FEI Tecnai F20 S/TEM: EDS system operation

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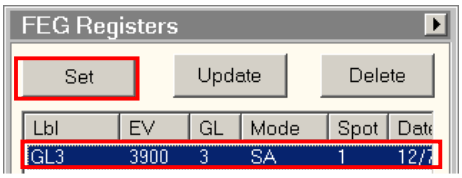
The user should already be familiar with operation of the instrument in STEM mode, use of the Microscope Control interface, and TIA.

1. Holder selection and plasma cleaning for EDS

    1.1. Either the double tilt or single tilt holders may be used for performing EDS. However, if the single tilt holder is used, it must be tilted a specific way to allow for efficient X-ray collection. It is also recommended that plasma cleaning of the specimen be performed (if permissible for the specimen) due to the high beam currents typically used for EDS.

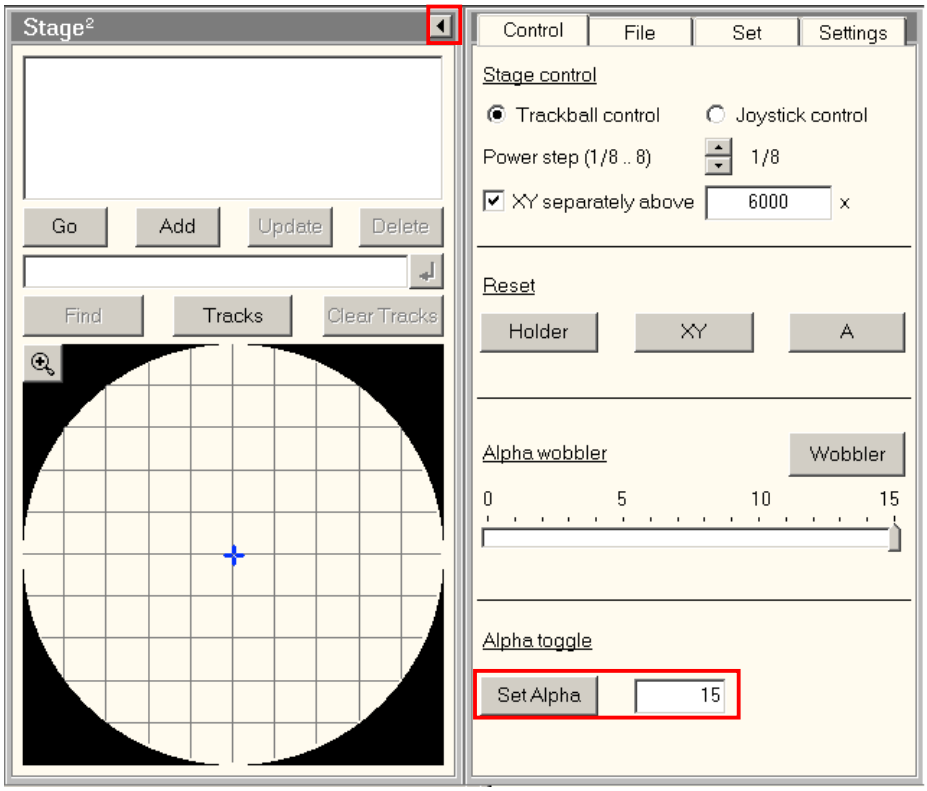
2. Instrument settings for EDS

    2.1. Select and apply the “GL3” FEG register.



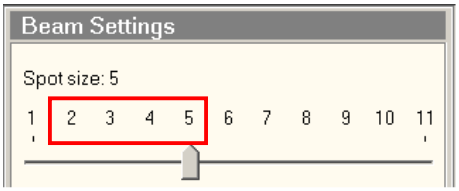
    2.2. Verify that the objective and SA apertures are both retracted (this will prevent overload to the EDS detector).

2.3. If using the single tilt holder, the holder must be tilted in a specific way to allow efficient X-ray collection. In Microscope Control, select the Stage tab; in the "Stage^2" control panel, select the flap-out arrow to expand the panel; under "Alpha toggle", input a value of α = +15° and then select "Set Alpha". Select the flap-out arrow to collapse the panel when finished.



2.4. After setting α = +15°, reset eucentric height for the region of interest.

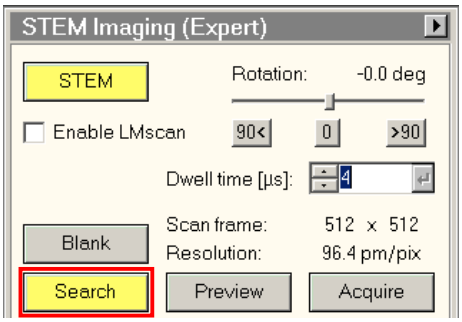
2.5. In Microscope Control, select the STEM tab and enter STEM mode; set spot size = 2 – 5 (after entering STEM mode); align the probe using the same procedure described for general STEM operation (use the #1 C2 aperture).



2.6. Set the camera length as desired (100 mm will produce a HAADF-STEM image) and center the Ronchigram inside the inner rim of the STEM detector.

3. Finding an area of interest

3.1. In Microscope Control, select the EDS tab; navigate to the “STEM Imaging” control panel and select “Search” to acquire a live image in TIA; adjust the magnification to change the field of view and the joystick to move around as needed.

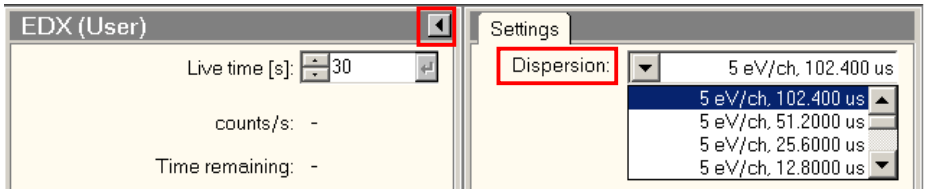


3.2. After locating an area of interest, adjust the scan rotation, focus, and condenser stigmators, if needed.

4. Inserting the EDS detector; detector settings

4.1. In RTEM Control, select “IN” to insert the EDS detector; the “OUT” button will turn gray and the “IN” button will turn red.

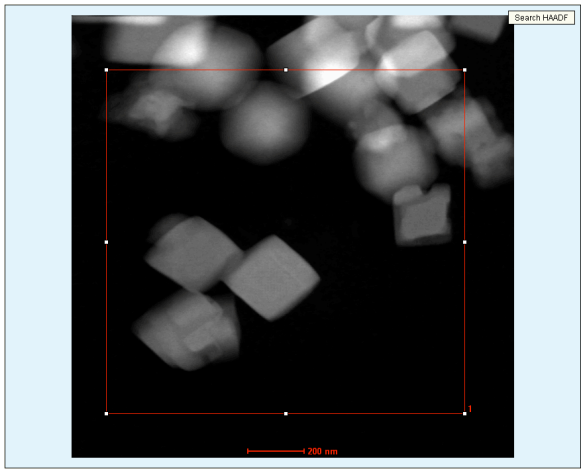
4.2. Microscope Control, select the EDS tab; navigate to the “EDX” control panel and select the flap out arrow to expand the panel. Select “Dispersion” and select from the list of available combinations of eV/ch and process times; 5 eV/ch dispersion will allow detection of X-rays up to 20 keV while 10 eV/ch dispersion will allow detection of X-rays up to 40 keV; longer process times will provide better energy resolution and more accurate processing of low energy (< 2 keV) X-rays, but at the expense of increased dead time. Once a setting has been selected, select the flap out arrow to collapse the panel.



