

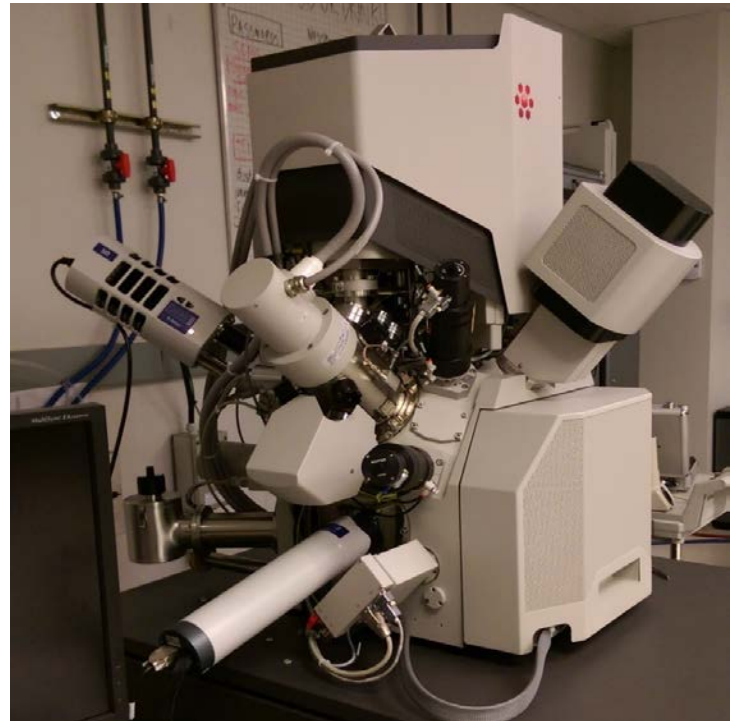
Oxford Instruments Energy-Dispersive Spectroscopy (EDS)

Operating Manual

This document is intended to describe the operation of the Oxford EDS system and its capabilities. In order to gain access on the tool, a formal qualification by staff is required. FIB Scios and the EDS system have two separate qualifications, so training for the FIB Scios does **not** qualify members to use the EDS system. Please contact: ravicha@ucdavis.edu for further questions.

Description

The Scios is a combination of a Scanning Electron Microscope (SEM) and a Focused Ion Beam (FIB) system. The integration of both systems yields a powerful analytical tool for obtaining any data from any sample in three dimensions. Energy Dispersive Spectroscopy (EDS) Analysis provides qualitative elemental (Be to Cf) and chemical microanalysis. The Scios is equipped with the X-MaxN 50mm² EDS system to measure sub-surface (0.3-3 μ m) with a detection limit of \sim 1 atom%. Schematic of system as it relates to the components of the Scios are pictured in Figure 1.



Always

- Make sure that the microscope is enabled through your Badger account
- Wear clean gloves or use tweezers when handling samples inside the vacuum chamber
- **Check vac states** before and after starting your session
- Report problems immediately to staff to ensure speedy repair

Never

- Wear headphones, listen to music, or use your cell phone when using the microscope
- Upload/install or insert USB devices into control PC
- Use the plasma cleaner with the detectors inserted into the chamber
- Use the microscope for samples that will generate particles, or have poor vacuum compatibility. If in doubt, please check with staff.
 - Wet photoresist, loose powders/flakes, liquids, biological samples, etc

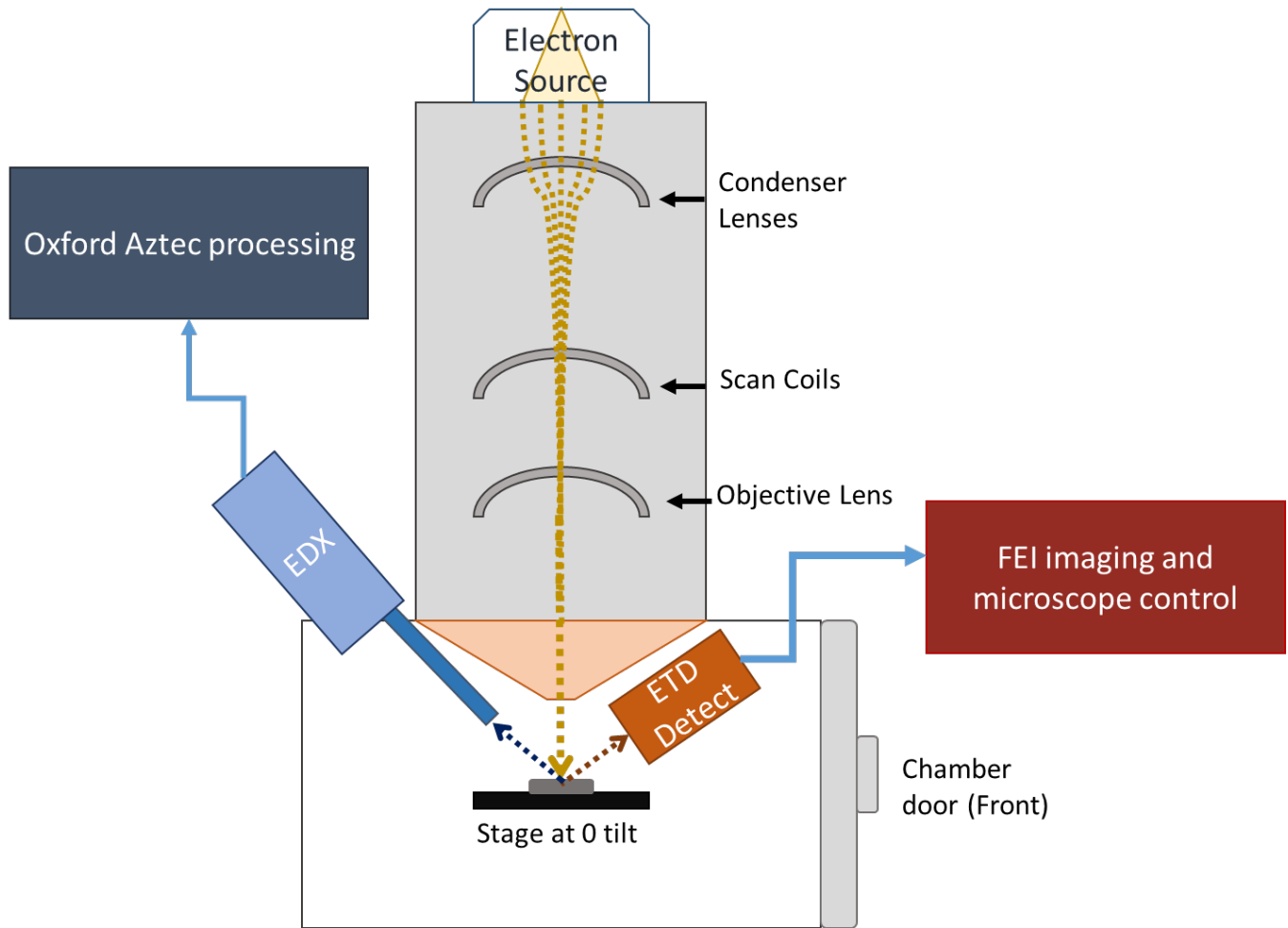
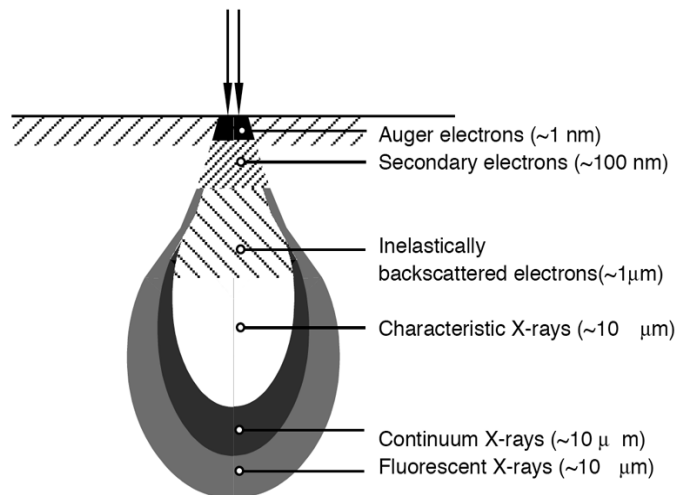


