1.0 **Equipment Purpose**

1.1 The Oxford Instruments X-MaxN 50 mm$^2$ EDS system is used in conjunction with the leo scanning electron microscope (SEM) to qualitatively and quantitatively determine the elemental composition of materials.

1.2 It is a sub-surface (0.3 - 3 um) elemental analysis technique with a best case detection limit ~ 1 atom%. It has a detection range of elements from Beryllium (Be) to Californium (Cf) with a resolution > 127 eV.

1.3 In addition to the standard analysis software, the system has been outfitted with the LayerProbe package, a minimally invasive technique to measure the composition and thickness of individual layers (i.e. thin films). It has a best case lateral and thickness resolution down to 200 nm and 2 nm, respectively.

2.0 **Material Controls & Compatibility**

2.1 Samples that are delaminating, flaking, or particle generating are not permitted.

2.2 Do not use the system to inspect photoresist, PMMA, spin-on based materials, or other organic based materials. This includes UV baked/stabilized, hard baked, and other fully cured organic materials.

2.3 Materials that out-gas, have poor vacuum compatibility, or can form loose particles are prohibited. If you have any doubt, ask a staff member for approval of your material.

2.4 Materials that are wet, sticky, greasy, or are in a form of a powder are not allowed. This includes: paste and tape of any kind, anything touched without clean gloves, and any material that might have flakes or shows flake-like delamination or particulation.

2.5 Contact Nanolab Staff if you are unsure your sample is compatible with this system.

3.0 **Applicable Documents**


4.0 **Definitions & Process Terminology**

4.1 Acquisition Rate - The rate at which the system is accumulating spectrum counts. Note the acquisition time is dependent on the process time.

4.2 Deadtime - The percentage of time the pulse processor is unavailable for further counting, i.e. the ratio of input rate to acquisition rate.

4.3 EDS/EDX - Energy dispersive spectroscopy

4.4 Input Rate - The approximate rate of photons striking the detector.

4.5 Interaction Volume - A teardrop shaped volume where incident/primary electrons interact within the sample of interest.
4.6 Spectral Line/Characteristic Peak - A known or measured photon energy, exhibiting a distribution when measured, resulting from the transition of electrons energy states, i.e. binding energy.

4.7 Operation Mode - Analysis techniques available in the AZtec software.

4.8 Process Time - The length of time spent reducing noise from the x-ray signal coming from the detector during processing.

4.9 Pulse Pile Up - When more than one photon is detected as a single photon. Occurs more often during high count rates.

4.10 Qualitative Analysis - A method generally used to determine the presence of an element in a given sample.

4.11 Quantitative Analysis - A method generally used to determine the amount of an element in a given sample.

5.0 Safety

5.1 Tool Qualification Policy

5.1.1 Only edx qualified members are permitted to use the EDS system. Qualification on the leo SEM does not qualify members for edx use - they are two separate qualifications.

5.1.2 Lab members must be qualified, demonstrate acceptable use, and have experience on the leo SEM to be eligible for qualification on the EDS system.

5.1.3 To become qualified, read and understand the online manual (this document), and attend a training session run by Nanolab staff. Contact Jeff Clarkson (jclarks <a tt>berkeley.edu) to register for the class.

5.2 Personal Safety

5.2.1 Do not touch the EDS detector connected to the SEM system. The unit is controlled with the AZtec software. This reduces the hazards of electrical shock and pinch points.

5.3 Tool Precaution

5.3.1 Refer to section 2.0 for criteria to determine what sample types are allowed. All samples must be stable under imaging and analysis conditions. Immediately reduce the EHT if you observe any indication of sample degradation/instability or an uncharacteristic change in system vacuum levels.

5.3.2 Never touch the EDS detector connected to the SEM system. This can cause misalignment and/or damage to the system.

5.3.3 Never pump or vent the main system chamber of the SEM with the EDS probe inserted (In position). Always move the probe head to the Out position before pumping or venting.

5.3.4 Never perform an O₂ plasma clean with the EDS probe inserted (In position). O₂ cleans may be run when the the probe head is retracted in the Out position.

5.3.5 Always turn on the live video feed of the main system chamber using the SmartSEM software and inspect the sample and its surroundings to verify it is safe to insert the detector.

5.3.6 Always position samples at a working distance of 5 mm and obtain an image before inserting the EDS probe (In position) and performing analysis.

5.3.7 Never tilt samples when using the system. All portions of the stage, sample holder, and sample must be completely below a working distance of 5 mm when the probe is inserted (In position).

5.3.8 If there is any uncertainty, discontinue use of the system and contact Nanolab Staff for assistance.
5.3.9 Qualified members are responsible for all samples placed in the system - including the samples of unqualified users during training and supervised use.

5.3.10 Always use the Sheeva Drive to copy data onto a USB drive. Never use the any other USB port.

6.0 Process Data
6.1 N/A

7.0 Available Processes, Gases, Process Notes
7.1 Calibration standards should be of the highest quality. For Si, do not use pieces from the scrap bin. Check out a new test or prime grade wafer from the Nanolab Office and store it in a 6” wafer shipping tray.