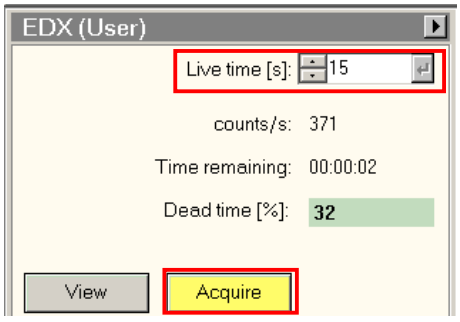
5. Acquiring a survey spectrum

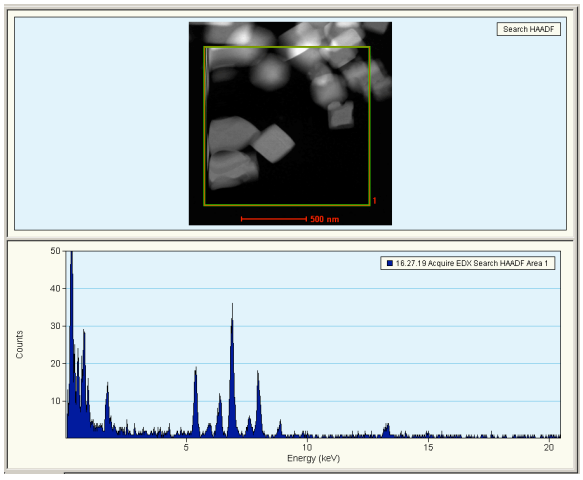
5.1. Make sure you are acquiring a live image using “Search”; otherwise, you cannot collect a survey spectrum.

5.2. In TIA, select the image selection tool and draw a box around the region in the image where a survey specimen is to be collected; the box may be repositioned and resized as needed.



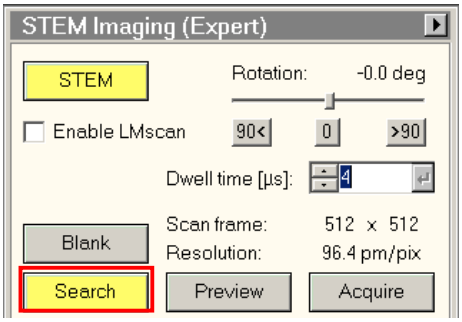
5.3. In Microscope Control, navigate to the EDS tab; under the “EDX” control panel, select “Acquire” to collect a survey spectrum from the defined area (outline will turn green); adjust the “Live time” (the time the detector is actively collecting X-rays) as needed (after starting the acquisition); the survey spectrum will appear below the STEM image in TIA and automatically update until complete.





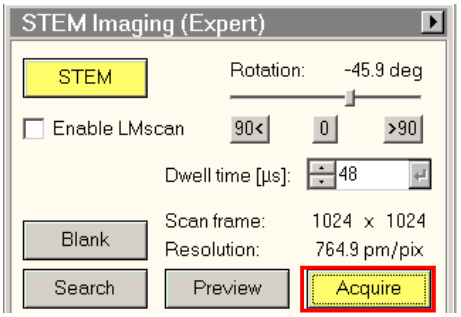
5.4. In TIA, select save to save the display (STEM image and spectrum); then select close to close the display.

5.5. The beam will freeze once the acquisition is complete. To restart the live image, go to Microscope Control and select the EDS tab; navigate to the "STEM Imaging" control panel and select "Search" to start acquiring a new live STEM image.

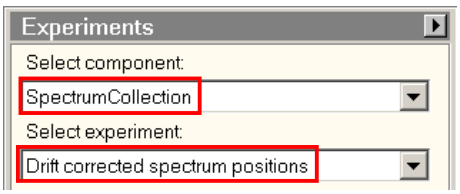


6. Point analysis

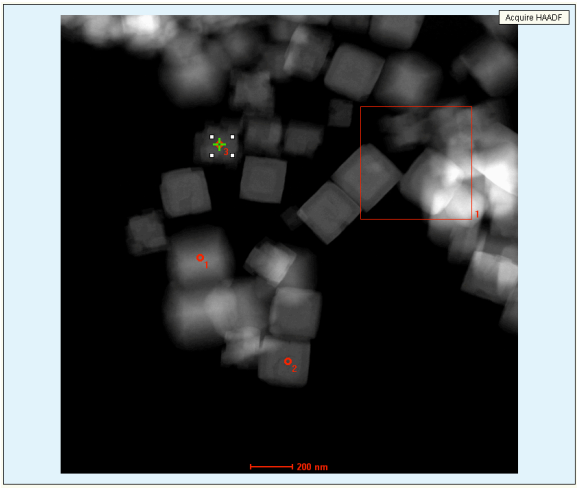
6.1. In Microscope Control, select the EDS tab; navigate to the "STEM Imaging" control panel and select "Acquire" to acquire a STEM image (you must use "Acquire" to collect the image or point analysis won't work).



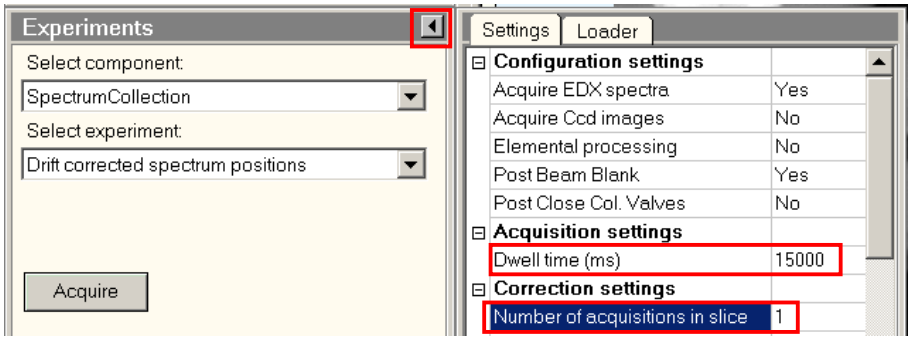
6.2. Navigate to the "Experiments" control panel; under "Select component", select "SpectrumCollection" and under "Select experiment", select "Drift corrected spectrum positions".