Fig 1. Cross section of the Scios, and a mapping of detector locations relative to the chamber door and pole piece. Electrons (FEG source) travel through various imaging coils and hit the sample. This sample is placed at a 0-degree tilt and at a eucentric height (or WD) of 7-10mm from the pole piece. As the electron beam scans, the scattered electrons from the sample's surface create an image that is collected by the ETD detector, and also simultaneously releases characteristic x-rays (see image below) that are collected by the EDX. The ETD image is processed by the FEI software, and the EDX data is processed by the Aztec software from Oxford. Additionally, the EDX is controlled by the Oxford Aztec software, and the imaging conditions are set by the microscope control software.

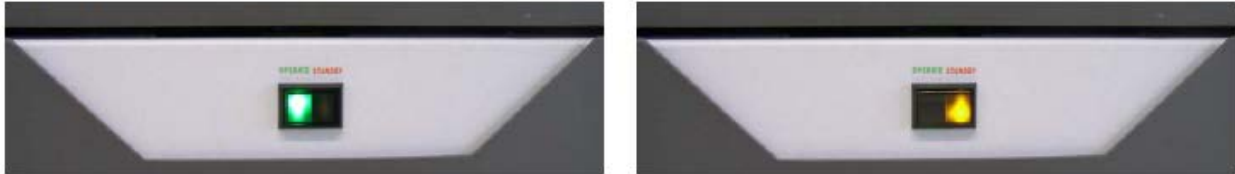


1. Microscope and Measurement Set up

System Checks

1.1 Is the machine on?

FIGURE 2-2 Control Panel Power Button



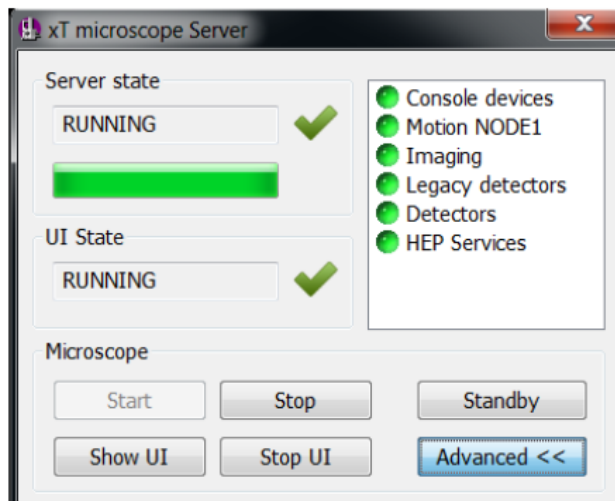
Green: Operation: Normal

Amber: Standby: Abnormal

****DO NOT USE THE TOOL IF THE LIGHT IS YELLOW****

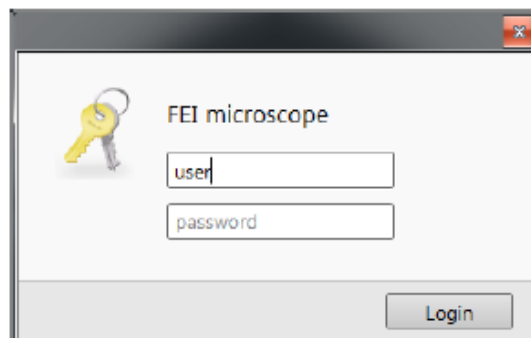
1.2 Enable FIB on Badger, this will turn on the computer monitor for the system.

- The computer should be logged into the Supervisor account (login information is posted)
- Microscope UI should be running, if not then click on the shortcut “XT Microscope Server”
- When starting the server, all the status bubbles should turn green on start-up



The Microscope XT UI will automatically start from this point, then prompting a login screen.

1.3 Login to microscope server with your username and password (created during training)



1.4 Open **sample exchange menu** and check status of all vacuum segments. They should all be bright green as shown below (Fig. 2). If any boxes look orange or grey colored, then this indicates a vacuum failure in the system and it should be reported immediately on Badger. Check the following vacuum values to ensure that the system is in good condition:

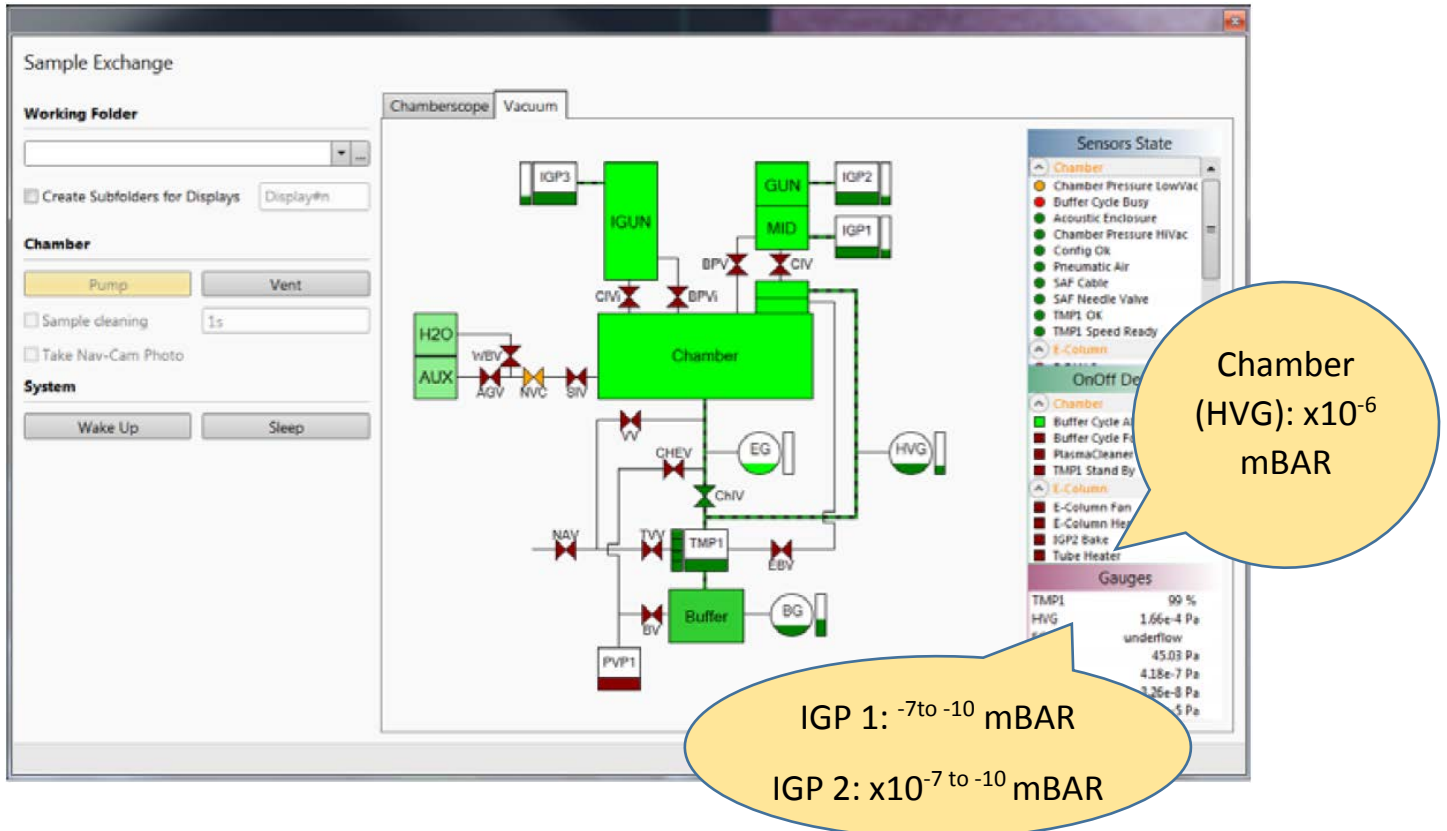


Fig. 2. It is important to make sure the vacuum segments are in spec and to check for potential leaks.

