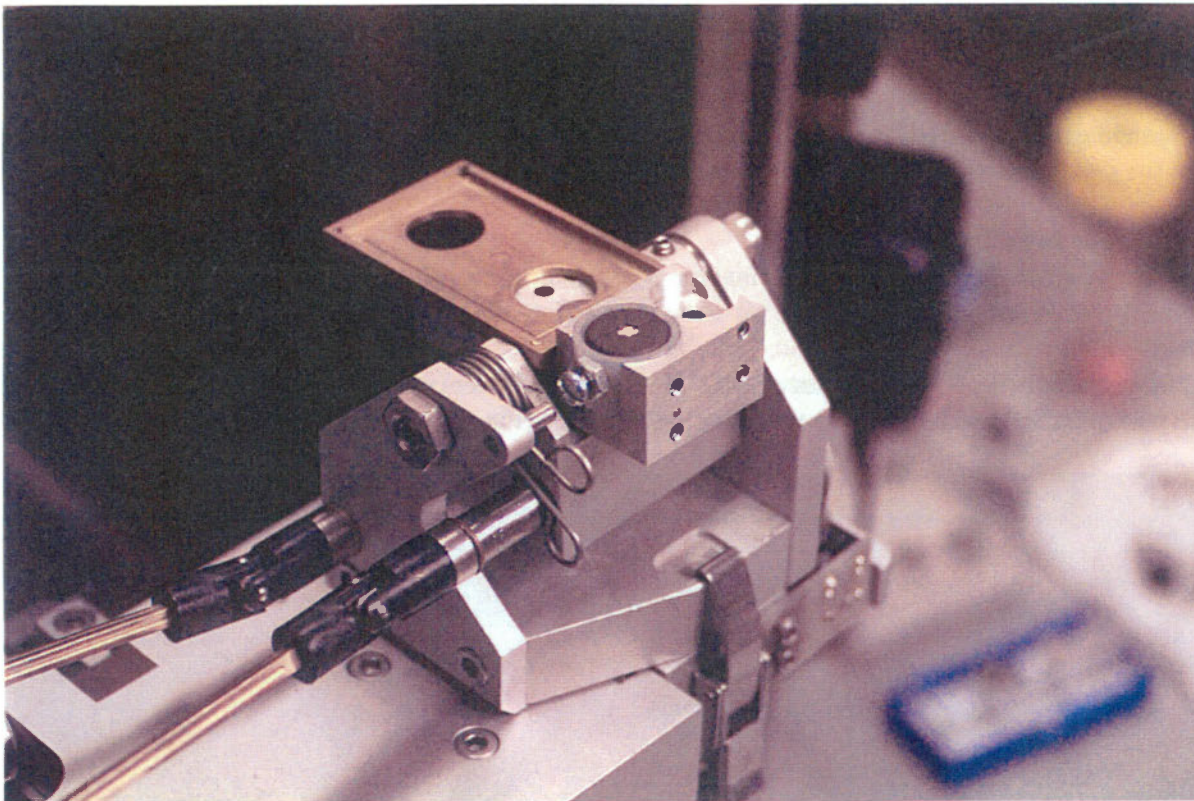


Figure 3.5b Photograph of the ESEM stage in the chamber with the TSH installed.

stem at the base of the mount. Ensure that the race slides all the way up to the top. The TSH fits transversally, with the brass thin section holder on the left (towards the door of the chamber; Fig. 3.5 b). Ensure that the mount fits snugly, and place the second bearing race, screw and washer using the 9/64 (hex) Allen key. Close the door by guiding the stage roller into the groove at the bottom of the chamber. Use the window to check that each stage of this operation is performed smoothly. The switch for the chamber light is just below the window.

If performing EPS analysis, remove the cover from the EDS detector (inside the sample chamber).

3.6 Filament

Check the [Filament] reading (Fig. 3.3). The value should be in the range 1.88 - 1.89A. If necessary, adjust the value with the \uparrow or \downarrow buttons. This should be monitored frequently while the ESEM is being operated. The value has a tendency to climb, oversaturating the filament and shortening its life.

