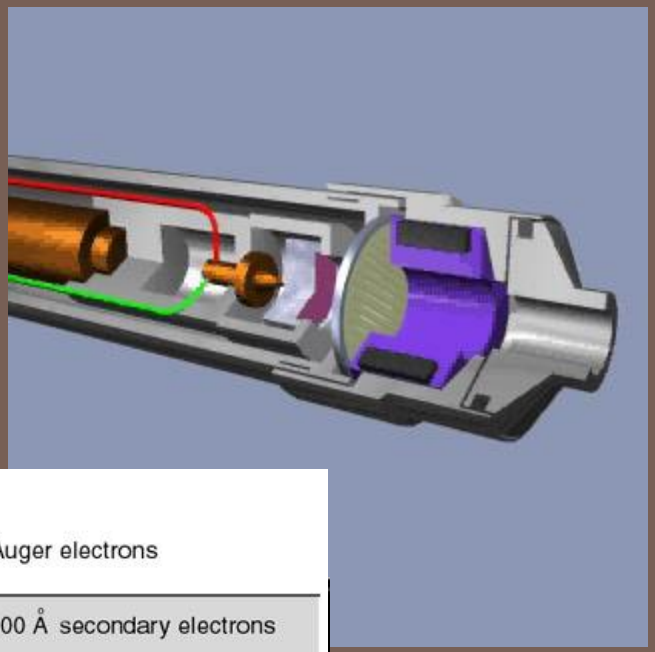
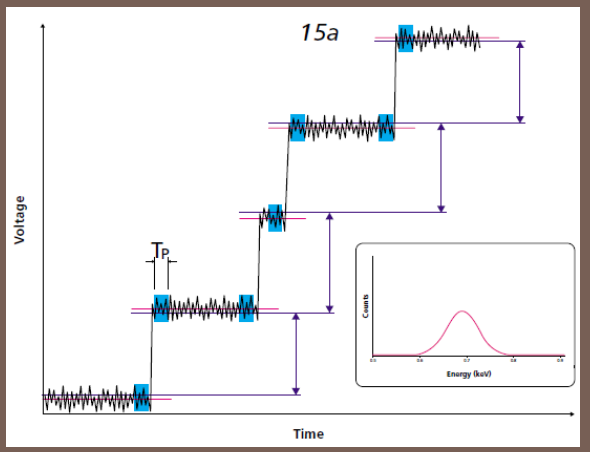
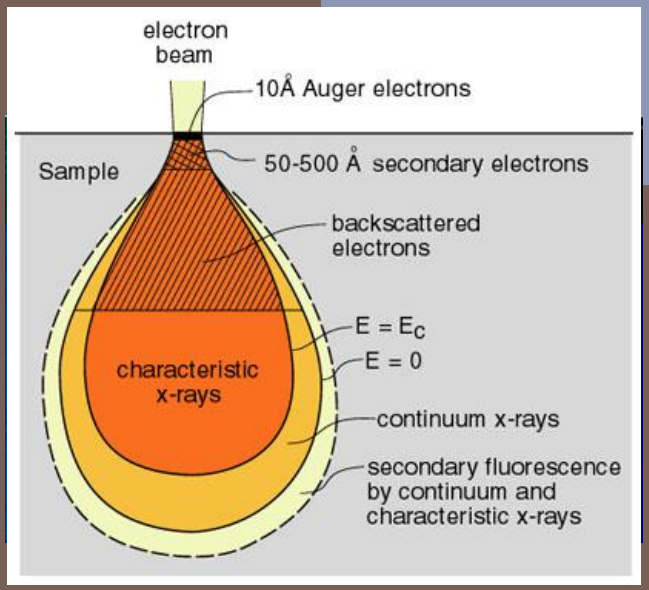
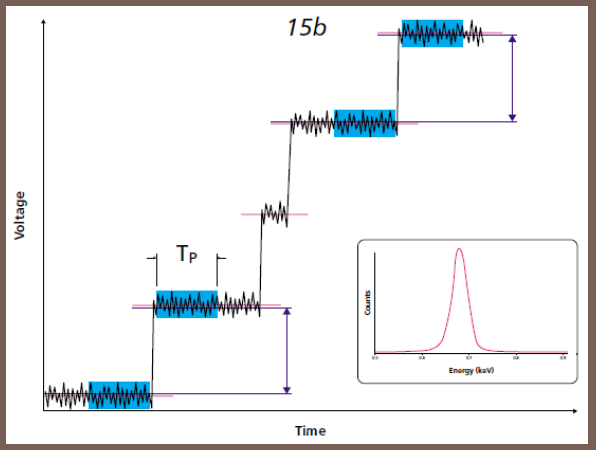