8.0 Equipment Operation
8.1 Methodology

The method of analysis is highly depended on inspection goals, materials involved, and sample architecture. The block diagram below is a workflow that serves as a guide for navigating this manual. Parenthetic values refer to the manual section to be used. e.g. (8.4) refers to section 8.4 of this document.

```
Initial Start-Up (8.2)

Point & ID (8.3)  Mapping (8.4)  Linescan (8.5)  LayerProbe (8.6)
Elemental analysis at a single point(s).
Elemental analysis over the entire surface of a sample.
Elemental analysis over a single line, typically located over a feature of interest.
Elemental analysis in the z-axis, i.e. determination of film thickness and elemental composition.

Normalized Quantification (8.7)  Unnormalized Quantification (8.8)
Analysis based on an internal library of standards, i.e. no standards are required. The software assumes all elements are identified in the sample.
Analysis based on normalized beam current for the sample of interest and standards. i.e. a standard(s) are required.

Reporting and Saving Data (8.9)

Session Closeout (8.10)
```

8.2 Initial Start-Up
8.2.1 Ensure the SmartSEM user interface is running and the sample of interest is being imaged at a working distance of 5mm.

8.2.2 Double click on the Remcon32 shortcut on the leo computer desktop.

8.2.3 From the pull down menu in the RemCon32 window, select COM 2. Then in the top toolbar, select Comms and then Open Port. A string of communication text will be continuously updating in this window when you open the AZtec software in the next step.
8.2.4 Power up the edx computer and log-in as usr: oxford, pw: oxford.
8.2.5 On the edx computer desktop, double click on the AZtec shortcut.
8.2.6 Click on New Project and enter a project name. Save the project in C:\Users\Oxford\Desktop\User Data\< your mercury login name, all lowercase here>. 
8.2.7 Click on FACTORY profile and then OK.
8.2.8 In the SmartSEM user interface, navigate to and frame the region of interest and adjust the following imaging conditions:
   - Working distance = 5 mm
   - Aperture = 60 um
   - EHT = 20 kV

***WARNING!!!
Make sure your sample is stable under such conditions. Immediately reduce the EHT if you observe any indication of sample degradation or instability.

8.2.9 In the AZtec software ensure step Describe Specimen is highlighted. Then choose Specimen is coated. Set Coating element to Carbon, Thickness from 2.00 - 10.00, and Density to 2.25. This accounts for hydrocarbon contaminants that are likely present on the sample's surface.

8.2.10 Turn on the live video feed of the main system chamber using the SmartSEM software. Inspect the sample and its surroundings and verify it is safe to insert the detector. If there is any uncertainty, discontinue use of the system and contact Nanolab Staff for assistance.

8.2.11 Once it's safe to insert the detector, at the bottom right of the AZtec window, click on the detector icon (Control of the EDS detector: Xmax50). Select Position, and then In.

***WARNING!!!
Never pump or vent the main system chamber with the EDS probe inserted (In position). Always move the probe head to the Out position before pumping or venting.

8.2.12 Return to the SEM image and adjust beam settings until dead time is < 65%. A dead time from 40% - 65% will provide the best acquisition rate, however it is still possible to use a dead time lower than 40%.

8.3 Point and ID Operation

This is one of the quickest way to analyze samples and will collect information from specific locations within an SEM image area. See Figure 1 in section 10.0 for an image showing this mode of operation.

8.3.1 At the top left of the AZtec window use the pull down menu to select Point & ID.
8.3.2 Image the region of interest on your sample and verify the working distance is 5 mm.
8.3.3 In the AZtec software, click on Scan Image, New Site, and then Start.
8.3.4 Select Acquire Spectra and then click locations on the scanned image where spectra is to be acquired. The EDS will begin acquiring spectra and each selected location will be listed on the top right hand side of the AZtec window.
8.3.5 Click on Confirm Elements and then choose the spectrum to be analyzed.
8.3.6 The AZtec software will autoID the spectra and present an analysis in a graph showing the acquired spectra in yellow and the proposed fitted spectra, based on which elements are included in the analysis, in pink.

8.3.6.1 The view of this graph can be modified by using the mouse scroll wheel, and/or clicking and dragging the left mouse button.

8.3.6.2 Inclusion and exclusion of elements in the analysis may be modified by double clicking on any element in the periodic table. Elements in green are included, whereas elements in red are excluded.

8.3.6.3 It is best practice to review the spectra by zooming in and looking at the fit between the fitted and acquired spectra. Start with the highest energy peaks first. Candidate elements can be investigated by enabling the question mark icon (left side of AZtec window) and double clicking on any peak in the spectrum. Upon selecting a peak, proposed elements can be reviewed by clicking on the small arrow located above the Candidate Elements and then clicking on each proposed candidate element. Double click on any candidate element to include it in the fitted spectra and list of confirmed elements. Double click it a second time to exclude it from the list of confirmed elements.

8.3.6.4 Occasionally it is desirable to include or omit specific spectral lines in the analysis. To do this, select the element of interest from the periodic table, click on the small arrow above Peak Labels, and then manually select peaks to be included.

8.3.6.5 Analysis of the spectra can be reset by clicking on the Clear all and then AutoID buttons located at the bottom of the AZtec window.

