

# Oxford Instruments Energy-Dispersive Spectroscopy (EDS)

## Operating Manual

### Always

- Make sure that you are logged in with your CRiS ID
- Wear clean gloves and use tweezers when handling samples inside the vacuum chamber
- Report problems immediately to staff to ensure quick diagnostic and repair

### Never

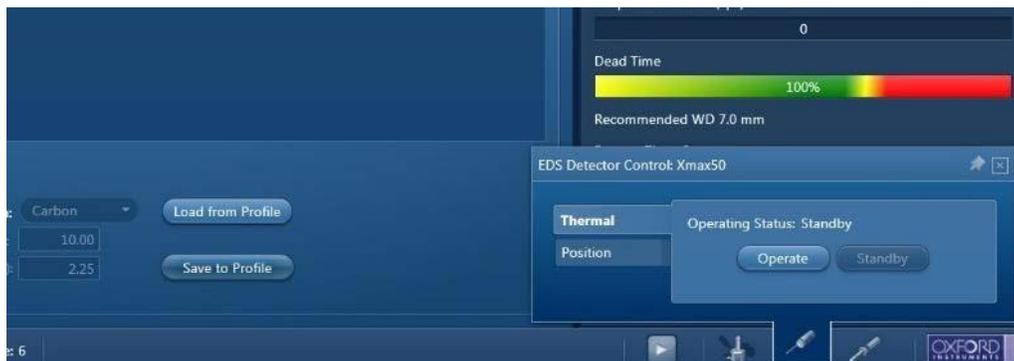
- Use the microscope for samples that will generate particles or have poor vacuum compatibility. If in doubt, please check with staff.
  - Wet photoresist, loose powders/flakes, liquids, biological samples, etc

1. Wearing gloves mount your sample on the appropriate SEM stub.
2. Start "SmartSEM" app and Login to microscope with your username and password (created during training)
3. Press **Vent**. A dialogue box will appear to confirm the "vent" operation. It takes approximately 5 mins for the chamber to vent completely.
4. **\*\*Do not insert or retract the EDS detector while the microscope is venting or pumping\*\***
5. 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.
6. While watching the CCD camera, close the chamber door. **Make sure there is enough clearance between your sample and the pole piece!**
7. 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.
8. While waiting for the chamber to pump down you can:
  - a. Move the stage up to the 7mm marker (7-10mm range is preferred for EDS)
  - b. Set your imaging parameters for the SEM (acc volt, beam current, resolution, etc)
  - c. Make sure the detector settings are appropriate for E-beam (.....)
  - d. Move to the Aztec software and begin setting up the project folder
9. 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 use memory stick.

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.