3.7 Vacuum

To reseal and evacuate the chamber after installing the TSH, push lightly on the chamber door. Then go to the [Vacuum] menu and hit the [Wet] button while keeping the door closed with your left hand. You will hear the chamber depressurize. You can then remove your hand from the door, and turn the pressure knob (located at the base of the column) to a reading of 0.9-1.0 (red digital readout). The LH

video display (where the sample image will appear) contains a line of information at the bottom of the screen

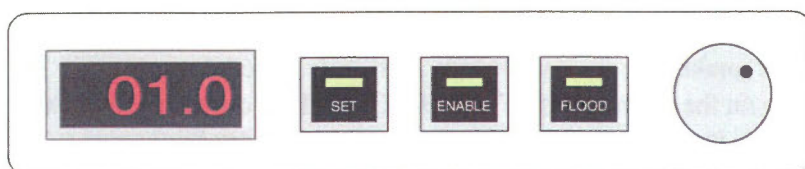
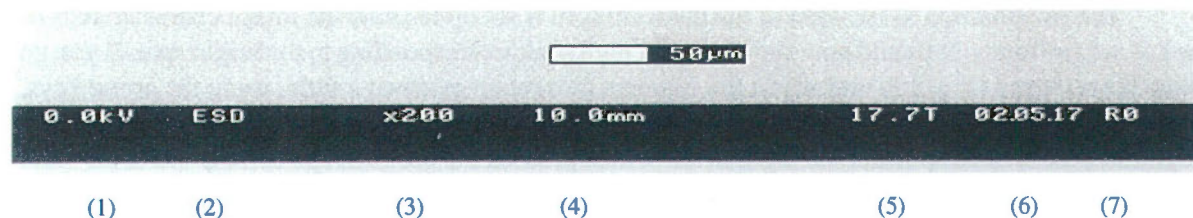


Figure 3.7 Chamber pressure controls.



This is an example of the data that appears at the bottom of the LH video display:

- (1) Accelerating Voltage
- (2) Environmental Secondary Detector
- (3) Magnification
- (4) Working Distance
- (5) Chamber pressure
- (6) Date: yy.mm.dd.
- (7) Record Number

Once the chamber pressure reaches 0.9T, press the flood button on the chamber pressure controls and hold, until the P value indicated on the screen reaches a value of 10T. Then, release the button, and allow the pressure to return to base value. Repeat three times. This procedure clears all the remaining air from the chamber, and replaces it with water vapour (Section 1)

Once this procedure is complete, check the **[Filament]** reading on the **[Gun]** menu (Fig. 3.3). Adjust to 1.88A if necessary.

3.8 Chamber Pressure

Set the pressure used for analysis by turning the knob located at the base of the column (Fig. 3.7) The value can be set at 2.0 to start, and adjusted later according to the nature of the sample or the type of analysis.

3.9 BEAM KeV

Turn on the beam: On the lower left corner of the RH video screen, hit the **ON** button and use the **↑↑** (Fig. 3.2) to set a value of 20.0 KeV (industry standard for EDS analysis). The **↑↑** button steps the KeV up in increments of 1.0, and should not be done too quickly. One step per second is a good rate of speed. If only imaging the sample, consult with MAF staff. A lower KeV is probably sufficient.

3.10 Source

SOURCE mode allows the ESEM user to see the beam in cross-section, and to check that the filament is properly saturated. The ideal saturation level or [SetPoint] of the filament is 1.88A (Fig. 3.3), however, this value may change slightly over time as the filament ages.

In order to perform this check yourself, make sure the MAGNIFICATION is turned down to 100 x, then press the SOURCE button on the main console. Turn BRIGHTNESS down a turn or two, and CONTRAST up a few turns, until the image on the LH video display shows a very bright oval spot. This spot may or may not be centered on the screen. As you turn up the CONTRAST, the spot should gradually become brighter relative to the dark background, indicating a properly saturated filament (i.e. emitting the maximum number of electrons).

The oscilloscope to the right of the main console is set up to show the image characteristics of the LH video display. It should now show a sharp, high peak, corresponding to the bright spot. If you go to the [Gun] menu (Fig. 3.3) and adjust the filament current up or down a little, using the arrow keys, you will note this effect on the bright spot and its corresponding expression on the oscilloscope. The brightest, best defined spot and the highest, narrowest peak on the oscilloscope correspond to the ideal set point.

Once you're confident of the gun saturation, turn SOURCE off and re-adjust CONTRAST and BRIGHTNESS to restore the image on the LH video display

3.11 Stage Calibration

Open the [Stage] menu (Fig. 3.7) and you will notice that the machine defaults to a 13 mm size stub, also adequate for analysing a thin section. Change this setting only if using a larger stub size.

Then, click on [Calibrate], and this will move the mount to a position directly below the beam.

