FEI Themis Z S/TEM: STEM-EDS
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ANALYSIS OF RADIOACTIVE SPECIMENS IS <u>STRICTLY</u> PROHIBITED

This document assumes the user is familiar with and competent in operation of the Themis Z S/TEM in STEM mode.

1. Instrument voltage

    1.1. The default voltage setting is 200 kV. If you need to perform STEM-EDS with the instrument at a different voltage (60 or 300 kV), please contact staff for assistance setting up the instrument.

        1.1.1. The example shown in this document was performed while operating the instrument at 300 kV.

2. Instrument alignment/settings for STEM-EDS

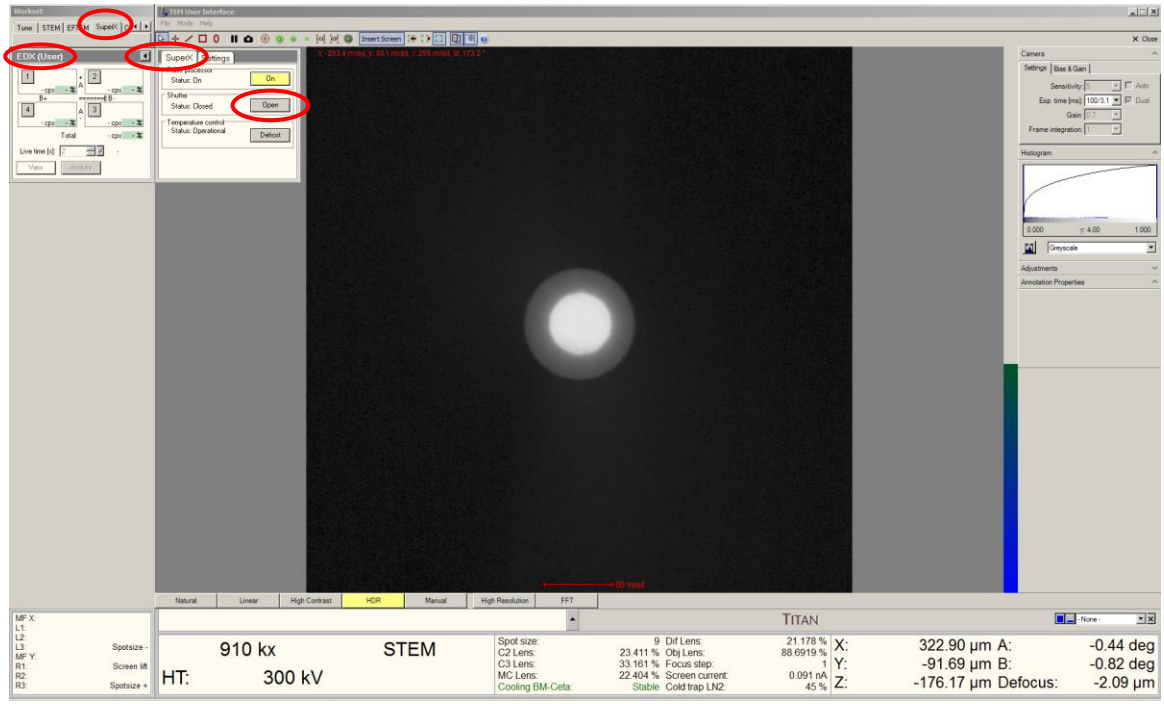
    2.1. Perform the same alignment in STEM mode using a HAADF-STEM image noting the following:

        2.1.1. Set the probe current to 50 – 150 pA; the less sensitive the specimen, the higher the probe current that may be used; in general for STEM-EDS, the higher the current, the better, so if the specimen can tolerate a probe current > 150 pA, feel free to adjust the probe current accordingly.

3. Opening the EDS shutters

3.1. In Microscope Control, select the “SuperX” tab, navigate to the “EDX” control panel, and select the flap-out arrow; select the “SuperX” tab and select “Open”.

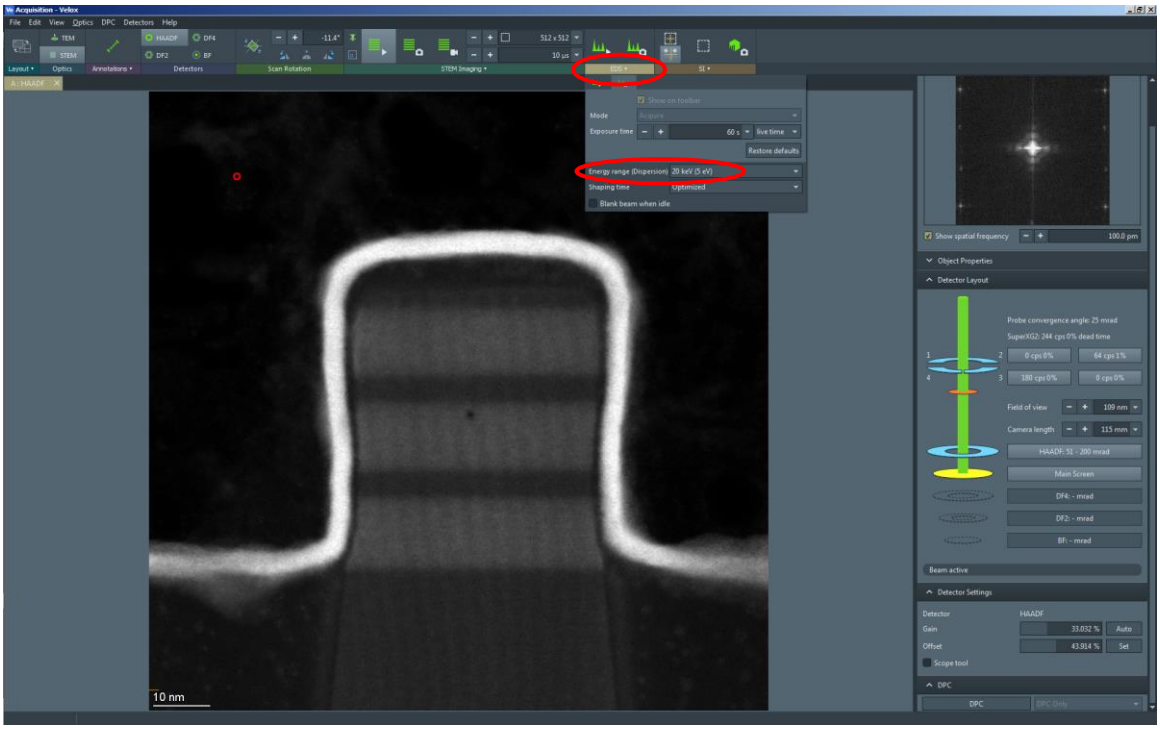
3.1.1. NOTE: the EDS shutters should only be open when EDS is ***actively*** being performed.