1.5 Check the emission currents, and note these values in the log book. If you see major changes, be sure to notify staff. The ion beam emission current tells you whether the ion source is heated or cooled. When in the nano-amps range the ion source is cooled and when in the ~ 2.0 micro-amp range the ion source is heated. Make sure to put the system to sleep if you are the last person to use the microscope.

1.6 Check **application status** by double clicking on the icon on the bottom right hand side of the screen. This window displays any alerts on the system. Take note of the messages, and be sure to check with staff before proceeding to use the machine. Here are examples of possible errors:

The screenshot shows the 'Application Status' window with the following data:

Time	Description	Source
Today - Thu, Mar 30		
10:35:19	The GIS W dep temperature has reached the set point 53.92 °C	GIS Patterning
10:35:10	The GIS W dep temperature has reached the set point 53.85 °C	GIS Patterning
10:35:03	The GIS C dep temperature has reached the set point 29.87 °C	GIS Patterning
10:30:58	FIB Lens 2 interlock failure.	FIB Lens
10:30:53	The GIS 'W dep' heater is switched off due to poor vacuum.	GIS Patterning
10:30:53	The GIS 'C dep' heater is switched off due to poor vacuum.	GIS Patterning
10:19:10	The FIB source is not operational.	FIB Source
09:31:11	The FIB source is working properly.	FIB Source
09:26:48	The FIB source is in the acquiring state.	FIB Source
09:24:50	The GIS W dep temperature has reached the set point 53.82 °C	GIS Patterning
09:24:37	The GIS W dep temperature has reached the set point 53.19 °C	GIS Patterning
09:24:30	The GIS C dep temperature has reached the set point 29.87 °C	GIS Patterning

At the bottom of the window, there is a dropdown menu set to 'Error' and three icons: a red 'X', a yellow warning triangle, and a blue information 'i'. 'Clear' and 'Hide' buttons are also present.

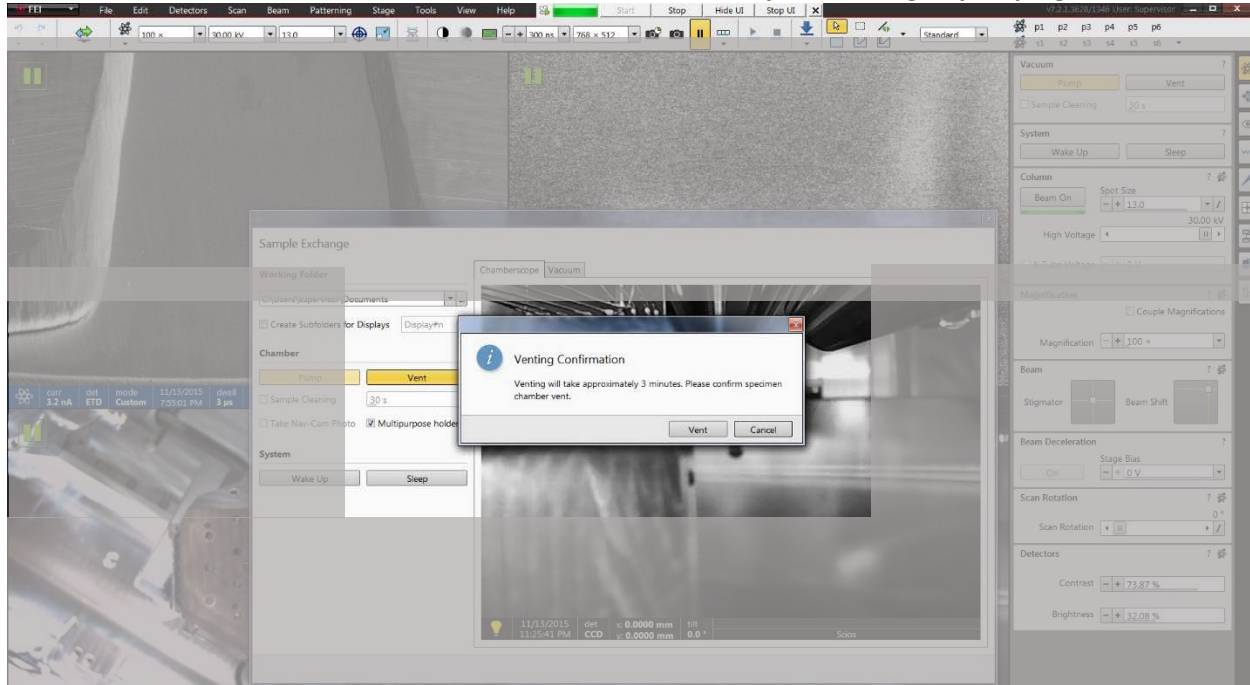
A red callout bubble points to the blue information 'i' icon in the bottom right corner of the main interface, with the text: "Application Status Button (double click here)".

The bottom status bar shows: 0 pA | Emission Current: 126.94 μA | 42.72 nA | Electron Source Pressure: 2.16e-9 mbar | Nav-Cam | 3/30/2017 11:29:31 AM | det CCD | x: 0.0000 mm | tilt 0.0 ° | y: 0.0000 mm | Scios

Sample Loading

1.6 Press **Vent**. A dialogue box will appear to confirm the “vent” operation. It takes approximately 3 mins for the chamber to vent completely.

****Do not insert or retract the EDS detector while the microscope is venting or pumping****



1.7 Once the chamber has completed its venting cycle. Pull the chamber door open, and while using stub forceps load your sample into one of the holes in the stage. Make sure to use the flat 0-degree holes of the stage.

1.8 While watching the CCD camera, close the chamber door. **Make sure there is enough clearance between your sample and the pole piece!**

1.9 Press “pump” on the vacuum module, with one hand on the chamber door. The chamber has to be under high vacuum ($< 10^{-4}$ mbar) in order to turn on the beam and start imaging.