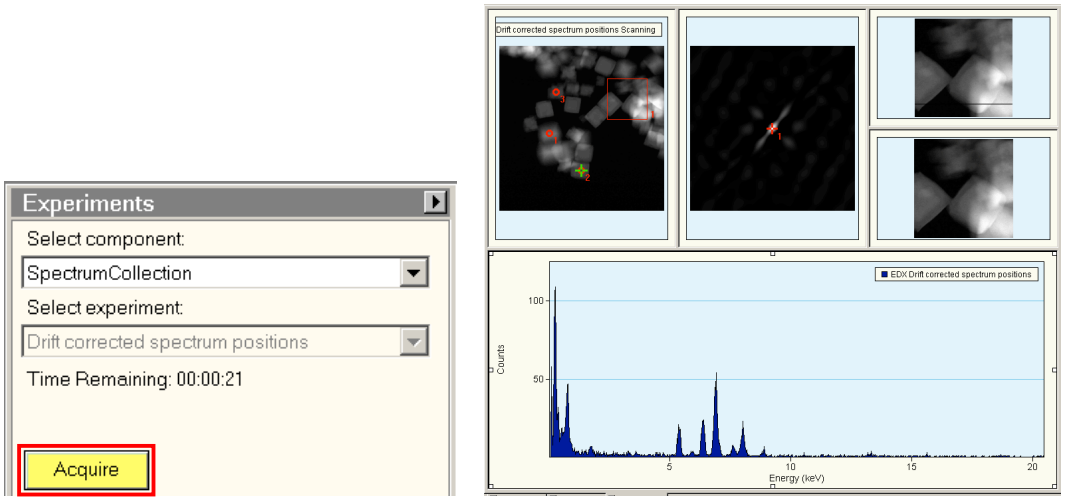
6.3. In TIA, a box and two beam position markers will appear on the image; position the markers where the spectra are to be collected; if you want to add another point, select the beam position marker tool  and click on the image to add another point (repeat for any additional points); then position the box around a distinct region in the image, not containing any of the markers (this will be used as the reference for drift correction).



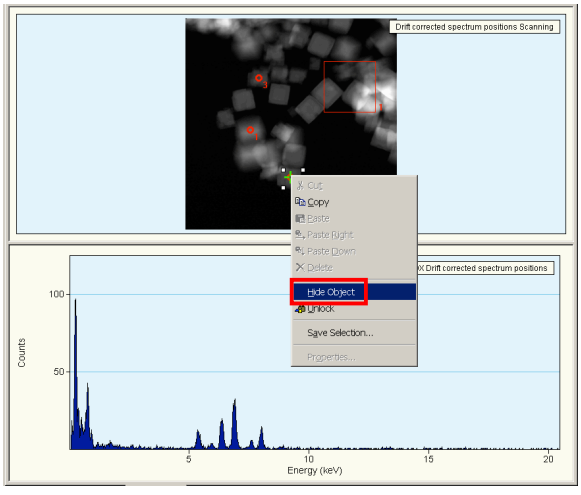
6.4. In Microscope Control, navigate to the “Experiments” control panel and select the flap out arrow  to expand the panel; under “Acquisition settings”, enter the dwell time to be used for each point; under “Correction settings”, make sure “Number of acquisitions in slice” is set to 1; when finished, select the flap out arrow  to collapse the panel.



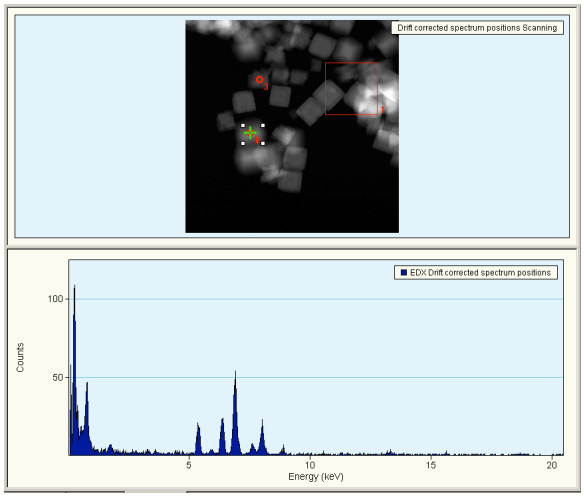
6.5. In the “Experiments” control panel, select “Acquire” to start collecting the spectra at the defined positions; after each spectrum is collected, it will be displayed below the STEM image in TIA (the final spectrum shown will be the one collected from the last point).



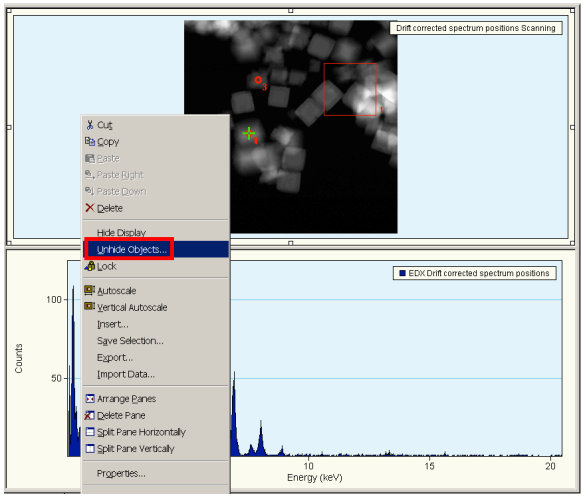
6.6. Once acquisition is complete and you want to view the individual spectra collected at the different points, you must first hide the beam position markers. To do this, simply right click on a beam position marker and select “Hide Object” and repeat for all remaining beam markers.



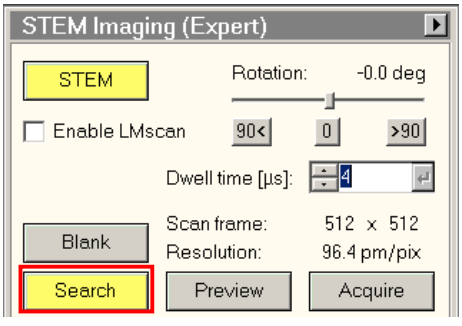
6.7. In TIA, select the image position marker tool and click anywhere on the image; then hold down the “Alt” key and click and drag it onto the spectrum below the image (the actual dark blue portion, not the background); the marker will now snap to a beam position marker and the spectrum for that point will appear. You can now simply click and drag the image position marker and it will snap to all the beam position markers and show the spectrum for each position.



6.8. To reveal the beam position markers again, right click on the light blue background in the panel containing the image and select “Unhide Objects”.

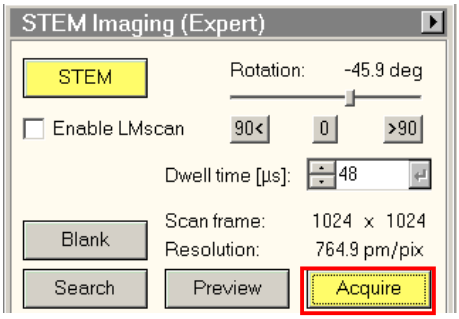


6.9. To restart the live image after acquisition, navigate to the “STEM Imaging” control panel and select “Search” to start acquiring a new live STEM image.

