

Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

Drexel Zeiss SUPRA 50VP - Oxford Aztec EDS

Manager: Kate Vanderburgh
Research Instrumentation Specialist

Contact: Drexel University
Bossone 212
3126 Market Street
Philadelphia, PA 19104
Mobile: 908-303-9142
kv434@drexel.edu

Latest Update: 2022-03-18

These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.

Before using the MCC, please review the MCC User Handbook available through our website.

Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

Sample Preparation:

- Mount sample on sample stub using double sided tape (copper or carbon) or a stub with clips.
- Make sure the sample is secure, does not hang over the edge of the holder, and does not have loose particulates.
- Contact Kate Vanderburgh for sample-specific sample prep questions.

SEM Operation:

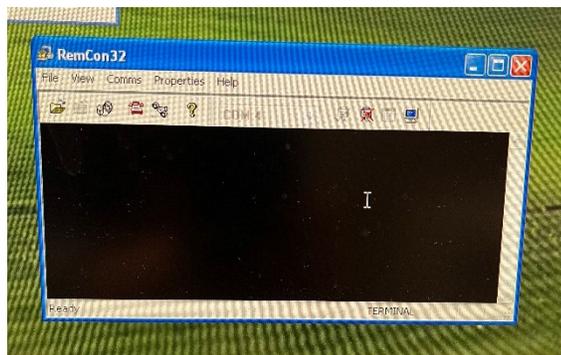
- Complete preliminary checks
- Log into paper logbook and iLab
- Vent chamber, LN2 red ball valve open
- Load samples
- Gently close chamber, make sure door is sealed, LN2 red ball valve closed, pump chamber
- When chamber is under vacuum, turn EHT on
- Set accelerating voltage (kV) to 10 kV or higher (this will be sample dependent)
- Set aperture to 60 μm or higher (sample dependent)
- Set working distance to 15 mm
- Find region of interest
- Perform focus, astigmatism correction, and aperture alignment as you would for imaging

EDS Operation:

1. Open RemCon32. This allows for communication between Smart SEM and Aztec Oxford EDS software. The icon is on the desktop two above that of SmartSEM.



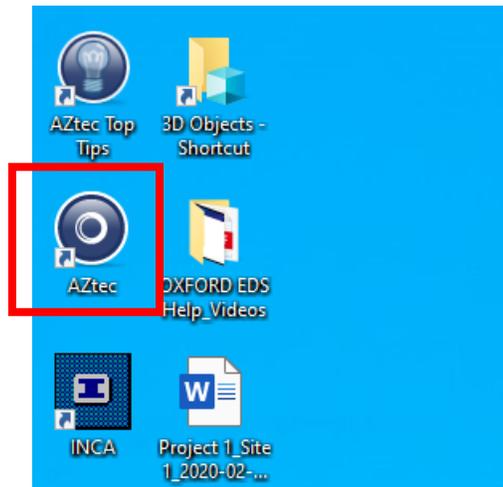
2. A command prompt/communication window should appear as shown below.



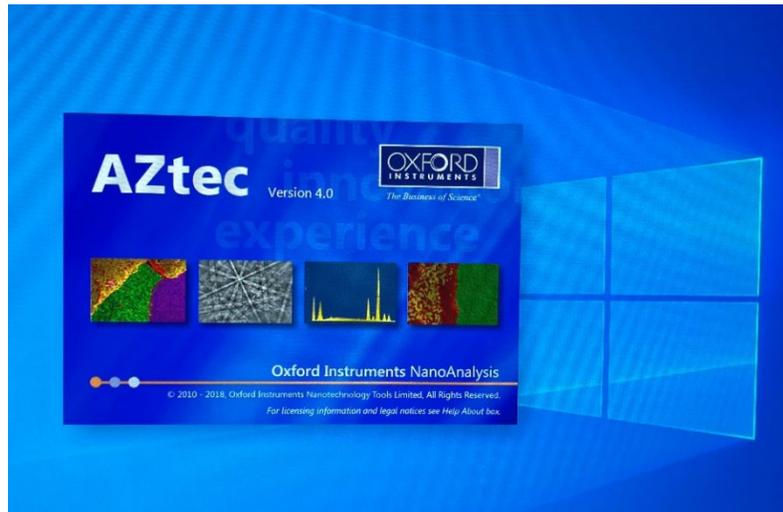
Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

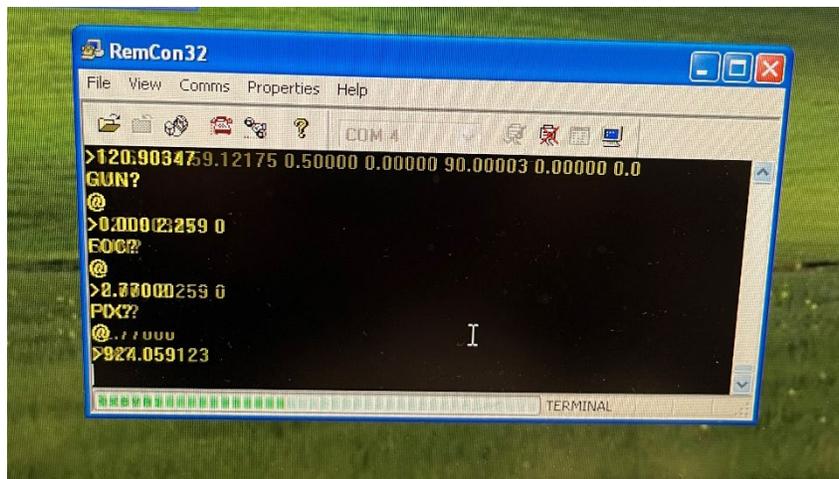
3. On the top monitor, select the Aztec software icon. (you will need to use the other mouse & keyboard)



the Aztec software should open as follows:



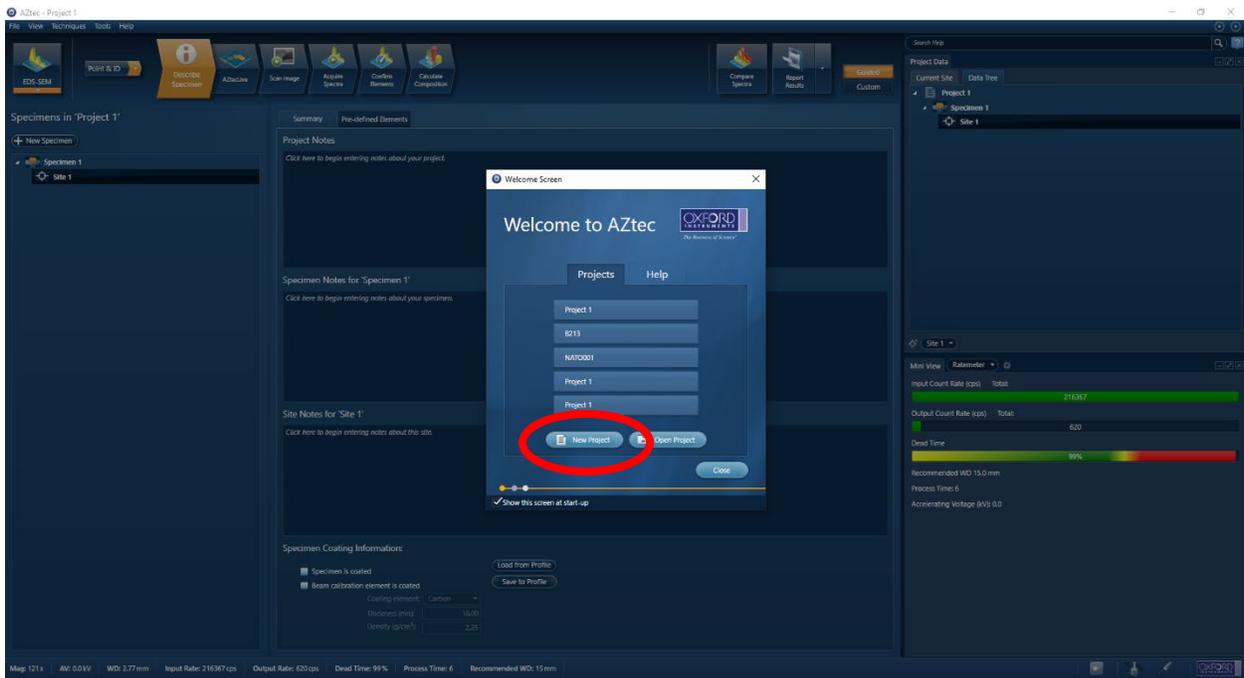
The RemCon communication window should connect as shown below.



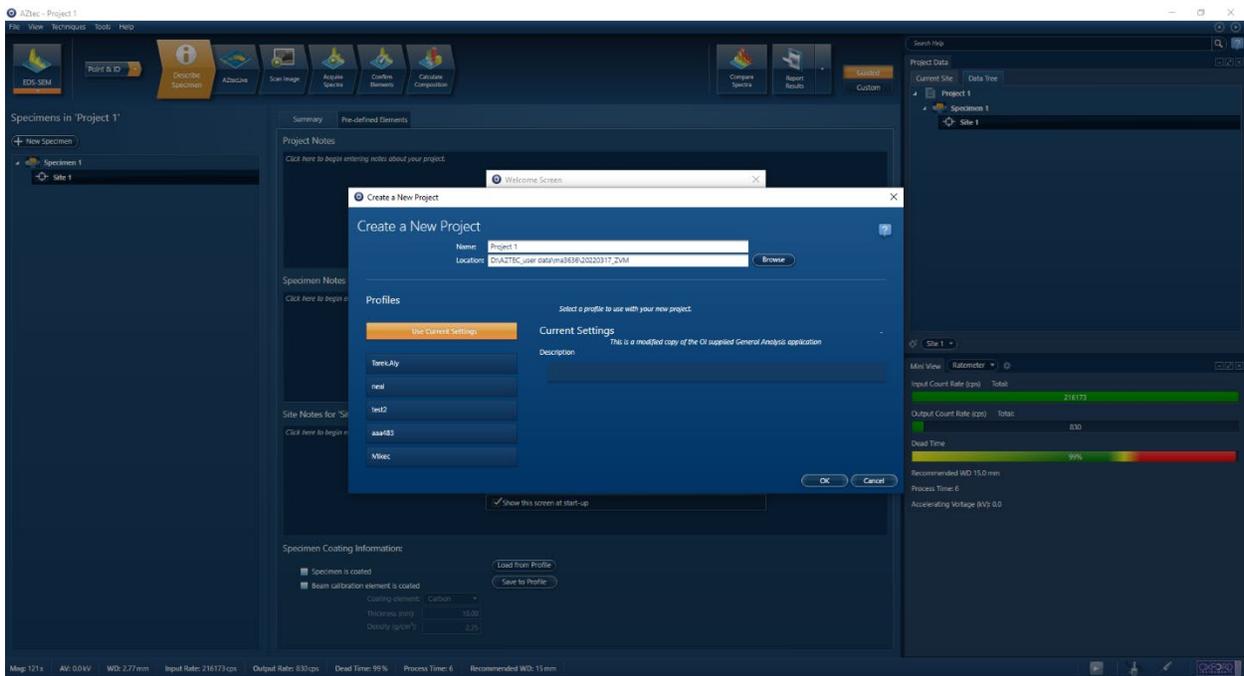
Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

And Aztec should open as follows:



4. Click New Project

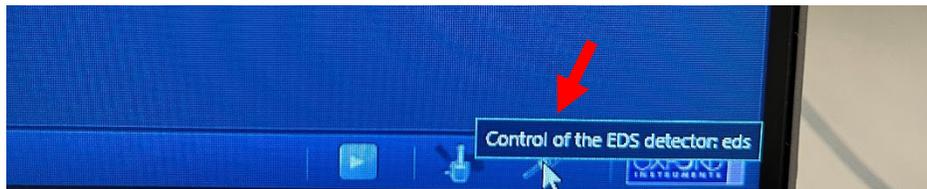


5. Name the project and change the location to your folder. If you are a new user, create your folder under D:\AZTEC_user data

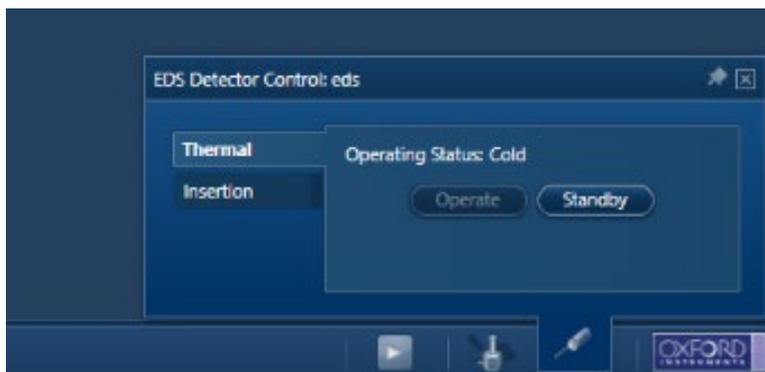
Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

6. Insert the detector by clicking on the Control of the EDS detector:eds (bottom right corner of Aztec software)



This will open the control of the EDS detector. In the thermal tab, the Operate button should be greyed out, indicating that the detector is already in Operate mode. The operating status should be cold.



The blue and green lights on the side of the physical EDS detector should be illuminated solid blue and solid green. If they are blinking, the detector needs ~5-10 minutes to cool down to working temperatures. Detector should always remain in Operate mode. If Standby happens to be selected, the lights on the detector will turn to orange. Select Operate to cool detector down and wait until blue and green lights are solid and Operating status: Cold before inserting detector.



Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

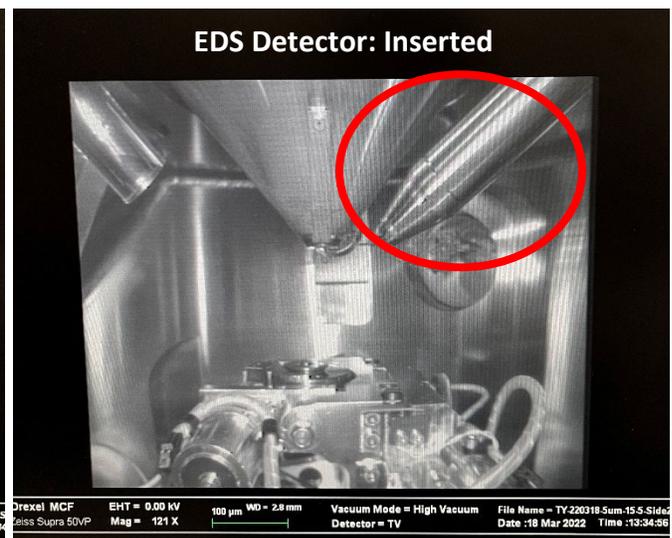
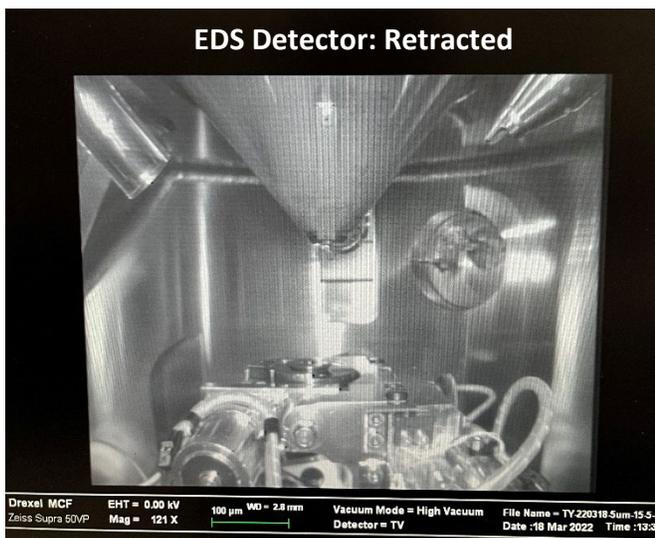
Click on the Insertion tab in the EDS Detector Control:eds.



Ensure that the detector is fully retracted and not moving. Insert the detector by clicking In.



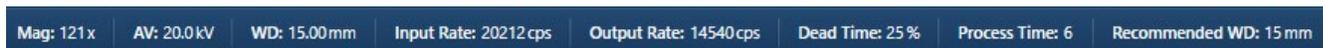
Once inserted, the State: Fully Inserted and Activity: Not Moving. In the Chamberscope view on SmartSEM:



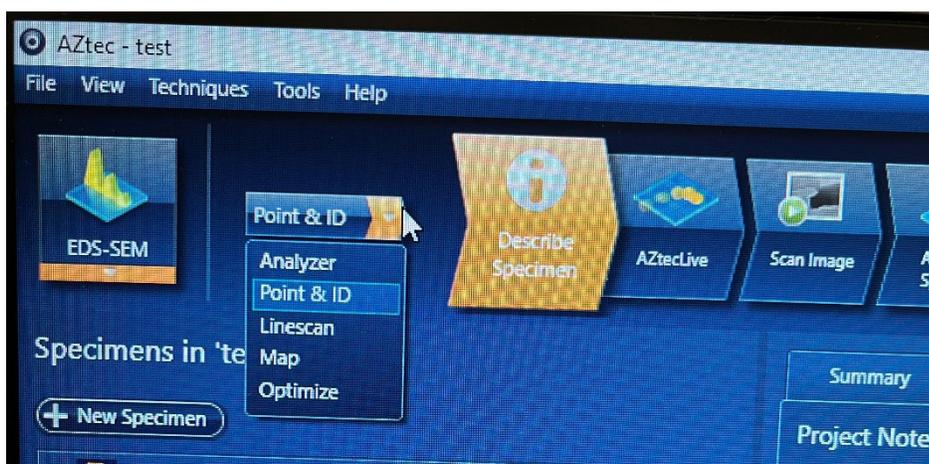
Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

7. The status bar in the bottom left corner of Aztec should show the selected magnification, accelerating voltage, working distance, input and output rates, and dead time.

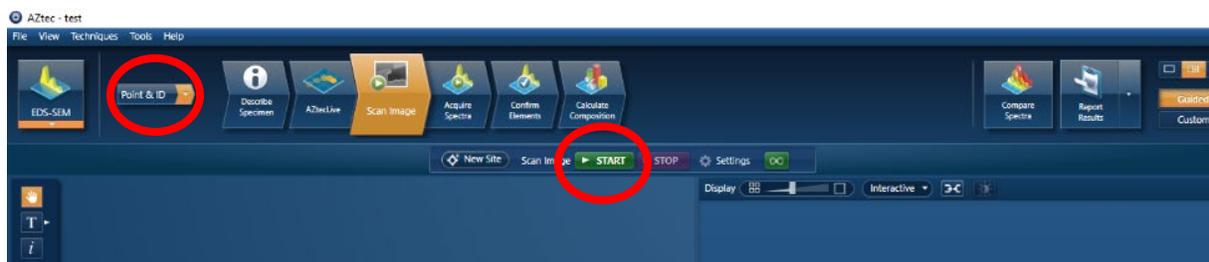


- Input rate: rate at which x-rays are collected by the detector
 - Output rate: rate at which system is processing counts
 - Dead time: ratio between input and output rates. Should be ~10 – 50% and represents the time spent processing the counts.
 - Process time: amount of time to reduce signal noise from incoming x-rays into the detector. Longer process time, longer acquisition, lower the noise, more dead time.
8. In the drop-down menu in the top left of Aztec, you have the option to select Analyzer (data analysis), Point & ID (spectrum acquisition), Linescan, Map (spectrum and elemental mapping), and Optimize.



Point & ID Data Acquisition:

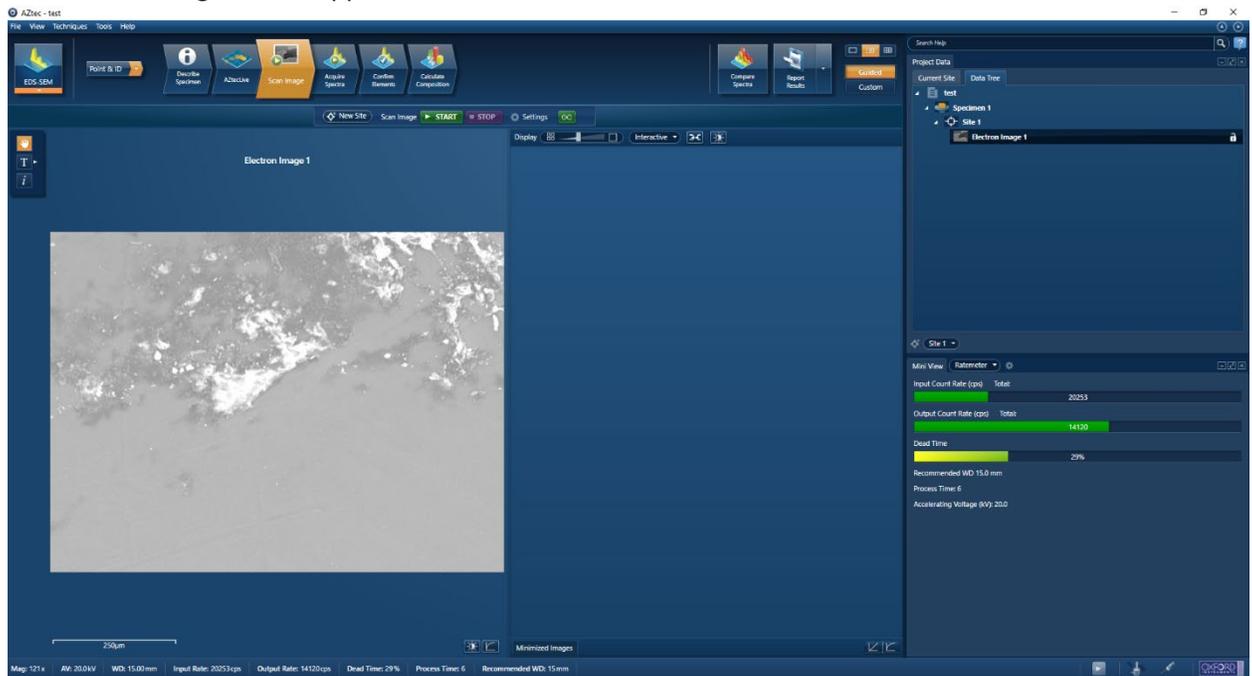
1. In the top-left drop-down menu, select Point & ID. First select the Scan Image window. Then select START to acquire an SEM image in Aztec.