For EDS analysis, it is recommended to raise the stage until a working distance of 19 mm is shown on the LH screen (after calibration). Simple stage movements are accomplished with the joystick mounted on the left side of the console. If x-y is selected on the stage control menu (Fig. 3x), the stage can be moved in any direction side-to-side. If y-z is selected the stage can be raised or lowered. Then, use the

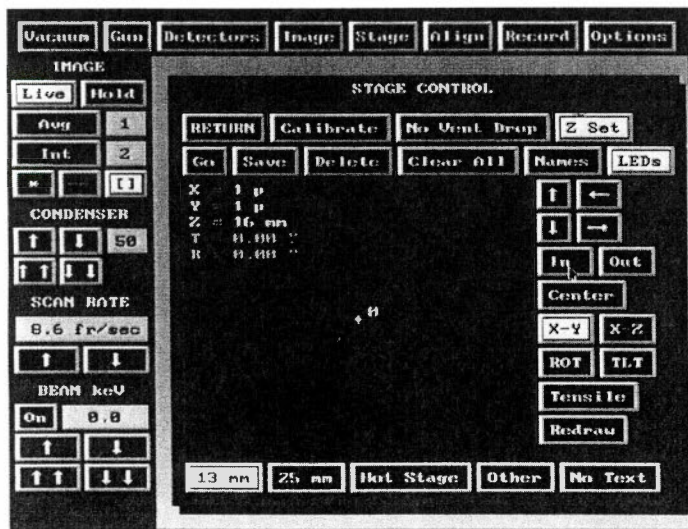


Figure 3.x Stage control menu.

[Focus] knob on the right part of the console to obtain the clearest image possible. Now, press the [X-Z], then the [Z set] on the stage menu. Ignore the warning message in red, and simply press [Z set] again.

Choosing the appropriate working distance (WD) is critical for imaging or EDS analysis. As mentioned above, a WD of at least 19 mm is necessary in order to accommodate the EDS detector. This means that the stage must be lowered nearly or all the way to its limit. For imaging, however, a much shorter WD, such as 11 - 13 mm is better. For the best resolution at higher magnifications ($> \sim 8000\times$), use a WD of 10 - 11 mm. For samples requiring less magnification, especially those with greater topographic relief, a 12 - 14 mm WD will provide better depth of focus, at the expense of resolution. Chamber pressure must be adjusted to accommodate different working distances - very short WD's will require greater pressures ($\sim 2-3$ torr); while longer ones, especially as in EDS work, will require pressures of 1 torr or less. The reason for these adjustments is simple - on its path through the sample chamber, the beam must encounter a given number of water molecules in order to generate an image. A shorter path (WD) through the chamber will need a greater concentration (higher pressure) to meet that number. Even so, the operator will notice a reduction in image quality as the WD lengthens.

If there is a certain location on the sample that you may wish to return to later, for more imaging or for EDS analysis, click the SAVE button on the Stage Control Menu. The location will be assigned a number, starting with 1. Up to 15 locations can be stored in this way.

3.12 Centering the Beam

There are three large silver knobs at the top of the ESEM column (Fig. 1.1) used to centre the beam. Turn each knob while looking at the image on the screen to obtain an image that is as bright as possible. Next, go to the smaller knobs half-way down on the front of the column for fine-tuning. These knobs control the position of the projection aperture(s) and are used in conjunction to maximise the brightness of the beam. This procedure will have to be repeated at intervals (20-30 min.) for the first 2 or 3 hours of operation, less frequently after that.

4. Image Display

4.1 Image Acquisition – Set Up

Voyager is a Unix system so the computer is switched on permanently. To begin, turn on the monitor.

Login: voyager

Password: ***** (for current password, see MAF staff)

The default screen shows the [Spectral Display] window. Close this window.

Right-click on the mouse anywhere on the blue background, and scroll down to [Imaging Tools], and choose [Image Display] from the menu.

Figure 4.1 shows the [Image Display] window (and the last image stored in the system).

Go to [Acquire], [Electron Grey Parameters], [Set Up].

