





# Standard Operating Procedure for EDS on SEM3 (Oxford Instruments X-Max-N 150)

Before beginning, ensure you have an active reservation for SEM3 in CoreResearch@Duke


- **Preparing to Acquire EDS Spectra:** Perform these steps in the Apreo User Interface
  - Load and image your sample per the Standard Operating Procedure for SEM3
  - Ensure that the FWD is linked. Set WD = 10.0 mm
  - Select desired Accelerating Voltage and Beam Current (e.g. 20kV and 0.8na), you can adjust later if necessary
  - Adjust focus, brightness, contrast, etc. as needed to achieve desired electron image
- **Acquiring EDS Spectra with AZtec Software:**
  - Click the AZtec icon in the Windows Taskbar 
  - Click New Project -OR- you can opt to open an existing project of yours (if creating a new project, select SMIF-USERS-PROFILE in the pop-up window)
  - Ensure the AZtec software is successfully communicating with the microscope PC:
    - In the bottom of the AZtec UI, there should be readouts for **Mag**, **AV**, and **WD**. These values should match what is displayed in the Apreo UI.
    - The **Mag** readout in the AZtec UI should update when you adjust the magnification knob on the SEM3 control panel.
  -  Confirm that WD = 10.0 mm; visually confirm no portion of the sample extends any closer to the pole piece than 10.0mm via the chamber camera in the Apreo UI
  - Insert the EDS detector:
    - Click “Control of the EDS Detector: EDS1” 
    - Click the **Position** tab then click the **In** button (you can watch the detector being inserted in the Apreo UI chamber view)
    - Close this pop-up window by clicking [x]
  - Pause the live chamber view in the Apreo UI (the chamber lights interfere with the EDS)
  - In the AZtec UI:
    - If adding to an existing project, click New Specimen: 
    - Change the default specimen name (e.g. “Specimen 1”) if desired
    - Edit Summary (Project Notes, Specimen Notes, Site Notes) as desired

- Verify x-ray Counts & Dead Time:
    - Make sure the chamber view is paused in the Apreo UI
    - Un-pause electron imaging in the Apreo UI
    - In the Mini View tab at the right of the AZtec UI:
      - Check the Rate Meter and ensure there are adequate counts (adjust beam current to increase / decrease counts)
      - Verify Dead Time is between 20%-40% as recommended by Oxford (click cog and adjust Process Time to achieve desired Dead Time)
    - Pause electron imaging in the Apreo UI
  - Select desired acquisition mode from drop-down menu: **Point & ID**, **Linescan**, or **Map**
  - Follow the steps in the Navigator bar at the top of the UI. Note the tips section located at the bottom-right of the UI window. These tips can assist you through each step of the Navigator
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- Example Procedure for **Point & ID** mode:

- Click Scan Image in the Navigator:



- Click the settings cog:  and edit **Image Scan Size**, **Dwell Time**, and **Number of Frames** as desired (default settings are typically adequate)

Note: select the secondary electron check box  **SE** (this simply sets brightness and contrast; it has no effect on the actual image you acquire; you must assign the detector in the Apreo UI)

- Close the settings window

- Click Start to initiate Scan Image:




- Wait for image acquisition to complete or press Stop if running continuous scan

- Click Auto Gamma to adjust image:  (and/or adjust brightness & contrast)


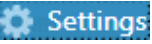



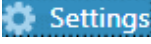

- Click Acquire Spectra in Navigator:




- Click settings cog and set desired scan settings: 



- Select the region of interest (ROI) using one of the tools on the toolbar at the left (Point, Rectangle, Elliptical, Freehand)
  - Spectrum acquisition should begin when you draw the ROI
  - Wait for acquisition to complete
  - Click Confirm Elements in the Navigation bar: 
  - You can now analyze your data, export spectra, save reports, etc.
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○ Example Procedure for **Linescan** mode:



- Click Scan Image in the Navigator: 
- Click the settings cog:  and edit **Image Scan Size**, **Dwell Time**, and **Number of Frames** as desired  
 Note: select the secondary electron check box  **SE** (this simply sets brightness and contrast; it has no effect on the actual image you acquire; you must assign the detector in the Apreo UI)
- Close the settings window
- Click Start to initiate Scan Image: 
- Wait for image acquisition to complete or press Stop if running continuous scan
- Click Auto Gamma to adjust image:  (and/or adjust brightness & contrast)
- Click Acquire Line Data in Navigator: 
- In *Acquire Line Data* box, choose: **Line**, **Truline** or **Quantline** (recommended)
- Click settings cog and set desired scan settings: 
- Draw desired line scan over the electron image (re-adjust settings if desired)
- Click Start to begin spectra acquisition: 
- Wait for the scan to complete (if running a continuous scan, press Stop after desired time elapses)



- Click Construct Linescans in the Navigation bar: 
- You can now analyze your data, export spectra, save reports, etc.