8.3.7 To perform quantitative analysis and determine elemental concentration, go to section 8.7 and/or 8.8.

8.3.7.1 It is important to ensure the interaction volume of the primary electron beam is completely within a portion of the sample that is homogeneous and representative of the material of interest. See section 11.1 and 11.2.

8.4 Map Operation

Mapping is used to determine the location of elements across the SEM image field. See Figure 2 in section 10.0 for an image showing this mode of operation.

8.4.1 At the top left of the AZtec window use the pull down menu to select Map.

8.4.2 Image the region of interest on your sample in the SmartSEM window and verify the working distance is 5 mm.

8.4.3 In the AZtec software, click on Scan Image, New Site, and then Start.

8.4.4 Select Acquire Map Data and then Start. A map for each element detected will be created and a list of all elemental maps will be listed at the top right hand side of the AZtec window.

8.4.4.1 The acquisition is continuous and may be ended by clicking the Stop button.

8.4.4.2 If imaging conditions are stable the acquisition can be left running. This will significantly increase the count at each pixel and improve the accuracy of analysis.

8.4.5 Click on Construct Maps.

8.4.5.1 The top right quadrant shows individual elemental maps. Each map can be included/excluded from the layered map by clicking on the red diamond icon located in the top left corner of each map.
8.4.5.1.1 Use the Display slider to increase and decrease the size of individual elemental maps.

8.4.5.2 The top left quadrant shows a map consisting of all elemental maps selected for overlay.

8.4.5.3 The bottom left quadrant shows the acquired spectra.

8.4.5.3.1 Further analysis for determining elemental composition can be performed by clicking on Confirm Elements. This opens the Point & ID operation. Information on using this operation can be found in section 8.3.6. To return back to the map, use the pull down menu located at the top left of the window and change the operation from Point & ID to Map.

8.4.5.4 The bottom right quadrant is often minimized. Click on the small up arrow next to Map Details to enlarge it.

8.4.5.4.1 In this quad, elements can be included or excluded in the map by double clicking on them in the periodic table. Elements in green are included, whereas elements in red are excluded.

8.4.5.4.2 Analysis of the map can be reset by clicking on the Clear all and then AutoID buttons located at the bottom of the AZtec window.

8.4.6 Ensure there are an adequate number of counts within the imaging field before performing quantitative analysis. Click on Construct Maps, Confirm Elements, Calculate Composition, and then Spectrum Details. Verify the Spectrum Area (counts) is > 500,000.

8.4.7 To perform quantitative analysis and determine elemental concentration, go to section 8.7 and/or 8.8.

8.4.7.1 It is important to ensure the interaction volume of the primary electron beam is completely within a portion of the sample that is homogeneous and representative of the material of interest. See section 11.1 and 11.2.

8.5 Linescan Operation

Linescan is used to profile elements across a user defined line within the SEM image field. See Figure 3 in section 10.0 for an image showing this mode of operation.

8.5.1 At the top left of the AZtec window use the pull down menu to select the Linescan operation.

8.5.2 Image the region of interest on your sample and verify the working distance is 5 mm.

8.5.3 In the AZtec software, click on Scan Image, New Site, and then Start.

8.5.4 Select Acquire Line Data, click and drag a line within the scanned image where spectra is to be acquired, and then click Start. Acquisition of spectra will begin and linescan data for individual elements found in the scan will be listed on the top right hand side of the AZtec window.

8.5.4.1 The acquisition is continuous and may be ended by clicking the Stop button.

8.5.4.2 If imaging conditions are stable the acquisition can be left running. This will significantly increase the counts at each pixel and improve the accuracy of analysis.

8.5.5 Select Construct Linescans

8.5.5.1 The top right quadrant shows the line scan for each individual elemental found.
8.5.5.1.1 Use the Display slider to increase and decrease the size of individual linescan.

8.5.5.1.2 The default display is Vertical Tiles. To place all linescans on a single graph, select Stacked from the pull down menu. To normalize the data, right click on the graph key and choose Normalize Y-Axis.

8.5.5.1.3 The Binning Factor may be used to control the number of measurements included along the linescan. This is often used to smooth the graph data lines.

8.5.2 The top left quadrant shows the SEM image field and location of where the linescan measurement is taken.

8.5.3 The bottom left quadrant shows the acquired spectra.

8.5.3.1 Further analysis for determining elemental composition can be performed by clicking on Confirm Elements. This opens the Point & ID operation. Information on using this operation can be found in section 8.3.6. To return back to the linescan, use the pull down menu located at the top left of the window and change the operation from Point & ID to Linescan.

8.5.4 The bottom right quad is often minimized. Click on the small up arrow next to Linescan Details to enlarge it.

8.5.4.1 In this quad, elements can be included or excluded in the linescan by double clicking on them in the periodic table. Elements in green are included, whereas elements in red are excluded.

8.5.4.2 Analysis of the map can be reset by clicking on the Clear all and then AutoID buttons located at the bottom.

8.5.6 Ensure there are an adequate number of counts along the linescan before performing quantitative analysis. Click on Construct Linescans, Confirm Elements, Calculate Composition, and then Spectrum Details. Verify the Spectrum Area (counts) is > 500,000.