4. Setting up and acquiring the spectrum image

4.1. In the Acquisition window of Velox, select “EDS” from the toolbar and specify the “Energy range”.

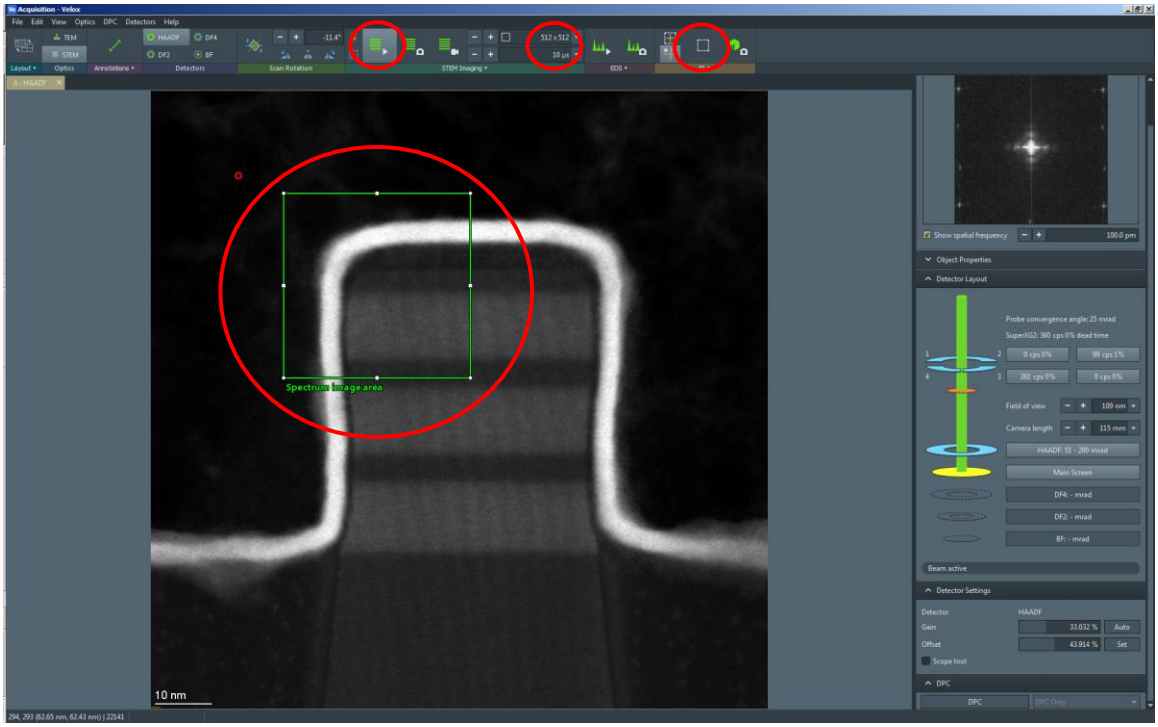
4.1.1. NOTE: if you are unsure of an appropriate energy range, “20 keV” is a good default value.



4.2. In Velox, start acquiring a *live* STEM image (512×512-pixel resolution); adjust the scan rotation, magnification, and focus as needed; the dwell time should be ≥10 µs.

4.3. Select “Spectrum Image Area” from the toolbar; click and drag on the live STEM image to draw a box to define the “Spectrum Image area”.

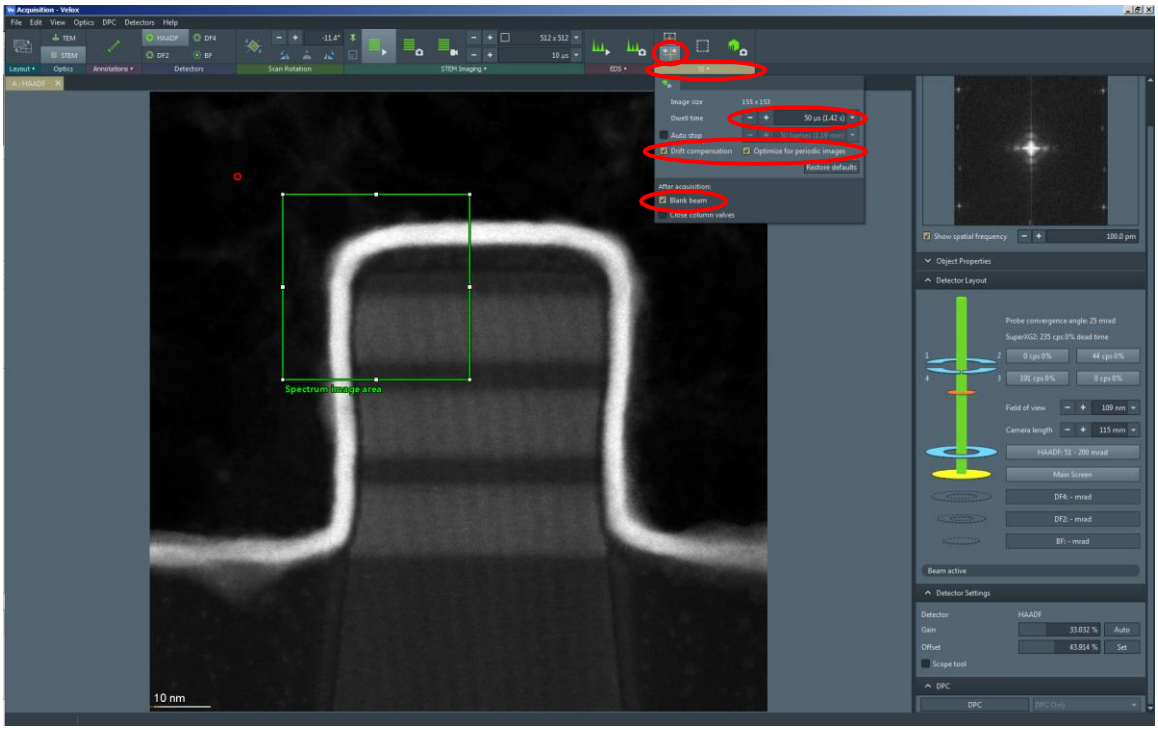
4.3.1. NOTE: the *pixel resolution* of the live STEM image will define the pixel resolution of the resulting spectrum image; *atomic-resolution* spectrum imaging requires indicated magnifications where atomic-level detail is readily observed in the live STEM image.