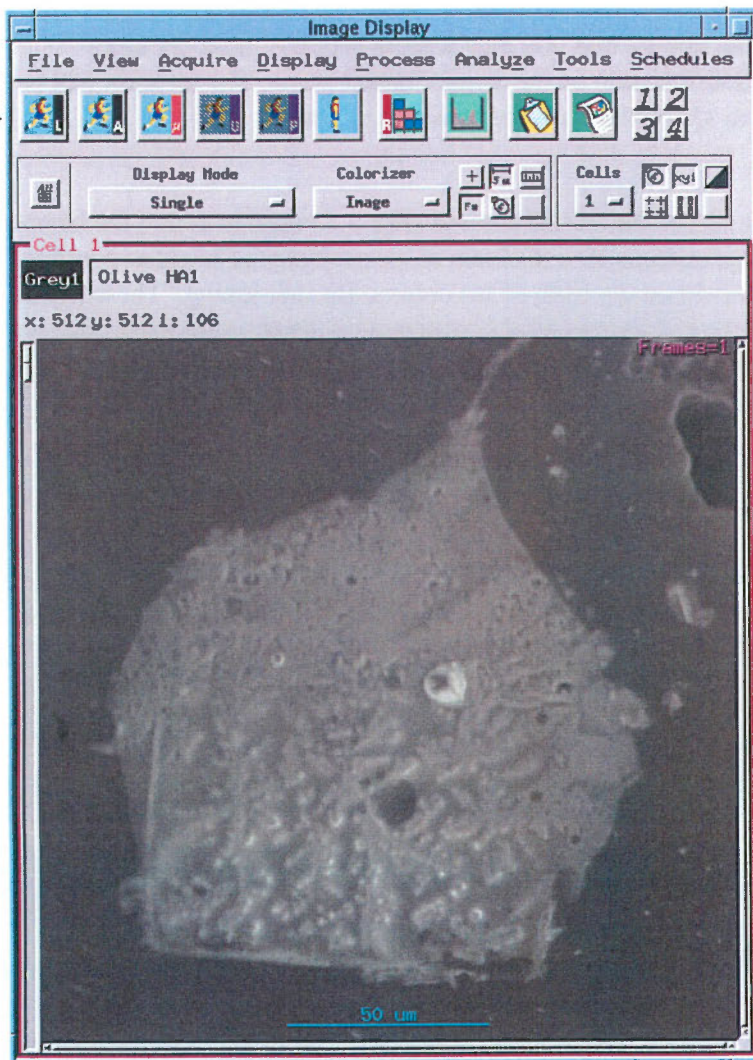


Figure 4.1 View of the Image Display window on Voyager, showing the last image stored.

The Set Up display takes a while to appear but eventually shows two windows: a small “IPA” window, and a “Prompt” window containing information on [Averaged Acquisition Parameters] and [Live Acquisition Parameters].

4.1.1 Averaged Acquisition Parameters

In the upper part of the window, set the [Averaged Acquisition Parameters] as follows:

Dwell Time: is set to 20000 by default.

Resolution: is set to maximum by default.

Number of Frames: default value is [1], we suggest you write in [5].

Averaging Mode: is set to kalman by default.

Weighting Factor: is set to 0.25 by default.

4.1.2 Live Acquisition Parameters

In the lower part of the window, set the [Live Acquisition Parameters] as follows:

Dwell Time: is set to 20000 by default.

Resolution: is set to 256 by default.

Number of Frames: is set at 0.

Please note: As a result, live grey acquisition will run an infinite number of frames until the process is halted by the user.

Click on [OK] to set the parameters. Then [Close] the “IPA” window.

4.1.3 Column Parameters

Go to [Tools], [Column Tool].

a) Pioneer

In the “Pioneer” section, **leave all parameters as is.**

b) Column Information

New information **must be entered in the “Column Information”** section.

Acc. voltage: The kV value can be obtained from either video display on the ESEM.

Magnification: This value is read from the LH video display.

Working distance: This value is read from the LH video display.

Spot size: Optional. Can be entered as condenser setting on the RH video display.

c) Beam Current Information

For the purpose of Image Acquisition, no value is required for the Beam Current.

d) Stage Information

X-Y coordinates can be read directly from the RH video display.
Other values can be entered at the user's discretion.

4.1.4 Other Adjustments

On the ESEM, click [\[Image\]](#) on the RH video display, to obtain the [\[Image Control\]](#) menu.
Go to [\[Ext Scan\]](#) and click. This hands over the control of the scanning process to Voyager.

4.2 Image Acquisition - Run

4.2.1 Step 1

The arrow on Figure 4.1 indicates where command icons are located in Voyager's [\[Image Display\]](#) window.



Click on this icon. This initiates the acquisition of the image displayed on the LH video display of the ESEM console. The image appears in the window. Note that the scale is in micrometers.

Throughout the live acquisition process, greyscale adjustments can be made using CONTRAST and BRIGHTNESS controls on the ESEM console. When fine-tuning of the image is completed,



click on this icon. This ends the [\[Live Acquisition\]](#) process.

4.2.2 Step 2



Click on this icon.

This step initiates the acquisition of 5 frames to produce the [\[Averaged Electron Image\]](#). This type of acquisition is slower because of the higher resolution of the image.

A total of 5 frames may not be necessary, depending on the goals and subject of analysis. In some cases, *the imaging process affects the integrity of the sample*. Consult with MAF staff for details. Also, try to avoid bumping the stage or any part of the column while the acquisition is taking place.



Click on this icon to end the imaging process (the current frame will be completed).

4.3 Image Acquisition - Save and Print procedures

4.3.1 Saving Files

Click on [File], [Save As] in the Image Display window. The image display can be saved in any of three formats: *.grey, *.tiff, or *.ras. We recommend that the image be saved as both *.grey and *.tiff.

