FEI Talos F200i S/TEM: EDS in STEM mode (points, line scans, maps)
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ANALYSIS OF RADIOACTIVE SPECIMENS IS *STRICTLY* PROHIBITED

The user should be familiar with operation of the instrument in TEM and STEM modes, use of the Microscope Control interface, and Velox; the user should also be familiar with basic operation of the EDS system as outlined the basic operation SOP.

1. Plasma cleaning for EDS

    1.1. It is highly recommended that plasma cleaning of the specimen be performed (if permissible for the specimen) to prevent contamination build up, which may compromise EDS analyses.

2. Instrument settings for STEM-EDS

    2.1. In general, it is preferable to use the double-tilt holder when performing STEM-EDS; particularly if you intend on analyzing any type of non-curved interface (e.g. film/substrate interface).

    2.2. EDS may be performed at either 80 or 200 kV (depending on specimen beam voltage tolerances); select a voltage, apply alignments and select the appropriate FEG register for STEM at said voltage (described elsewhere).

    2.3. While still in TEM mode, find an area of interest, set it at eucentric height, and align any interfaces of interest parallel with the incident beam (described elsewhere).

    2.4. Enter STEM mode and perform STEM alignment using spot size 1 with the 70 µm C2 aperture (described elsewhere).

        2.4.1. At 200 kV, these settings will produce a probe size of ~1 nm with probe current of ~800 pA, which is enough for most STEM-EDS applications requiring spatial resolution ≥1 nm.

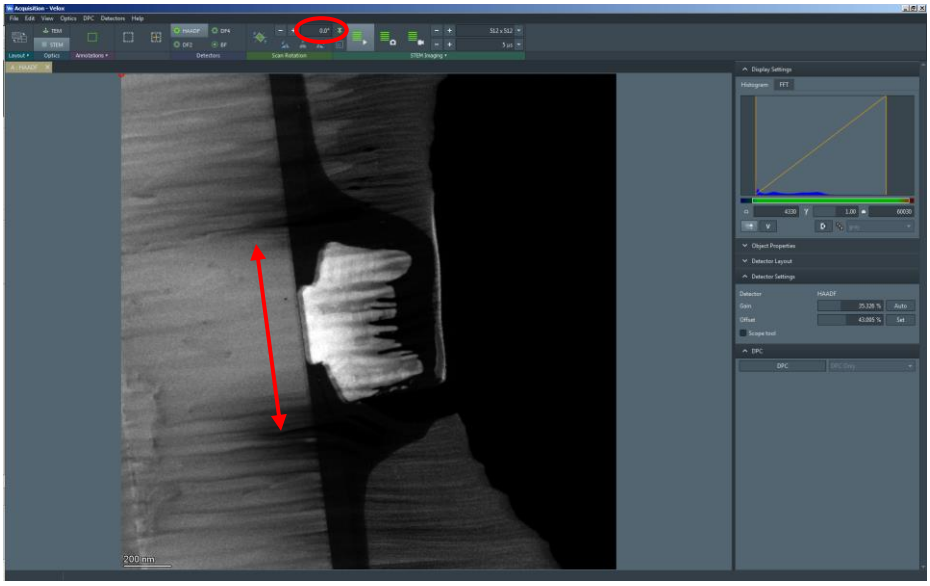
        2.4.2. If desired, a larger C2 aperture can be inserted for higher probe current, but at the expense of a larger probe.

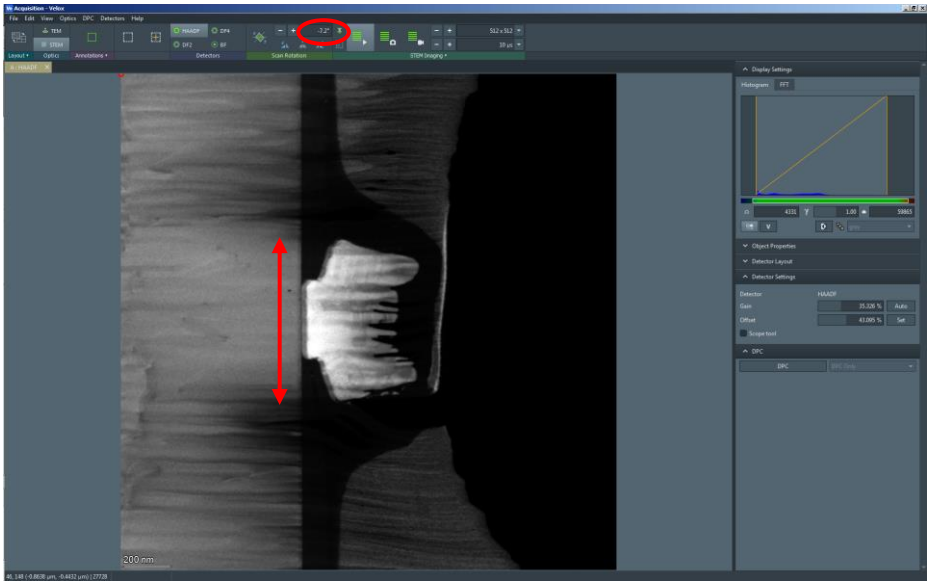
        2.4.3. In contrast, higher numerical values of spot size may be used to produce smaller probes (and potentially improve spatial resolution), but this will reduce probe current.

3. Finding an ROI/setting up the EDS system

3.1. Acquire a live STEM image in Velox; adjust contrast/brightness, scan rotation, focus, and condenser stigmators as needed (described elsewhere).

3.1.1. If intending on performing linescans or mapping, it is best to adjust the scan rotation so interfaces of interest run vertically in the STEM image.

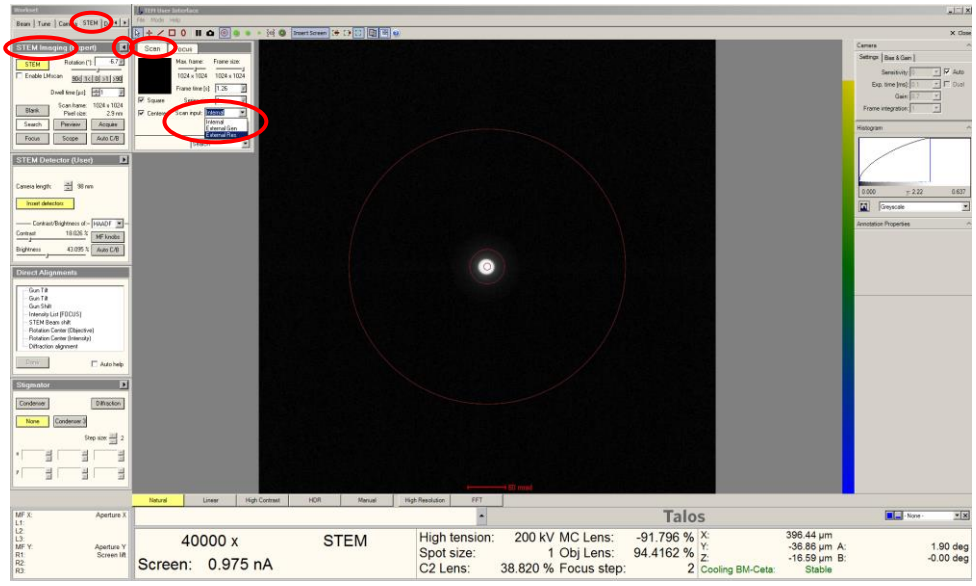




3.1.2. You may also need to perform additional tilting to align interfaces of interest to be parallel with the incident beam; if additional tilting is performed, be sure to refocus the STEM image by adjusting the Z axis of the stage so eucentric height is maintained.

3.2. In Microscope Control, select the “STEM” tab and navigate to the “STEM Imaging” control panel; expand the flap-out arrow and select the “Scan” tab; in the “Scan input” pull-down menu, select “External Res”.