4.4. Select “Drift Compensation” from the toolbar; then select “SI” from the toolbar.

4.4.1. Set “Dwell time” = 20 – 50 µs such that the individual frame time is (at most) a few seconds.

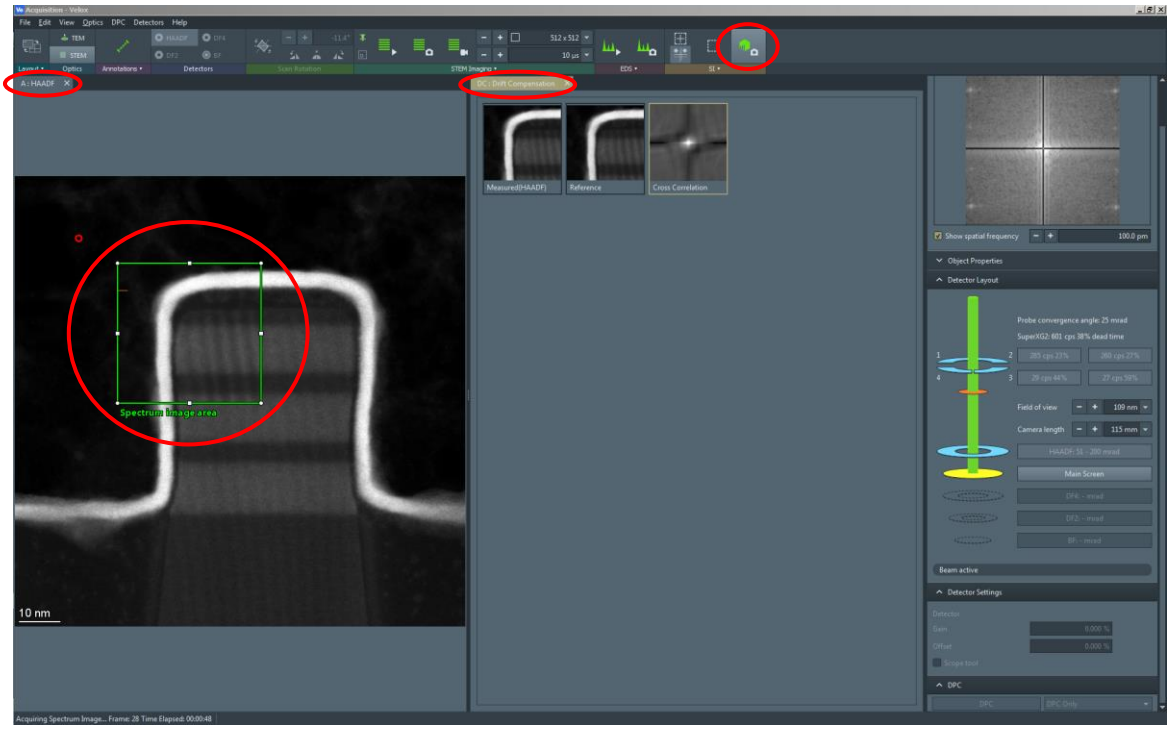
4.4.2. Check the “Drift compensation”, “Optimize for periodic images”, and “Blank beam” boxes (all other boxes should be *<u>unchecked</u>*).



4.5. While *live* STEM imaging, select “Spectrum Imaging” from the toolbar to start acquiring the spectrum image; the initial STEM image with the “Spectrum Image area” will be transferred to the “HAADF” panel; a “Drift Compensation” panel with new images related to drift compensation will also be generated.

4.5.1. While spectrum imaging is proceeding, you may adjust the focus (as needed) while observing the live STEM image in the “Spectrum Image area”.

4.6. When ready to stop collecting frames for the spectrum image, select “Spectrum Imaging” from the toolbar to stop the acquisition (same as previous step).



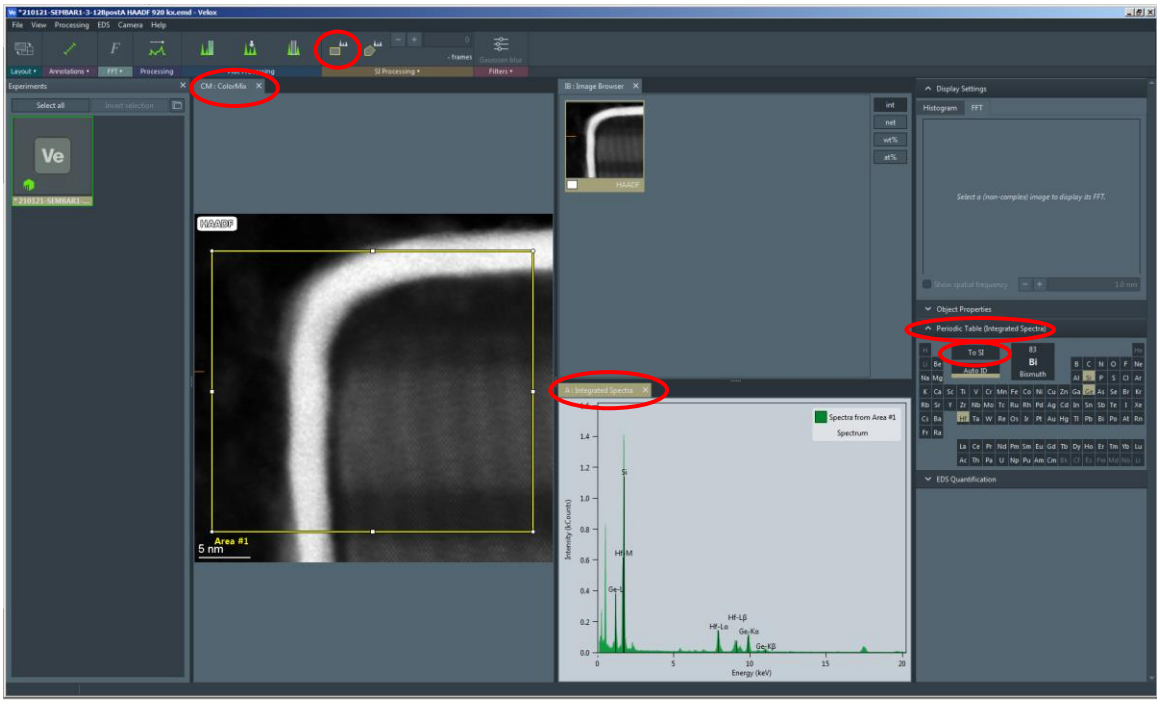
5. Intensity maps (possible while live spectrum imaging)