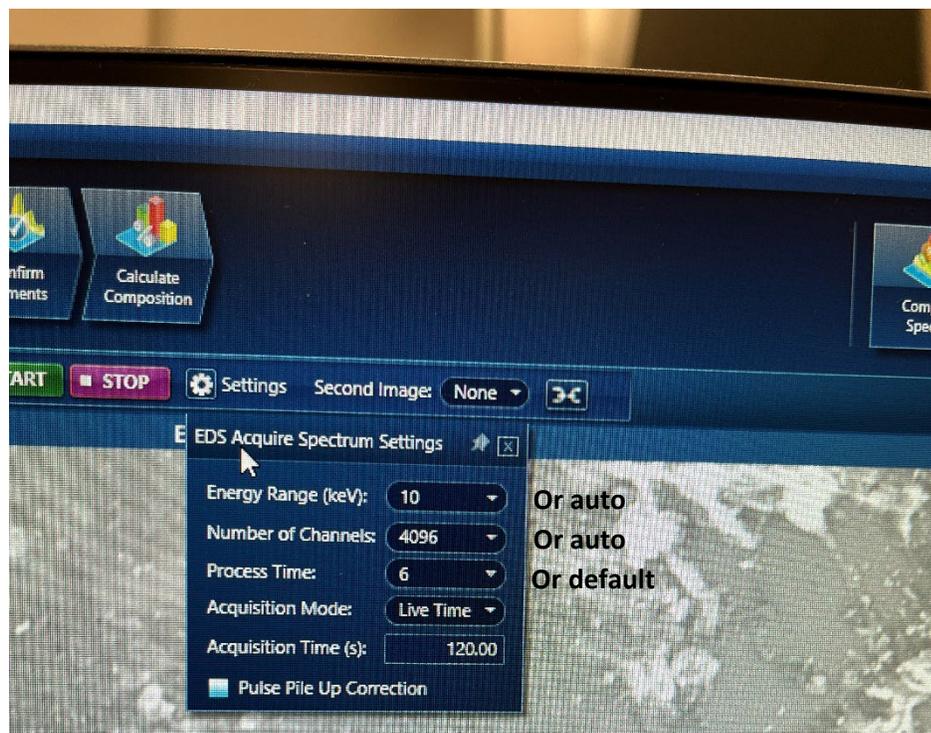
Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

An electron image should appear



2. Select the Acquire Spectra tab. Select the Settings Icon to select the spectrum acquisition settings.

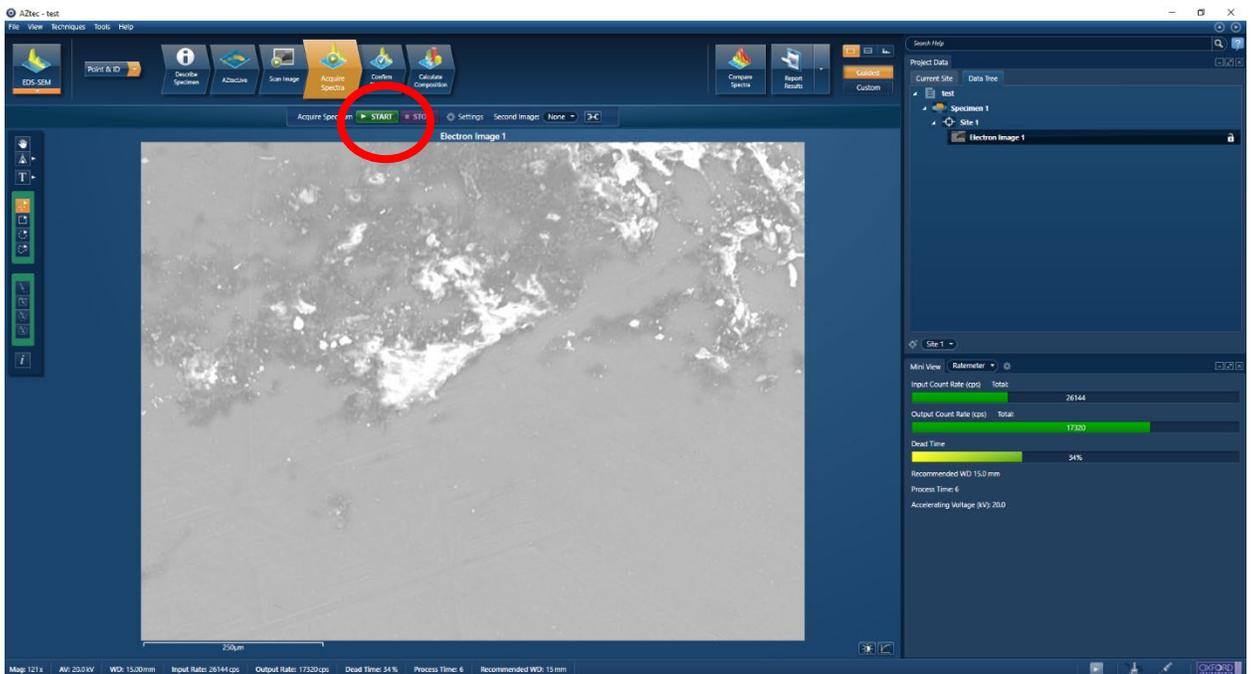


Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

- Energy range: select energy range entered in SmartSEM or auto will automatically set to appropriate value.
- Number of channels: depends on energy range. select channel to display spectrum or select auto which will take into account selected energy range and choose suitable number of channels.
- Process time: amount of time to reduce signal noise from incoming x-rays into the detector. Longer process time, longer acquisition, lower the noise, more dead time.
- Acquisition mode: Auto, Live Time, counts. Auto will acquire data until sufficient counts are collected for quantification. Live Time will continue until you decide to stop data collection. Counts allows you to enter the time for data collection.