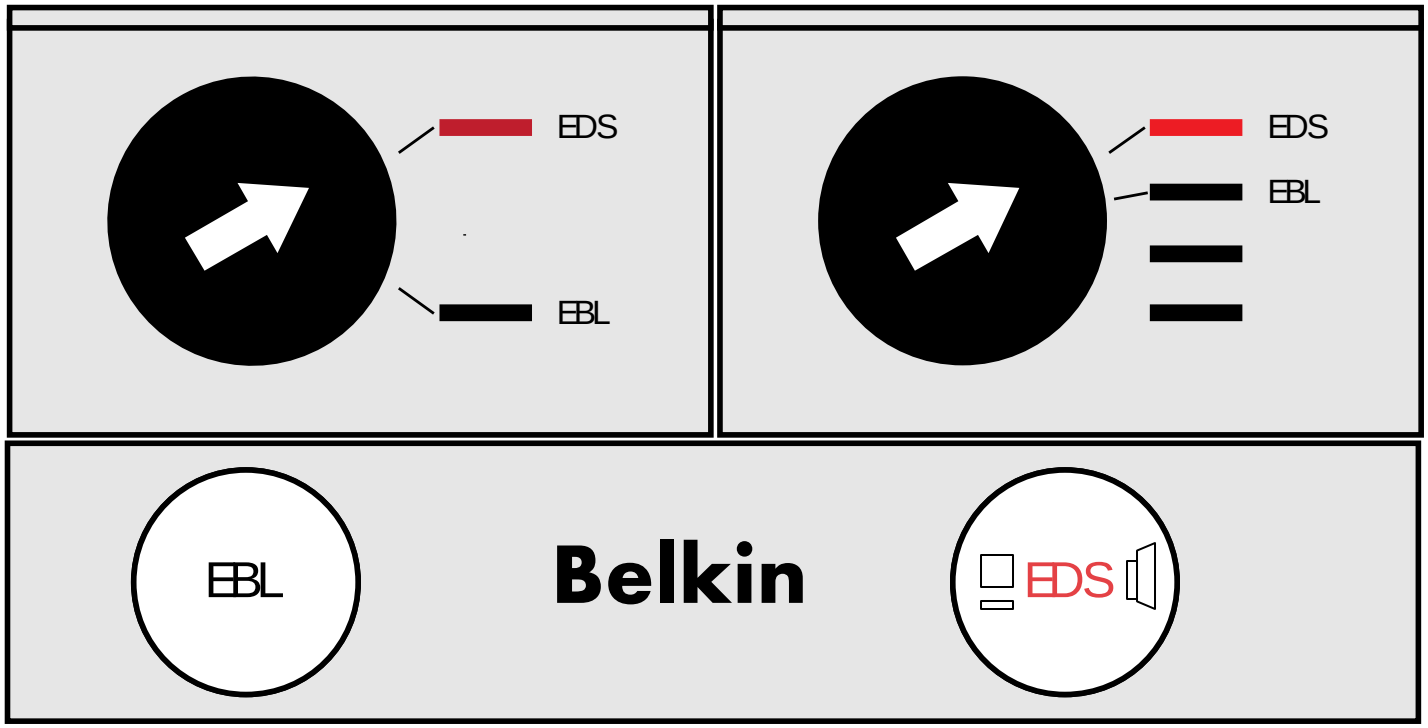


SIRION EDS

Rev. 0814

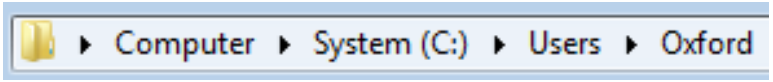
Enable the Oxford EDS

- Enable the Oxford PC under SEM Tools in Coral
- Switch KVM and switch boxes to EDS/Oxford

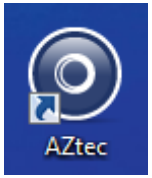


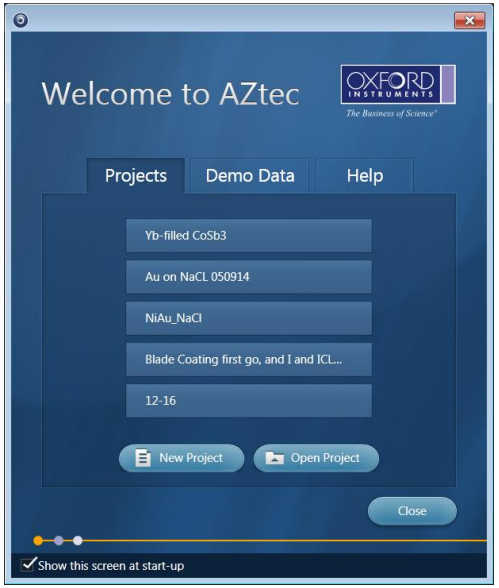
Start an Aztec Project

- Launch Aztec software.
- Create a new project or open an existing one.
- User projects are kept on the local drive in the Oxford Users folder.