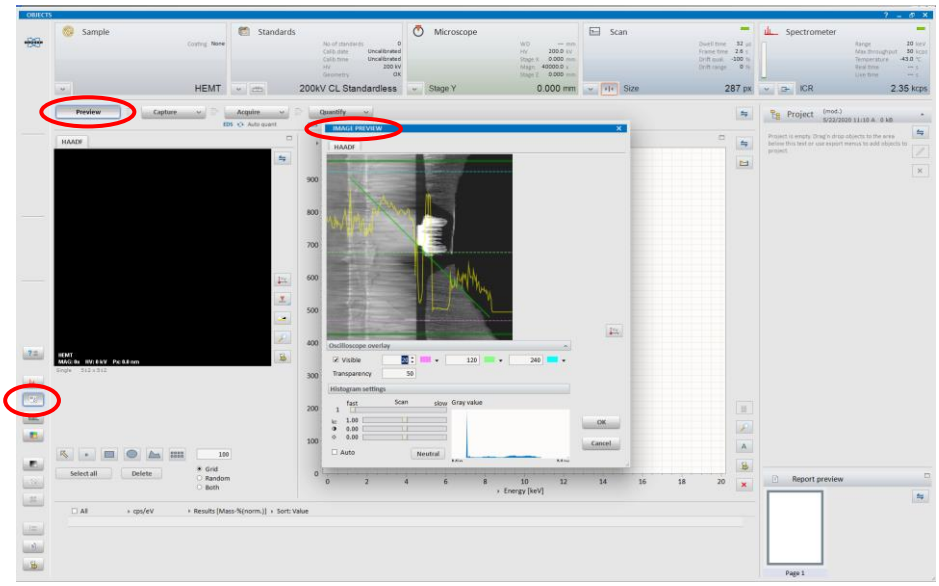
3.2.1. An error message may pop up in Velox; it is not problematic if this happens, simply clear the message, and proceed.



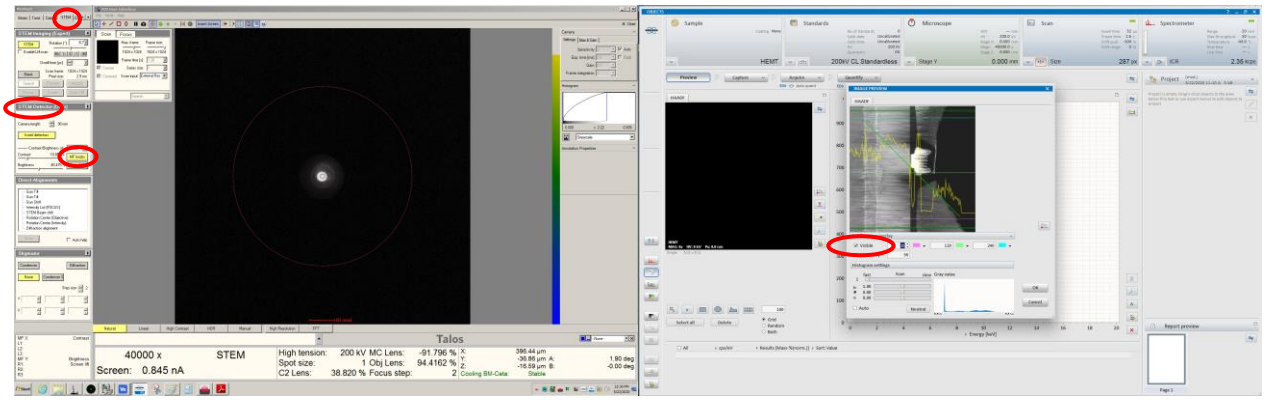
3.3. In ESPIRT 2, open a new project file; then insert the EDS detector, and select appropriate detector settings (as described elsewhere).

4. Point analysis

4.1. In ESPIRT 2, select “Objects” from the options list on the left panel; select “Preview” to open the “IMAGE PREVIEW” window and starting acquiring a live STEM image.

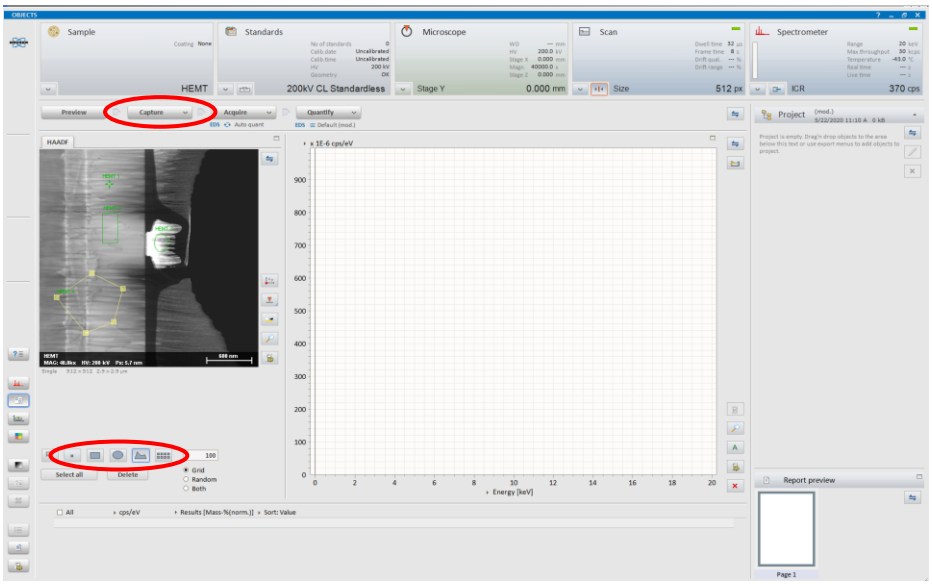


4.2. If the contrast/brightness of the preview image is unsatisfactory, it must be manually adjusted; in the “IMAGE PREVIEW” window, check the box next to “Visible” to see the plotted STEM signal; then return to Microscope Control, select the “STEM” tab, and navigate to the “STEM Detector” control panel; select “MF knobs” and use the “Multifunction” knobs to adjust the plotted STEM signal as desired.

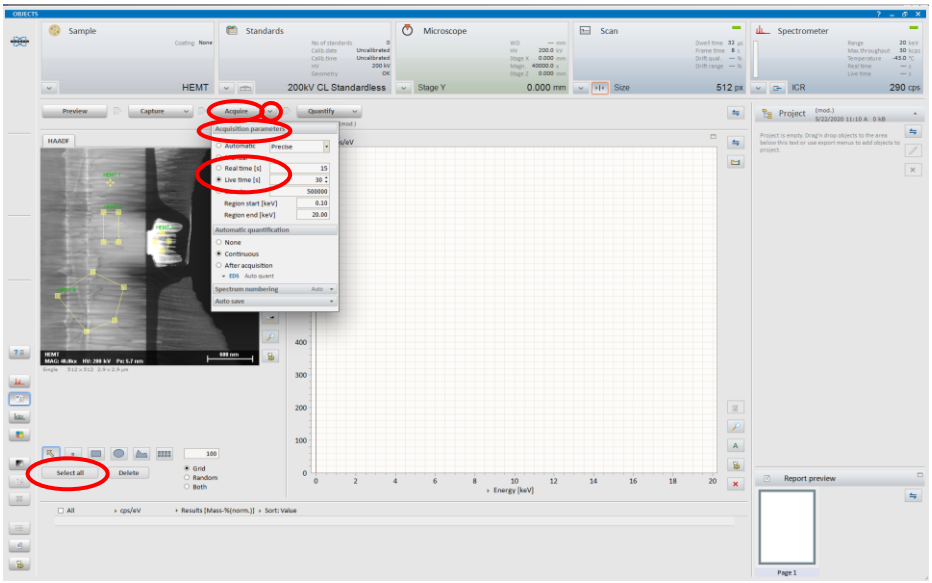


4.3. The focus will typically slightly (but noticeably) change as a result the EDS detector being inserted; while viewing the live image the “IMAGE PREVIEW” window, refocus the image with the “Focus” knob.