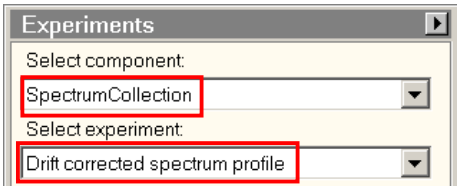


7. Spectrum profiles (line scans)

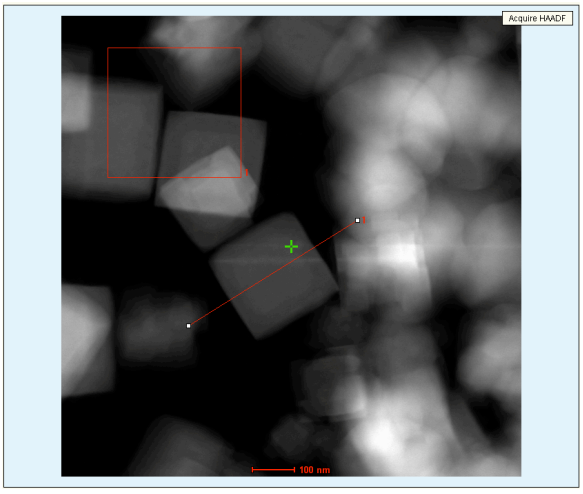
7.1. In Microscope Control, select the EDS tab; navigate to the “STEM Imaging” control panel and select “Acquire” to acquire a STEM image (you must use acquire to collect the image or spectrum profiles cannot be collected).



7.2. Navigate to the “Experiments” control panel; under “Select component”, select “SpectrumCollection” and under “Select experiment”, select “Drift corrected spectrum profile”.



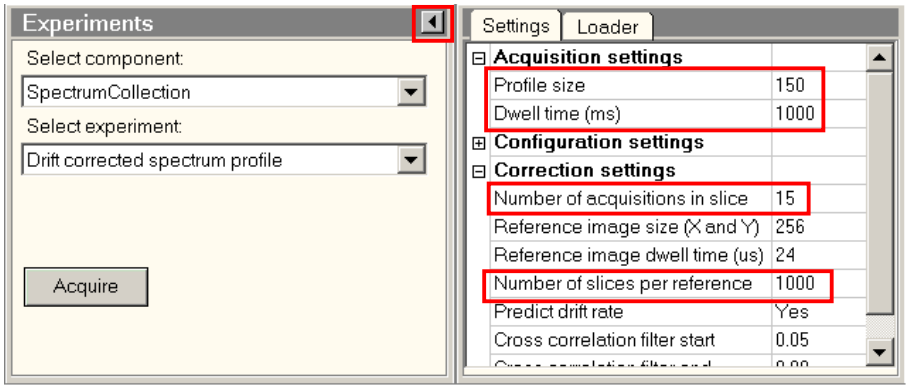
7.3. In TIA, a box and line marker will appear on the image; position/resize the line to where you want to collect the spectrum profile; then position the box around a distinct region in the image, not containing line marker (this will be used as the reference for drift correction).



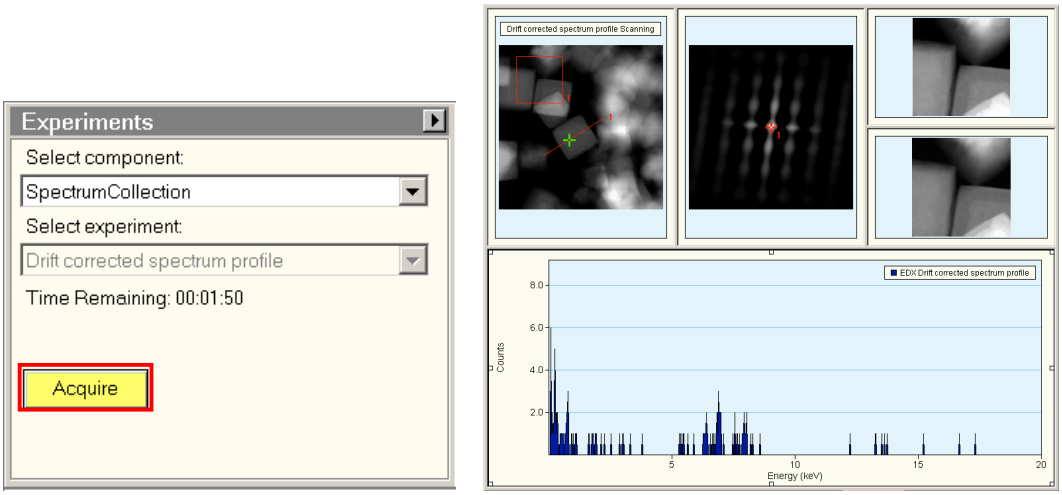
7.4. In Microscope Control, navigate to the “Experiments” control panel and select the flap out arrow to expand the panel

7.4.1. Under “Acquisition settings”, enter the desired “Profile size” (number of points in the spectrum) and the “Dwell time” (how long the probe collects a spectrum at each point);

7.4.2. Under “Correction settings”, “Number of acquisitions in slice” should be set so a reference image is collected at least every 10 – 15 s (at most); “Number of slices per reference” should be equal to the profile size; when finished, select the flap out arrow to collapse the panel.

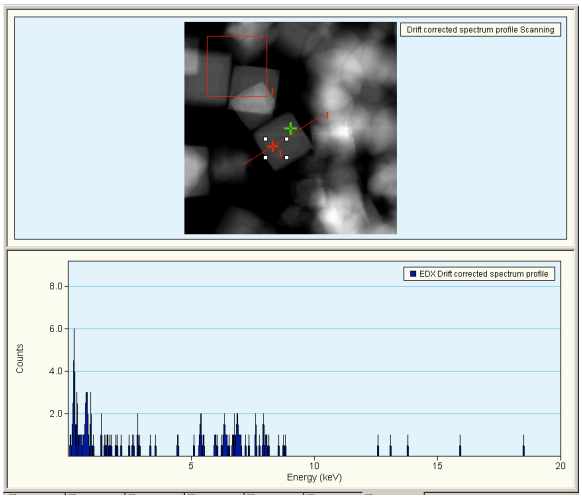


7.5. In the “Experiments” control panel, select “Acquire” to start collecting the spectrum profile; after each spectrum at each point is collected, it will be displayed below the STEM image in TIA (the final spectrum shown will be the one collected from the last point in the profile).

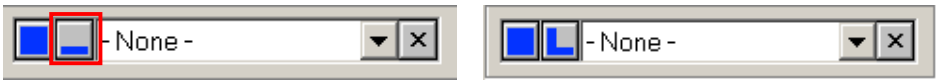


7.6. Once acquisition is complete you can view the spectrum collected at any point on the profile; select the image position marker tool and click anywhere on the image; then hold down the “Alt” key and click and drag it onto the spectrum below the image (the actual dark blue portion, not the background); the marker will now snap to the line marker and the spectrum for that point will appear. You can now simply click and drag the image position marker along the line and it will snap to all the beam position markers and show the spectrum for each position.