1.10 While waiting for the chamber to pump down you can:

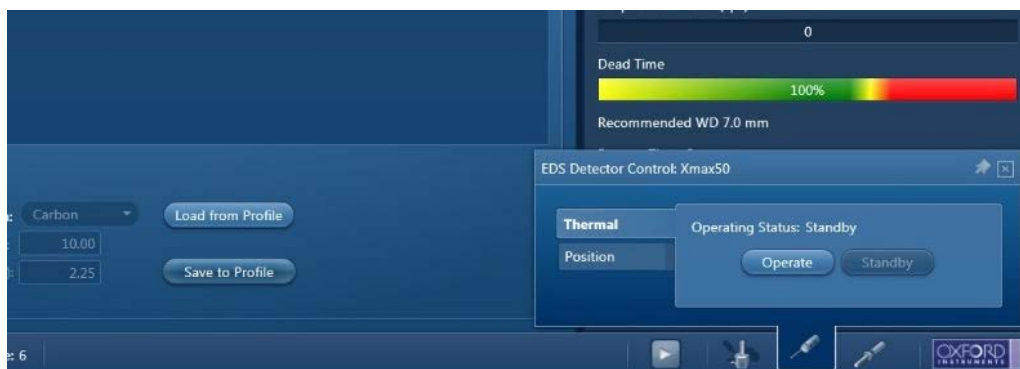
- Move the stage up to the 7mm marker (7-10mm range is preferred for EDS)
- Take a nav-cam photo after you have raised the stage to the 7mm marker
- Set your imaging parameters for the SEM (acc volt, beam current, resolution, etc)
- Make sure the detector settings are appropriate for E-beam
- Make sure post-processing parameters have not been adjusted (digital brightness (1), digital contrast (0), and gamma(1))
- Move to the Aztec software and begin setting up the project folder

1.11 AZTEC: You will be prompted to either open an existing project or to create a new one. This project file is where all of your data and reports will be saved, which is a local file. In order to copy files from this computer, you will need to move the files first to the shared data folder.

After selecting a project folder local file pathway, the main screen will be available to set up your data acquisition. Project file will automatically your progress and data.

1.12 Once the chamber has finished pumping down, you can begin setting up the EDS detector by using the controls found at the bottom of the screen.

- a. Insert EDS detector into chamber
- b. Switch EDS from “standby” into “operating” mode
- c. Wait until the blue flashing light on the detector goes a solid blue light—detector is ready



1.13 Switch back to the Microscope controls and begin imaging by pressing the **HV button** to turn on the beam. Once the sample is in place under the beam, pause the CCD camera because it will interfere with the EDS acquisition.

1.14 Select Beam parameters based on the energy chart (**APPENDIX A**) and the Dead Time:

- a. Select an accelerating voltage that is 2-2.5x the critical excitation energy to generate x-rays
- b. The apertures are selected automatically based on spot size
- c. The spot size of the beam should be adjusted so that the Dead Time (on the Aztec software) is between 30-70%. Note: To view the Dead Time make sure the Ratemeter is set on the Mini view

1.15 Focus, stigmatize, and align the beam to 2-3x the desired magnification/feature size on the sample surface. Then move back to the desired magnification and switch over to the Aztec software. General overview of AZtec Software is shown in Figure 3.

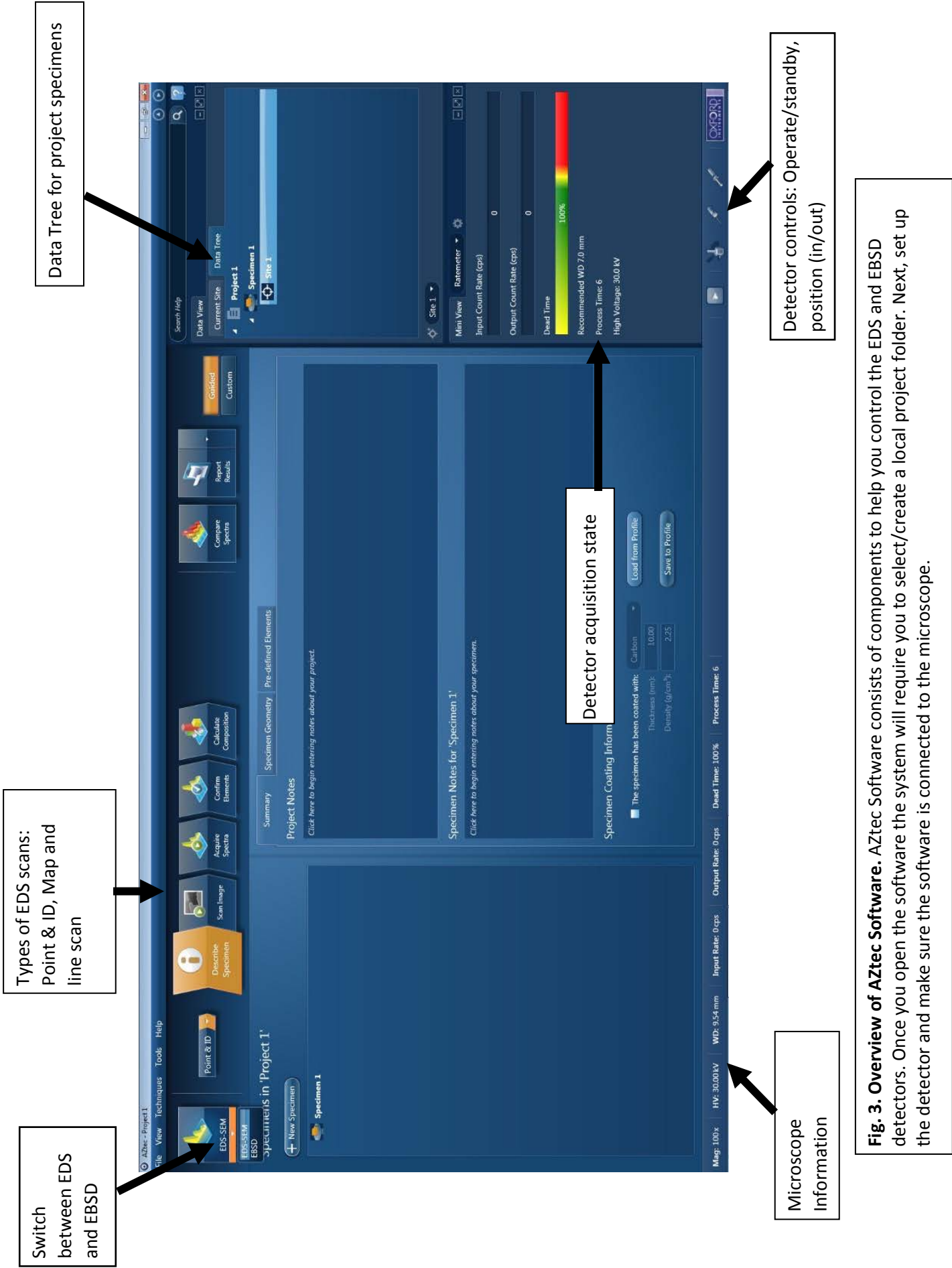
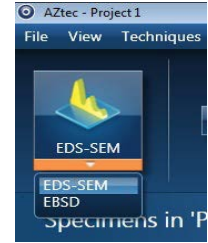


Fig. 3. Overview of AZtec Software. AZtec Software consists of components to help you control the EDS and EBSD detectors. Once you open the software the system will require you to select/create a local project folder. Next, set up the detector and make sure the software is connected to the microscope.

1.16. Make sure the software is set to EDS-SEM instead of EBSD.

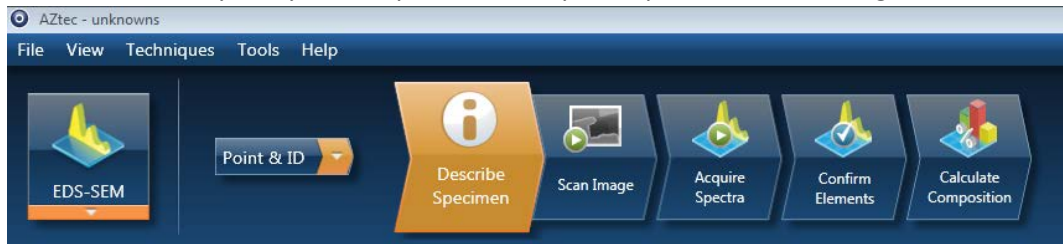


1.17. From the EDS menu select 1 of the 3 different scan options:

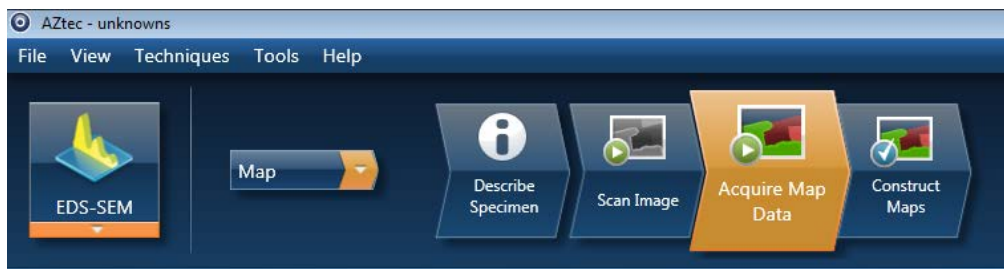
**Depending on which scan option you choose, it will affect the subsequent steps available. These options will be discussed in more depth in the following sections. To switch between scan options use the drop down menu on the front page.