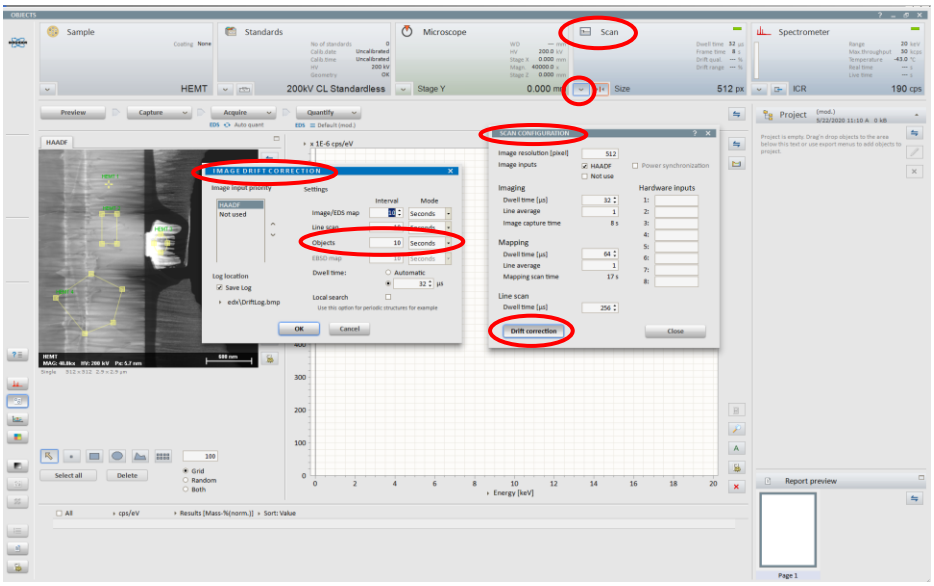
4.4. Return to ESPRIT 2 and close the “IMAGE PREVIEW” window; select “Capture” to acquire the STEM image to be used as the reference for point analysis; the use the marker tools to indicate which points/areas you want to analyze in the image.



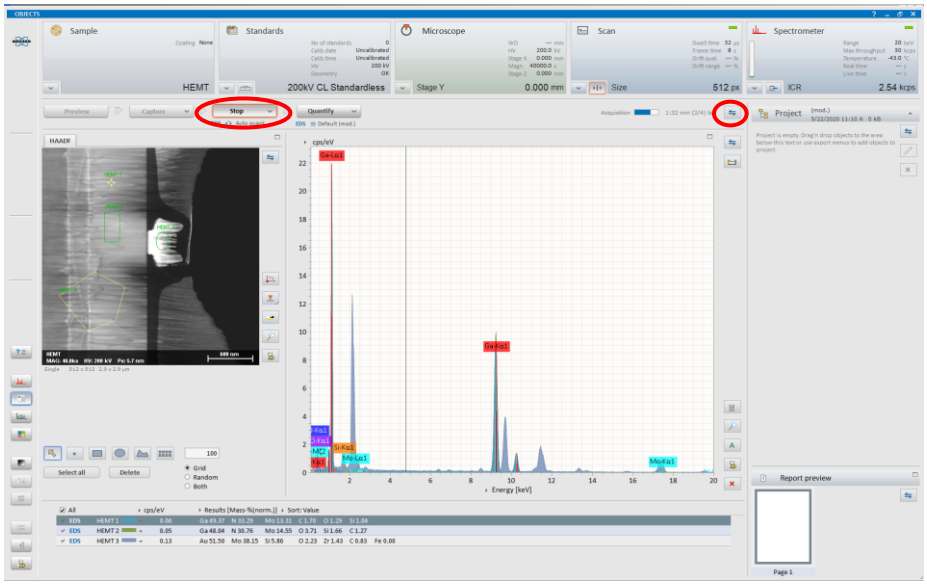
4.5. Select “Select all” to select all points/areas drawn on the image; the select the downward facing arrow on “Acquire”; input your acquisition parameters for each point/area on the image (typically, fixed real or live time).



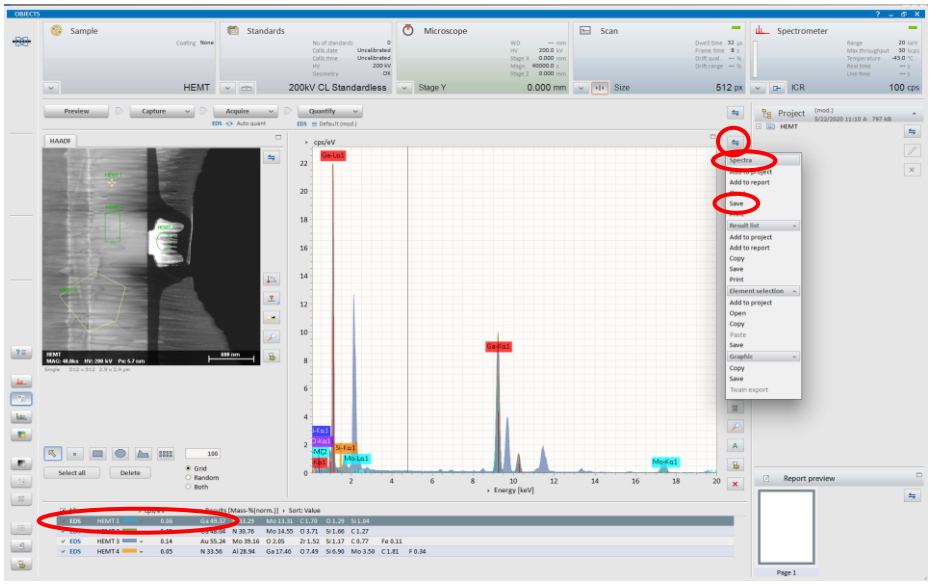
4.6. Navigate to the “Scan” configurator and select the downward facing arrow; in the “SCAN CONFIGURATION” dialogue box, select “Drift correction”; in the “IMAGE DRIFT CORRECTION” dialogue box, input the time interval to be used between corrections for “Objects” (10 – 20 s is usually sufficient), then select OK and close any remaining open dialogue boxes.



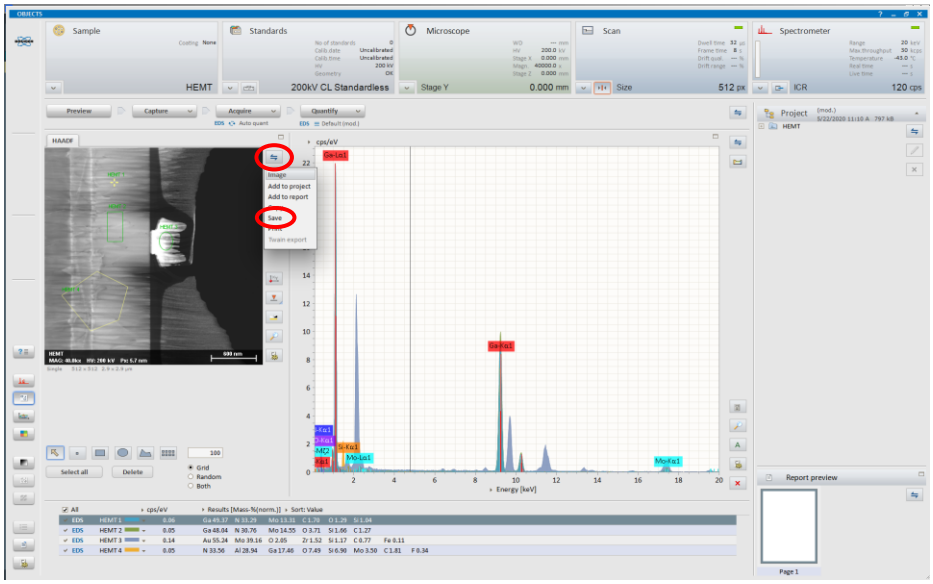
4.7. Select “Acquire” to start acquiring the spectra (button will now say “Stop”); when finished, select the I/O icon to send the data to the project.