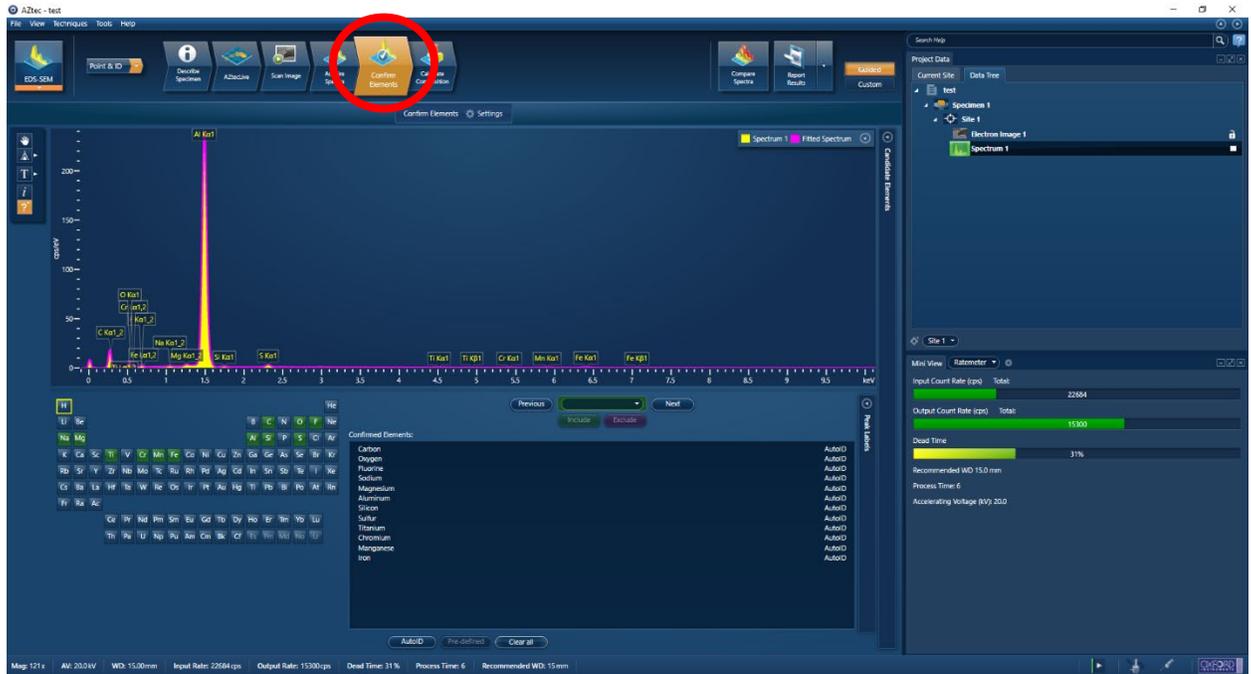
Select START to begin data acquisition.



Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

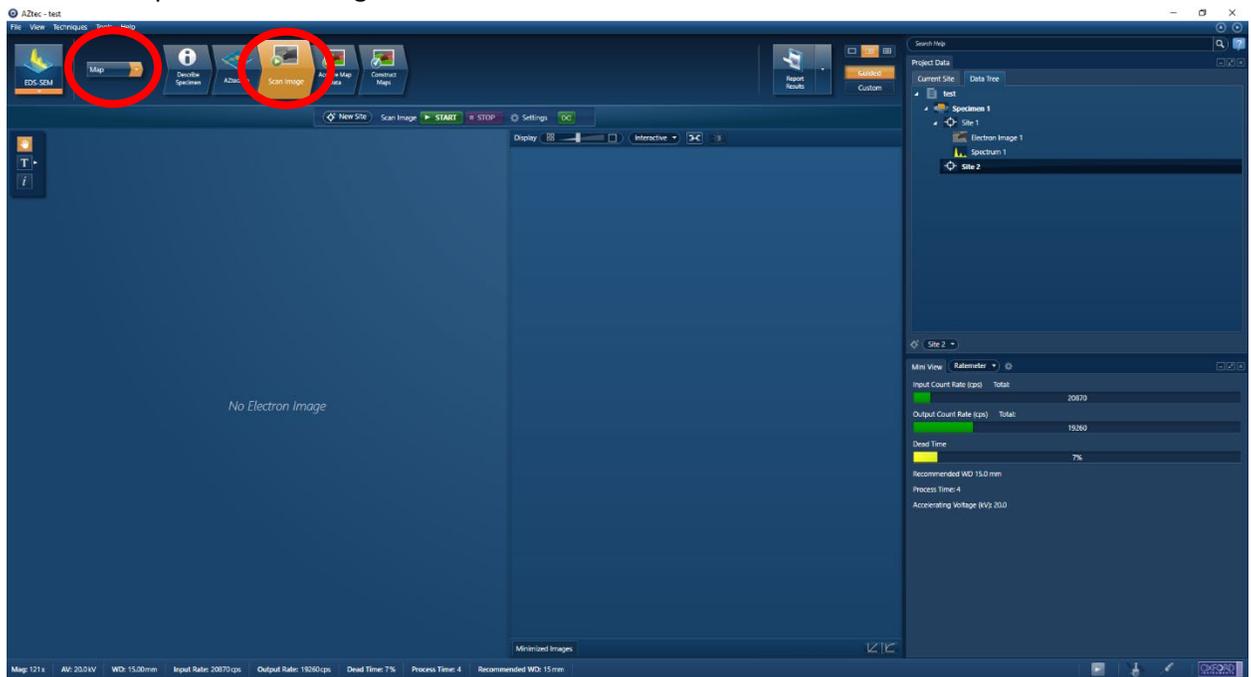
3. Go to the Confirm Elements tab



Include/Exclude elements using the periodic table.

Mapping Data Acquisition:

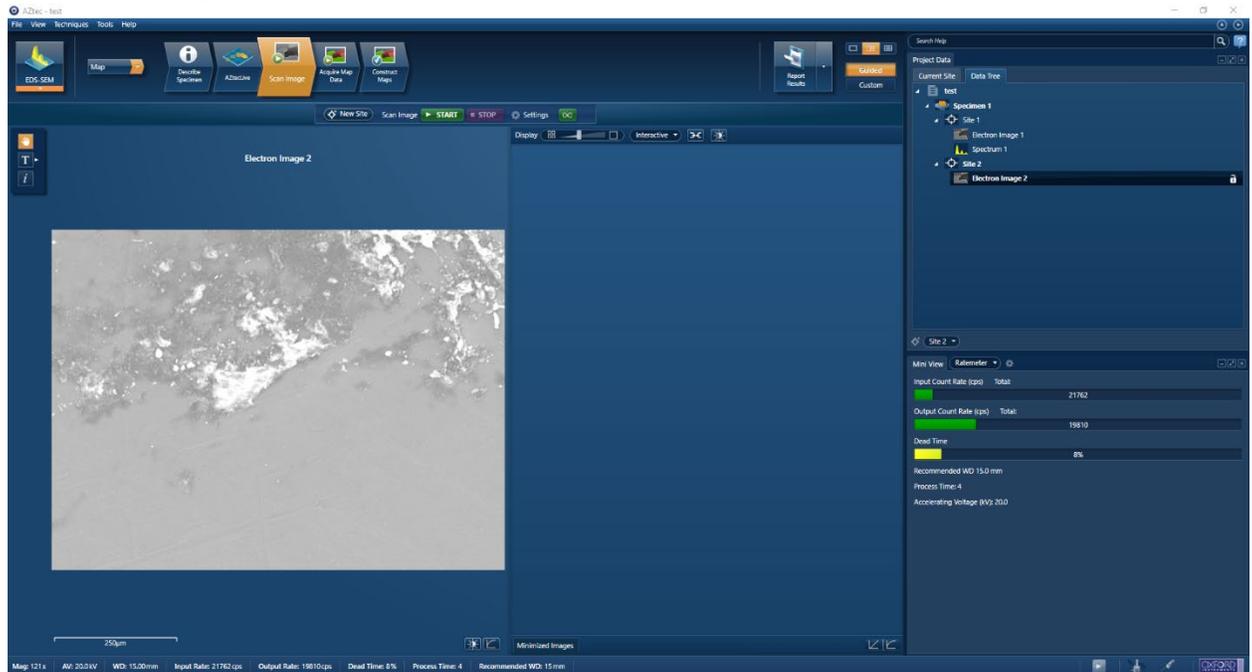
1. In the top-left drop-down menu, select Map. First select the Scan Image window. Then select START to acquire an SEM image in Aztec.