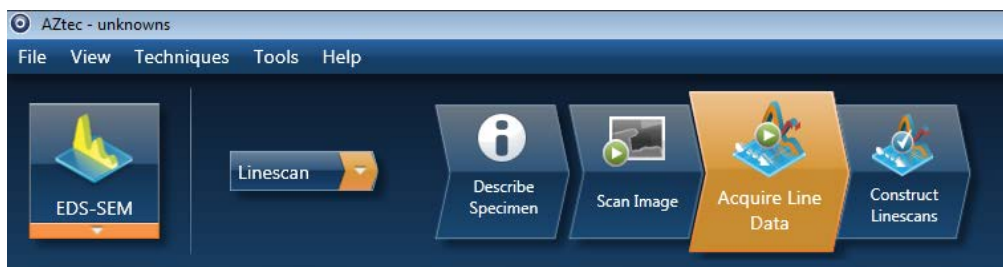
a. Point & ID: Quickly analyzes composition at a specific point or across a region of interest.



b. Map: Marks the location of elements across a region of interest by overlaying different colors. If there is intense or brighter the color at a specific location, than higher concentration of that element is present.

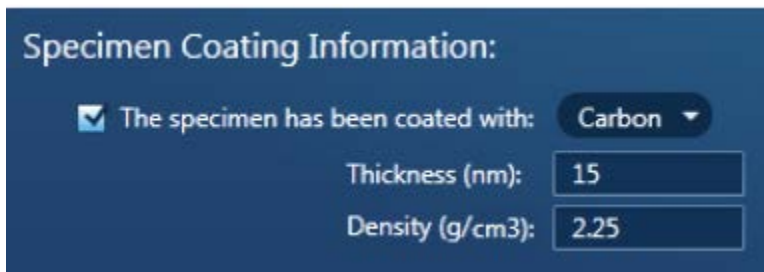
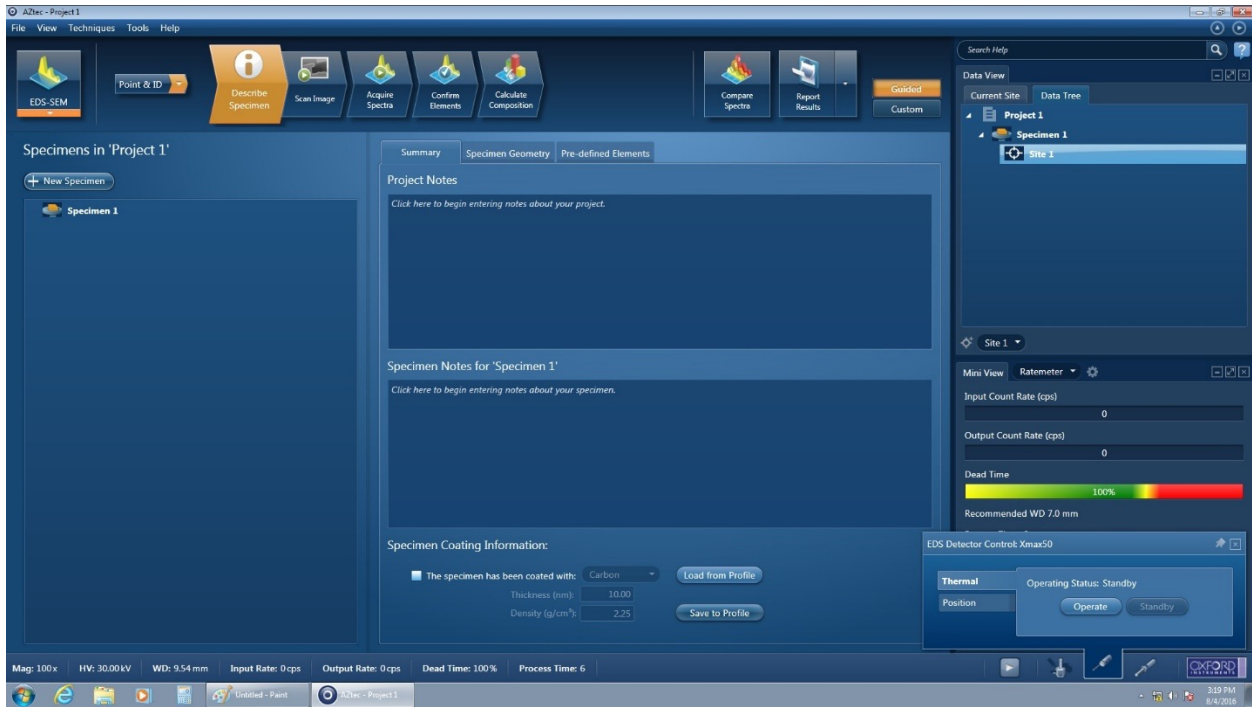


c. Linescan: Determines the concentration of elements across a user-defined line



1.18. After you choose an option, on the first page you will be asked to describe the specimen. You can write notes about the sample and the parameters used which will be included in any saved reports. Make sure you also include any information about a specimen coating (ex. Carbon). Spectral data from the sample coating will automatically be screened out by the software.

See APPENDIX B: WHY ARE SPECIMENS COATED? For more information about what materials to use and their effects on microanalysis.



In the second tab for specimen geometry make sure “pre-tilted specimen holder” is not selected.



Now go to the last tab for Pre-defined Elements and choose elements of interest. You can also choose “Perform Auto ID” to, compare all spectra peaks to an internal library, and have the software propose fitted solutions.

The screenshot shows the 'Pre-defined Elements' tab in the AZtec software. It features a periodic table of elements. Three callout boxes provide instructions: 1) 'Predefine your elements from the Periodic table. Click on an element first to select it and then press the Include button.' 2) 'Press this to load a profile for your analysis.' pointing to the 'Load from Profile' button. 3) 'Save the Predefined Elements to the User Profile.' pointing to the 'Save to Profile' button. At the bottom, there is a checked checkbox for 'Perform Auto ID During Acquisition' and 'Include' and 'Clear' buttons.

Summary Specimen Geometry Pre-defined Elements

Predefine your elements from the Periodic table. Click on an element first to select it and then press the Include button.

Press this to load a profile for your analysis.

Save the Predefined Elements to the User Profile.

Check this box to enable AutoID during the spectrum acquisition.