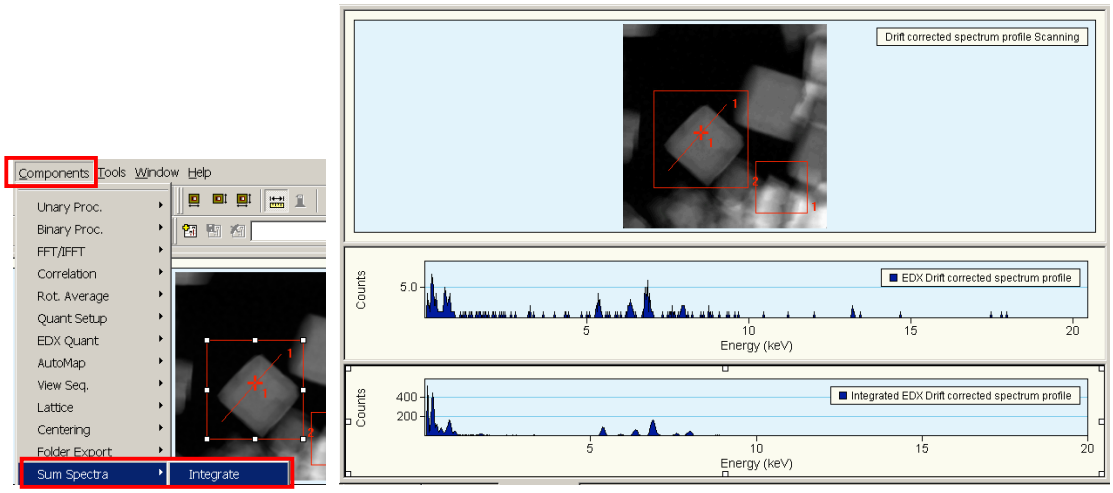
Do not delete the pane containing the spectrum corresponding to the image position marker; otherwise, the information needed to generate the profiles will be lost.



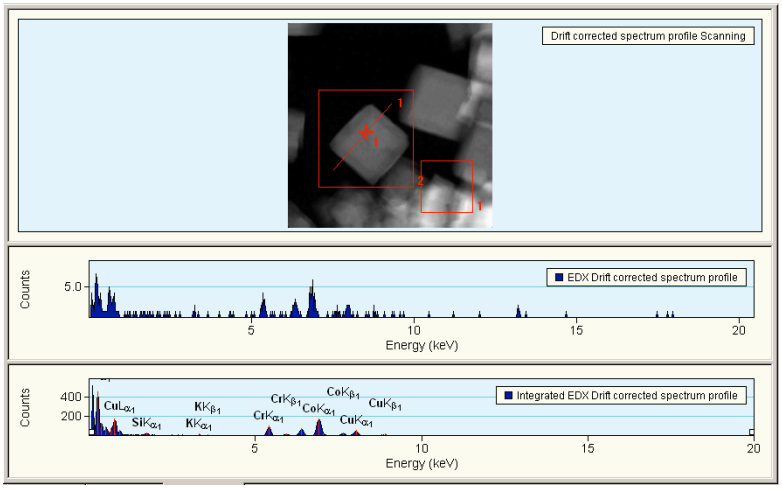
7.7. To generate the profiles for the elements of interest, the data must first be saved and TIA must then be expanded to analysis mode; select from the lower information panel; the button will change to and TIA will be in analysis mode.



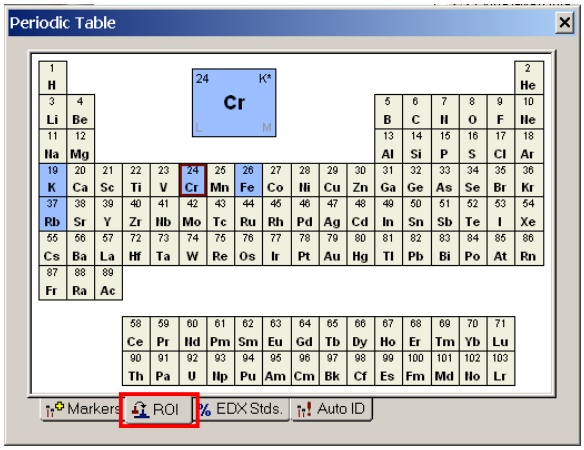
7.8. It will be difficult to identify individual peaks in a spectrum from a single point, so the sum spectrum from all the points should be used for this. To generate the sum spectrum, select the image selection tool and draw a box around the entire line on the STEM image; then select “Components” from the pull-down menu, then “Sum Spectra” and then “Integrate”; the sum spectrum will be generated in a new pane below the pane containing the spectrum from a single point.



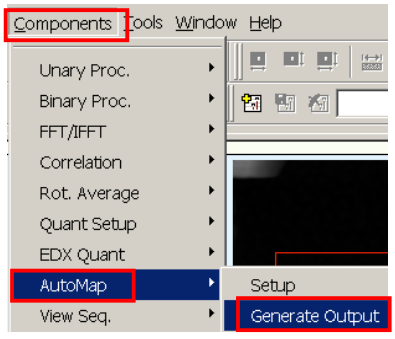
7.9. Select the sum spectrum and then select auto ID to automatically identify the different peaks (this is usually sufficiently accurate) in the sum spectrum; you can also select KLM labels button to further characterize the peaks by transition.

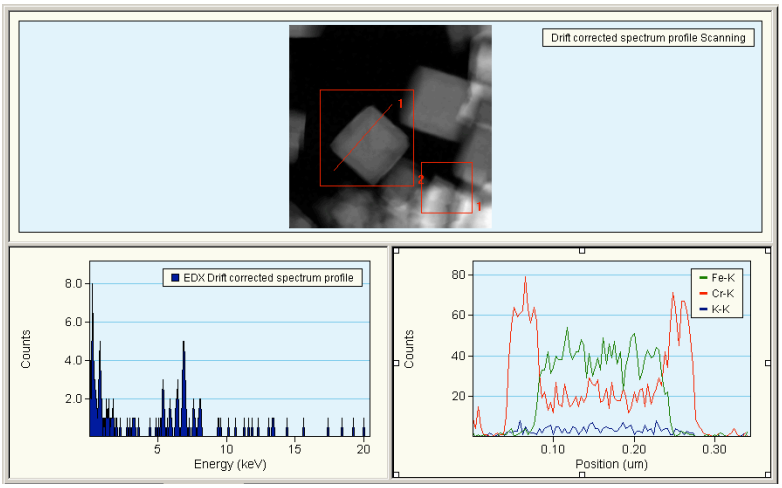


7.10. Select the periodic table button ; a periodic table dialogue box will pop up. Select the “ROI” tab ROI to choose which elements will or will not be used to generate the profiles. Each time you select an element, you can select which X-ray peak(s) to use for mapping by selecting from the K L M buttons