10. 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

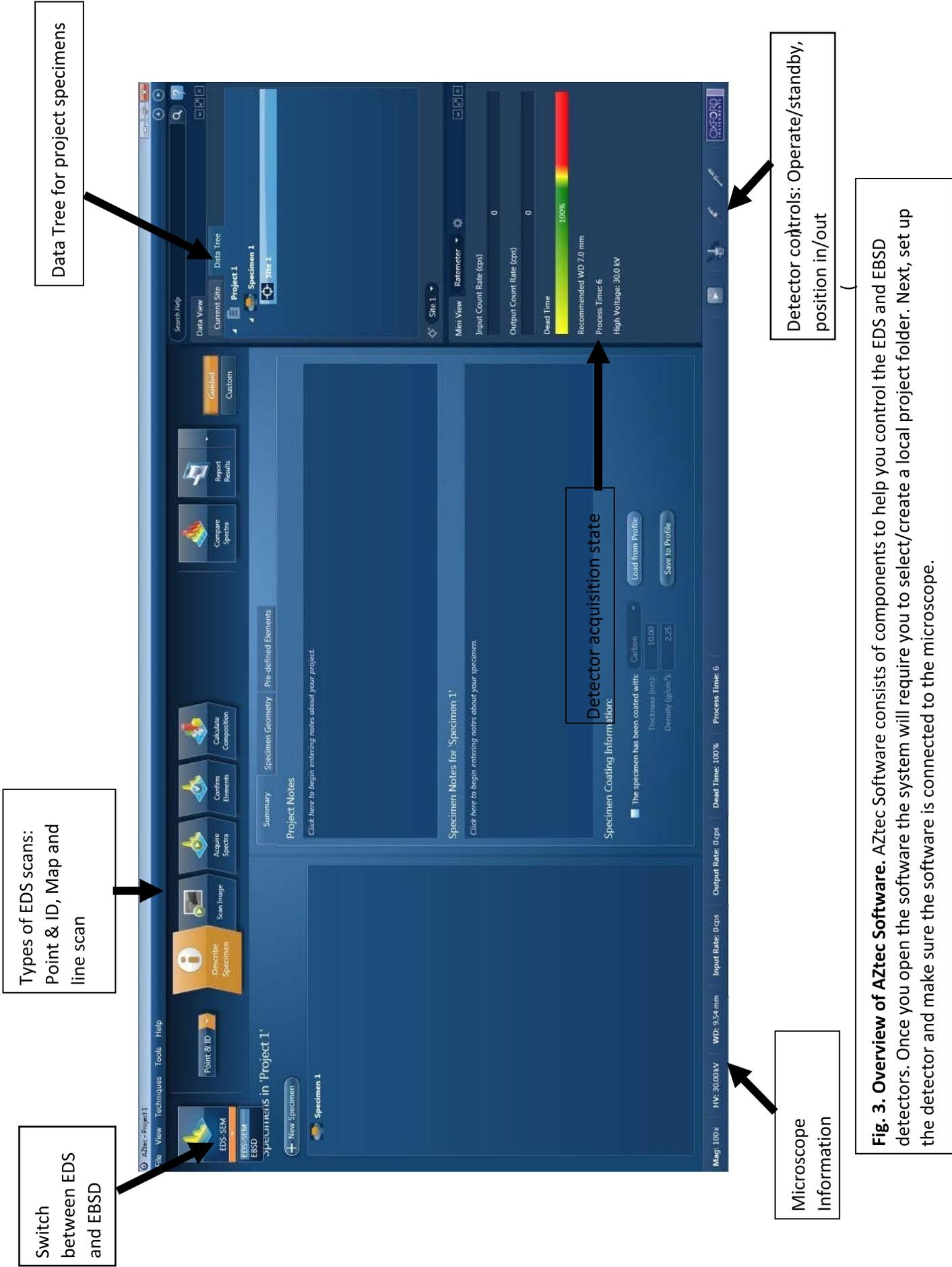


11. Switch back to the Microscope controls and begin imaging by pressing the **EHT button** to turn on the beam.

12. 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

13. 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.

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



15. 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.