- Creating a new project will automatically create a folder of the same name in your folder.
- The projects will auto-save each time you collect a new spectrum.

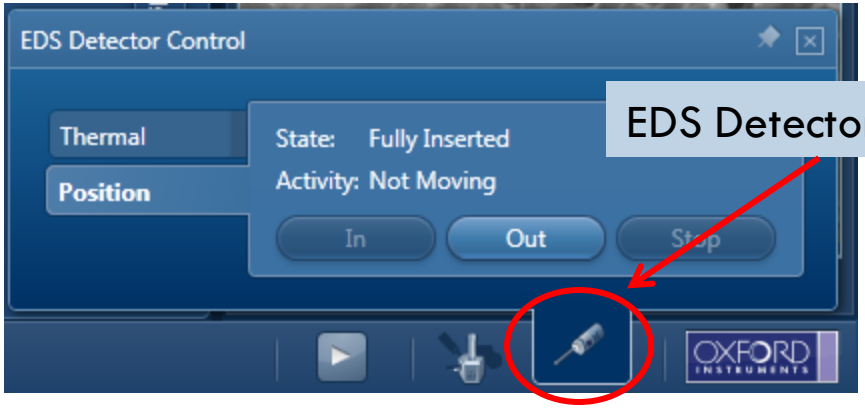




Prepare the Chamber

The EDS detector should be “Out” when not in use.

- Raise the sample to 5mm working distance.
- Use the control window to insert the detector.

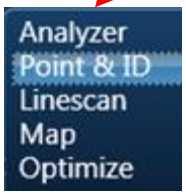


- Turn off the CCD camera at the toggle switch

  The CCD camera emits IR light which excites the x-ray detector.

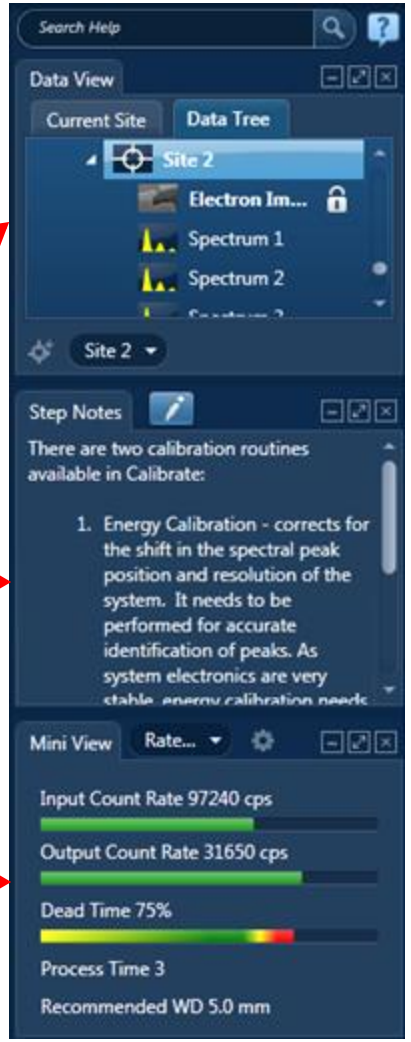
Navigation & Support Panels

Navigation Panel



Step-Through Guide changes for each mode

Support Panel



Data Tree organizes samples, sites, images and spectral data

Step Notes give more detailed info about the operation

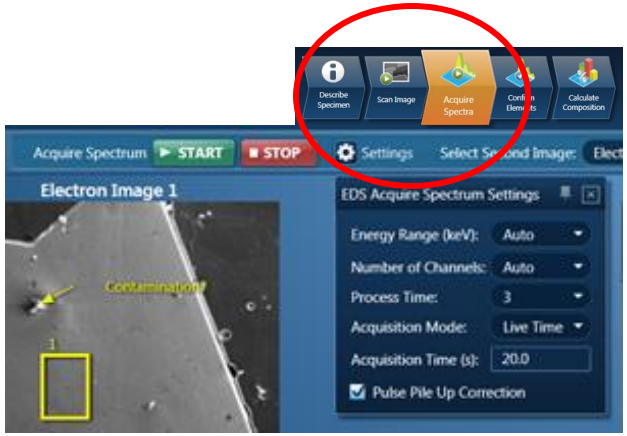
Real-time X-ray signal processing shows in the Ratemeter