4.3.2 Creating *.grey files

The *.grey file is proprietary to NORAN, and cannot be imported into other graphics or image analysis

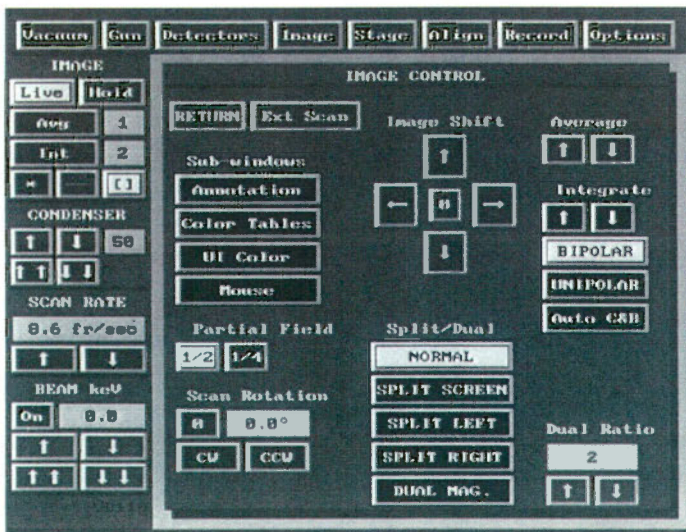


Figure 4.1.4 Image control menu.

applications. However, it does incorporate the Column Information, and allows for notepad comments.

Go to [File], [Save As].

In the window [Save Image As], select a personal Voyager directory, or create a new one. Consult MAF staff for this.

Go to [Selection] and write a filename at: .../dir/filename.

The *.grey extension is added automatically. There is no real limit on the number of characters in a filename, but certain characters such a / or - should be avoided. Use underscore _ instead.

Press [OK] to complete the saving procedure.

4.3.3 Creating *.tiff or *.ras files

Go to [File], [Save As].

In the window [Save Image As], select a personal Voyager directory.

Click on [TIFF] or [RAS].

Go to [Selection] and write a filename at: ../dir/filename. Note that (.tiff) and (.ras) images will not contain a scalebar. See MAF staff for conversion to (.jpg), with a scalebar.

Press [OK] to complete the saving procedure.

When a set of images is completed, MAF staff can copy it to a CD, or FTP them to users.

4.3.4 Creating a Print File

Right-click with the MENU button on the mouse, over a blue area of the screen. [VOYAGER Menu] appears. Scroll down to [Printing], select [VOYAGER Print Tool].

Release the button and go to [Screen Printing Tool].

Printer: [sem5]

Mode: Select [Export-RAS]

Go back to the [Image Display] window click on [File], and [Plot].

Operations are displayed in the [Info-VOYAGER Console] window.

The command line reads: **Exporting to implot.*.ras.**

At each [Plot] command, an *implot.*.ras* file is written to the /tmp/ directory located off the root directory.

****Note ! A maximum of 10 plot files can be created sequentially before system overload. Please ensure that all plot files are removed at the end of a session.**

To move *implot.*.ras* files from the /tmp directory to a personal directory, please follow these instructions:

White box, upper left screen: [Info - VOYAGER Console]:

First go to the root directory. Root can be accessed by successive [cd ..] commands, and is shown as :/.

The tmp directory is accessed by typing: cd tmp.

To copy files to a personal directory, the following command line is used:

: cp implot.*.ras [dir], where [Dir] is the personal directory.

4.3.5 Plotting a Print File

In the [Info-VOYAGER console] directory, type the following command line:

Psprint implot.*.ras, then press Return.

In a few moments the print will appear on the LaserJet 5M printer.