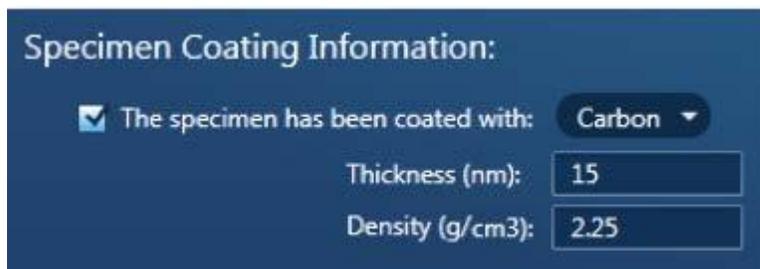
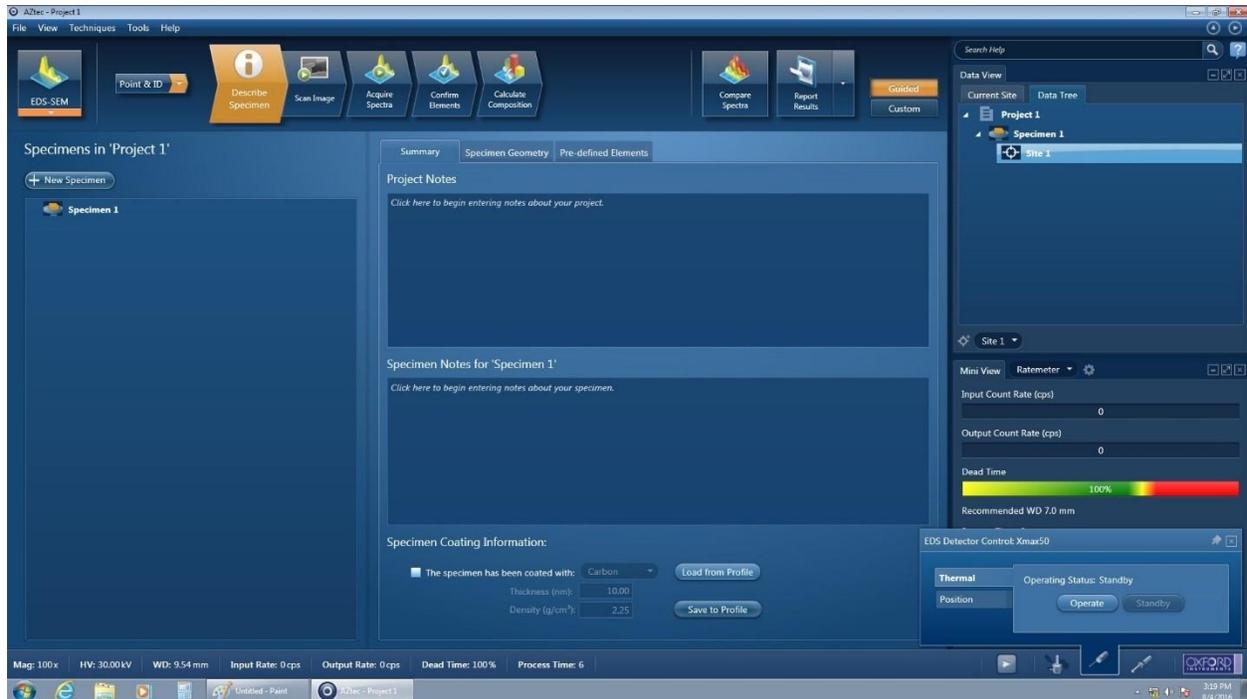