5.1. An intensity map for a given element uses all X-rays within one or more specified energy ranges to generate the map; no background subtraction or deconvolution with X-rays from other elements occurs.

5.2. Navigate to the Processing window of Velox; the STEM image collected from the analysis area will be transferred to the “ColorMix” panel. Select the “Spectrum Integration Rectangle” from the toolbar and click and drag on the STEM image in the “ColorMix” panel; an “Integrated Spectra” panel with the integrated spectrum will be generated.

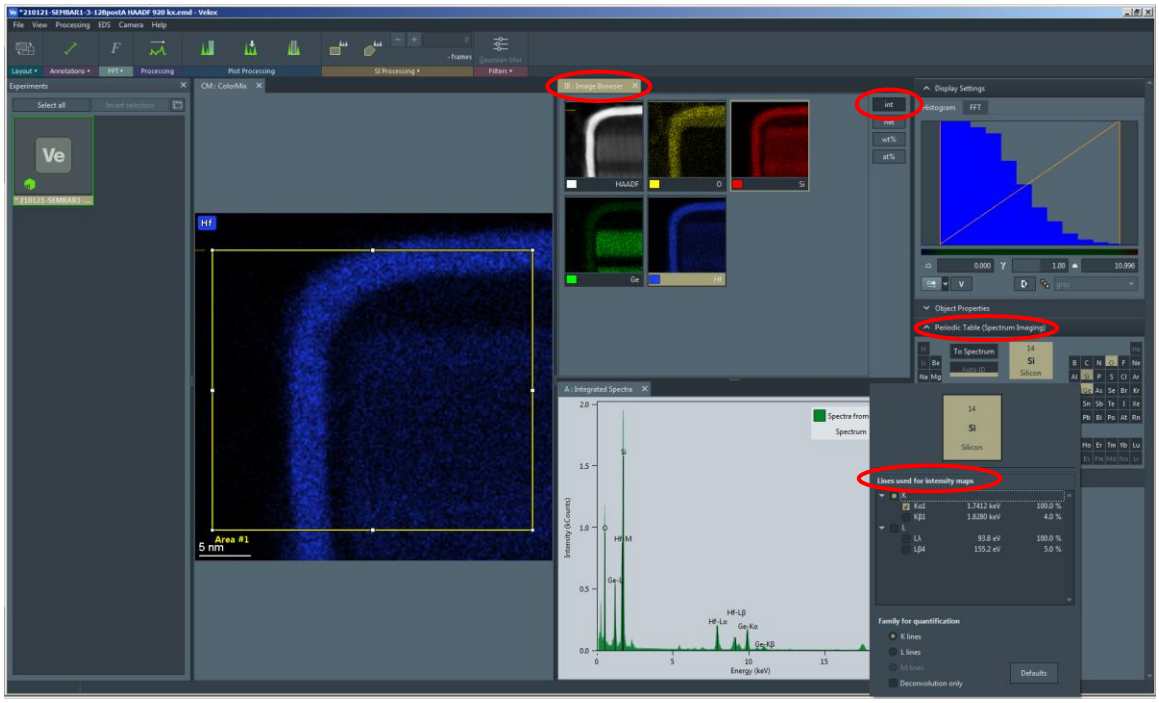
5.3. Select the “Integrated Spectra” panel, then select the “Periodic Table (Integrated Spectra)” side panel and select the elements of interest from the periodic table; then select “To SI”.

5.3.1. The X-ray peaks for the selected elements will now be labelled in the integrated spectrum.



5.4. A set of intensity maps for each element will be generated in the “Image Browser” panel; to add/remove/change the X-ray peaks contributing to a particular element; select the “Image Browser” panel, then select the “Periodic Table (Spectrum Imaging)” side panel; right click on an element in the periodic table to see the options for X-ray peaks.

5.4.1. More than one X-ray peak may be used to generate an intensity map for a particular element to improve signal (if more than one line is generated by the element).