8.5.7 To perform quantitative analysis and determine elemental concentration, go to section 8.7 and/or 8.8.

8.5.7.1 It is important to ensure the interaction volume of the primary electron beam is completely within a portion of the sample that is homogeneous and representative of the material of interest. See section 11.1 and 11.2.

8.6 LayerProbe

LayerProbe is used to measure the composition and thickness of thin films. Typically, either the thickness or elemental composition needs to be known such that a model may be created and used to solve for the remaining unknown variable(s). In addition to the sample of interest, an additional calibration sample must be included to perform the analysis. A Si chip is usually sufficient.

8.6.1 At the top left of the AZtec window use the pull down menu to select LayerProbe.

8.6.2 Click on Describe Model to create a model describing the thin film stack on the sample of interest.

8.6.2.1 In the Model region, click on Add Above and then define the substrate material by selecting Substrate.
In the Layer Details region, select Manual Entry, Number of Atoms, and enter the elemental composition in the Composition field. The composition should be entered as a chemical formula. E.g. Silicon dioxide is entered as SiO$_2$.

If the elemental concentration of the substrate material is known, set the Concentration to Fixed and Tolerance (%) to 0.0.

Add subsequent thin films to the model as above.

If the concentration of any of the elements in the films are not known, set the Concentration to Unknown and enter a Density and film thickness for each film.

If the thickness of any films are unknown, select Unknown thickness and enter Density and elemental Composition for each film.

Confirm the Status region is OK. If not, go back and verify all necessary information is entered into the Model.

At the pull down menu located at the top left of the AZtec window, select Optimize.

Set the Routine to Beam Measurement and then choose an Element that will be used for optimization.

Under Settings, set the Process time to 4 or 5 while making sure the Dead Time is between 40% - 65%. A dead time at this setting will provide the best acquisition rate, however it is still possible to use a dead time lower than 40%.

Ensure the magnification of the SEM image field is greater than 1000X and then focus on a calibration sample using the same column conditions that will be used for performing LayerProbe analysis on the sample of interest.

When the sample it in focus, ensure the working distance is at 5 mm.

Click on the Start button and wait for the spectra acquisition to complete.

Click Yes to save the beam measurement.

The stability of the beam can be checked by running successive spectra acquisitions. If two acquisitions, run immediately after one another, vary by more than +/- 3% file a problem report in Mercury. Ideally, the beam current should not vary by more than +/- 1%.

At the top left of the AZtec window use the pull down menu to select LayerProbe.

Click on Set Up Solver to determine the recommended EHT to use.

Verify the Elevation (deg) field reads 35.0.

Verify the Model reflects that of the sample of interest.

In the Settings region, select User list and enter 5 10 15 20 25 30.

Set the Target count rate (kcps) to 50.0 and the Live time (s) to 20.0.

Click on Update.

Under the Available Lines for Solubility Calculation, ensure all boxes are checked for each element and kV.

Click on Calculate Solubility.

Take note of the Selected acquisition conditions.
8.6.6.8.1 If 5 kV is suggested, make sure to use the 5 kV Standards Library found in the Settings menu during Calculate Composition.

8.6.7 Return to Optimize and run the Beam Measurement again using the EHT recommended by LayerProbe in the Solubility Calculation Results area.

8.6.8 Immediately move the stage to the sample of interest and acquire a well focused image field with a working distance of 5 mm.

8.6.8.1 At the top left of the AZtec window use the pull down menu to select LayerProbe.

8.6.8.2 Click on Scan Image, New Site, and then Start.

8.6.8.3 Select Acquire Spectra and then click locations on the scanned image where spectra is to be acquired. The EDS will begin acquiring spectra and each selected location will be listed on the top right hand side of the AZtec window.

8.6.8.4 Click on Confirm Analysis and then choose the spectrum to be analyzed.

8.6.8.5 If using an EHT < 10 kV, it may also be appropriate to use the Quat Standardizations(5 kV). This may be done by going to the Options Menu and selecting Factory, followed by Quat Standardizations(5 kV).

8.6.8.6 The Analysis region is located at the bottom right of the AZtec window and shows the calculated elemental composition in each layer.

8.6.8.7 If the Fitted and Theoretical Spectrum do not fit the measured spectrum well, the Simulate Spectra tool can be used to gain more insight on the sample of interest and to make adjustments to improve the model.

8.6.8.7.1 Once a better fit has been achieved, the solver can be re-run by going to the Calculate Layers area and clicking on Requatify.

8.6.8.8 Click on Calculate Layers to see a summary of the quantitative and film thickness results.

8.7 Calculating Normalized Elemental Composition

8.7.1 After attempting to confirm elements by achieving a fit between the acquired and fitted spectra, it is possible to perform a quantitative composition analysis. Accuracy of composition depends on many factors and much care must be taken to determine the accuracy of results.

8.7.1.1 It is important to ensure the interaction volume of the primary electron beam is completely within a portion of the sample that is homogeneous and representative of the material of interest. See section 11.1 and 11.2.