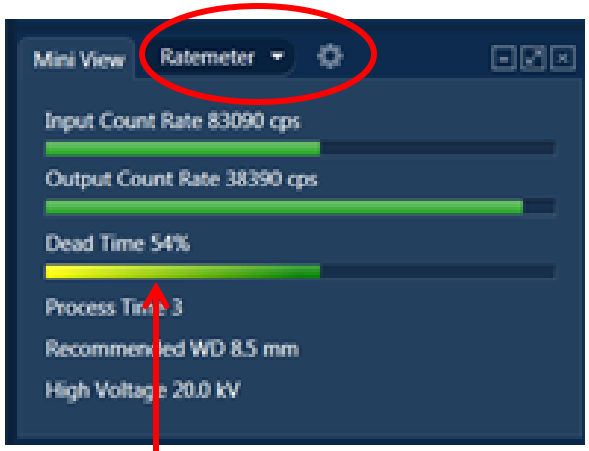
Operating Modes:

- Point & ID gives the most accurate element quant
- Linescan is a 1D spatial analysis
- Maps are 2D spatial analyses

Select kV and Spot Size

- Set the SEM beam energy at 2X the highest energy characteristic x-ray you want to quantify.
- The SEM is aligned at 5, 10, 20, and 30kV. Choosing an aligned beam will generally make operation easier.
- In Acquire Settings or Rate Meter Settings set Process Time 3 or 4.



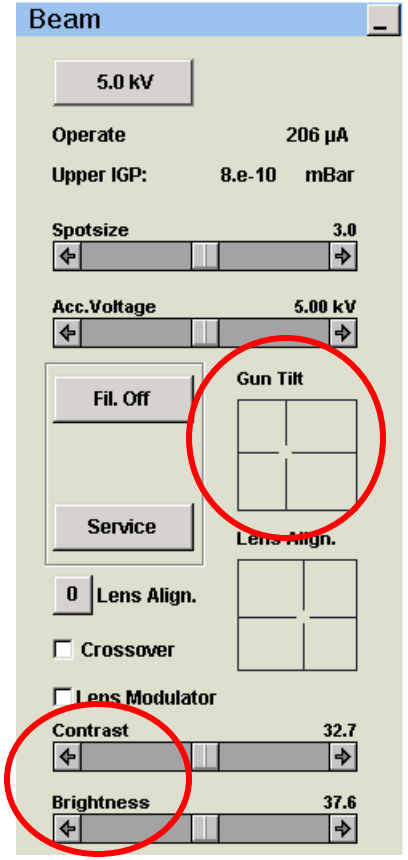


- Adjust the SEM spot size to achieve 25 – 50 % Dead Time.
- Spot 6 and 7 may need a Gun Tilt adjustment for maximum signal (next page.)

Optimize Beam

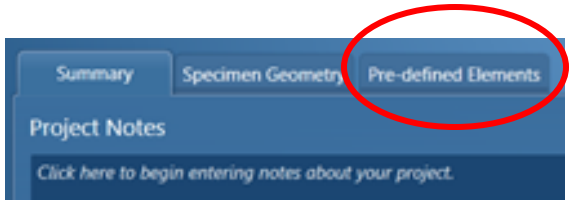
To maximize x-ray output using spot 6 or 7, optimize the "Gun Tilt" in Microscope Control "Beam" submenu.

1. Adjust contrast/brightness to see the image.
2. Left-click in the Gun Tilt box and adjust it to the brightest position.
3. Reduce contrast to correct over-saturation.
4. Repeat Gun Tilt and contrast/brightness.
5. Focus and stigmate for the best final image resolution.

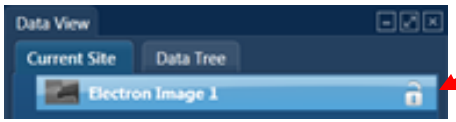


Describe Specimen

- Right-click on Specimen 1 to rename.
- Any Project or Specimen Notes you add will show on the '.doc' reports you generate for the project.
- Because of carbon contamination in the chamber, the default "Specimen Coating Info." is set to exclude C from quant.
- Elements of particular interest can be added to the AutoID in the "Pre-defined Elements" tab.



Scan Image



- Open the Image Settings menu.
- Select the Scan Size (1025 pix suggested).
- Set Dwell Time (5 us works for many samples).
- Select the correct detector SE or BSE (note BSE must be installed by a staff person at the beginning of the SEM session).
- Close settings and click “Start” to acquire.
- To protect an image select the padlock icon and it will not be overwritten by new images at the same site.