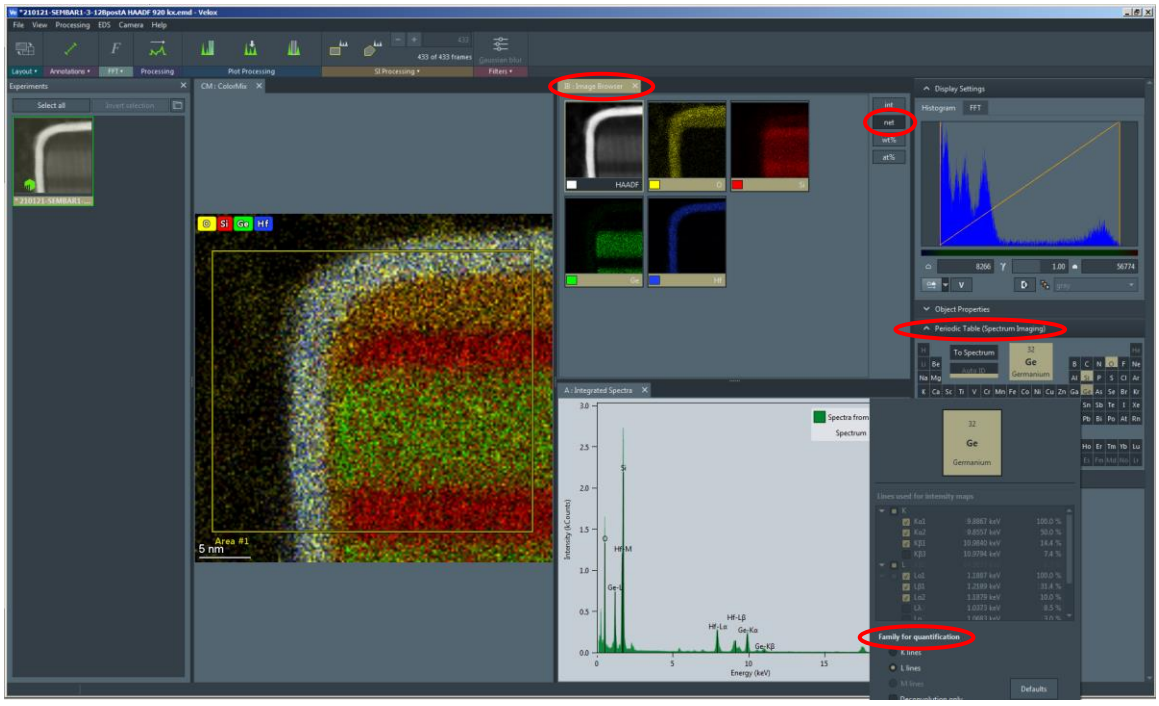


6. Net, wt%, and at% maps; map filtering; area quantification

6.1. Net maps remove background signal contributions and attempt to deconvolute overlapping X-ray signals but can only use <u>one X-ray peak family per element</u>; net maps serve as the basis to generate wt% and at% maps.

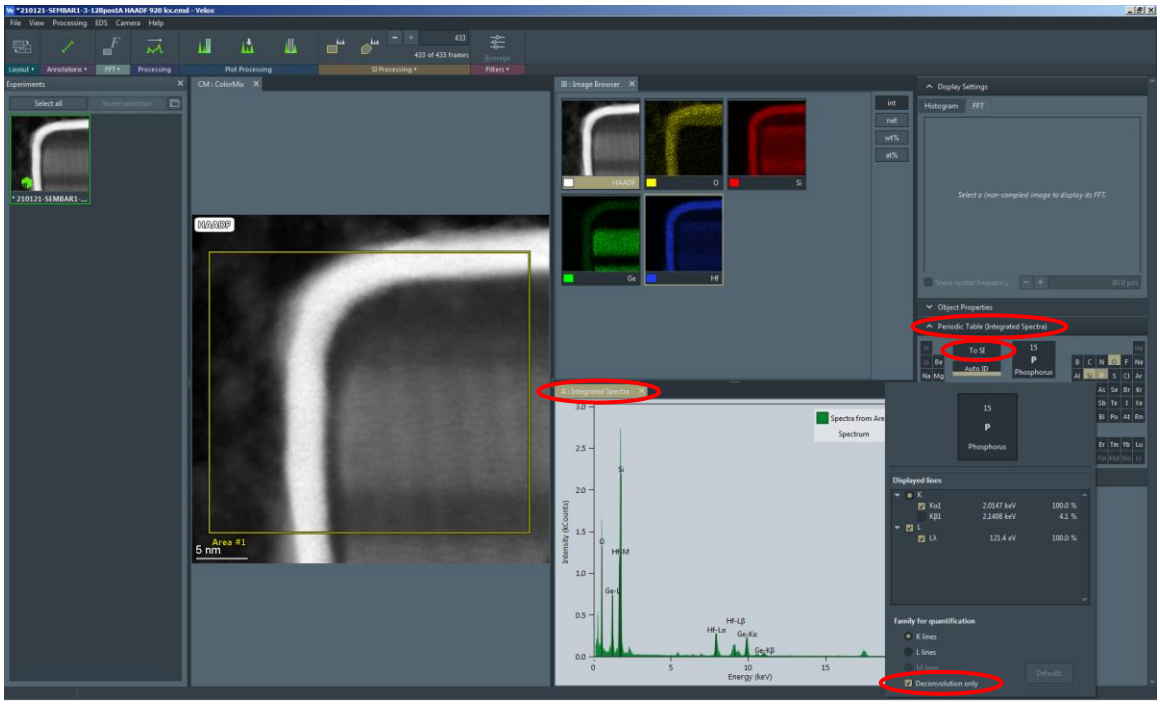
6.1.1. NOTE: net maps can be generated while live spectrum imaging but wt% and at% maps can only be generated once live spectrum imaging is ***<u>stopped</u>***.

6.2. On the right side of the “Image Browser” panel, select “net” generate net maps; to select the contributing X-ray peak family for a particular element; select the “Image Browser” panel, then navigate to the “Periodic Table (Spectrum Imaging)” side panel; right click on an element in the period table to see the X-ray peak family options.