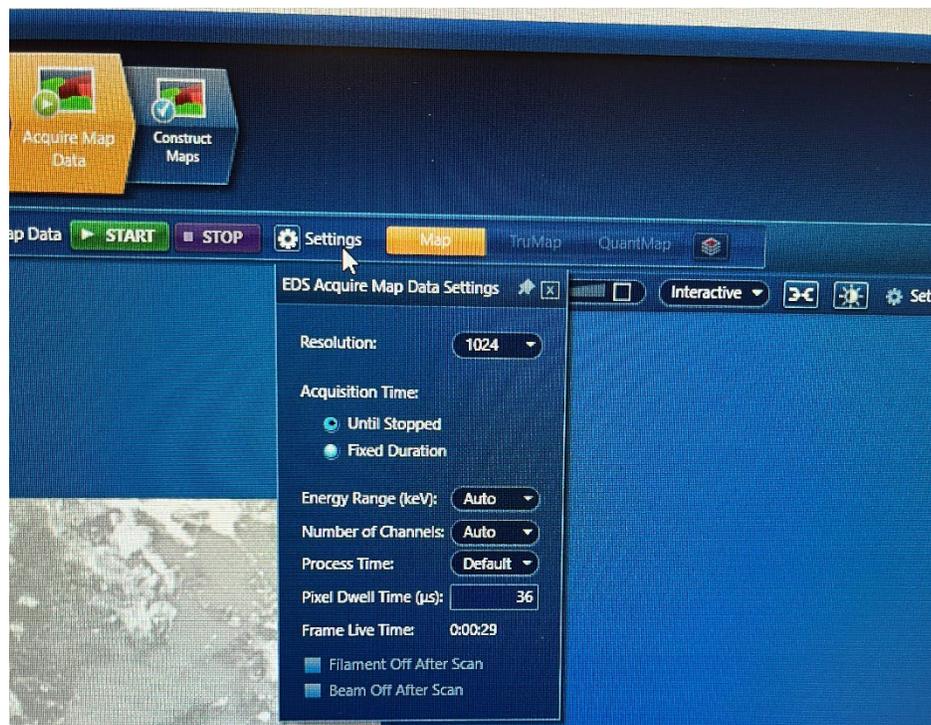
Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

An electron image should appear



2. Select the Acquire Map Data tab. Select the Settings Icon to select the spectrum acquisition settings.

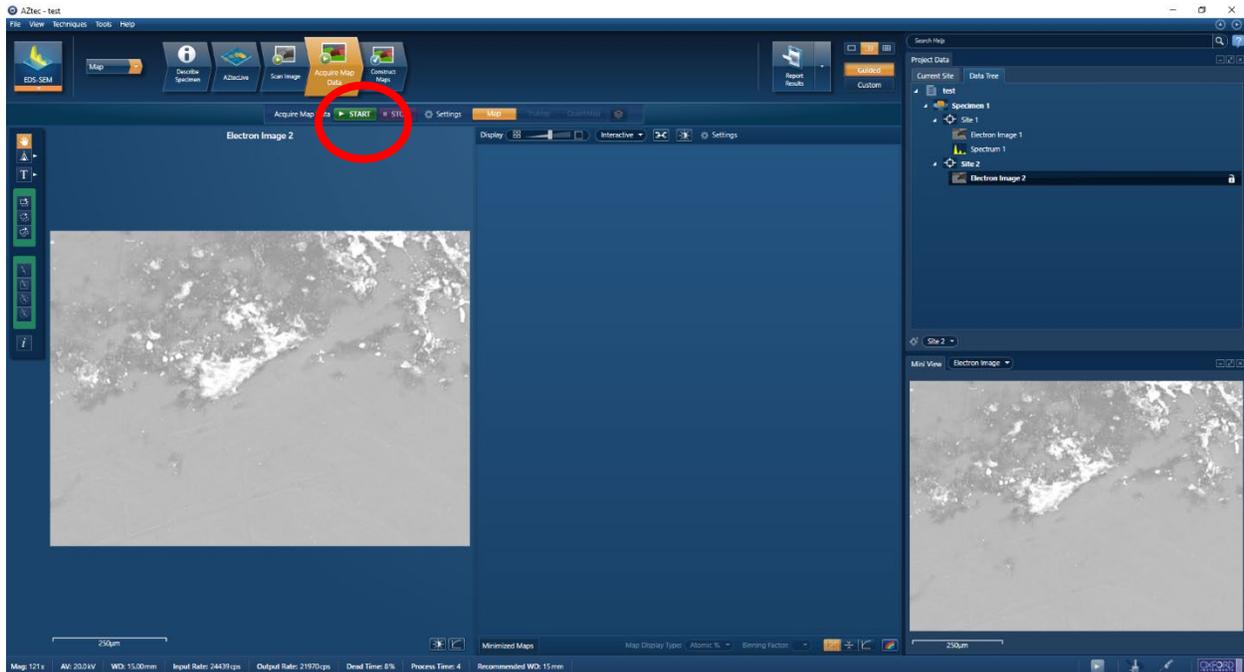


Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

- Resolution: 1024
- Acquisition time: until stopped or fixed duration. Fixed duration – enter time for data collection.
- Energy range: select energy range entered in SmartSEM or auto will automatically set to appropriate value.
- Number of channels: depends on energy range. select channel to display spectrum or select auto which will take into account selected energy range and choose suitable number of channels.
- Process time: amount of time to reduce signal noise from incoming x-rays into the detector. Longer process time, longer acquisition, lower the noise, more dead time. Set to default.
- Pixel dwell time: 36 μ s

Select START to begin data acquisition.