Load from Profile

Save to Profile

Clear All

Include Clear

Perform Auto ID During Acquisition

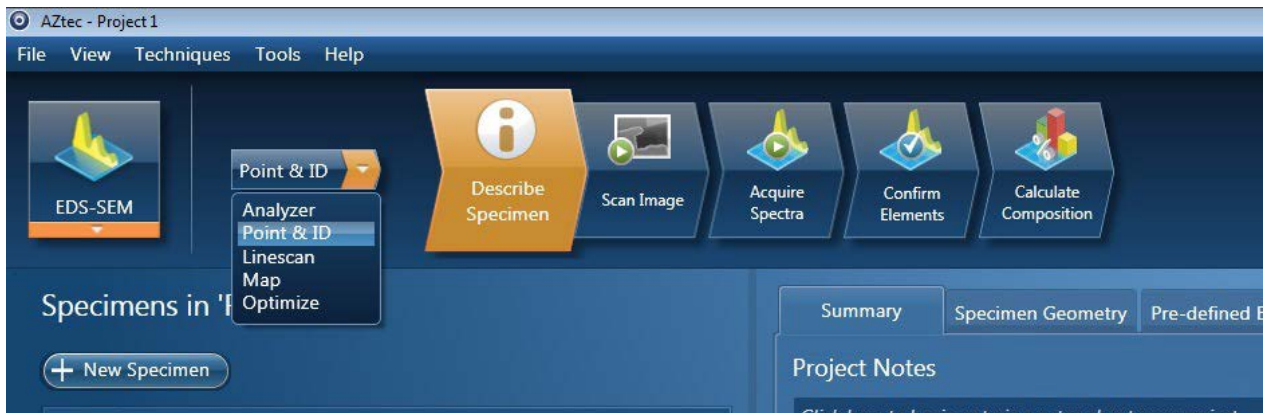
1.19. Next, choose a region of interest and make sure the area is in focus on the SEM. The detector and imaging modes do not matter. Make sure the beam parameters are set such that the Dead Time on the detector is between 30-70%. Under the **Scan Image** tab, take a picture of the region of interest.

It is best practice to create a “New Site” for each new electron image for each new area found under the SEM. These images act as a reference point for the EDS acquisition. Therefore, you should have no more than one electron image per site, but you can have multiple EDS scans per site.



1.20. The following sections will discuss the different scans available: **Point & ID**, **Map**, and **Line Scan**. To switch between any of these types of scans use the drop down menu.

The **Analyzer** function can be accessed at any point for a more in-depth statistical analysis.



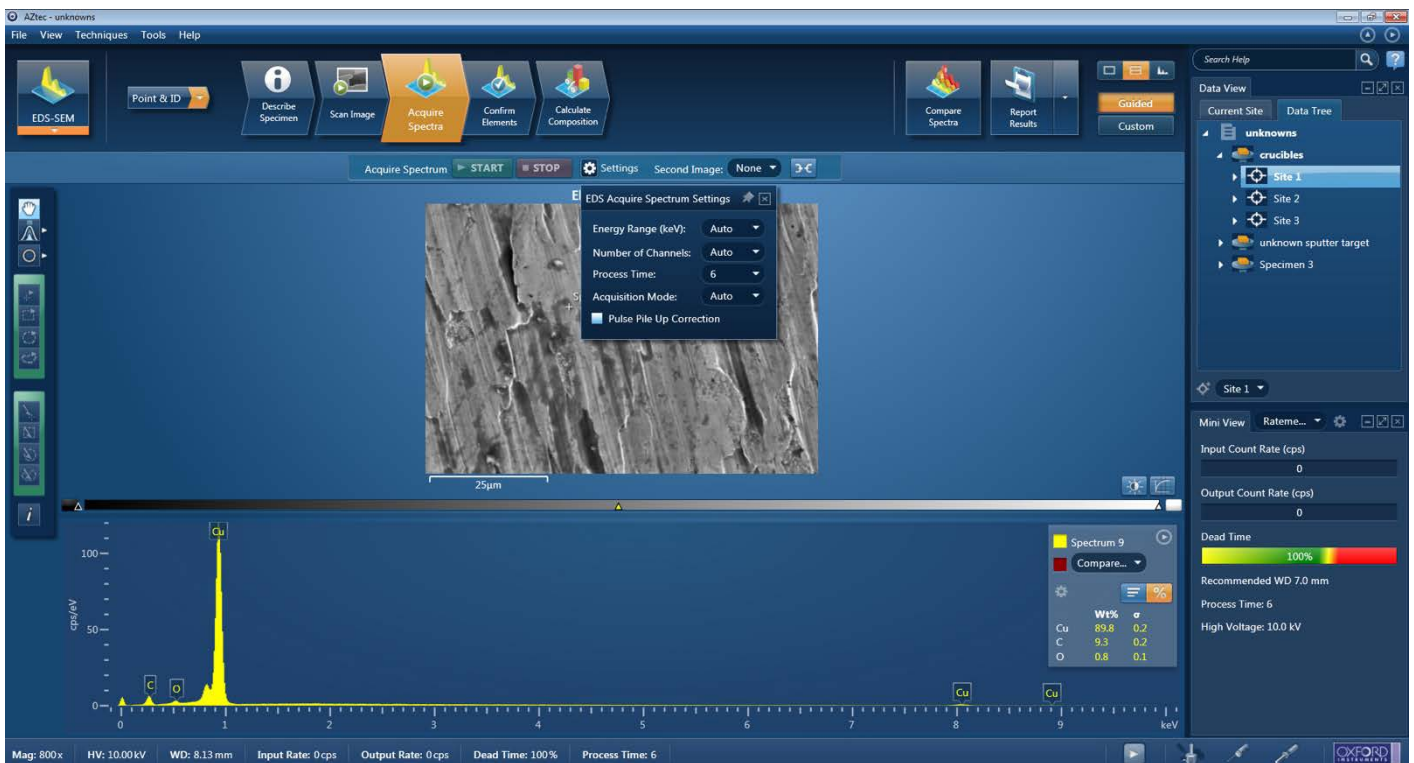
2.0 Point & ID (Quick Scan)

Follow steps 1.1-1.20 as described above.

See **APPENDIX B: DEFINITIONS OF KEY TERMS** for information about terminology used in the following sections about the various EDS parameters

2.1 After taking an electron image of the area of interest, move to the Acquire Spectra tab.

- Press **start** at the top to scan the entire area as displayed by the electron image
- Use the **point** icon on the left-side menu to gather information about a specific spot on the image
- Use the **area** icon on the right side menu to draw a region of interest from within the image, and analyze that area



2.2 The status bar at the bottom of the screen will show the progress of the scan.

2.3 At the end of the scan, move to **Confirm Elements** to analyze the results in wt % or at%. In this menu, you can:

- Inclusion and exclusion of specific elements by double-clicking on the labels of the spectrum peak. This will display candidate elements on the right side. Alternatively, you can select the element from the periodic table or from the drop down list. This will highlight the areas or overlay theoretical data onto the given spectrum.
- Elements in green are included either from the user or through the auto-id function. Elements in red are excluded by the user.

NOTE: It is best practice to compare the data with the library of information and look at the best fit. It is also important to ensure the interaction of the primary electron beam is completely representative of the material of interest.

- To reset the analysis, press **Clear all** and use the **Auto-ID function** to relabel the peaks.
- Switch between wt %, at%, and combination elements. A further mathematical analysis can be done under the Calculate composition tab.
- If you'd like to plot the spectra data through other methods, you can right-click on the spectra to save this information as a text file

See **APPENDIX D: EXPORTING SPECTRA DATA** for more information about how to export data into CSV or text files.