○ Example Procedure for **Map** mode:

- Click Scan Image in the Navigator: 
- Click the settings cog:  and edit **Image Scan Size**, **Dwell Time**, and **Number of Frames** as desired


Note: select the secondary electron check box  **SE** (this simply sets brightness and contrast; it has no effect on the actual image you acquire; you must assign the detector in the Apreo UI)

- Close the settings window
- Click Start to initiate Scan Image: 
- Wait for image acquisition to complete or press Stop if running continuous scan
- Click Auto Gamma to adjust image:  (and/or adjust brightness & contrast)

- Click Acquire Map Data in Navigator: 
- In *Acquire Map Data* box, choose: **Map**, **Trumap** (recommended) or **Quantmap**
- Click settings cog and set desired scan settings: 
- Select area to map:

- Select the region of interest (ROI) using one of the tools on the toolbar at the left (Rectangle, Elliptical, Freehand); Map acquisition should begin when you draw the ROI

– OR –

- Select nothing to map the entire field of view → click Start: 
- Wait for acquisition to complete (if running a continuous scan, press Stop after the desired time elapses)

- Click Construct Maps in the Navigation bar:
- You can now analyze your data, export spectra, save reports, etc.



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- **Ending your EDS session:**

- Retract the EDS detector:

- Click “Control of the EDS Detector: EDS1”
- Click the **Position** tab then click the **Out** button  
(you can watch the detector being retracted in the Apreo UI chamber view)
- Close this pop-up window by clicking [x]
- Visually confirm the detector is fully retracted



The detector must be retracted before venting the SEM to reduce the risk of damage to the detector

- Save your project: File > Save Project
- Close the AZtec software: File > Close AZtec
- Turn off the EDS computer monitor
- You can continue to image your samples in SEM3 -OR- end your session as per the SEM3 Standard Operating Procedure

# Appendix

## **Linescan notes (Line vs TruLine vs QuantLine):**

The data can be processed in several ways:

- Line, also known as Window Integral, obtains the counts in the element energy windows including the background. Line gives a fast and simple representation of the Xray energies.
- TruLine, also known as Filtered Least Squares (FLS), applies further processing. Sometimes the standard X-ray mapping (Line) gives misleading results because some elements have overlapping energy windows. For example, a Titanium linescan might include Barium information. The TruLine option eliminates the problem by comparing the X-ray line series with the expected peak shape for each element. The linescans are corrected for peak overlaps and any false variations due to X-ray background.
- QuantLine further processes the data, showing the atomic or weight percentages of elements at every point on the line. If the "Oxygen by Stoichiometry" option is selected, oxide percentages are also available. The apparent concentration is available in EDSSEM mode.

At a minimum TruLine is recommended, as erroneous results are common in Line mode. See considerations below...

### **Considerations when using QuantLine:**

If you switch linescan modes from QuantLine to Line (the standard linescan) or TruLine and then back to QuantLine, the linescans are instantly regenerated - you do not have to reprocess the data. However, if you switch linescan modes from Line or TruLine to QuantLine, the data are lost. If you want to switch back, you must press the Reprocess button. Any changes to QuantLine settings, binning factor or the element list affect the validity of the results. Therefore, if you make a change, you must press the Reprocess button. The software deletes the existing container and creates a new "SEM Line Quant Results" container.

### **Map notes (Map vs TruMap vs QuantMap):**

The data can be processed in several ways:

- Map, also known as Window Integral Map: Historically, Window Integral Maps have been the standard mode for X-ray maps. These are ideal when there are no overlapping peaks, and you are not looking for trace elements in your specimen (does NOT remove background).
- TruMap is ideal for specimen containing elements with overlapping peaks, and removes false variations due to X-ray background.
- QuantMap allows you to acquire quantitative elemental maps to visualize the spatial distribution of elemental concentrations in the specimen.

At a minimum TruMap is recommended, as erroneous results are common in Map mode. See considerations below...

### **Considerations when using QuantMap:**

QuantMap mode can be prohibitively slow under certain conditions, e.g. when scanning a large area at high resolution. The user must determine if the computation time is acceptable. When you switch mapping modes from QuantMap to the standard mapping or TruMap and then back to QuantMap, the map images are instantly regenerated without having to reprocess the data. In the case of standard maps and TruMaps, the data are lost when you switch mapping mode.