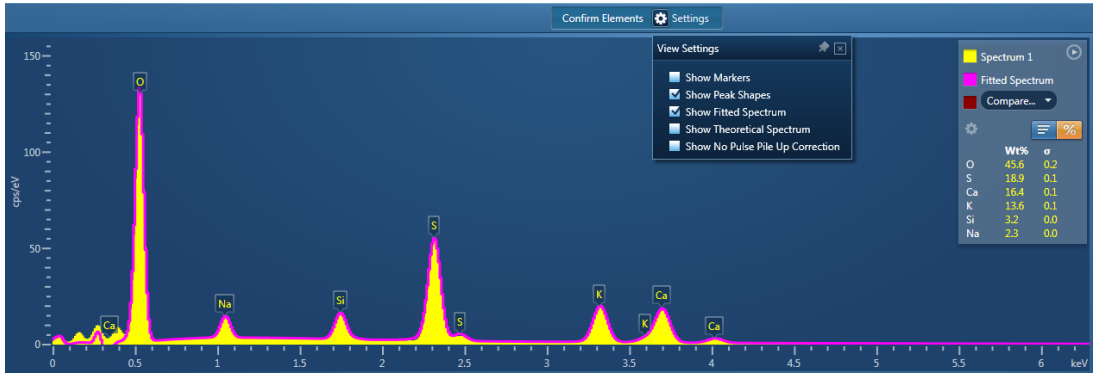
Acquire Spectra

- Define a region of interest on the image with the tool bar at left or use the entire frame.
  - Options are spot, square, circle, and freehand area
  - Do not define a region to scan the entire imaged area
- Open the Spectrum Settings to switch Acquisition Mode between Live Time and Counts.
  - For good statistical significance chose at least 300,000 Counts
  - 20 sec Live Time also yields good results on most samples
- Click "Start."

Confirm Elements

Point & ID only

- Review the element IDs and add/remove elements to achieve the best agreement between the spectral data in yellow and the fitted spectrum in magenta.



PAN

ANNOTATE

INFO

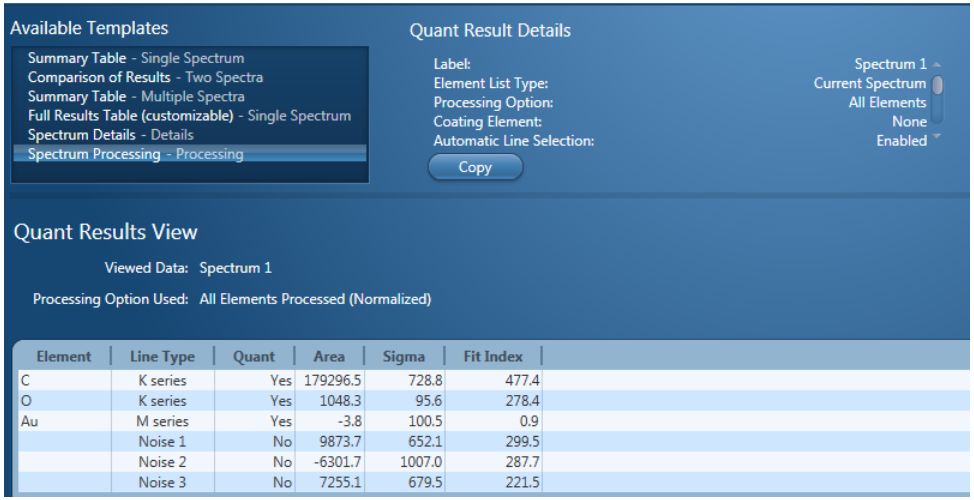
SUGGEST ELEMENT

Calculate Composition

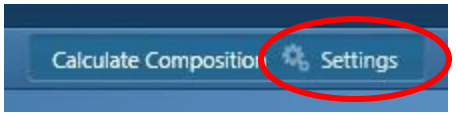
Point & ID only

- Calculate Composition after element IDs are confirmed.
- Use available templates to view quant results, spectrum details, and compare multiple spectra.
- Threshold for AutoID is normally set to 3x the sigma value (~1% by wt.)