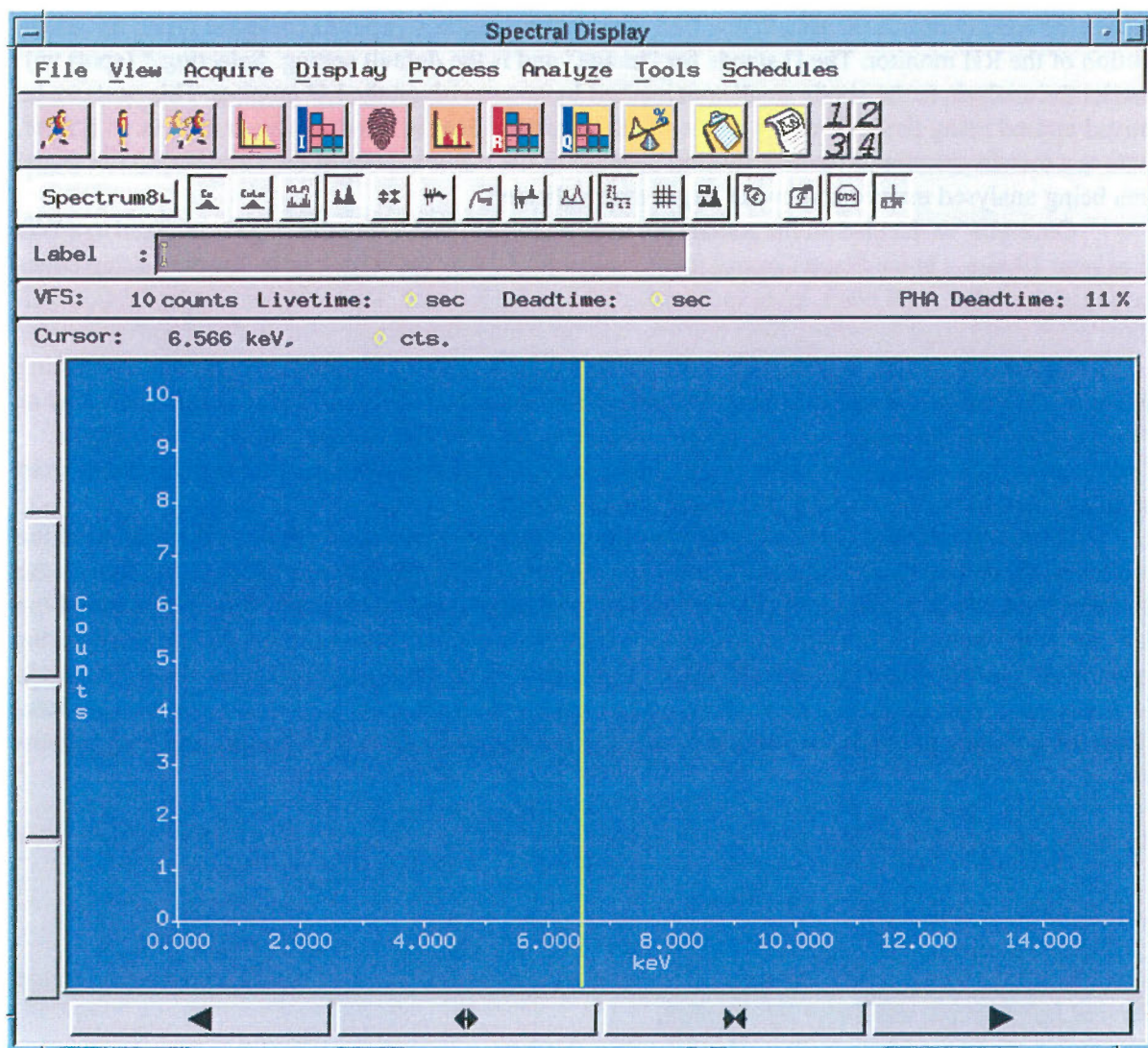


Figure 5.1 Spectral Display window.

5. Qualitative Spectrum Acquisition

5.1 Set Up

An explanation of EDS spectra and examples of their use in natural sciences are presented in Section 1. The following section explains the protocol for acquiring spectra using samples mounted on stubs, thin sections, or glass plates. Figure 5.1 shows the Spectral Display window on Voyager.

Find the spot on the thin section sample or stub that you wish to analyse. Basically, whatever view you have in the LH monitor is the area that will be analysed as long as you have [] selected under the Image section of the RH monitor. The [] stands for “image” and is the default setting. Selecting * (spot) will restrict the analysis to the single small spot marked by an asterisk on the LH monitor. This spot can be moved around using the X Y offset knobs on the console. Bear in mind, however, that in an ESEM, there is a certain amount of beam scattering because of the water vapour in the chamber, so the actual area being analysed may be somewhat larger than indicated.

Once you’ve decided on the actual spot to be analysed, make sure the stage is lowered to a WD of at least 19 mm. On the Stage Control menu, select X-Z. Now the stage can be lowered using either the joystick or the small black lever located just above the joystick. Reducing the magnification will allow more rapid stage lowering. Note that you will have to reduce the pressure in the chamber in order to retain a useable image. If you wish, once you have the sample at the proper height and everything else is ready, you may turn the chamber pressure down towards 0. This will reduce beam scattering as much as possible; the image will darken and disappear, but that will have no effect on the analysis. Realistically, for a variety of reasons, the maximum magnification at which a reliable EDS analysis can be undertaken on this instrument is probably about 15000x.

Manually roll the EDS detector into position, using the small hand crank on the side, until the pointer on the detector slide indicates 5 cm. Then open the specimen chamber window, and turn on the chamber light just below it. (At this point Voyager will begin beeping because of the saturation of the detector with photons - this will stop when the light goes out.) Continue slowly rolling the detector towards the sample while watching through the window, until the pointer indicates about 3.5 cm. If your sample is very large, you may not be able to get the detector much closer. **Do not, at any time, allow the detector to touch the sample or any part of the stage - it is very fragile and very expensive.**

Go to [Spectral Display], [Tools].

5.1.1 Column Parameters

Select [Column Tool]. This command opens up a window with *Pioneer, Column Information, Beam Current Information, and Stage Information.*

Ensure that the Stage Menu is up on the RH video display.