8.7.2 To see the proposed composition, ensure Point & ID operation is selected, click on Calculate Composition and select Full Results Table (customizable) under Available Templates.

8.7.2.1 Click on Settings, select Normalize results, and then choose Factory followed by Quant Standardizations(Extended Set) from the pulldown menu. If using an EHT < 10 kV, selecting Quat Standardizations(5 kV) may also be appropriate.

8.7.3 To view atomic concentrations, click on Edit Columns, and check the Atomic % box.

8.7.4 To compare multiple spectra, choose Comparison of Results from the Available template field.

8.7.4.1 Spectra to be compared may be selected within the pull down menu located in the key of the spectra graph.
8.8 Calculating Unnormalized Elemental Composition

Unnormalized quantification utilizes the measurement of beam current on a calibration sample. Ideal samples include, Si for EHT = 5 kV, Ti for 10 kV and Cu for 20 kV. Si is readily available and may be used for all EHT settings.

8.8.1 Image the sample of interest by performing a complete alignment of the column at an appropriately selected EHT. Ensure a working distance of 5 mm is used.

8.8.1.1 *It is important to ensure the interaction volume of the primary electron beam is completely within a portion of the sample that is homogeneous and representative of the material of interest see section 11.1 and 11.2.*

8.8.2 At the pull down menu located at the top left of the AZtec window, select Optimize.

8.8.2.1 Set the Routine to Beam Measurement and then choose an Element that will be used for optimization.

8.8.2.2 Under Settings, set the Process time to 4 or 5 while making sure the Dead Time is between 40% - 65%. A dead time at this setting will provide the best acquisition rate, however it is still possible to use a dead time lower than 40%.

8.8.2.3 Ensure the magnification of the SEM image field is greater than 1000X and then focus on the calibration sample using the same column conditions that will be used for performing unnormalized quantification on the sample of interest.

8.8.2.4 When the sample is in focus, ensure the working distance is at 5 mm.

8.8.2.5 Click on the Start button and wait for the spectra acquisition to complete.

8.8.2.6 Click Yes to save the beam measurement.

8.8.2.6.1 The stability of the beam can be checked by running successive spectra acquisitions. If two acquisitions run after one another vary by more than +/- 3% file a problem report in Mercury. Ideally, the beam current should not vary by more than +/- 1%.

8.8.3 Immediately move the stage to the sample of interest and acquire a spectra with the desired operation mode. Ensure the working distance is at 5 mm.

8.8.4 Return to the calibration sample and run the optimization as before to check beam stability. As the current deviates from 100% of the original value, more error will be present within the quantization.

8.8.4.1.1 Performing the optimization on a standard that has material identical to that of the sample of interest will reduce error.

8.8.5 Return back to the spectra acquired from the sample of interest and choose the desired operation mode. To see the proposed composition, click on Calculate Composition and select Full Results Table under Available Templates.

8.8.5.1 Click on Settings, make sure Normalize results is not selected, and then choose Factory followed by Quant Standardizations(Extended Set) from the pulldown menu. If you are using an EHT < 10 kV, selecting Quat Standardizations(5 kV) may also be appropriate.

8.8.5.2 To view atomic concentrations, click on Edit Columns, and check the Atomic % box.

8.8.5.3 To compare multiple spectra, choose Comparison of Results from the Available template field.

8.8.5.4 Spectra to be compared may be selected within the pull down menu located in the key of the spectra graph.
8.9 Reporting and Saving Results

8.9.1 After analysis is complete, click on the down arrow next the the Append/Save Report or Report Results icon and choose site Report.

8.9.1.1 Select file Type, Page Size, Location, and File Name.

8.9.1.1.1 Save the report in C:\Users\Oxford\Desktop\User Data\< your mercury login name in lowercase here>.

8.9.1.2 The format of the report to be exported can be modified by clicking on the down arrow next the the Append Report icon and choosing Report Templates.

8.9.2 Always use the Sheeva Drive to copy data onto a USB drive. Never use the any other USB port.

8.9.2.1 Insert a small volume (≤ 4 Gb recommended) and wait 10 seconds for the Sheeva Drive to scan the volume.

8.9.2.2 Copy and paste data onto the USB volume. A shortcut to the networked Sheeva Drive is located on the desktop of the edx computer.

8.9.2.3 Use the Sheeva Manager, shortcut located on the edx computer desktop, to safely remove the USB volume by opening the manager and choosing option 3.

8.10 Ending the Analysis Session and Closing Out the System

8.10.1 Turn on the live video feed of the main system chamber using the SmartSEM software. Inspect the sample and its surroundings and verify it is safe to retract the detector. If there is any uncertainty, discontinue use of the system and contact Nanolab Staff for assistance.

8.10.2 At the bottom right of the AZtec window, click on the detector icon (Control of the EDS detector: Xmax50). Select Position, and then Out.

!!!WARNING!!!

Never pump or vent the main system chamber with the EDS probe inserted (In position). Always retract the probe head to the Out position before pumping or venting.