7.11. Click on the sum spectrum (dark blue portion); then select “Components” from the pull down menu, then “AutoMap”, then “Generate Output”; a new pane will be generated containing the profiles for each of the selected X-ray peaks. After doing this, it is a good idea to save the data

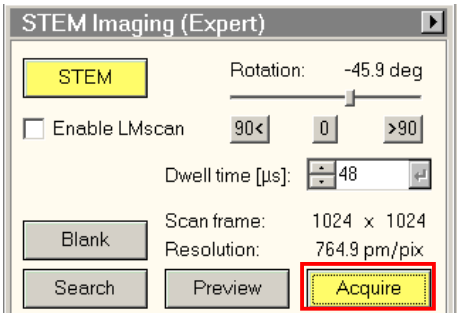




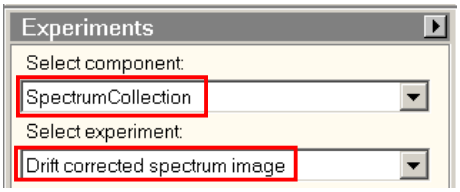
7.12. When finished, select to switch from analysis mode back to acquisition mode .

8. Spectrum imaging (mapping)

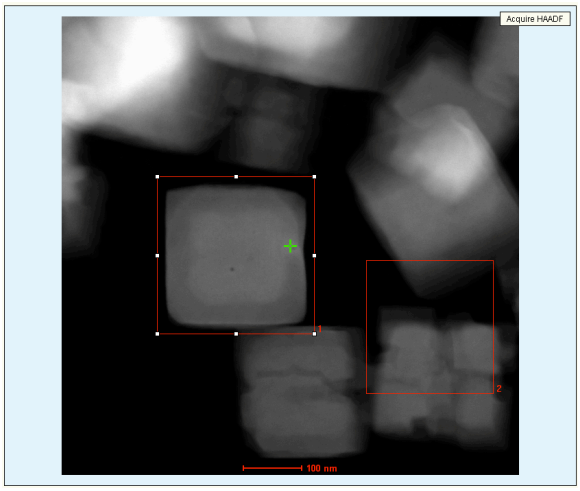
8.1. In Microscope Control, select the EDS tab; navigate to the “STEM Imaging” control panel and select “Acquire” to acquire a STEM image (you must use acquire to collect the image or spectrum imaging cannot be performed).



8.2. Navigate to the “Experiments” control panel; under “Select component”, select “SpectrumCollection” and under “Select experiment”, select “Drift corrected spectrum image”.



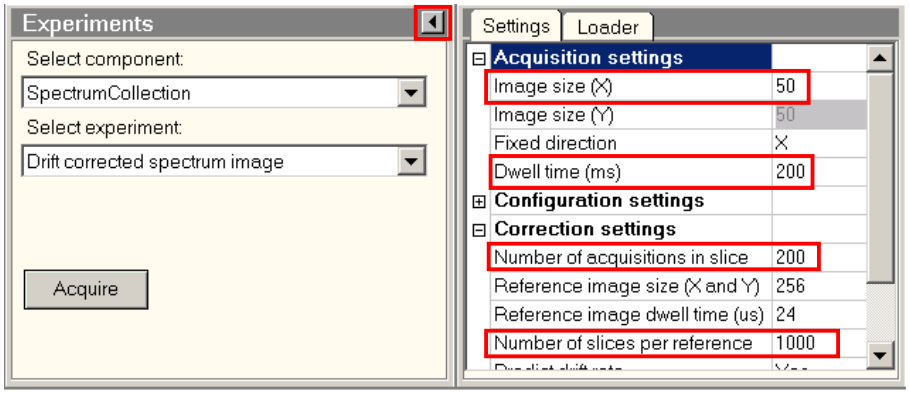
8.3. In TIA, 2 boxes will appear on the image; the large box is the area where spectrum imaging will be performed and small box is the area that will be used as the reference for drift correction; resize and reposition the boxes as necessary (the two boxes should not overlap and the box used for drift correction should contain distinct features).



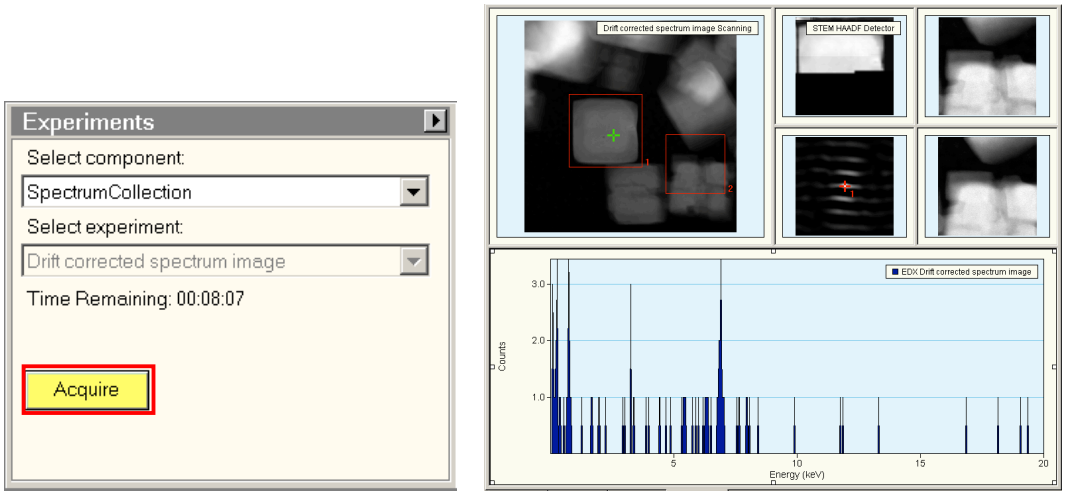
8.4. In Microscope Control, navigate to the “Experiments” control panel and select the flap out arrow to expand the panel; under “Acquisition settings”

8.4.1. Enter the desired “Image size” (how wide the imaged area is in terms of points) and the “Dwell time” (how long the probe collects a spectrum at each point)

8.4.2. Under “Correction settings”, “Number of acquisitions in slice” should be a multiple of “Image size” and be chosen so a reference image is collected at least every 60 s; “Number of slices in reference” should be equal to the total number of pixels in the map; when finished, select the flap out arrow to collapse the panel.