(a) Pioneer

The data already visible in the Pioneer Tool include the *Take-off angle, Solid angle, Default Slide Position, and Window Material.*

Read off the slide position on the scale located below the Liquid Nitrogen dewar, and write in as the new *Slide Position*. Other parameters are automatically recalculated.

(b) Column Information

All new information must be entered.

Acc. voltage: is the keV read from the RH video display.

Magnification: is read from the LH video display.

Working distance: is read from the LH video display.

Spot size: is optional. This value can be read from the [Condenser] setting on RH monitor.

(c) Beam Current Information

For the purpose of qualitative spectrum acquisition, **no value is required**.

(d) Stage Information

The X-Y coordinates, if required, can be read directly from the RH video display.

5.1.2 Deadtime

Go to the Spectral Display window. PHA Deadtime is near the upper right corner, and is shown as a percentage value.

Deadtime should be in the range **20-35%**. It represents the amount of time that the Pulse Height Analyser (PHA) is too busy to process data. A range of 20-35% is optimal - it means that lots of signal is coming in, but not too much is wasted. If it's higher than that, acquisitions will take longer, since deadtime is subtracted from total acquisition time before livetime is counted. If deadtime is too low, insufficient signal is coming directly from the sample spot, and results may be suspect. Deadtime can be adjusted in several ways. It can be decreased by backing the detector away from the sample or by turning up the condenser to a higher value. Trying to analyse locations on the right side of "hills" or in deep "valleys" on samples with high topographic relief will also result in deadtimes too low for good analyses.

5.1.3 Acquisition Livetime

The default Acquisition Livetime is 100 seconds, a value that is adequate for most applications. The value is indicated on the same line of the *Spectral Display* window as the *Deadtime*. Modify at [Acquire], [Parameters], [Set Up]. If, for instance, you may be particularly interested in an element which may only be present in very small amounts, a longer acquisition time (200 - 600 seconds) may be more appropriate. Consult with MAF staff.

5.2 Spectrum Acquisition

Once you have the deadtime in the optimal range, and have entered the necessary information (KeV, Magnification, Slide Position, Working Distance) in the Column Tool window, you may begin to acquire a spectrum.



Go to the menu in the Spectral Display window and click on this icon to start the acquisition process.



Once the spectral acquisition is complete, (you will hear a beep) click on this icon. This initiates Voyager's peak identification software.

A small Results window appears Fig. 5.3, and the peaks are labeled on the spectral display.

Note: Elemental peaks can be misidentified by Voyager because of peak overlap for two or more elements; "Escape" peaks; etc. The results obtained using the automatic peak identification software must be confirmed using additional information (tables, etc). See MAF staff for details.

The complete procedure for fully quantitative analysis of materials, using standards of known composition, will be described in a subsequent publication. The techniques outlined in this section will produce a spectrum in which the elemental composition of the sample is revealed, and rough estimates can be made of relative amounts of its constituent elements. This is often sufficient for the sort of "reconnaissance" survey most often performed on this equipment.

5.3 Save, Plot and Print Procedures

5.3.1 Saving Files

The spectral display information is saved either as an *.eds file or *.emsa file.

a) Saving the spectrum as an *.eds file (VOYAGER)

Go to [File], [Save As] in the Spectral Display window.
Select a *Directory* in the LH pane of 'Save Spectrum As' window.

Go to *Selection* and write a *filename* at: **.../dir/filename.**
The *.eds extension is added automatically.

Press [OK] to complete the saving procedure.

b) Saving the spectrum as an *.emsa file (EMSA)

Go to [File], [Save As].
Select [EMSA]. Re-enter the filename as above: **.../dir/filename.**

Press [OK] to complete the saving procedure.

5.3.2 Plotting and Printing Files



The spectrum and results are printed using distinct procedures.

a) Plotting and Printing the Spectral Display



Click with the MENU button on the mouse, over a blue area of the desktop.

Printer: sem5

Mode: Export-RAS

Click on this icon.

Operations are displayed in the window. The command line to /tmp/splot1.ras is explained below:

When using this icon, a *.*.ras file is written to the /tmp/ directory located off root.
A total of 5 plot files can be created sequentially before splot.1.ras is overwritten.

To move plot files from the tmp directory to a personal directory:

First go to the ROOT directory. Root can be accessed by successive [cd ..] commands, and is shown as :/.

The tmp directory is accessed by typing: cd tmp.

To copy files to a personal directory, the following command line is used:

: cp splot.*.ras [dir]).

Go to the VOYAGER Console. Type the following command line in the /usr/home/voyager directory:

Psprint splot.*.ras [Return]

b) Plotting and Printing the Results Window

Expand the window **to view all the Results.**

Date, Time, Livetime, Technique and List of Elements are listed.

Go back to the Screen Printing Tool and click on [Window]. The cursor becomes cross-hairs at this point. Move the cursor back to the Results window.