To report composition, first arrange data view using a template and available options. Then generate the corresponding Word or Excel report (more pg.18)

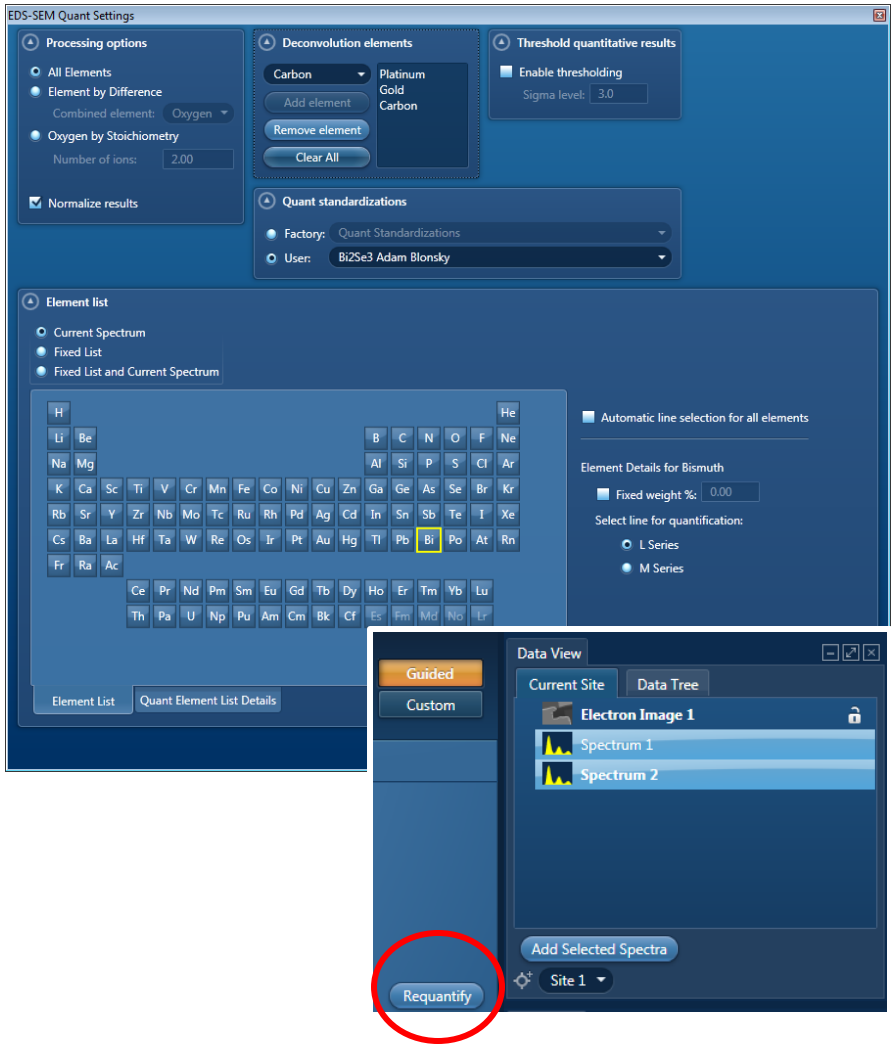


Calc. Composition Settings



Options:

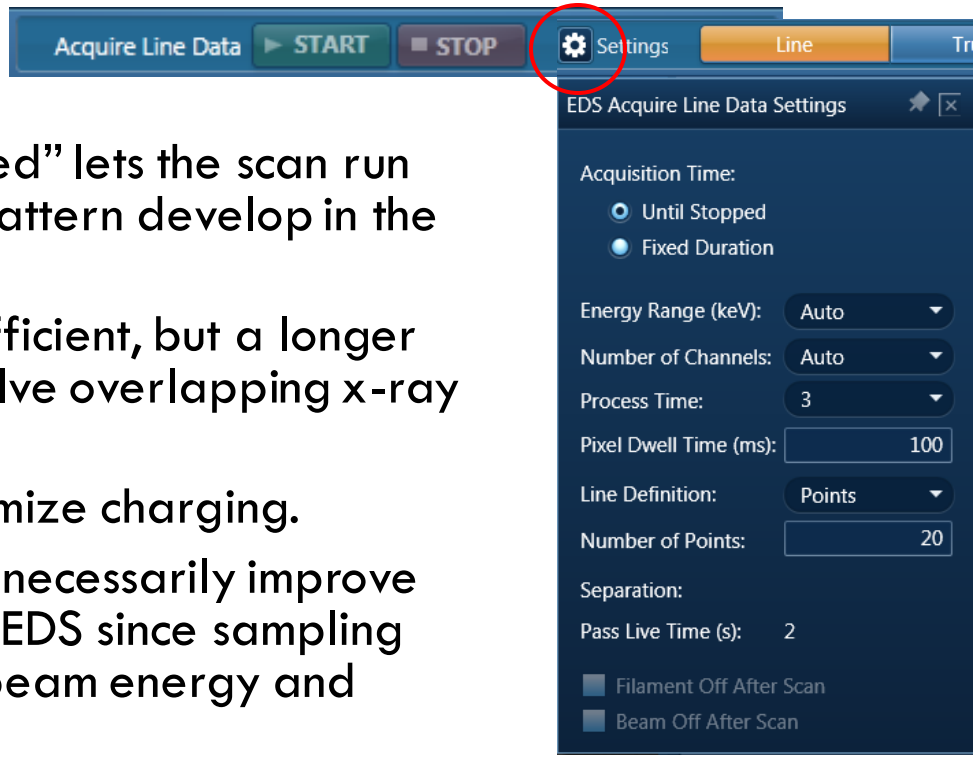
- Normalize composition to 100%
- Remove elements from the Quant. by adding them to the "Deconvolution Elements" list.
- Select an alternate peak series for Quantification by un-checking the "Automatic line selection" box and choosing the element.
- To apply your setting changes click Apply and Save, then highlight all spectra and click "Requantify."