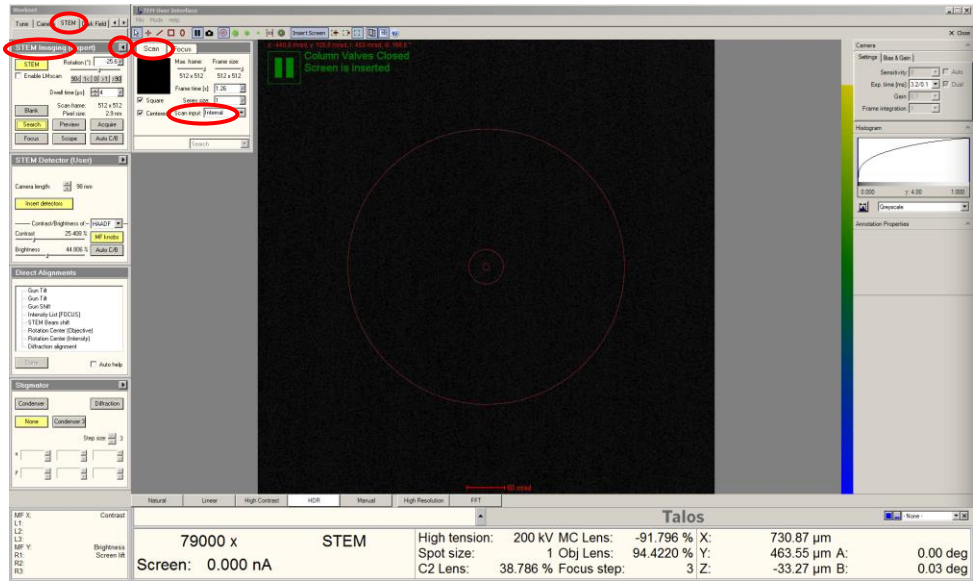
4.8. Element labels may be added/removed to the spectrum (as described elsewhere); select a spectrum from the spectrum list, and the select the I/O icon next to the spectrum; under “Spectra”, select “Save” to save the spectrum (as an image or raw data).



4.9. To save the STEM image with the indicated analysis points/areas, select the I/O icon next to the STEM and then “Save”.



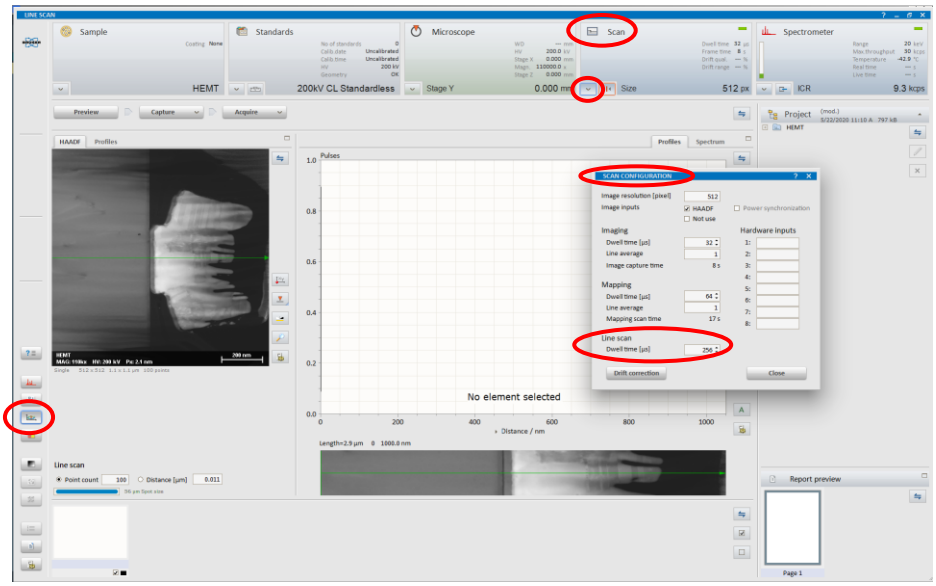
4.10. If you wish to move to a different ROI, or refocus, adjust stigmators, etc, it is easiest to do this using the live STEM image in Velox. Return to Microscope Control, select the “STEM” tab, and navigate to the “STEM Imaging” control panel; expand the flap-out arrow, select the “Scan” tab, and change the “Scan input” back to “Internal” (the live image should now be visible in Velox).



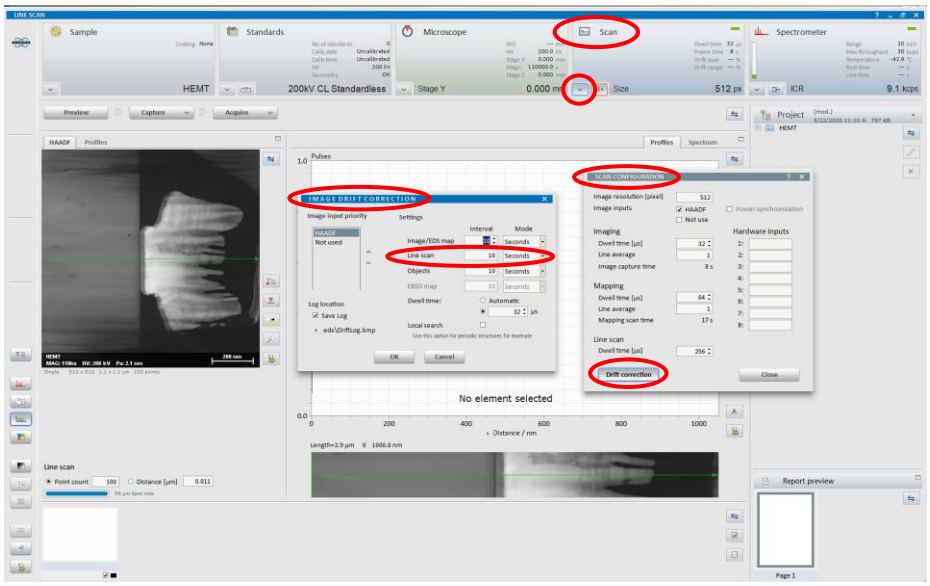
4.11. After making changes to the ROI and/or STEM image, be sure to change “Scan input” back to “External Res” to give control back to ESPRIT 2

5. Line scans

5.1. In ESPRIT 2, select “Line scan” from the options list on the left-hand panel, then preview and capture your reference STEM image (as described previously). Navigate to the “Scan” configurator and select the downward facing arrow; in the “SCAN CONFIGURATION” dialogue box; input a dwell time to be used for the line scan (256 µs is a recommend default value).

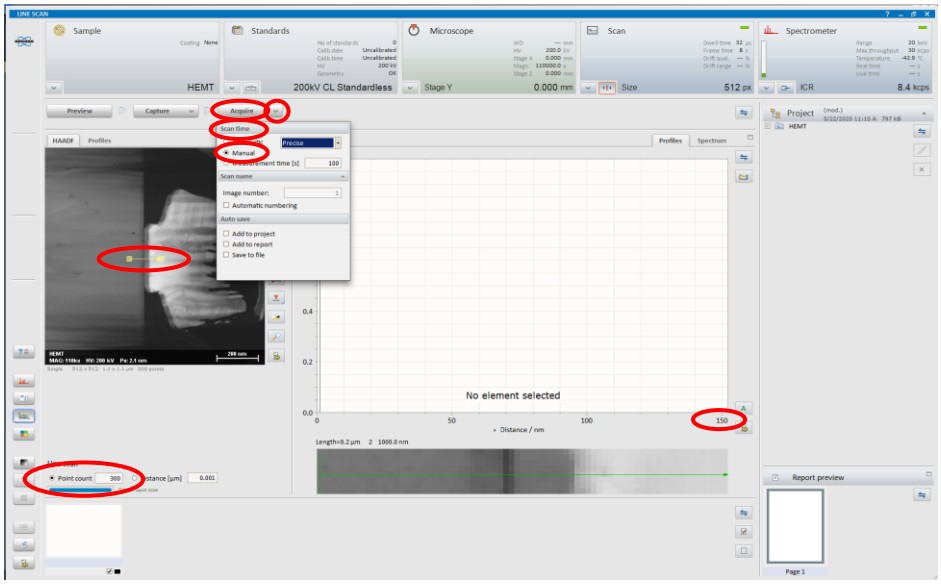


5.2. Remaining in the “SCAN CONFIGURATOR” dialogue box, select “Drift correction”; in the “IMAGE DRIFT CORRECTION” dialogue box, navigate to “Line scan” and input a time interval (10 – 20 s is usually sufficient); select “OK” and close any remaining open dialogue boxes.