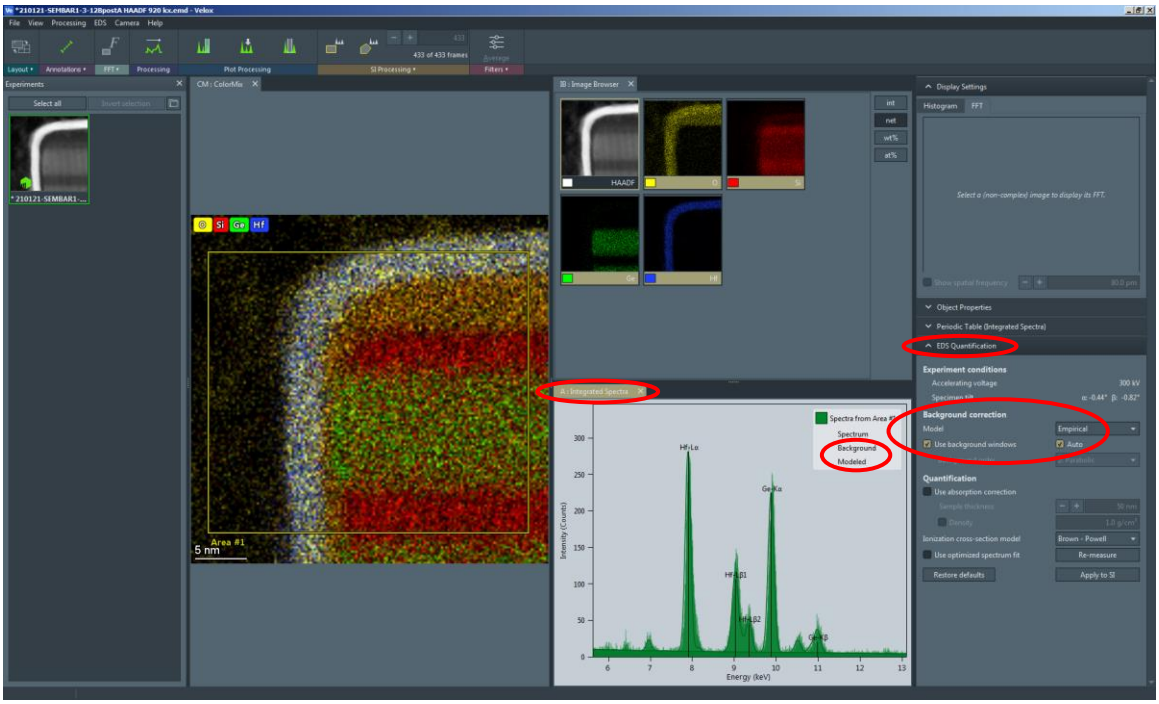


6.3. Select the “Integrated Spectra” panel and observe the integrated spectrum; if any X-ray peaks from elements not of interest are overlapping with a family of X-ray peaks of selected elements used for mapping, these should be identified for deconvolution only. Select the “Periodic Table (Integrated Spectra)” side panel and right click on an element for deconvolution only in the period table; in the element window, select “Deconvolution only”.

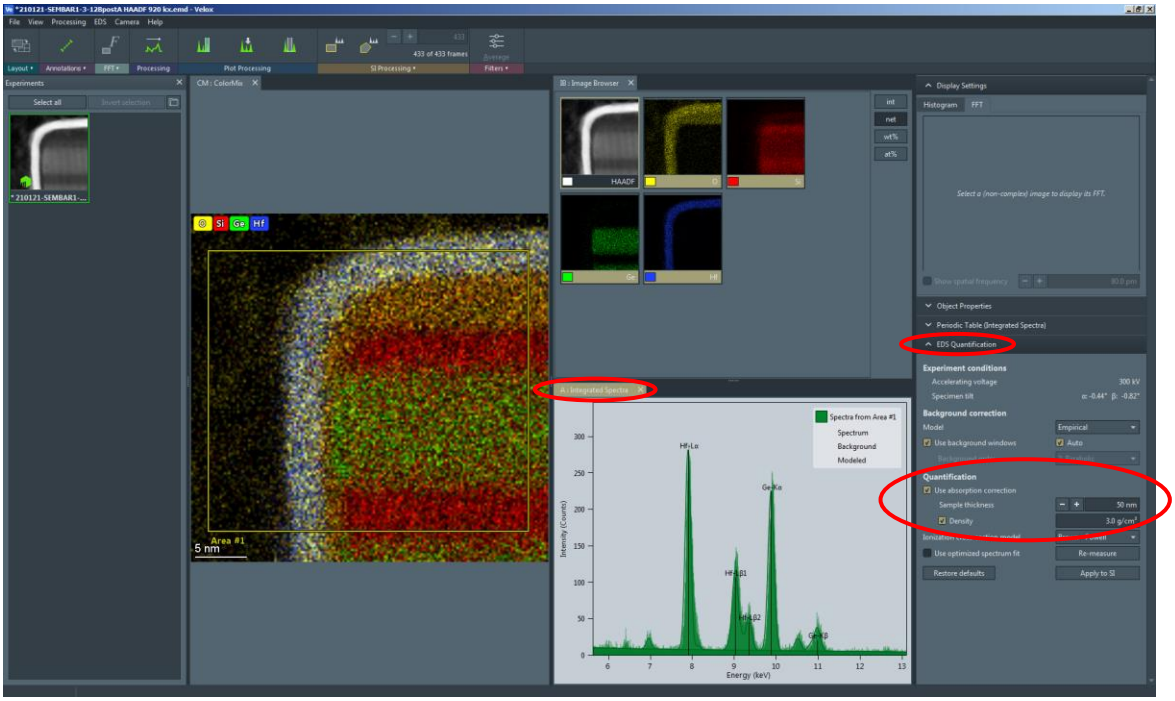
6.4. Remain in the “Periodic Table (Integrated Spectra)” side panel; once all elements for deconvolution only are identified and selected from the periodic table, select “To SI”.



6.5. Select the “Integrated Spectra” panel; mouse over the spectrum legend and activate “Background” and Modeled”. Then navigate to the “EDS quantification” side panel; for “Background correction”, select “Empirical” for the model and check “Use background windows” and “Auto”; verify the background model is accurate for the X-ray peaks of interest.