Acquire Line Scan

Chose the Linescan collection mode.

Line selection tool defines the line to scan on image.



- Acquisition "Until Stopped" lets the scan run until you see a spatial pattern develop in the spectrum.
- Process time 3 is most efficient, but a longer time will more fully resolve overlapping x-ray peaks.
- Shorter dwell times minimize charging.
- More points/line do not necessarily improve the spatial resolution of EDS since sampling volume is a function of beam energy and sample material.

Construct Lines

Acquire Map

Choose the Map collection mode.

Scan Image to capture the SE or BSE image.

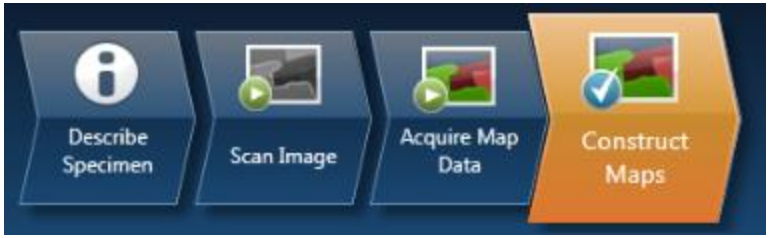
Open Map Settings for options:

- Select high spatial resolution for low magnification scans only. High resolution scans do not improve the spatial resolution of EDS.
- Acquisition "Until Stopped" lets the scan run until you see a spatial pattern develop.
- Select Process Time 3.
- Short pixel dwell times minimize charging.

Acquire Map: Click "Start" to map the whole area or draw a shape around the are of interest.

Mag	Resolution
< 500 X	4096
1000 X	2048
2500 X	1024
> 5000 X	512

Construct Map



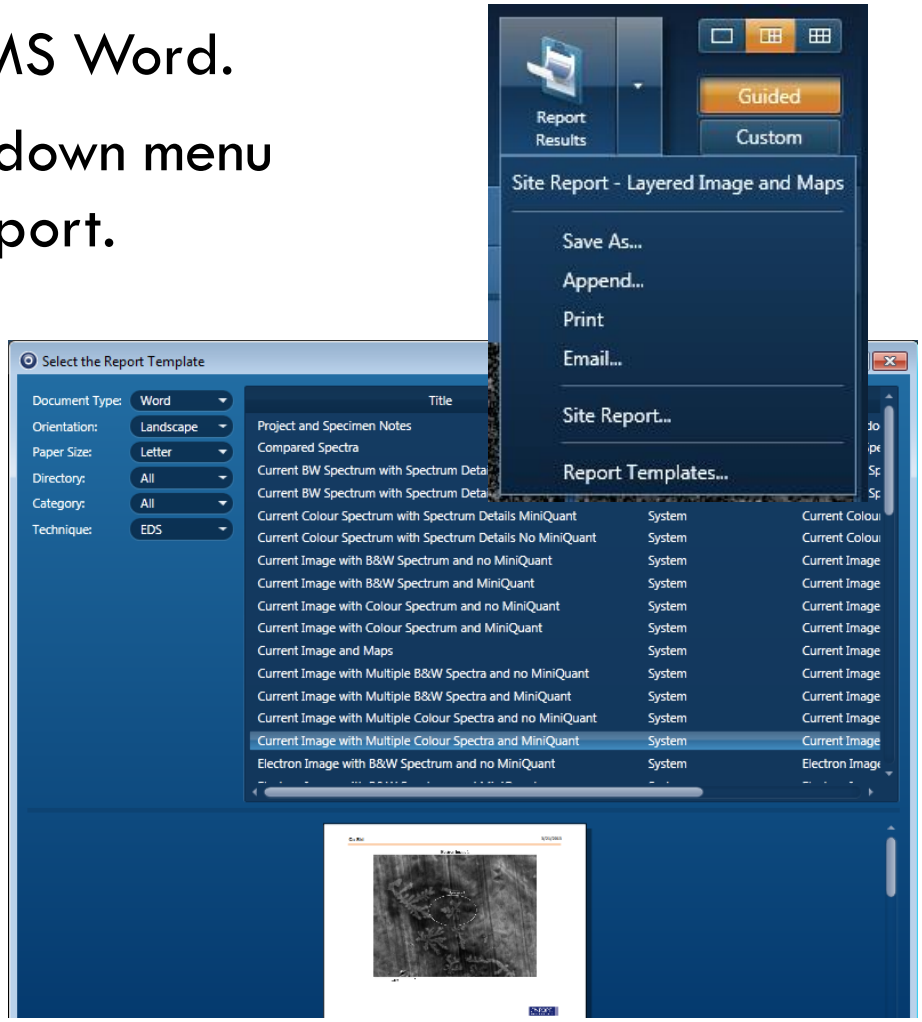
Click layer icons to overlay maps in the Layered Image