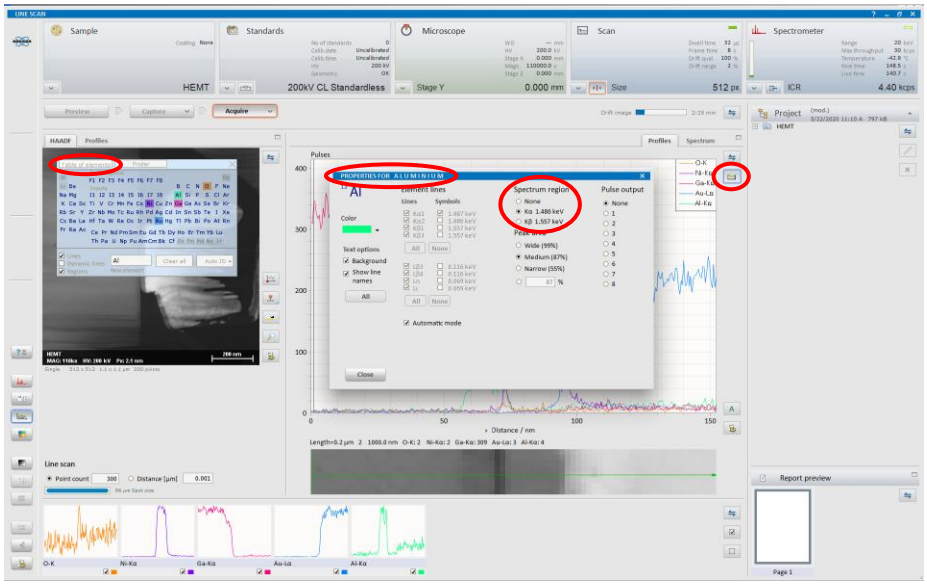
5.3. Click and drag on the arrowed line in the STEM image to select the position of the line scan (if the interfaces are vertical, the line should remain horizontal); adjust the “Point count” to set the pixel resolution of the line scan. Select the downward-facing arrow next to “Acquire”; under “Scan time”, select “Manual”; the select Acquire to start collecting the line scan.

5.3.1. NOTE: as the probe size is ~1 nm, there is little effective benefit in adjusting the point count such that the pixel resolution is <0.5 nm.

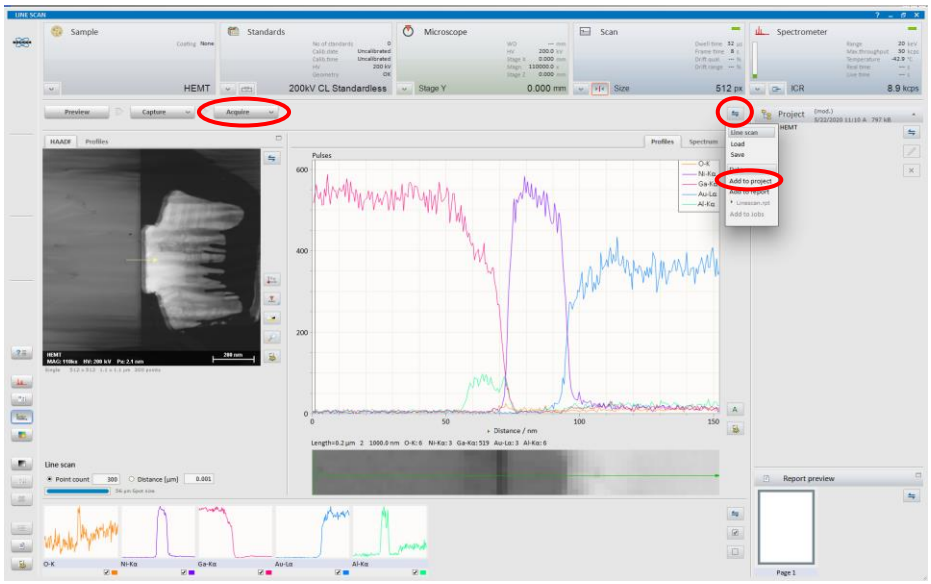


5.4. To manually select/deselect elements/peaks for the line scan, select the periodic table icon and then select the “Table of elements” tab; left click once on an element to add/remove it from the line scans.

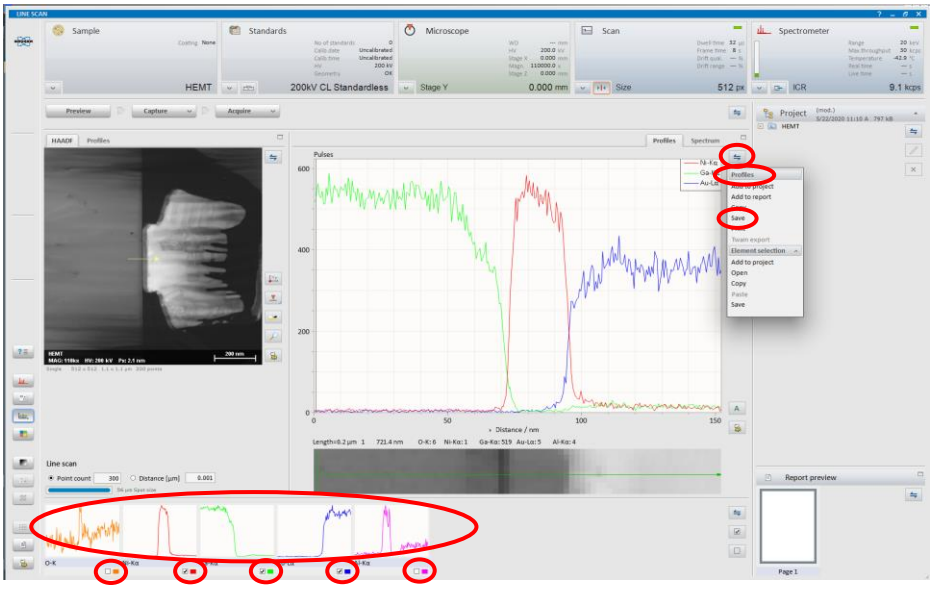
5.4.1. Additionally, you may double left click on an already selected element to view the “PROPERTIES” dialogue box for said element; under “Spectrum region”, you may manually select which peak to use for the line scan.



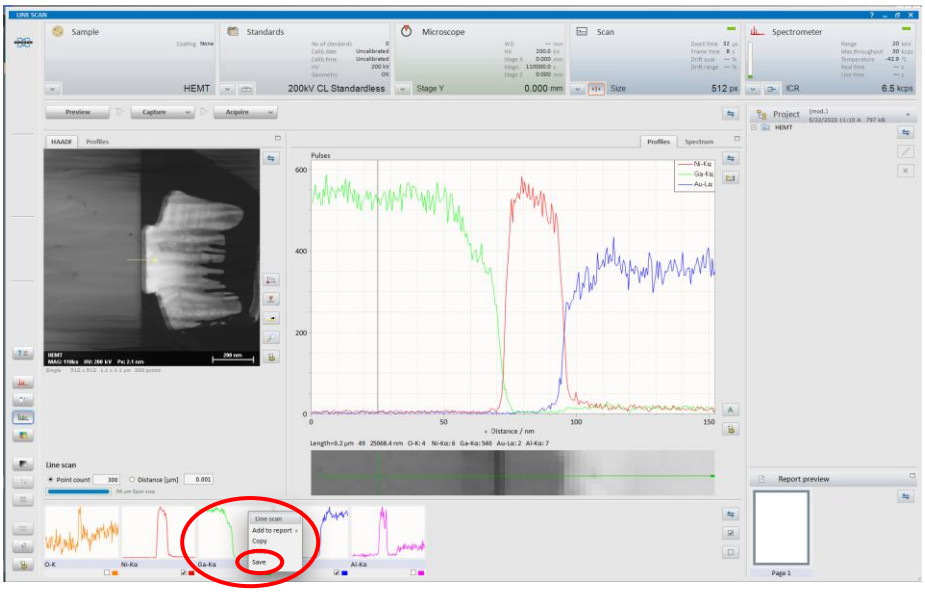
5.5. The line scan will run until manually stopped but be sure to add all necessary elements/select peaks before doing so as adding elements and/or selecting different peaks is not possible afterwards. Select “Acquire” to stop the line scan; then select the I/O icon and then “Add to project”.