c. Linescan: Determines the concentration of elements across a user-defined line

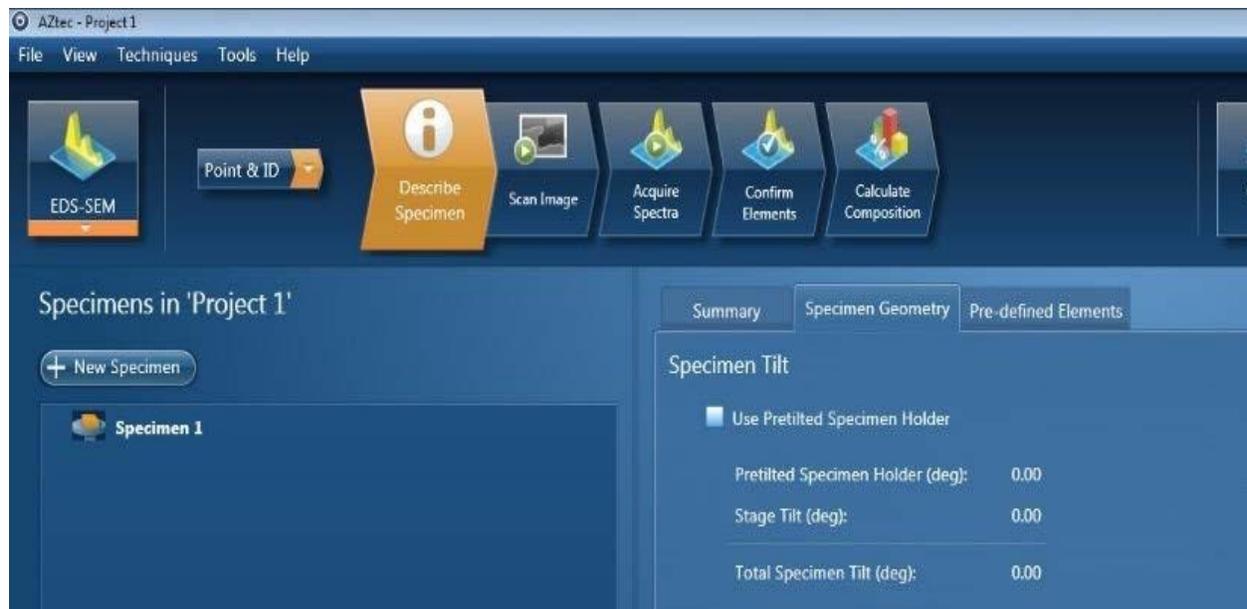


16. After you choose an option, on the first page you will be asked to describe the specimen. You can write notes about the sample and the parameters used which will be included in any saved reports. Make sure you also include any information about a specimen coating (ex. Carbon). Spectral data from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

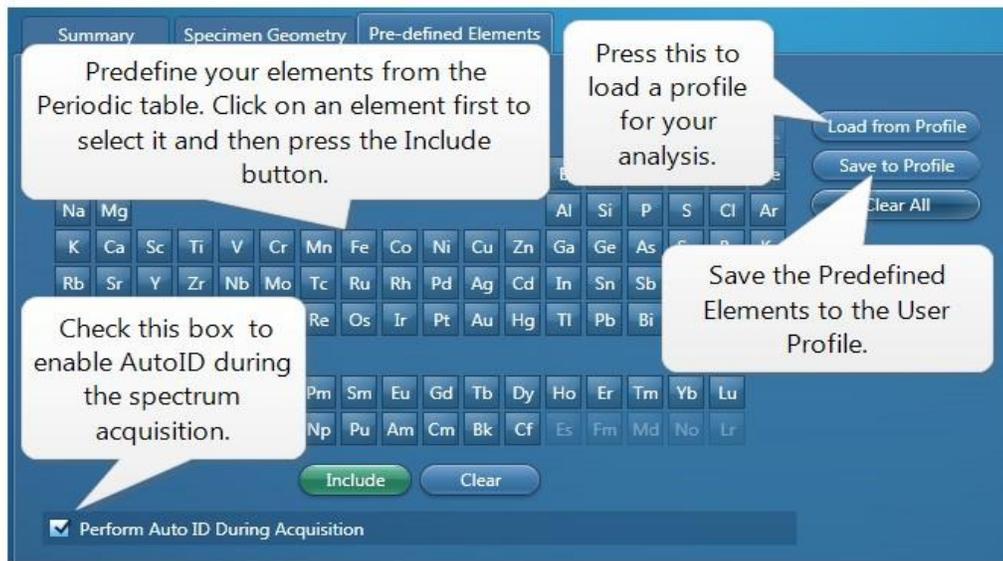
See **APPENDIX B: WHY ARE SPECIMENS COATED?** For more information about what materials to use and their effects on microanalysis.



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.



17. 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.



18. 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.