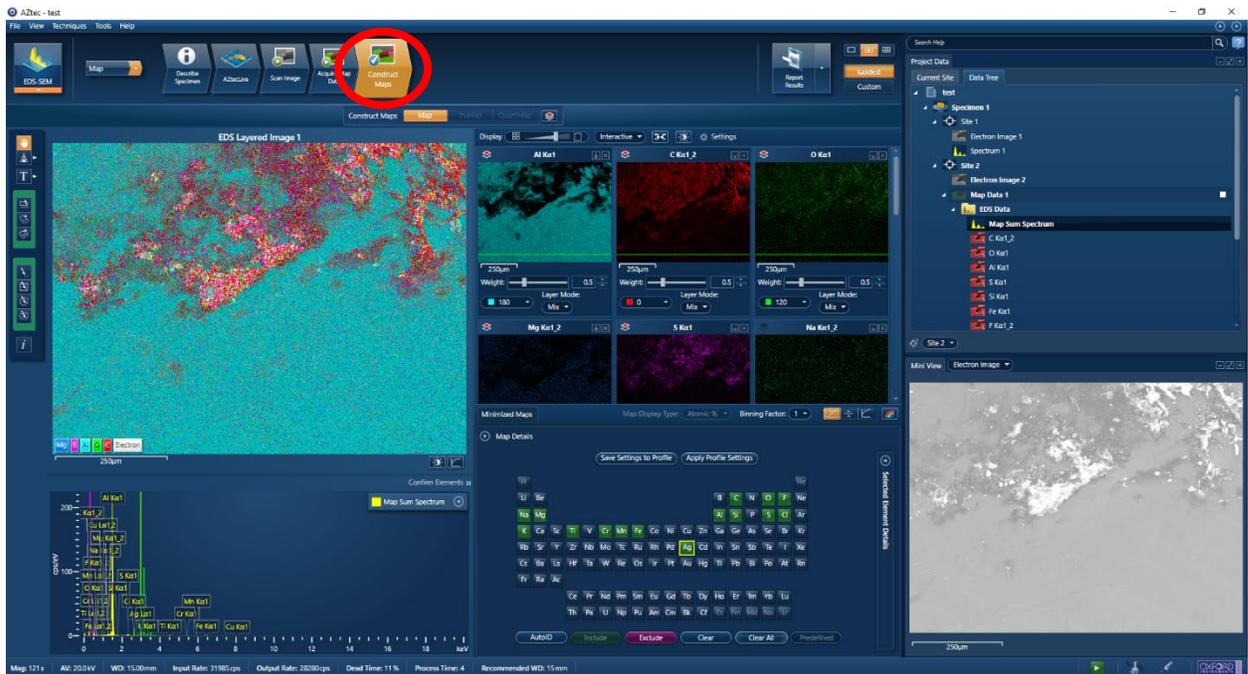
Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

Map acquisition should begin



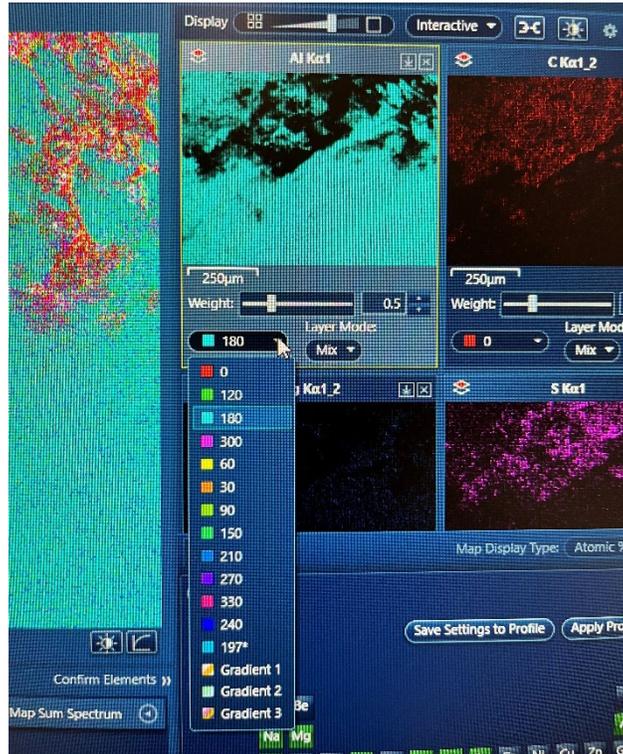
3. Select the Construct Maps tab to include/exclude elements and view the spectra



Materials Characterization Core at Drexel University

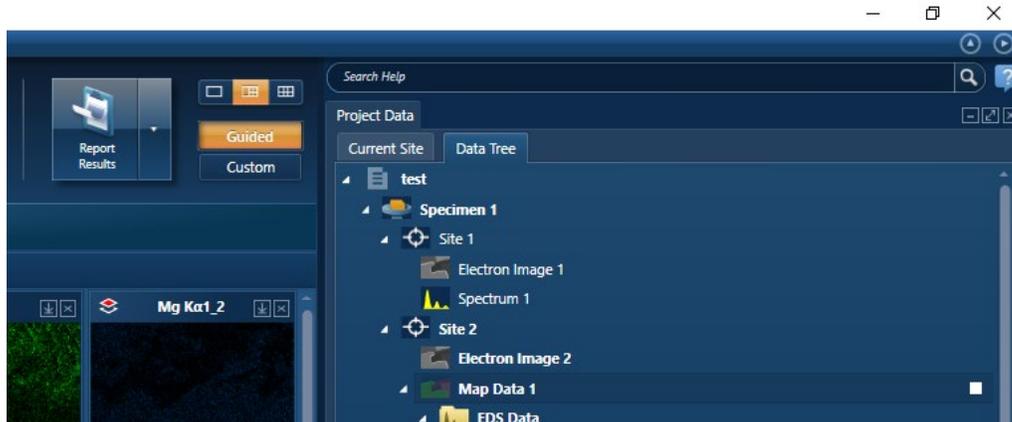
Training Library – Standard Operating Procedures

4. Change map colors by element



Saving Data:

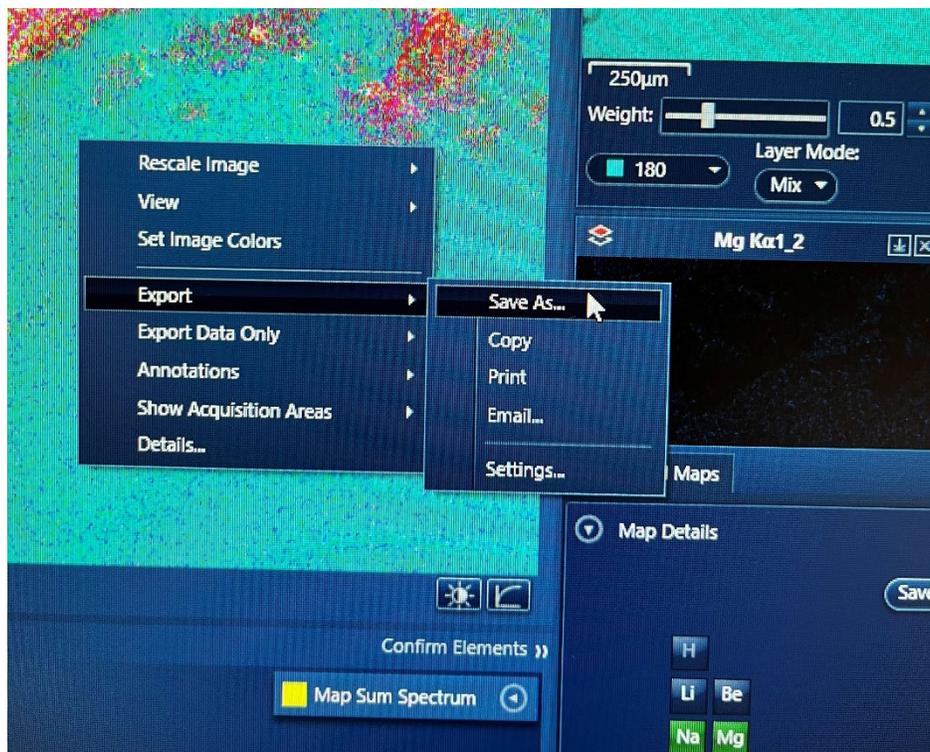
1. In the data tree, select desired site. Click Report Results to generate a Microsoft Word doc with a summary of the results from that site.