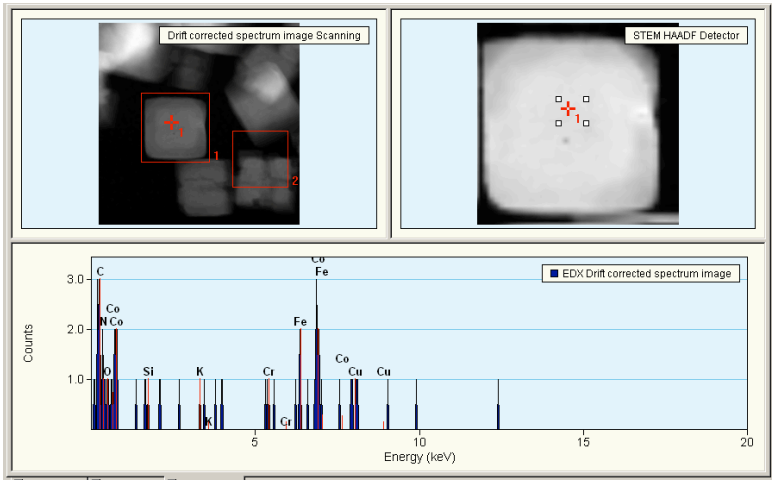


8.5. In the “Experiments” control panel, select “Acquire” to start collecting the spectrum image; after each spectrum at each point is collected, it will be displayed below the STEM image in TIA (the final spectrum shown will be the one collected from the last point in the defining region).

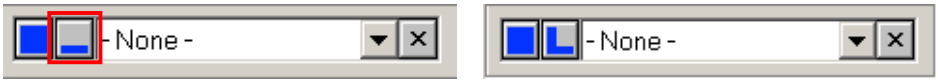


8.6. Once acquisition is complete you can view the spectrum collected at any point in the area used for spectrum imaging; simply click and drag on the image position marker tool in the area (you do not need to add a new marker) and the spectrum collected at that position will be shown below.

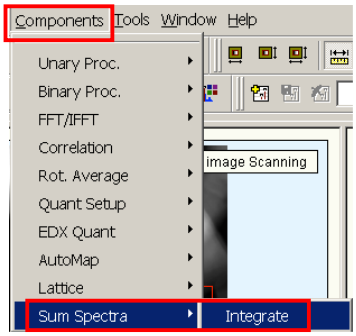
Do not delete the pane containing the spectrum corresponding to the image position marker; otherwise, the information needed to generate the maps will be lost.

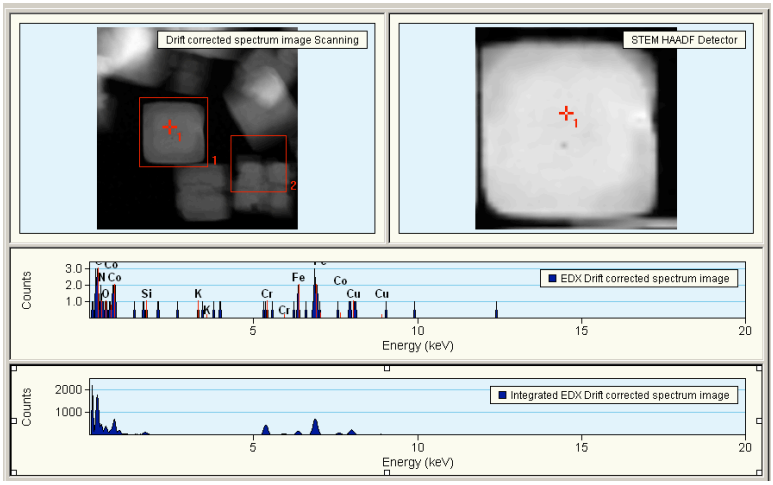


8.7. To generate the spectrum images for the elements of interest, the data must first be saved and TIA must then be expanded to analysis mode; select from the lower information panel; the button will change to and TIA will be in analysis mode.

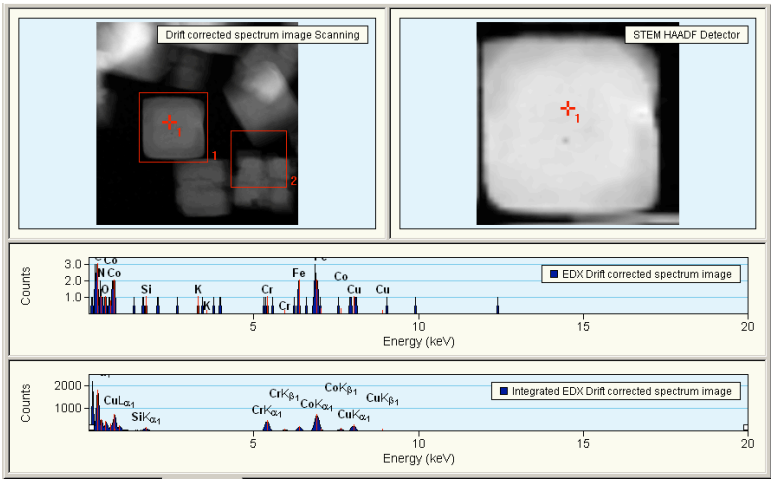


8.8. It will be difficult to identify individual peaks in a spectrum from a single point, so the sum spectrum from all the points should be used for this. To generate the sum spectrum, select the red square used to define the area for spectrum imaging; then select “Components” from the pull-down menu, then “Sum Spectra” and then “Integrate”; the sum spectrum will be generated in a new pane below the pane containing the spectrum from a single point.

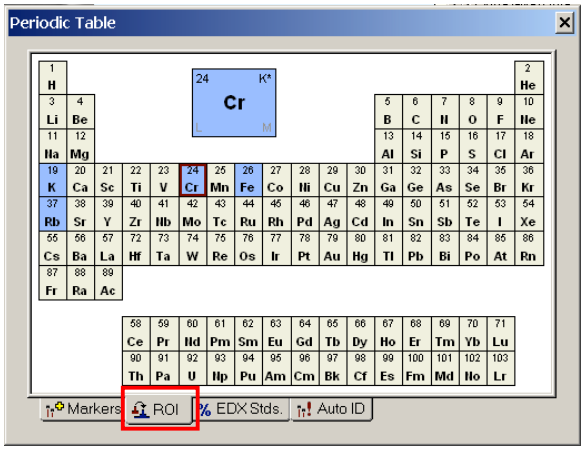




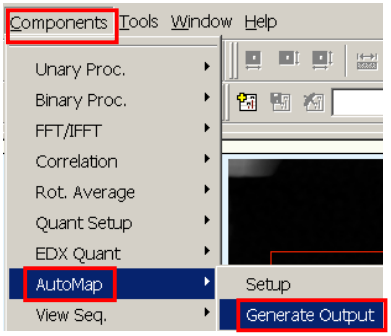
8.9. Select the sum spectrum and then select auto ID  to automatically identify the different peaks (this is usually sufficiently accurate) in the sum spectrum; you can also select KLM labels button  to further characterize the peaks by transition.