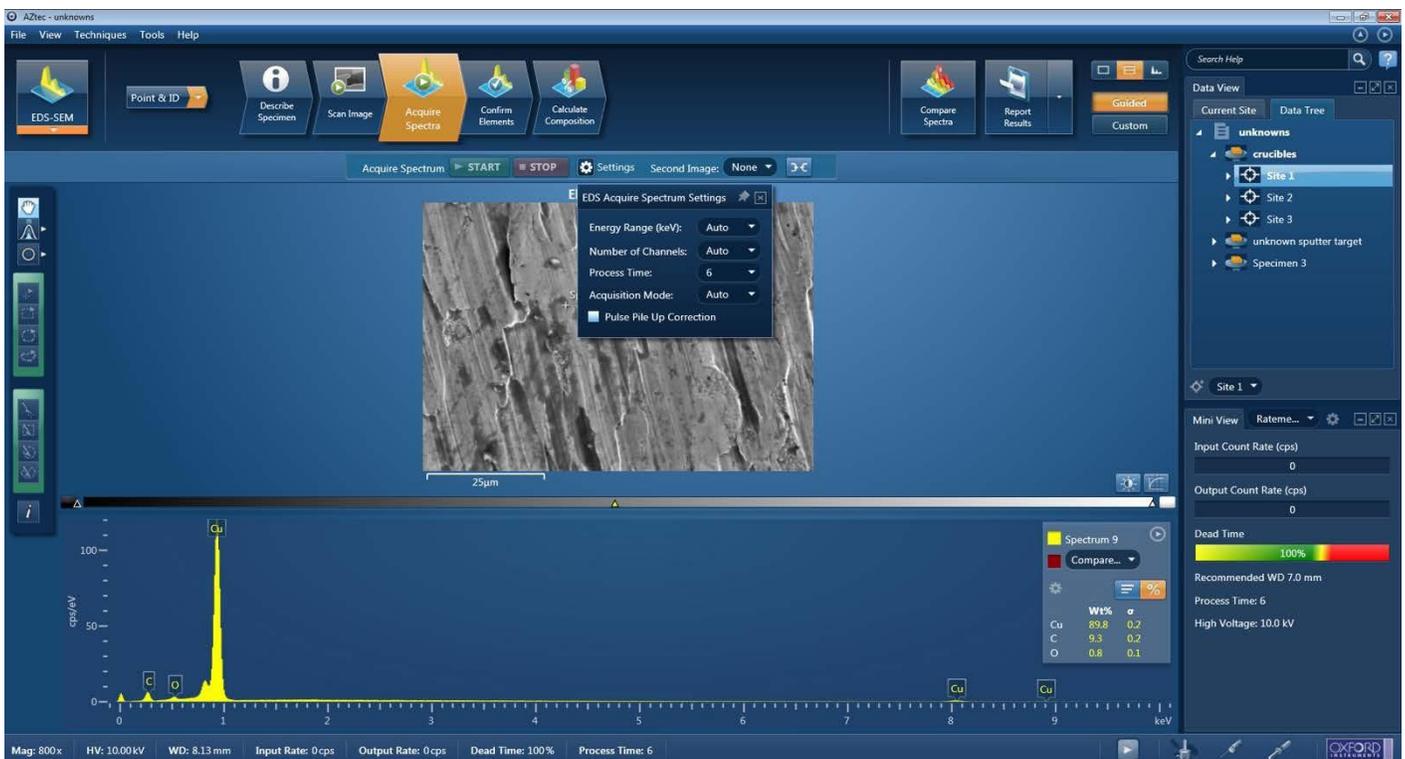


# Point & ID (Quick Scan)

Follow steps 1-18 as described above.

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

1. After taking an electron image of the area of interest, move to the Acquire Spectra tab.
  - a. Press **start** at the top to scan the entire area as displayed by the electron image
  - b. Use the **point** icon on the left-side menu to gather information about a specific spot on the image
  - c. Use the **area** icon on the right side menu to draw a region of interest from within the image, and analyze that area



2. The status bar at the bottom of the screen will show the progress of the scan.
3. At the end of the scan, move to **Confirm Elements** to analyze the results in wt % or at%. In this menu, you can:
  - a. 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.

b. 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.

c. To reset the analysis, press **Clear all** and use the **Auto-ID function** to relabel the peaks.

d. Switch between wt %, at%, and combination elements. A further mathematical analysis can be done under the Calculate composition tab.

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.