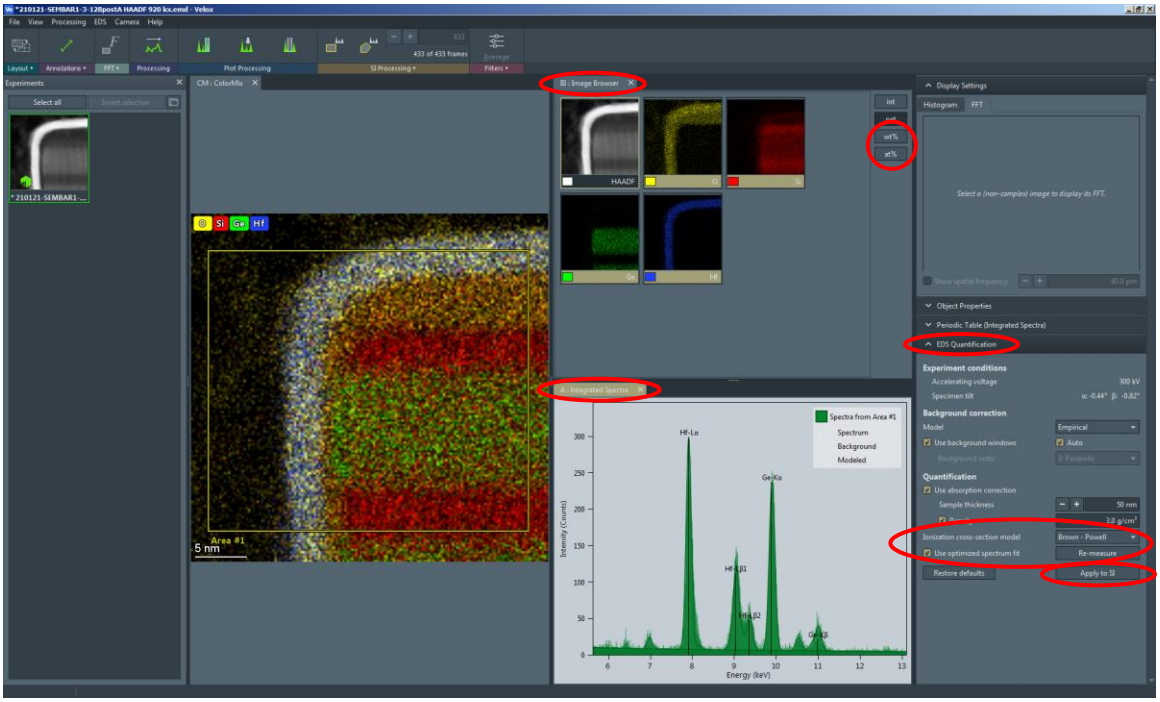
6.6. Remain in the “EDS Quantification” side panel; for “Quantification”, check “Use absorption correction” if the approximate specimen thickness and/or density are known and input accordingly.



6.7. Remain in the “EDS Quantification” side panel; for “Ionization cross-section model”, “Brown-Powell” will usually suffice (this is the default) unless working primarily with transition metals and/or metal oxides (in those cases, select “Schreiber-Wims”). Check “Use optimized spectrum fit”, then “Re-measure”, and finally “Apply to SI”.

6.7.1. On the right side of the “Image Browser” panel, it will now be possible to select “wt%” and “at%” options.

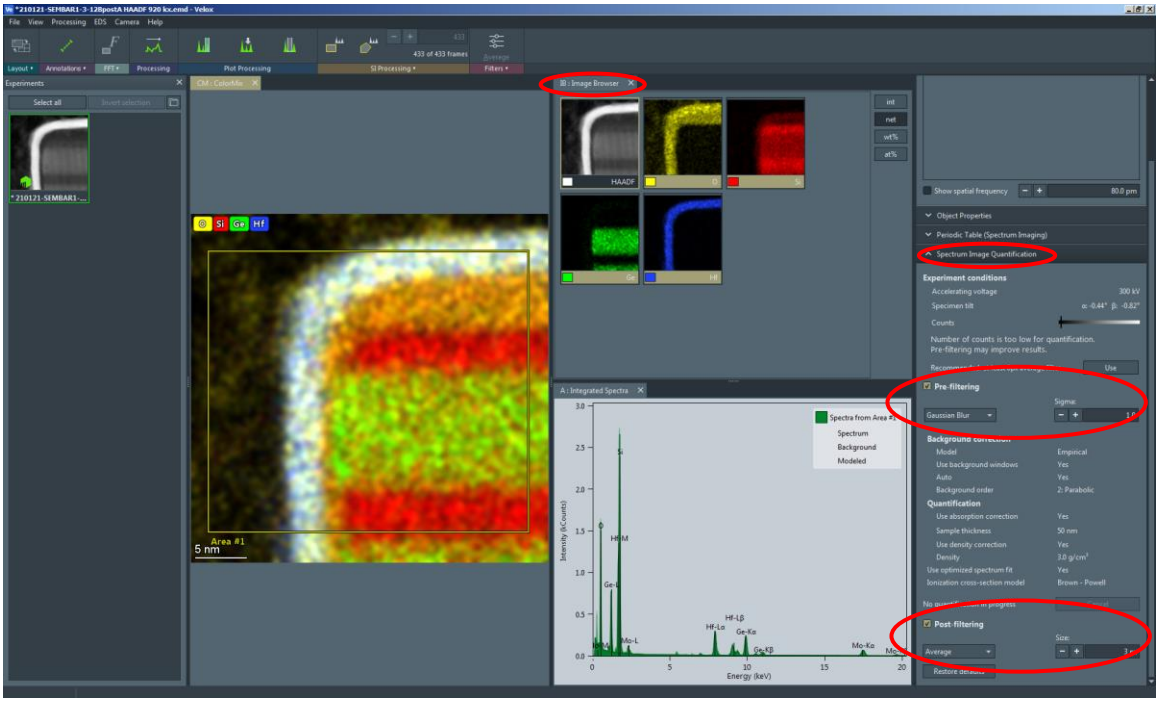
6.7.2. NOTE: wt% and at% maps should be avoided when part of the analysis area is vacuum; the accuracy of the wt% and at% maps are also *questionable when obtained using strong channeling conditions* (i.e., alignment along a major crystallographic zone axis).



6.8. The maps can also be “pre-filtered” to help improve quantification accuracy (particularly if the overall counts are low) and potentially produce higher quality maps. Select the “Image Browser” panel and then select the “Spectrum Image Quantification” side panel; check “Pre-filtering” and then select either “Average” or “Gaussian” blur with an appropriate kernel size.