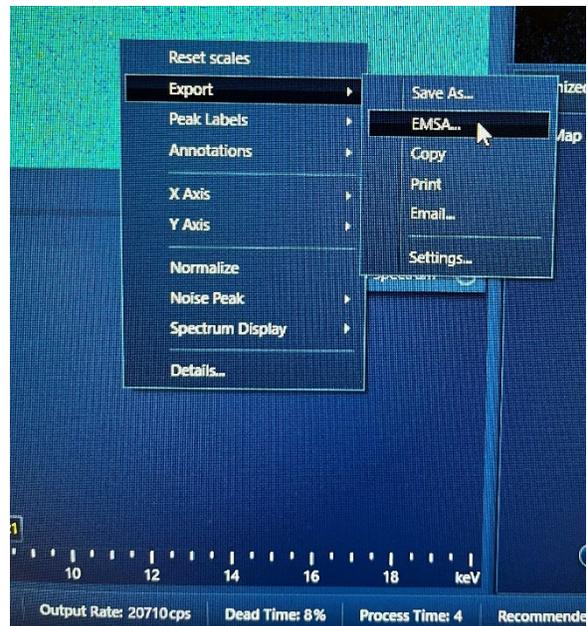
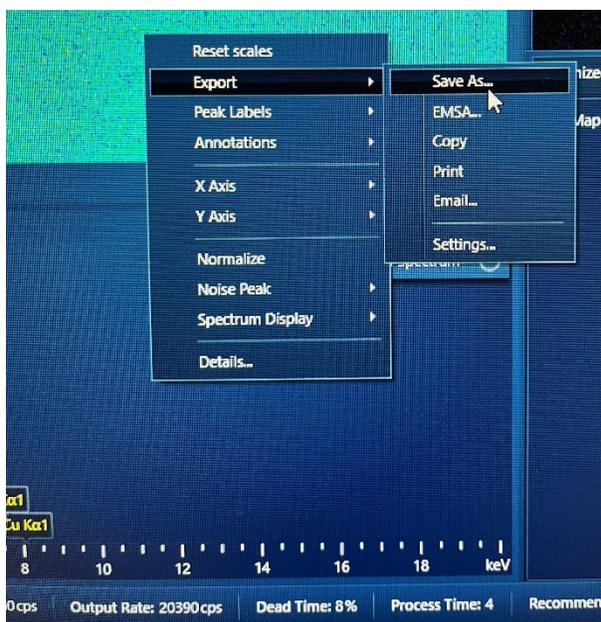
Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

2. Another option, right click < Export < Save As...



3. Make sure to export spectra of elements for each map as an image and .txt To export an image of the spectra, right click < Export < Save As... and to export the .txt right click < export < EMSA...

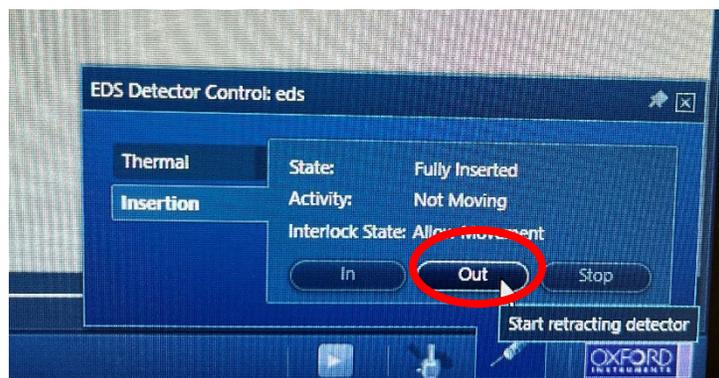


Shutdown:

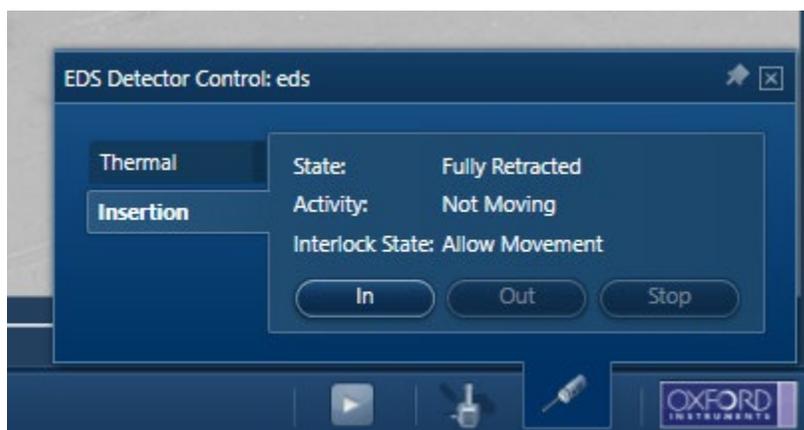
1. When you are finished, make sure no scans are in progress. The status icon should be greyed out.



2. Retract the detector. In the bottom right corner of Aztec select the EDS Detector Control and select the insertion tab. The detector should be Fully Inserted and Not moving. Select the Out option to start retracting detector. In the Chamberscope view on SmartSEM, confirm that the detector is retracted.



Once retracted, check that the EDS Detector Control State: Fully Retracted, Activity: Not Moving. In the Chamberscope view on SmartSEM, confirm that the detector is retracted.



3. Make sure to save your project.
4. Exit out of Aztec.
5. Close RemCon32.
6. Continue with standard SEM shutdown procedure.
 - Aperture: 30 μm
 - Scan speed: 1
 - Line averaging: N = 3
 - Store resolution: 1024x768 pixels

Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

- EHT off
- TV mode
- Vent (open LN2 red ball valve)
- Remove samples
- Pump – wait until vac check mark is green before logging out of SmartSEM (close LN2 red ball valve)
- Log out of SmartSEM software
- Log out of iLab
- Sign out of paper log book

This SOP serves as a guide for basic EDS detector operation and data acquisition. For questions regarding SEM EDS analysis or further analytical capabilities, please look to the Oxford Aztec EDS manual (red binder) or contact Kate Vanderburgh.

Kate Vanderburgh, Ph.D.
Drexel University
212 Bossone Research Center
3126 Market Street
Philadelphia, PA 19104
kv434@drexel.edu
(908) 303-9142