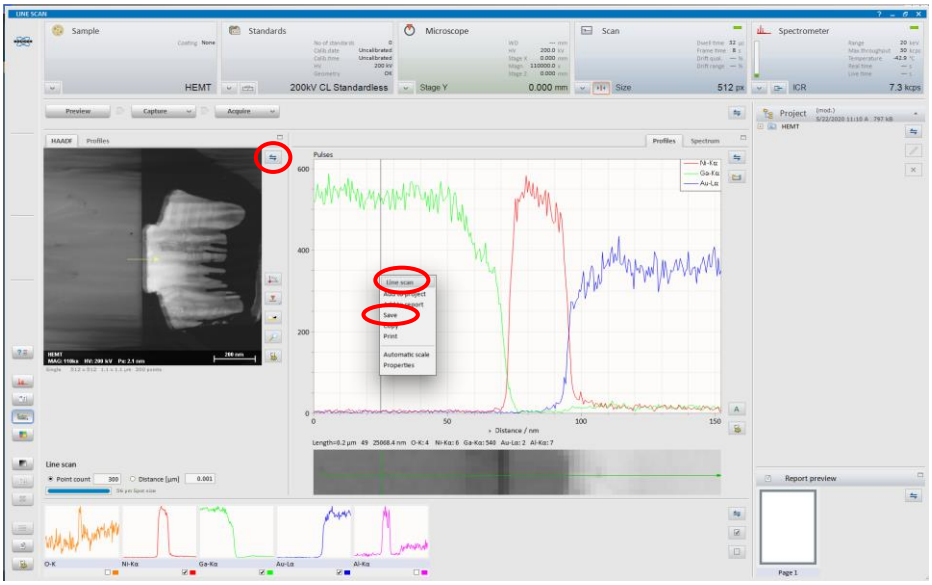
5.6. To add/remove elements from the composite, simply check/uncheck next to each individual line scan; to change the color of an element, select the box showing the current color to view additional options. Select the I/O icon next to the composite; under “Profiles”, select “Save” to save images of the composite, local scan area, or both.



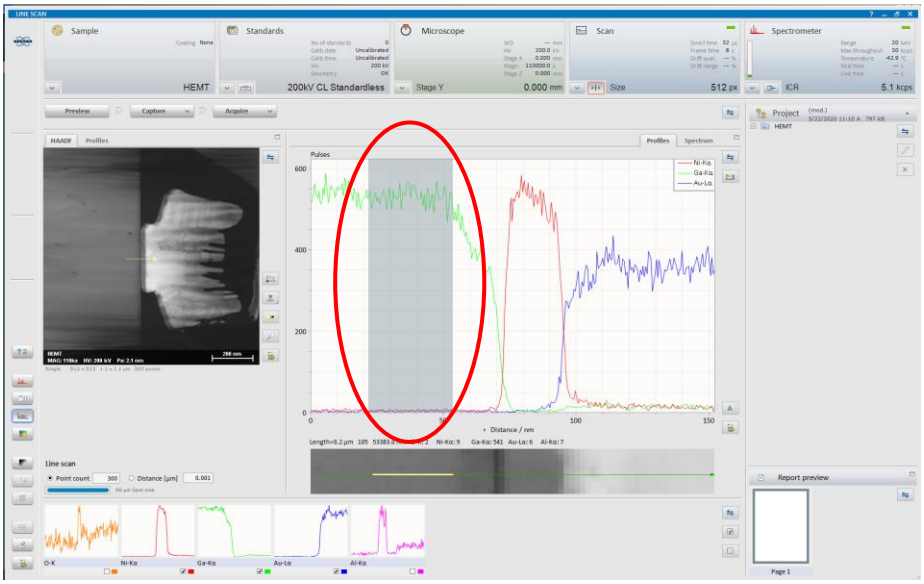
5.7. To save images of the individual line scans, right click on a line scan and select “Save”.



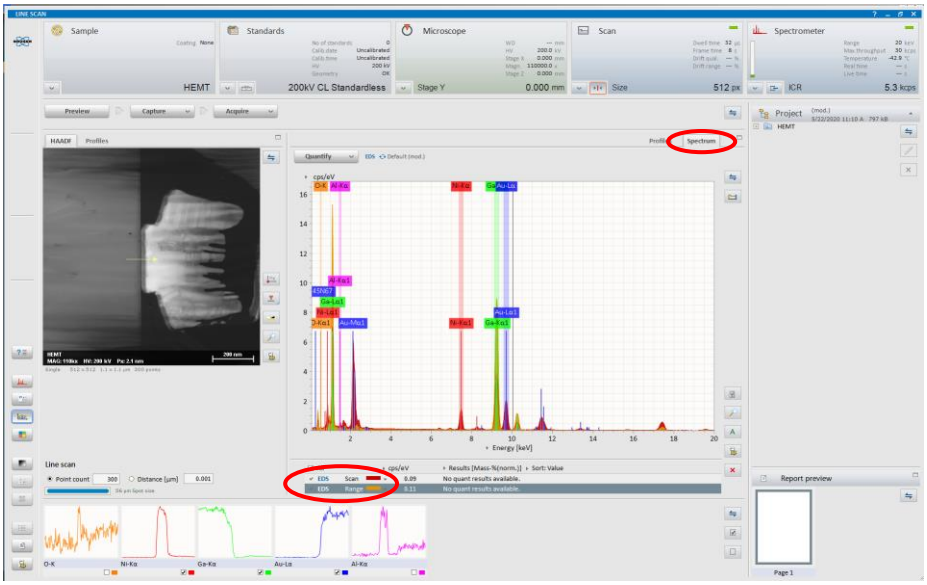
5.8. To save the raw line scan data; right click directly on the composite line scan and then select “Save” to see options for saving the raw data; the reference STEM image may also be saved by selecting the I/O icon at the upper right corner as described previously.



5.9. Spectral information may be extracted from all/part of the line scan. Simply right click and drag on a portion of the composite line scan to select that portion of the line scan for isolation.



5.10. Select the “Spectrum” tab to see the spectra isolated from the selected portion of the line scan (“Range”) and the whole line scan (“Scan”).

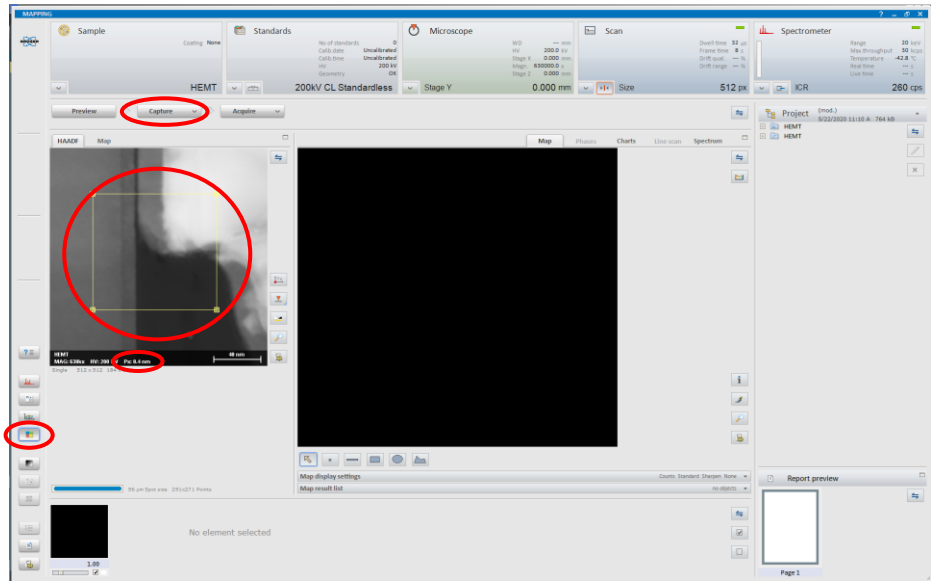


6. Mapping

6.1. Select “Mapping” from the options list on the left-hand panel; then preview and capture the reference STEM image as described previously; click and drag on the box in the STEM image to select the area to be mapped.

6.1.1. Note that pixel size of the STEM image (shown on the data bar) will determine the pixel resolution of the resulting maps; if this number is too low/high, preview the STEM again and adjust the magnification accordingly, then capture a new STEM image.