Click with the ADJUST button on the mouse.

In the Info – VOYAGER Console window, you will read:

Exporting to /tmp/ptool.*.ras.

The same rules apply to the number of files created, as for the splot.*.ras files.

UNIX protocol for copying and saving these files is also identical.

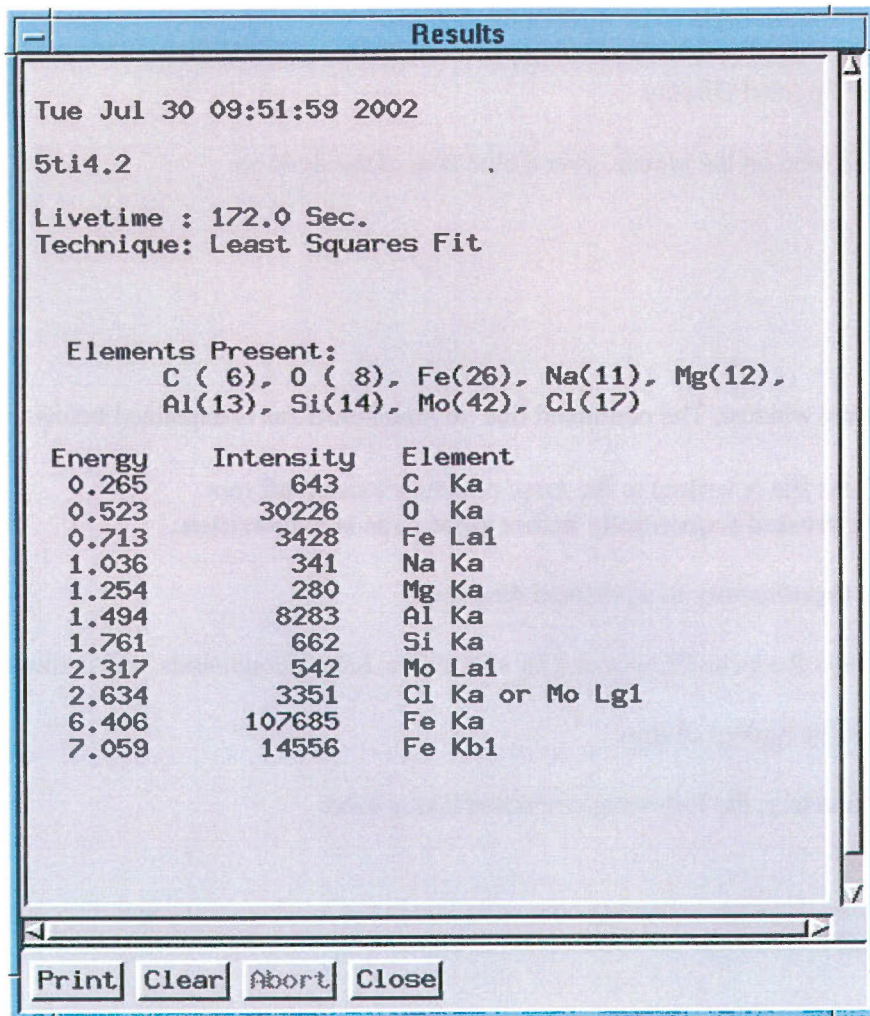


Figure 5.3 Print file from the Voyager.

In the VOYAGER directory, type the following command line:
Psprint ptool.*.ras [Return]

Figure 5.3 shows an example of the Voyager print file.

6. Shut Down Procedure

6.1 [ESEM Shut Down Protocol](#)

1. Reduce CONTRAST until the LH video display is black.
2. Reduce KeV to 0 by clicking on the $\downarrow\downarrow$ button.
3. On the [Gun Control Menu], click on [Cool Filament].
4. Set pressure to 1.0 torr on the PRESSURE CONTROL panel on the column base.
5. If the EDS detector has been in use, make sure it is fully retracted (pointer on 17 cm) before going any further.
6. On the [Vacuum Control] menu, click on [Vent Chamber].
7. When hissing has ceased and the chamber is fully vented, carefully open the chamber door and remove the sample stub.
8. While holding the door closed with the left hand, click [WET] on the [Vacuum Control] menu.
9. When the chamber has pumped down to 1.0 torr, click [STANDBY].
10. On the [Gun Control] menu, check that the filament has fully cooled to 0.0 mA.
This may take a few minutes.
11. Once the filament is fully cooled, you can hit the red CONSOLE OFF button.
12. Fill in log book.

6.2 [VOYAGER Shut Down Protocol](#)

Anywhere on the blue background, click right and select EXIT - Exit Session. (do not power down VOYAGER).

When the screen shows the white Voyager opening display, turn off the monitor. (Voyager, being a UNIX machine, normally stays ON all the time.)