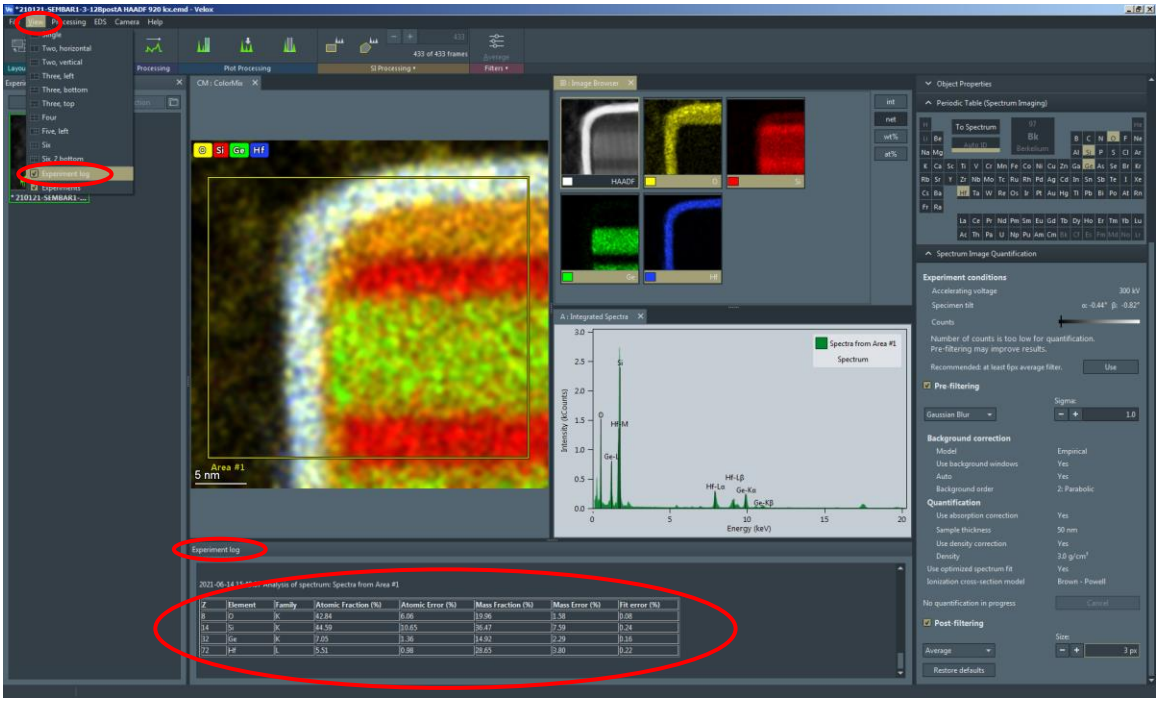
6.9. The maps may also be “post-filtered” to improve overall appearance without any impact on the quantification; remain in the “Spectrum Image Quantification” side panel, check “Post-filtering”, and select an appropriate filter and kernel size.

6.9.1. The “Radial Wiener” filter is generally only useful for atomic-resolution maps should only be used in analysis areas with *no vacuum regions present*.



6.10. To obtain quantitative information from the area selected in the “ColorMix” panel, navigate to the “View” pull-down menu and then check “Experiment log”; the “Experiment log” panel will open in the bottom of the window and display the quantitative results.

6.10.1. Again, it is best to *avoid strong channeling conditions and including vacuum regions in the selected area* to improve quantification accuracy.



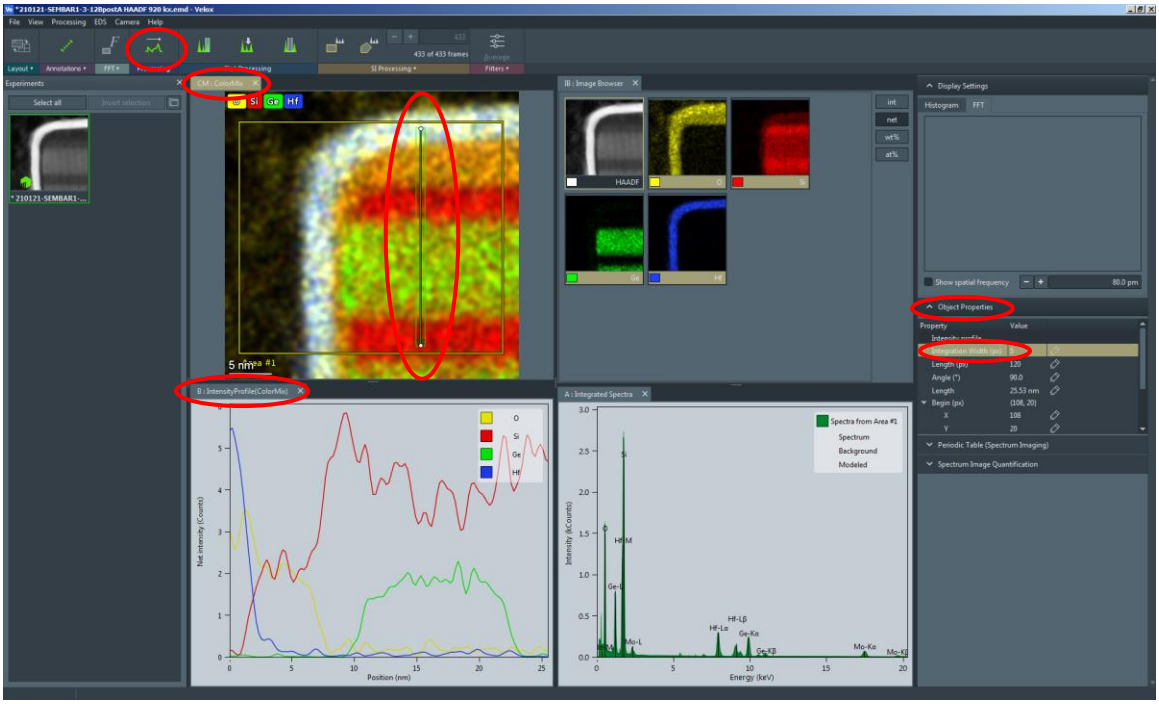
7. Extracting element profiles

7.1. Select “Intensity Profile” from the main tool bar; then click and drag on the image in the “ColorMix” panel to position the profile as needed; an “Intensity Profile (ColorMix)” panel with the profiles will be generated.

7.1.1. NOTE: the intensity profiles will be the same type selected for the maps in the “Image Browser” panel (e.g. “int”, “net”, etc.); any pre- and/or post-filtering applied to the maps will also be reflected in the profiles.

7.2. Select the intensity profile on the “ColorMix” panel, then navigate to the “Object Properties” side panel; adjust “Integration Width (px)” as needed.

7.2.1. This averages the intensity value *perpendicular* to the direction of the line and will improve profile accuracy across interfaces.



8. Closing the EDS shutters

8.1. When finished performing EDS, return to Microscope Control, select the “SuperX” tab, navigate to the “EDX” control panel, and select the flap-out arrow; select the “SuperX” tab; and select “Open” (button will be gray when the shutters are closed).

8.1.1. Again, the EDS shutters should only be open when EDS is ***actively*** being performed.