3.0 Mapping (of element to region)

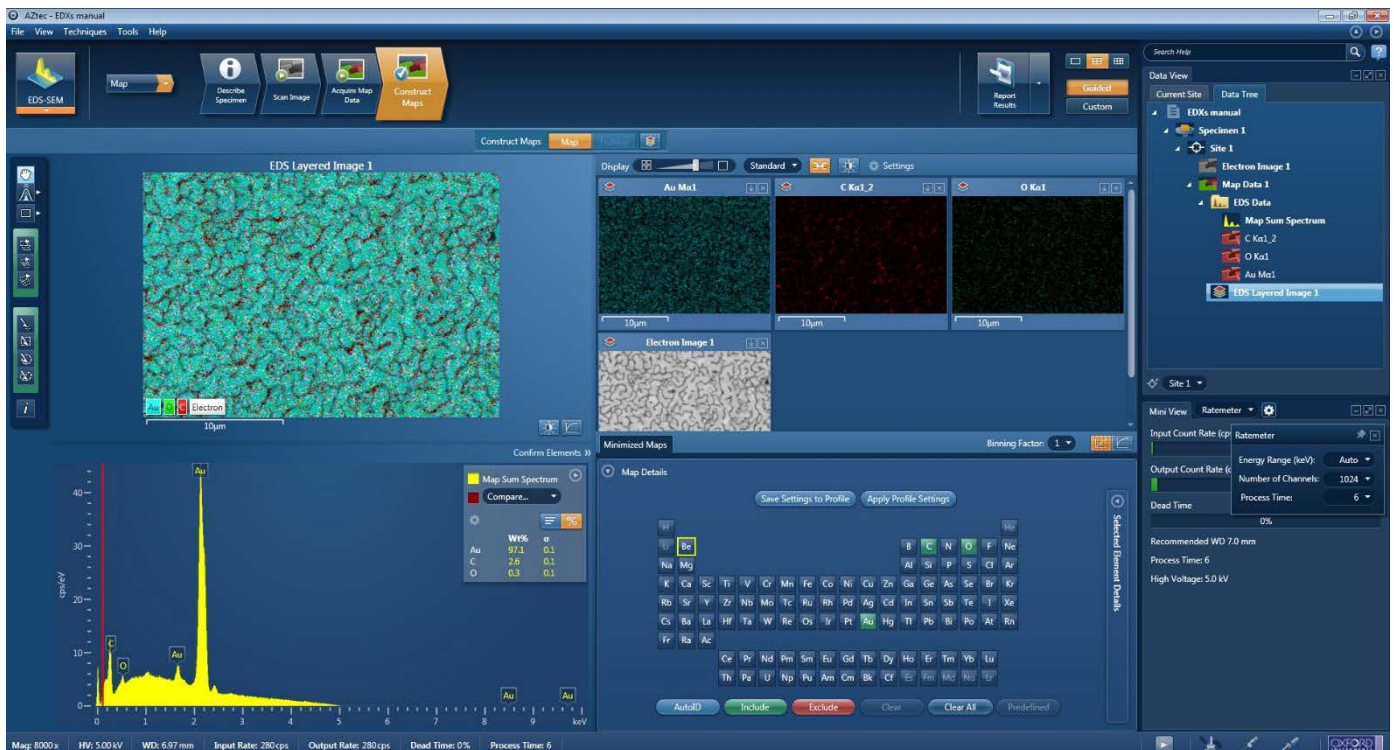
Follow steps 1.0-1.20 above

3.1 After taking an electron image of the area of interest, move to the Acquire Map data tab.

- Press **start** at the top to map the entire area as displayed by the electron image
- Use the **draw** icon on the right side menu to gather information about a specific spot on the image
- The scan can be continuous or fixed for a certain number of scans. As long as the beam is stable, the more scans mean that there are more counts per pixel which means that there is a higher accuracy in the software analysis.

3.2 After the data has been acquired, click on **construct maps**

- The top right-side of the screen will have a selection of images and elemental maps
- The top left-side of the screen will have the overlay of the selected images
- The bottom of the screen shows the acquired spectra and elements to be excluded or included from the spectra



d. The confirm elements option on this page will take you to step 2.3 for analysis.

e. If you'd like to plot the spectra data through other methods, you can right-click on the spectra to save this information as a text file

See **APPENDIX D: EXPORTING SPECTRA DATA** for more information about how to export data into CSV or text files.



Note: Use the drop down menu on the front page to switch back to Maps

4.0 Linescan (elements across a line)

Follow steps 1.0-1.20 above

4.1 After taking an electron image of the area of interest, move to the Acquire Line data tab

- Click and drag a line across the image to collect the spectra from that area
- Press **start** to start the acquisition of that data
- The scan can be continuous or fixed for a certain number of scans. As long as there is no drift, the more scans mean that there are more counts per pixel which means that there is a higher accuracy in the software analysis.
- The elements found in the scan will be displayed on the right-side of the window

4.2 To analyze and correct the data go to Construct Line scans

- The profile of the concentration of each element across the line will appear
- Adjust the binning factor to smooth the graph lines
- The left-side shows the SEM image at which the location of the scan was taken
- The spectra and concentration of elements can be recreated at individual points on the line by choosing the 'point' option on the left-side
- The confirm elements option on this page will take you to step 2.3 for analysis.
Note: Use the drop down menu on the front page to switch back to Line scan.
- If you'd like to plot the spectra data through other methods, you can right-click on the spectra to save this information as a text file
See **APPENDIX D: EXPORTING SPECTRA DATA** for more information about how to export data into CSV or text files.



5.0 SAVING FILES AND SHUTTING DOWN THE SYSTEM

5.1 To save files, the recommended format is “Site Reports”. This includes information about every type of scan taken on the electron image, the wt % and/or at%, and the SEM image of the area of interest. Alternatively, you may select other templates available depending on the type of information you are interested in.

****Make sure to go to “File” and “Save Project” before moving your documents, shutting down the software, or turning off the beam****

5.2 In order to save site reports, click on the site of interest. Then go to the “Report Results” button above the data tree area and select “site report” to save all the information from that site into a word doc or an excel sheet.

5.3 A second window will pop-up from here to confirm the location into which the site reports will be saved. This should be a local folder. The document will pop-up as it is saving all of your data, this is a preview of your document.

5.4 Then copy all your files from the local folder to the shared data folder. Then copy your files off the support PC with a USB or through email.

5.5 Once your files have been saved, and the project has been saved you may start shutting down the system.

5.6 First retract the detector and move the EDS to “standby”, then close out of the AZtec software once the detector has retracted completely.

5.7 Then move to the Microscope software and turn off the beam. You should hear a hissing sound (1-3 seconds) that indicates the system is now safe to vent.