8.10.3 Verify the probe has stopped moving by inspecting the detector attached to the outside of the SEM chamber.

8.10.4 Save any projects of interest in the AZtec software and then quit the program by closing the AZtec window.

8.10.5 Continue by ending the imaging session on the LEO SEM as normal.

9.0 Troubleshooting Guidelines

9.1 Common Questions and Answers

9.1.1 What are the limiting factors and tradeoffs that must be considered when performing energy dispersive spectroscopy?

There are many important aspects of EDS to consider. One of the most important considerations is the relationship between acceleration voltage (EHT in the case of the leo SEM), ability to generate x-rays, and shape of the interaction volume.

9.1.2 How do I determine what EHT to use?
There are many determining factors. Perhaps the ultimate trade off in EDS is between the size of interaction volume, as determined by EHT, and having sufficient electron energy to generate x-rays from the elements of interest. A rough starting EHT can be determined by multiplying the K-alpha line of an element of interest (or other characteristic lines for that matter) by 2.4. Refer to section 11.1 and 11.2 of this manual for more details.

9.1.3 Why is it important to have the interaction volume of the primary electron beam completely in my material of interest?

Photon production occurs within the interaction volume. By placing the volume completely within the material of interest, x-ray emission is representative of elements in that material (assuming its homogeneous).

If the interaction volume extends beyond the material of interest, x-ray production is likely to come from surrounding materials. In this case, the reported composition of elements will be incorrect because elements from the material of interest and surrounding areas will be reported. Refer to section 11.1 and 11.2 of this manual for more details.

9.1.4 How do I know if the interaction volume of the primary electron beam is completely in the material of interest?

Run a simulation using CASINO (see section 11.1 for instructions on how to run a simulation). After this, run a series of scans with varied EHT and see if the composition changes. As long as the EHT is large enough to generate x-rays from the elements present and the concentration of the elements do not change for different EHTs, the interaction volume is very likely in the material of interest.

9.1.5 What is process time?

Process time is the length of time spent reducing noise from the X-ray signal coming from the EDS detector during processing. The longer the Process time, the lower the noise. If noise is minimized, the resolution of the peak displayed in the spectrum is improved. Increased resolution results in peaks that are narrower and it becomes easier to separate or resolve peaks that are close in energy value. A process time of 4 is generally a good trade off between time to acquire spectra and resolution of peaks. If it is suspected that two peaks are overlapping, a process time of 5 may be used in an effort to resolve them. One example is Pb and S. A process time of 5 shows separate peaks for each element, however a process time of 4 shows nearly a single peak.

9.1.6 What is a k-ratio?

The k-ratio is Castaing’s first approximation (R. Castaing, PhD thesis 1951).

\[
\frac{C_i}{I_i} = \frac{C_{\text{pure}}}{I_{\text{pure}}}
\]

where \(C_i\) is the concentration of element \(i\), \(I_i\) is the x-ray intensity of element \(i\) in the sample, and \(I_{\text{pure}}\) is the x-ray intensity of a pure sample of the element.


During standardless analysis the k-ratio is calculated in the software based on an internal standardless library. For analysis using standards, the k-ratio is measured using
a reference sample with a known composition and then the same conditions are used for the sample of interest.

9.1.7 What is dead time and how do I adjust it?

Deadtime is the percentage of time the pulse processor is unavailable for further counting, i.e. the ratio of input rate to acquisition rate. Note the acquisition time is dependent on the process time. Below is a graph showing optimal acquisition rates for high and low process times in blue and red respectively.

![Graph showing acquisition rates](image)

Based on this graph it is seen that the maximum acquisition rates occur at a dead time of ~ 65%. The deadtime can be adjusted by changing the beam current (EHT, aperture size, high current mode on the leo SEM), and process time (1 - 5 in the AZtec software).

9.1.8 Are there recommended spectra acquisition conditions?

Yes, begin with an Energy Range = 20 keV, Number of Channels = 2048 ch, Process Time = 4, Acquisition Mode = Counts, and Count Limit = 500,000. The use of Pulse Pile Up Correction is highly recommended. For best results, these conditions should be tailored to meet the needs of specific analytical goals and sample types.

9.1.9 When should I use a conductive coating?

Coatings may be used to manage charge when a non-conducting (insulating) sample is under investigation. Much effort should be placed on preparing samples in the most conductive means without the use of a conductive coating. After this, the aperture and EHT should be adjusted on the leo SEM to reduce the beam current. If none of these techniques work, a conductive coating may be considered.

9.1.10 What type of conductive coating should I use?
C, Al, and Cr are candidate coating materials for EDS. C is the material of choice because there are no significant K-peak overlap of materials with $Z = 8 - 20$. Due to the low atomic number, amorphous carbon films are highly transparent to electrons. Carbon films used in EDS are typically 2 - 5 nm thick and exhibit good mechanical and chemical stability. The disadvantage of using C is that the high temperature during evaporation based deposition may cause damage to thermally sensitive samples. A pulse deposition mode is commonly used to reduce thermal load.

Carbon coating systems set up to evaporate sharpened carbon rod that are backed by a turbo pump result in high quality coatings. The use of rod, instead of fiber, allows for higher purity and better control over film thickness typically 1 - 50 nm thick. A turbo pump is used to achieve high vacuum ($< 7.5E^{-5}$ torr) which results in the deposition of an amorphous film, thereby eliminating grain structure in polycrystalline films.