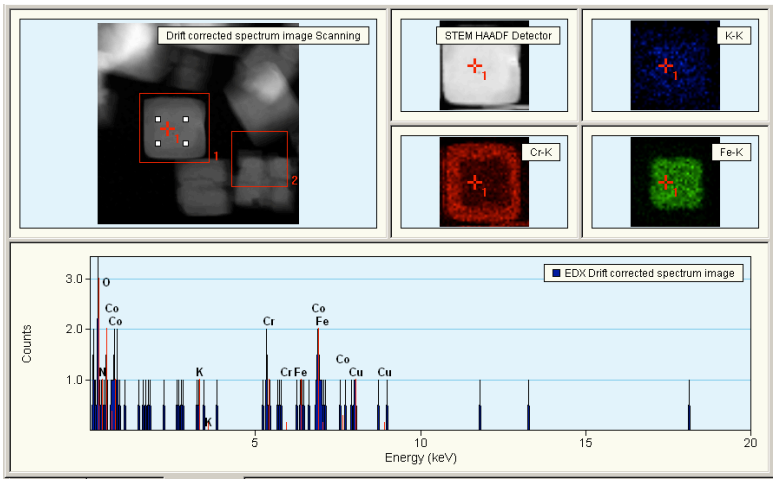


8.10. Select the periodic table button  ; a periodic table dialogue box will pop up. Select the “ROI” tab  to choose while elements will or will not be used to generate the spectrum images. Each time you select an element, you can select which X-ray peak(s) to use for mapping by selecting from the  buttons.



8.11. Click on the sum spectrum (dark blue portion); then select “Components” from the pull down menu, then “AutoMap”, then “Generate Output”; for each of the selected X-ray peaks, a new pane will be generated showing the X-ray map. After doing this, it is a good idea to save the data 

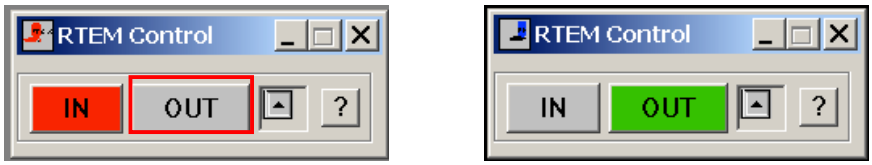




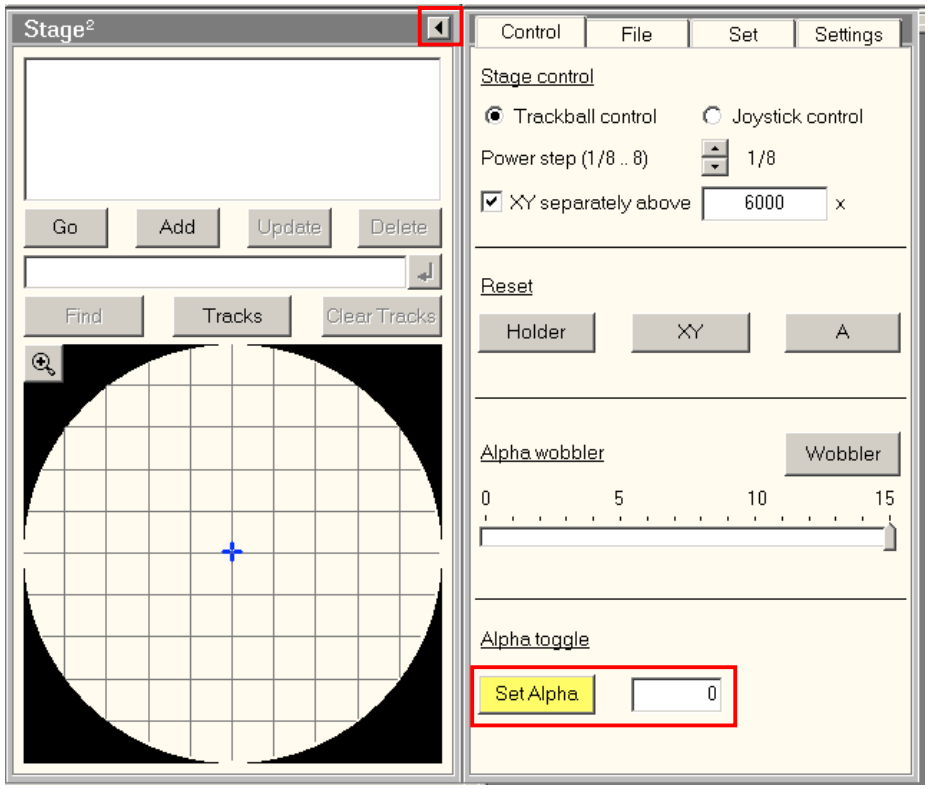
8.12. When finished, select  to switch from analysis mode back to acquisition mode  .

9. Finishing

9.1. In RTEM Control, select “OUT” to retract the EDS detector; the “OUT” button will turn green and the “IN” button will turn gray. Do not leave the EDS detector inserted unless it is actively in use.



9.2. If the single tilt holder was used, the alpha tilt needs to be reset. In Microscope Control, select the Stage tab; in the “Stage^2” control panel, select the flap-out arrow to expand the panel; under “Alpha toggle”, input a value of $\alpha = 0°$ and then select “Set Alpha”. Select the flap-out arrow to collapse the panel when finished.



9.3. In Microscope Control, select the STEM tab and exit STEM mode; follow all the other procedures for returning to TEM mode from STEM mode.

Appendix: shorthand procedure for performing EDS in STEM mode

1. Verifying objective/SA apertures retracted
2. If using single-tilt holder, set α = +15°
3. Bring sample to eucentric height
4. Enter STEM mode
5. Select spot size
6. Perform STEM alignment
7. Start STEM imaging
8. Insert EDS detector
9. Set EDS detector settings
10. Find area of interest and focus image
11. Acquiring survey spectrum
    a. Draw box around region of interest in live STEM image
    b. Select “Acquire” in EDX control panel
12. Point analysis
    a. Acquire STEM image
    b. Set experiment for “Drift corrected spectrum positions”
    c. Position/resize drift correction reference box
    d. Position and/or add beam position markers
    e. Set experiment settings
    f. Select “Acquire” in Experiments panel
13. Spectrum profiles
    a. Acquire STEM image
    b. Set experiment for “Drift corrected spectrum profiles”
    c. Position/resize drift correction reference box
    d. Position/resize line marker
    e. Set experiment settings
    f. Select “Acquire” in Experiments panel
    g. Save panel and switch to analysis mode
    h. Draw box around line marker and generate sum spectrum
    i. Open periodic table, select “ROI” tab, select elements/peaks of interest
    j. Generate profiles
14. Spectrum imaging
    a. Acquire STEM image
    b. Set experiment for “Drift corrected spectrum profiles”
    c. Position/resize drift correction reference box
    d. Position/resize spectrum image box
    e. Set experiment settings
    f. Select “Acquire” in Experiments panel
    g. Save panel and switch to analysis mode
    h. Select spectrum image box and generate sum spectrum
    i. Open periodic table, select “ROI” tab, select elements/peaks of interest
    j. Generate images
15. Retract EDS detector; if using single-tilt holder, set α = 0°,
16. Exit STEM mode