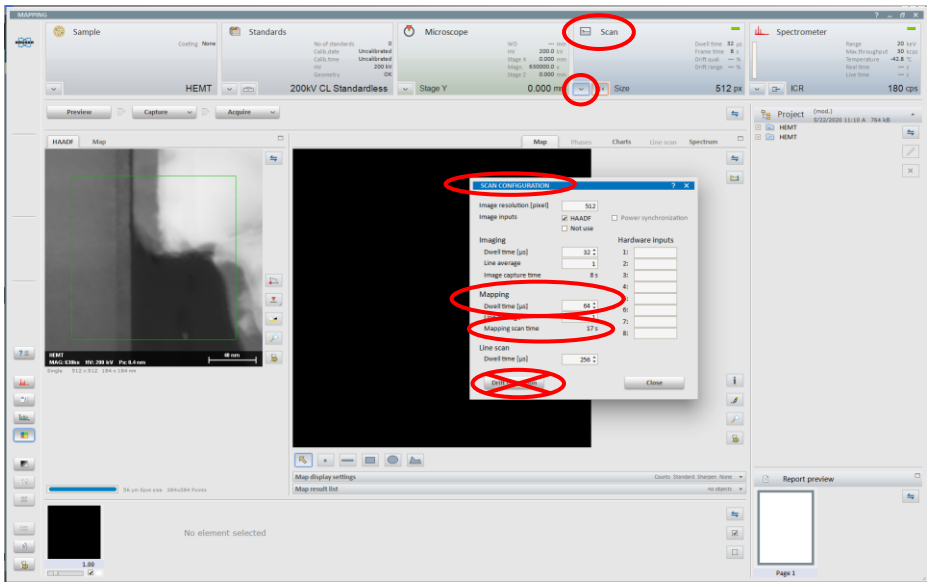
6.1.2. As the probe size is ~1 nm, there is little effective benefit in mapping with a pixel size <0.5 nm (this corresponds to a maximum indicated STEM magnification of ~700k×).



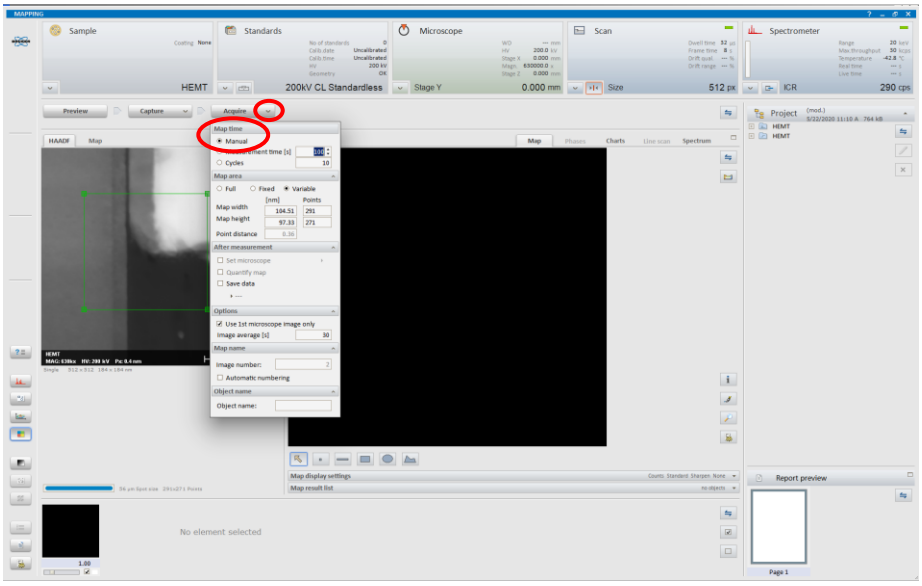
6.2. Navigate to the “Scan” configurator and select the downward facing arrow; in the “SCAN CONFIGURATION” dialogue box, input a dwell time for mapping (64 µs is a recommended default value)”.

6.2.1. NOTE: the time interval for drift correction while mapping is equal to the “Map scan time”, which is controlled by the dwell time for mapping; a “Map scan time” of ~20 s (or less) will usually suffice.

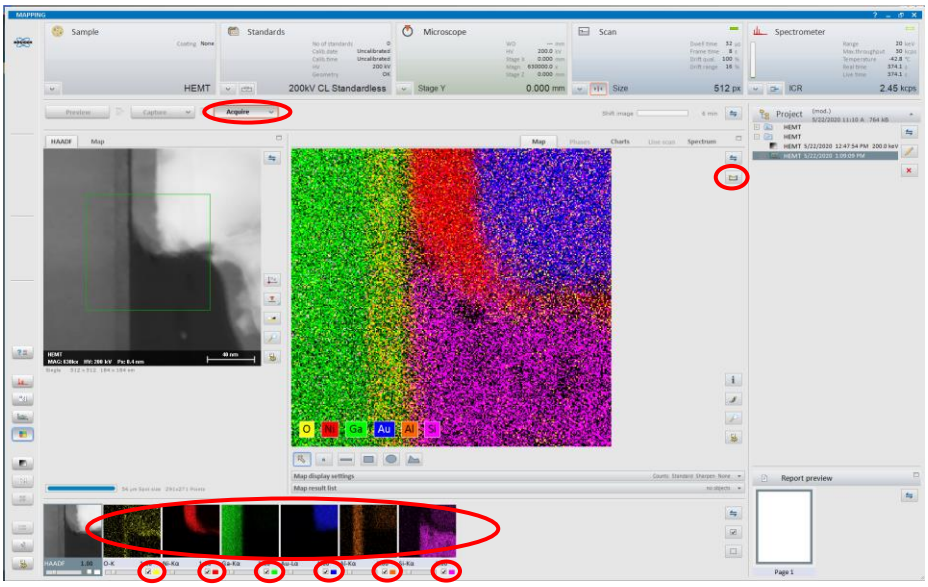
6.2.2. It is <u>not necessary</u> to open the “DRIFT CORRECTION” dialogue box and adjust any parameters for drift correction while mapping.



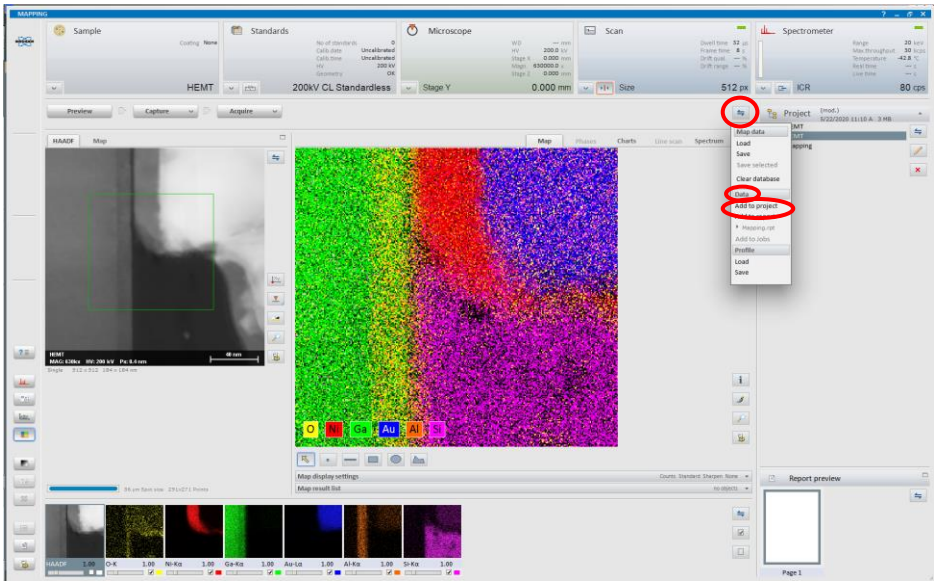
6.3. Select the downward facing arrow next to “Acquire”; under “Map time” select “Manual”.