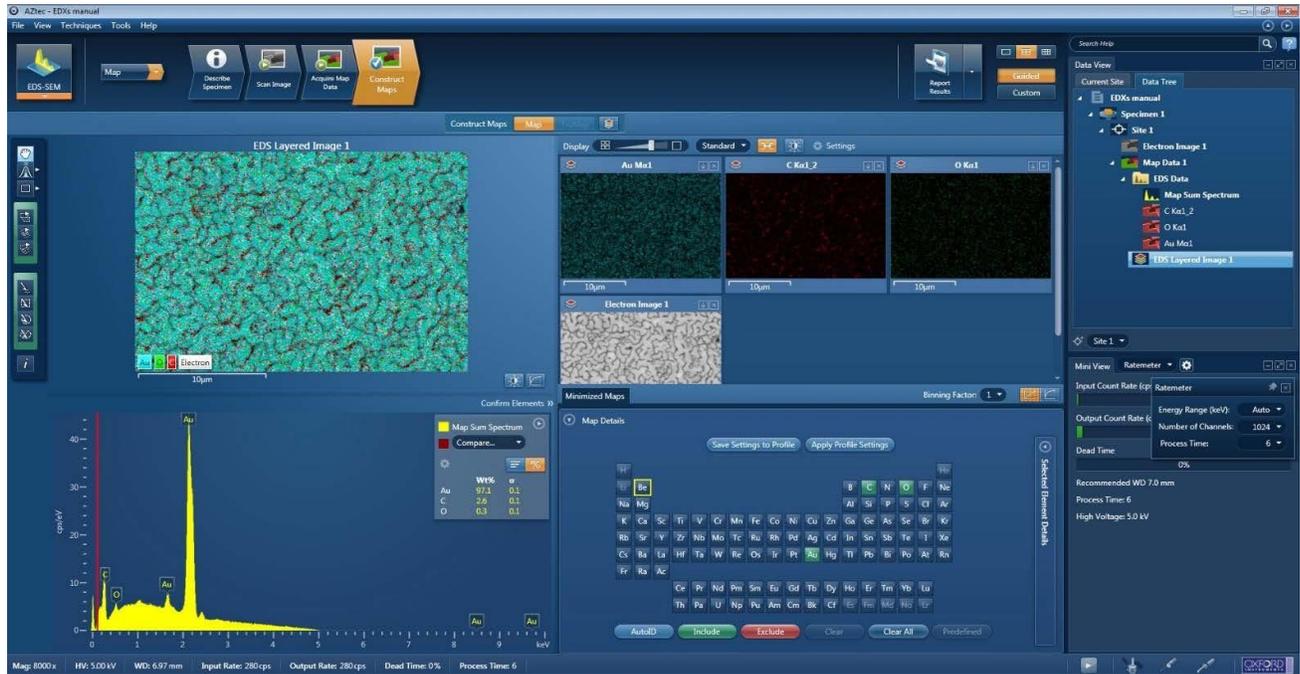


## Mapping (of element to region)

Follow steps 1-18 above

1. After taking an electron image of the area of interest, move to the Acquire Map data tab.
  - a. Press **start** at the top to map the entire area as displayed by the electron image
  - b. Use the **draw** icon on the right side menu to gather information about a specific spot on the image
  - c. 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.
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



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

# Linescan (elements across a line)

Follow steps 1-18 above

1. After taking an electron image of the area of interest, move to the Acquire Line data tab
    - a. Click and drag a line across the image to collect the spectra from that area
    - b. Press **start** to start the acquisition of that data
    - c. 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.
    - d. The elements found in the scan will be displayed on the right-side of the window
  2. To analyze and correct the data go to Construct Line scans
    - a. The profile of the concentration of each element across the line will appear
    - b. Adjust the binning factor to smooth the graph lines
    - c. The left-side shows the SEM image at which the location of the scan was taken
    - d. The spectra and concentration of elements can be recreated at individual points on the line by choosing the 'point' option on the left-side
    - e. 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.
    - f. 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.