9.1.11 Why is the measurement of length in the leo SmartSEM software more accurate than that of the Oxford AZtec software?

It is not possible to calibrate measurement length on the EDS system over a large range of magnification levels. Use the leo SEM SmartSEM software for all length measurements.

9.1.12 What are the recommended View Settings in Confirm Elements Mode?

Under Settings; Markers, Peak Shapes, and Fitted Spectrum should be selected.

9.1.13 How should I interpret the fit between the acquired and fitted spectra?

Interpretation is based on many factors and is different for each sample. In general, the fit of the highest energy peaks in the acquired spectra to known energy lines of an element should be very good. It is possible to see slight offsets between known spectral lines and acquired peaks at lower energy.

9.1.14 Are there common peaks that are known to be difficult to distinguish because of peak overlap?

Yes. perhaps the most common for ICs is Si, W, and Ta. When inspecting a sample it is best practice to look up energy lines for the elements of interest to determine how close they will be. The detector has a best case resolution of 127 eV (the Nanolab detector actually came in at 125 eV during installation validation). A process time of 5 should be used if peak overlap is of concern.

9.1.15 What should I do if I am trying to detect two peaks that overlap?

The acquired spectra is a product of the (number of atoms) x (probability of producing an x-ray). Increasing the process time to 5 and collecting more counts will help resolve peaks more clearly.

9.1.16 How do I quantify composition of an in-homogenous sample?

An in-homogenous sample (with respect to its 2D surface) must be given special consideration because the software analysis assumes the interaction volume of the primary electron beam is within a homogeneous region of the sample under investigation. Do not use the Analyze operation. Mapping and linescan operations may be used to observe in-homogeneity. Point and ID operation may be used to inspect the given phases throughout an in-homogeneous area of interest. Once the composition of all phases within the region of interest are understood, a field area composition may be calculated by renormalizing them over the field that is scaled by the fractional area of each phase present. For composition analysis in the z-direction, through the thickness of films stacked on a substrate, the layerprobe operation should be considered.
9.1.17 How should I go about analyzing rough surfaces?

It has been reported that significant differences in quantitative analysis occur between samples polished with 1 um diamond and 0.1um alumina polish. Signal peak to background experiences similar attenuation from surface roughness and the ratio between the two will therefore be unaffected. As such, normalized (standardless) analysis making use of a peak to background method should be used on rough samples.

9.1.18 What library of standards should be used when calculating elemental composition?

If using an EHT of 20 kV, the Quat Standardizations(Extended Set) should be used. The Quat Standardizations(5 kV) Set should be considered when using an EHT of < 10 kV.

9.1.19 The Mapping operation is awesome, why would I ever use any other option?

The photon count flux is higher when using other scan modes. Point & ID can be used to examine specific areas and observe differences in composition that would not otherwise be seen in Mapping.

9.1.20 Are there recommended map conditions?

Yes, begin with Resolution = 512, Acquisition Time = Until Stopped, and Pixel Dwell Time (us) = 50.

10.0 Figures & Schematics

Figure 1 - Aztec Software in Point & ID Operation
Figure 2 - Aztec Software in Map Operation
11.0 Appendices

11.1 CASINO Simulations for Primary Electrons; Determination of Sample and Interaction Volume Interactions

CASINO is an open source simulation package that may be used to determine if the interaction volume of the primary electron beam is completely within a targeted area of a sample. Examples of this include interaction volume placement inside a given phase of a non-homogeneous field or a thin film. Below are the steps taken to run a simulation.

11.1.1 Open CASINO by double clicking on the CASINO v2.48 shortcut on the edx computer desktop.

11.1.2 Click on Settings, Modify Sample.

11.1.3 Click on Add Layer twice.

11.1.4 Double click on Undefined (for both the Substrate and thin films created) and add the elemental composition in the Composition field. CASINO will try to determine the density and autofill a value in the Density (g/cm³) field.

11.1.5 Continue to add layers as needed making sure each layer has the elemental composition and density input as was for the substrate.

11.1.6 Set the thickness of each film by double clicking on the Thickness value.

11.1.7 Choose Settings, Set Up Microscope.

11.1.8 Enter the EHT of interest in the Start field. Adjust other parameters as needed and click OK.

11.1.9 To begin the simulation click on the green play icon located in the toolbar at the top of the window.
11.1.10 Once the simulation is finished, it is helpful to rescale the plot such that both the thin film(s) and substrate are shown. To do this, click on the intersecting arrows icon (located to the left of the yellow question mark icon) and input X- and Z-Axis values.

11.1.11 A plot showing the yield of x-rays can be opened by clicking on the folder labeled with the EHT that was used during the simulation, followed by X-Ray. This is located in the explorer/file navigator area on the left side of the CASINO window.

11.1.12 Continue a series of simulations with varied EHT to determine optimal electron interaction volume placement.

11.1.12.1 Remember the EHT must still be large enough to generate x-rays from the element(s) of interest. As such, a tradeoff between placement of the interaction volume and generation of x-rays must be considered.