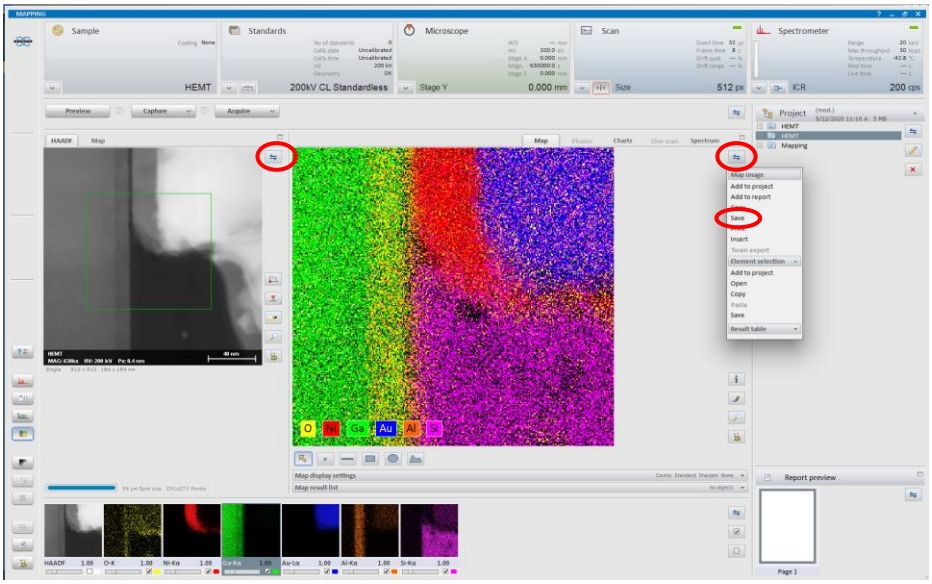
6.4. Select “Acquire” to start mapping; the process to add/remove elements, alter peak selections, and change individual map colors is identical to as described previously for line scans. When the maps are satisfactory, select “Acquire” again to stop mapping.



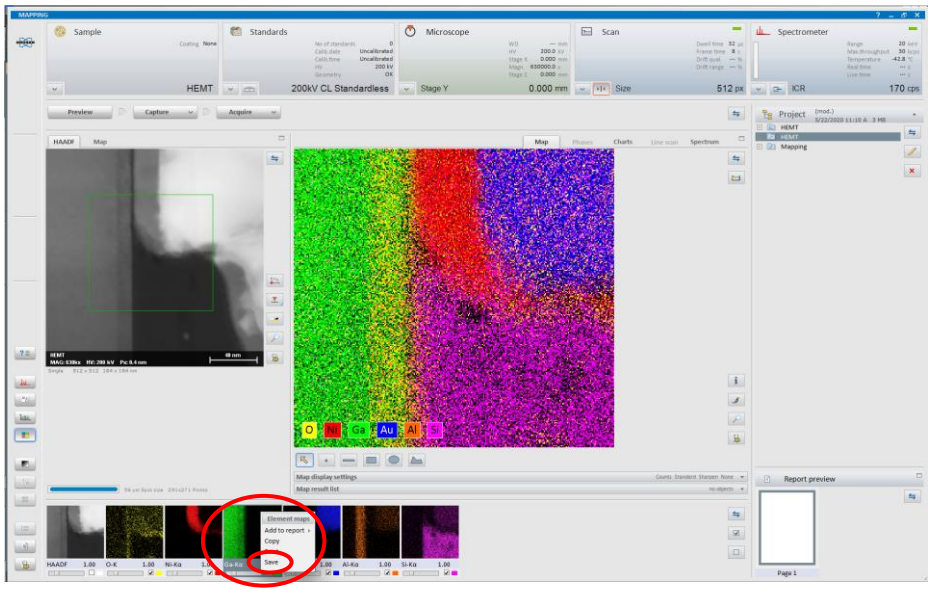
6.5. Be sure to add all necessary elements and/or select peaks before proceeding as adding elements and/or selecting different peaks will not be possible later. Select the I/O icon; under “Data”, select “Add to project”.



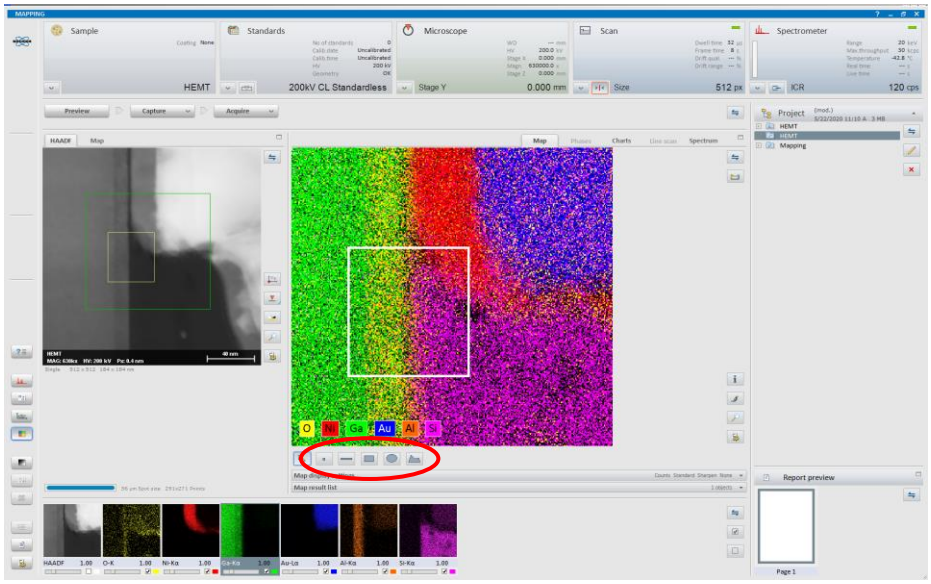
6.6. To save the composite map, select the I/O icon next to the map and then “Save” to save the composite image; to save the reference STEM image with the indicated map area, select the I/O icon next to the STEM image.



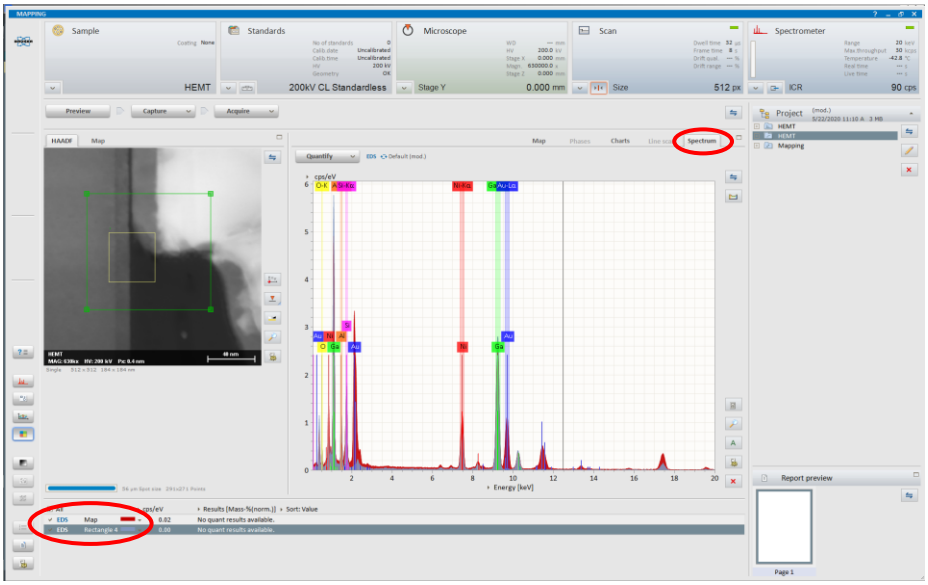
6.7. To save individual elements maps, simply right click on an individual map and select “Save”



6.8. Spectral information may be extracted from all/part of the map. Select an object (point, line, area, etc.) from the icons underneath the composite and draw/position it on the map to select the area to isolate.



6.9. Select the “Spectrum” tab to see the spectra isolated from the selected portion of the map (“Rectangle”, “Point”, etc.) and the whole map (“Map”).



7. Finishing

7.1. Retract the EDS detector (as described elsewhere).

7.2. In Microscope Control, select the “STEM” tab and navigate to the “STEM Imaging” control panel; expand the flap-out arrow and select the “Scan” tab; in the “Scan input” pull-down menu, select “Internal”.