## SAVING FILES AND SHUTTING DOWN THE SYSTEM

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

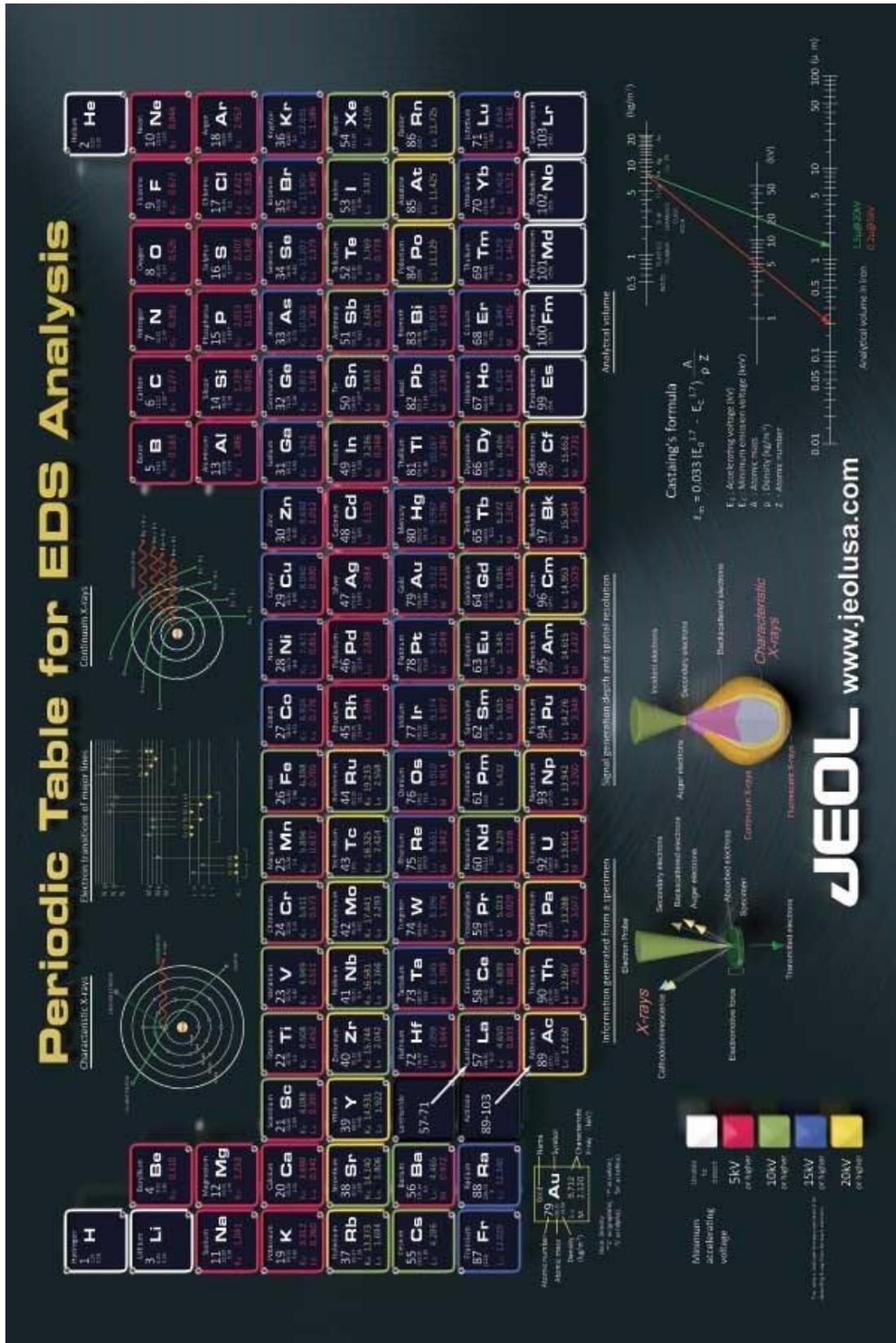
interest. Alternatively, you may select other templates available depending on the type of information you are interested in.

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

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.
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.
4. Then copy all your files from the local folder to the USB storage device
5. Once your files have been saved, and the project has been saved you may start shutting down the system.
6. First retract the detector and move the EDS to “standby”, then close out of the AZtec software once the detector has retracted completely.
7. Then move to the Microscope software and turn off the beam and lower the stage.
8. Vent the chamber
9. Pull out your sample from the chamber.
10. Pump down the chamber
11. When the system indicates that it has successfully pumped down log off



# 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.

Gold/Palladium is commonly used for coating specimens since it increases the secondary electron yield providing improved SEM image quality.

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, gold coated samples will always display a gold 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.