11.2 Case Study: Elemental Analysis of a 400 nm Si<sub>x</sub>N<sub>y</sub> thin film on Si

11.2.1 It is desired to determine the Si to N ratio of a 400 nm film of Si<sub>x</sub>N<sub>y</sub> on Si. The first consideration made is the EHT suitable to generate x-rays from the elements of interest, i.e. Si and N. This can be quickly estimated by multiplying the principal lines by 2.4:

\[
\begin{align*}
\text{Si K}\alpha & \rightarrow 1.740 \text{ keV} \times 2.4 = 4.176 \text{ keV} \\
\text{Si L}\beta & \rightarrow 0.092 \text{ keV} \times 2.4 = 0.221 \text{ keV} \\
\text{N K}\alpha & \rightarrow 0.392 \text{ keV} \times 2.4 = 0.942 \text{ keV}
\end{align*}
\]

Ideally an EHT greater than 4.176 keV will be used. Note, this estimate of using a factor of 2.4 can be extended to other elements as well.

Next, the location of the interaction volume must be considered. The location should be completely within the material of interest. In this case, CASINO simulations are run to determine what EHT will place the interaction volume completely within the Si<sub>x</sub>N<sub>y</sub> film.

Figure 4. CASINO simulation showing the cross-sectional view of electron trajectory at 20 keV. The sample consists of 400 nm Si<sub>x</sub>N<sub>y</sub> (assumed ratio) on a crystalline Si substrate. The path of primary and backscattered electrons are in blue and red respectively.
As seen in Figure 4, at 20 keV the incident electrons travel through the Si$_x$N$_y$ film, deep into the Si sample. In this case, x-rays are generated by both the film and Si substrate. If the Si:N was determined from a spectrum using this EHT, x-rays from the underlying Si substrate would cause the reported ratio to have an incorrectly high Si content.

![Figure 5: CASINO simulation showing the cross-sectional view of electron trajectory at 10 keV. The sample consists of 400 nm Si$_3$N$_4$ (assumed ratio) on a crystalline Si substrate. The path of primary and backscattered electrons are in blue and red respectively.](image)

Reducing the EHT to 10 keV, as shown in Figure 5, results in a smaller interaction volume, however primary electrons are still able to breach the film and travel into the Si substrate. A lower EHT is needed.
Figure 6. CASINO simulation showing the cross-sectional view of electron trajectory at 5 keV. The sample consists of 400 nm Si$_3$N$_4$ (assumed ratio) on a crystalline Si substrate. The path of primary and backscattered electrons are in blue and red respectively.

At 5 keV in Figure 6, the interaction volume is well placed within the Si$_3$N$_4$ film. A collected spectrum using this EHT will reflect the material composition of the film well (assuming it’s homogeneous) because it is well placed within the film and also large enough x-ray generation. The plots below show the intensity of x-rays as a function of depth into the sample.
Figure 7. Plots of x-ray intensity as a function of sample depth for Si (top) and N (bottom).

The plots in Figure 7 show the generation of x-rays are well within the film of interest. Peak intensity occurs at a depth of ~110 nm and falls off at 400 nm. Now that the simulation is complete, it is understood that an EHT of 5 kV should be used on the leo SEM.

12.0 **Online Test and Superuser Checklist**
12.1 This system does not have a test.
12.2 Superuser Checklist
   12.2.1 The super-user should ensure the new user has working knowledge and experience with the following key points:
   12.2.2 Absolutely no delaminating, flaking, or particle generating materials of any kind are allowed.
   12.2.3 Do not use the system to inspect photoresist, PMMA, spin-on based materials, or other organic based materials. This includes UV baked/stabilized, hard baked, or other fully cured organic materials.
   12.2.4 Materials that out-gas, have poor vacuum compatibility, or can form loose particles are prohibited. If you have any doubt, ask a staff member for approval of your material.
   12.2.5 Materials that are wet, sticky, greasy, or are in a form of a powder are not allowed. This includes: paste and tape of any kind, anything touched without clean gloves, and any material that might have flakes or shows flake-like delamination or particulation.
   12.2.6 All samples must be stable under imaging and analysis conditions. Immediately reduce the EHT if you observe any indication of sample degradation/instability or an uncharacteristic change in system vacuum levels.
12.2.7 Never touch the EDS detector connected to the SEM system. This can cause misalignment and/or damage to the system.

12.2.8 Never pump or vent the main system chamber of the SEM with the EDS probe inserted (In position). Always move the probe head to the Out position before pumping or venting.

12.2.9 Never perform an O$_2$ plasma clean with the EDS probe inserted (In position). O$_2$ cleans may be run when the probe head is retracted in the Out position.

12.2.10 Always turn on the live video feed of the main system chamber using the SmartSEM software and inspect the sample and its surroundings to verify it is safe to insert the detector.

12.2.11 Always position samples at a working distance of 5 mm and obtain an image before inserting the EDS probe (In position) and performing analysis.

12.2.12 Never tilt samples when using the system. All portions of the stage, sample holder, and sample must be completely below a working distance of 5 mm when the probe is inserted (In position).

12.2.13 Qualified members are responsible for all samples placed in the system - including the samples of unqualified users during training and supervised use.