Select a map and click contrast to adjust threshold.

True Image filters overlapping x-ray peaks out of the map images.

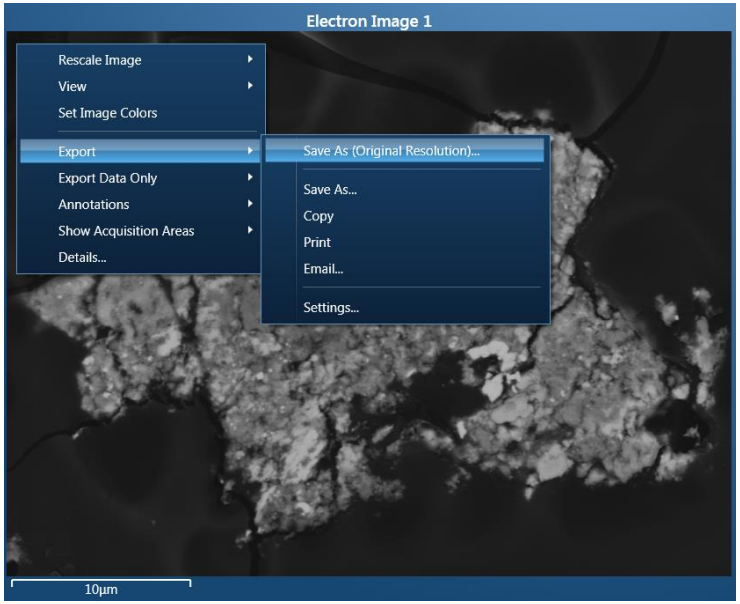
Report Results

- All reports are exported to MS Word.
- From the Report Results dropdown menu chose “Save As” for a site report.
- You can chose an alternate template from “Report Templates.”
- Select a template that includes all of the relevant results.
- Preview the report below.



Export Raw Data

Right click on any spectra and export as EMSA (.txt)



Right click on any image and export as “original resolution.”

Shutdown

- Turn the chamber Camera back on.
- Move detector to “Out” position.

- Save Project.
- Quant Settings to default
  - Clear the deconvolved elements list.
  - Check Autoline Selection.
- Describe Sample to default
  - Reset Specimen Coating to 10 nm Carbon.
  - Remove any Predefined Elements and “Save to Profile.”
- Disable Oxford in Coral.

Troubleshooting

The Detector control is not present or Aztec not reading mag or controlling beam sweep

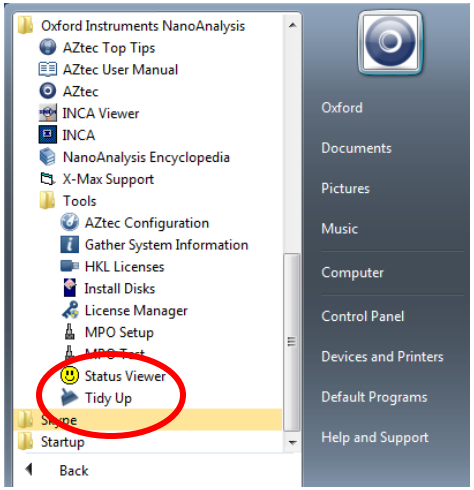
- Run the Tidy Up utility from the desktop

Rate meter >60% or strong Strobe

- Lower the spot size
- Process Time 3-4

Rate meter low Output Counts

- Working distance 5mm
- Unfreeze image
- Spot 3 - 6
- Correct Gun Tilt



Strong Strobe at 0eV