5.8 Vent the chamber

5.9 Pull out your sample from the chamber, lower the stage if necessary.

5.10 Pump down the chamber

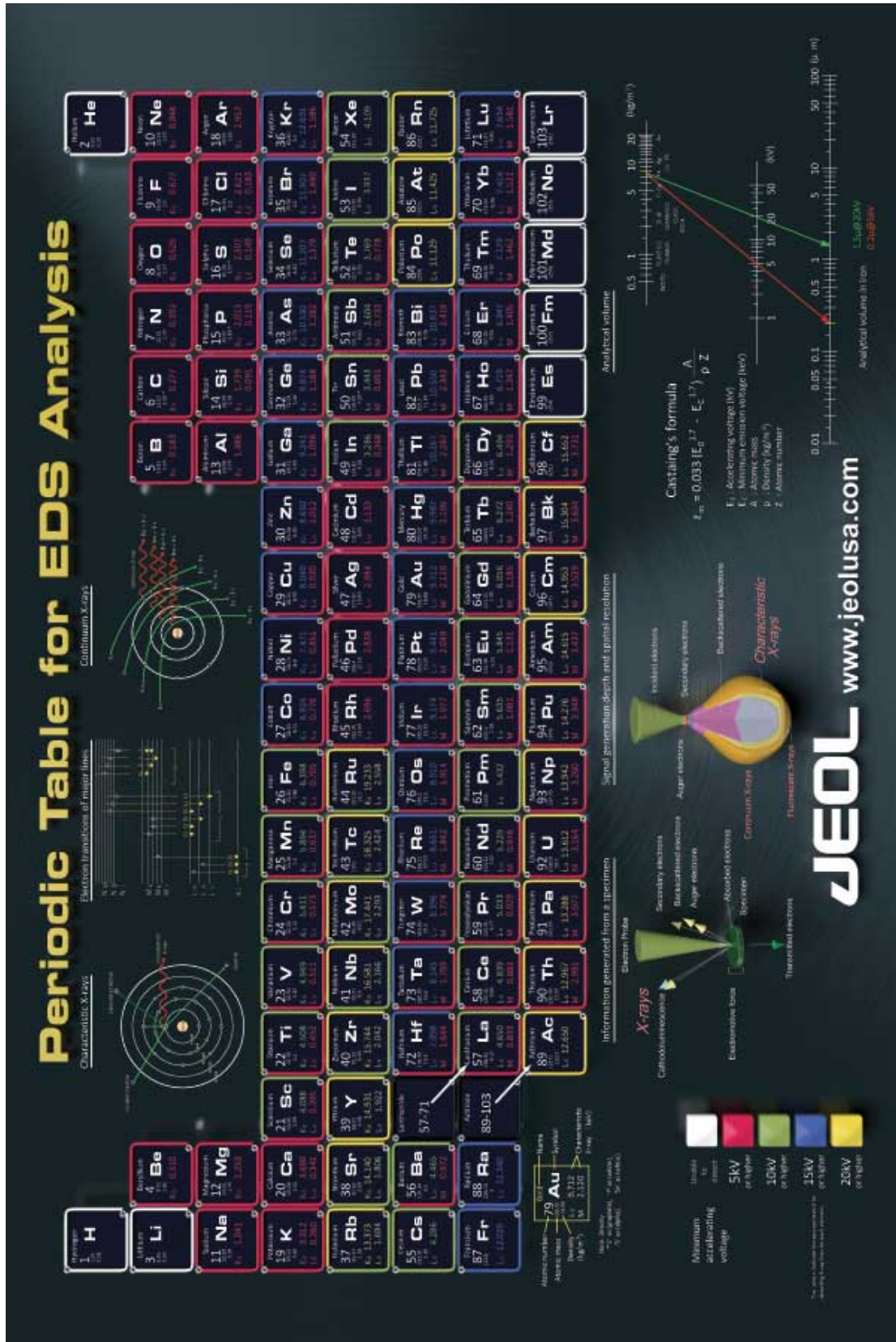
5.11 Home the stage

5.12 Make sure all vacuum segments in the “Sample Exchange” Menu are back to bright green. This indicates that the system has successfully pumped down and it is OK to walk away from the machine.

5.13 Log off the “Supervisor” account and disable the tool from Badger.



APPENDIX A: EDS ENERGY CHART FOR BEAM VOLTAGE PARAMETERS



APPENDIX B: DEFINITIONS OF KEY TERMS

Dead Time: The percentage of time the pulse processor is unavailable for further counting. The ratio between input rate to acquisition rate. The input rate is the rate at which x-rays are hitting the detector. The acquisition rate is the rate at which the system is processing the counts and formulating the spectrum. Ideally the dead time should be somewhere between 30-70%.

Energy Range: An appropriate energy range should be selected in conjunction with the current microscope accelerating voltage. If the accelerating voltage is above 10 kV, in order to view lines which may be excited above 10 keV, the 20 keV range should be chosen. Below 10 kV, it may be more appropriate to choose the 10 keV range since no lines above 10 keV will be excited. In the Auto mode, the software checks for the accelerating voltage set on the microscope and selects a suitable energy range in the software.

Number of Channels: The number of eV/channel will depend on both the energy range and the number of channels you select. Select number of channels from the drop down list of Auto, 1024, 2048 or 4096 (4K) select the channel you wish to display the spectrum. In the Auto mode, the software checks for the energy range selected and sets the appropriate number of channels.

Energy Range (keV)	Number of Channels	eV/channel
0-10	4096	2.5
0-10	2048	5
0-10	1024	10
0-20	4096	5
0-20	2048	10
0-20	1024	20
0-40	4096	10
0-40	2048	20
0-40	1024	40

Process Time: The Process time is the length of time spent reducing noise from the X-ray signal coming from the EDS detector during processing. By selecting different Process Times it is possible to reduce differing amounts of noise. The longer the Process Time, the lower the noise, the longer the acquisition time and higher the Dead Time. Select the Process Time from the drop-down list of Process Times: Default and 1 to 6. If Default is selected, the Process Time is automatically set to a suitable value

Pulse Pile-Up Correction: This setting is available under the “Acquire Spectra” tab. The pileup correction algorithm assumes that the count rate at every energy is constant throughout the analysis period. Therefore, the correction works best when analysis is performed on single pixels, points or areas of same composition. Bad results may be obtained if the beam is rastered over an area where composition is changing or if a spectrum is reconstructed from a SmartMap over a region where the composition is changing. Check Pulse Pileup Correction check box if you wish to automatically correct the spectrum for pulse pileup peaks. Uncheck the box if you wish to disable this correction.

Acquisition Mode: There are three options to terminate the acquisition, Auto, Live Time and Counts. If Auto mode is selected, acquisition continues until enough counts are collected in the spectrum for quantification. You can choose to terminate acquisition at the end of a preset Live Time. Enter the required time in seconds into the text box. This is the time for which the system is processing counts into the spectrum. The live time clock runs slower than the real time clock so that the acquisition for ‘100’ live seconds takes longer than 100 real seconds. This time is extended to compensate for the output rate being less than the input rate by the degree of Dead time. You can choose to terminate acquisition at the end of a preset number of counts. Enter the value in the Count Limit text box. The default value is 500,000.

APPENDIX C: WHY ARE SPECIMENS COATED?

Samples are sometimes coated with a thin conductive layer prior to observation in the SEM to ensure that there is a good electrical path to ground. This prevents non-conducting specimens, as well as the oxides which are present on the surface of many samples, from charging under the electron beam.

- Carbon is generally the preferred coating material for microanalysis applications because of its minimal effect on the X-ray intensities.
- Gold is also commonly used for coating specimens since it increases the secondary electron yield providing improved SEM image quality. However, the number of peaks produced by the gold coating can cause overlap problems with the peaks of interest.
- A guide for the desirable thickness range of coatings is of the order of 5-30 nm. To minimize the effect of your coatings aim to make them as thin as is practically possible. Thick coatings will, for some samples, result in poor analysis.

Effect of the Coating on the X-ray spectrum

The coating will have three main effects:

1. Energy loss of the primary electron beam as it passes through the coating.
2. Attenuation of the emerging X-rays.
3. The contribution of characteristic peaks to the X-ray spectrum. Thus, carbon coated samples will always display a carbon peak in the spectrum.
 - In the software, you can specify a coating element for a particular sample. During spectrum processing any X-ray peaks arising from this element are automatically **deconvolved** in addition to two other corrections (loss of X-ray intensity due to absorption of the emergent X-rays, reduction of primary KeV). Application of these corrections is particularly important for low kV spectra in particular (~ 5 keV).
 - The final quantitative results could have significant errors if no such corrections were applied.
 - Sample step - input of additional parameters : coating density and thickness
 - Standardization - here the normalized area of the standard ($I(\text{std}) / I(\text{optimization})$) is corrected for the coating. Full calculations - the quantitative results are corrected for the coating.

APPENDIX D: EXPORTING SPECTRA DATA

You can save your spectra data in CSV or as a text file format for any EDS scan.

In order to do so, go to the page that has your spectra. Right-click on the spectra itself and a smaller menu should pop-up.

Once you select “export” you can save the data either as a CSV or a text file depending on the scan itself.

From here, you can move this file to the shared data folder, and from there move the file to your USB or email.

