

## FEI Helios G4 PFIB CXe: EDS using EDAX APEX

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### **\*ANALYSIS OF RADIOACTIVE SPECIMENS IS STRICTLY PROHIBITED\***

This document assumes proficiency in basic operation of the instrument and Microscope Control. Additionally, this document only covers the use of APEX to collect typical EDS data, it does not otherwise exhaustively cover all the capabilities of APEX, including EDS data processing. The interested user is referred to the APEX EDS manual for more information (posted under the “Docs” page for the PFIB) or may contact RSC staff.

#### 1. Sample considerations

- 1.1. Specimen mounting and preparation considerations are identical to as described previously for general operation; again, use of well-dried C paint is recommended to improve specimen stability and minimize drift.
- 1.2. Samples with substantial topographical variations (> 1  $\mu\text{m}$ ) tend to be problematic for EDS due to shadowing effects preventing clear line of sight from the point of X-ray generation to the EDS detector. EDS can still be performed on rough samples, but certain areas may experience reduced or minimal EDS signal due to this shadowing.

#### 2. Specimen loading

- 2.1. Typically, specimens for EDS should be loaded in one of the 18 positions on the flat section of the multi-purpose holder; that being said, it is also possible to perform EDS on specimens loaded on the 45° pre-tilted section.
  - 2.1.1. This document otherwise assumes specimens are loaded on the flat section of the multi-purpose holder.
  - 2.1.2. If needing to perform EDS on specimens loaded on the 45° pre-tilted section, please ask for RSC staff assistance.



### 3. Plasma cleaning

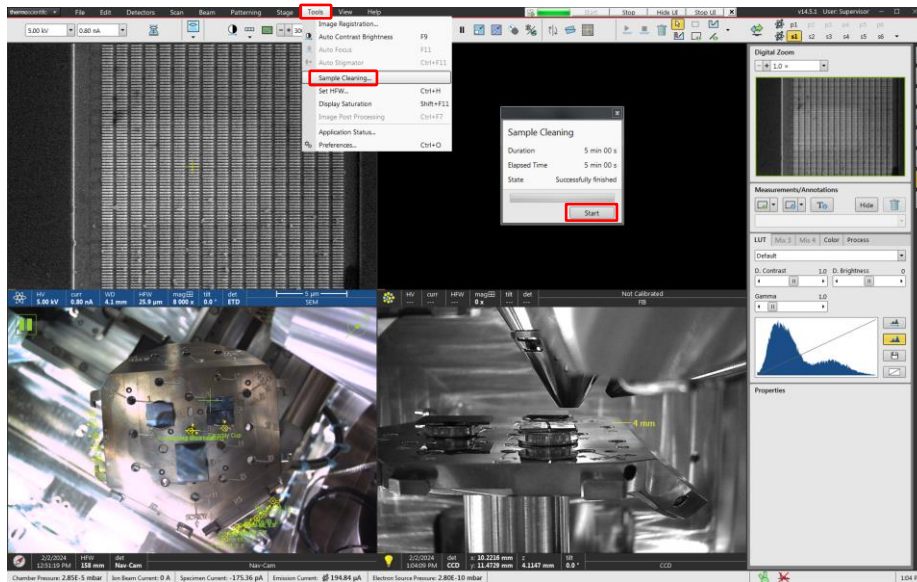
3.1. Once all specimens are loaded and the chamber returned to vacuum, it is recommended to plasma clean the specimens to minimize/prevent carbonaceous contamination build-up during EDS.

3.2. In Microscope Control, navigate to the “Tools” pull-down menu, then select “Sample Cleaning”.

3.2.1. In the “Sample Cleaning” window, select “Start”; the process will take about 10 min total to complete with 5 min of actual cleaning time. If desired, additional cleaning cycles may be employed.

3.2.2. NOTE: even with plasma cleaning, specimens may still experience carbonaceous contamination build-up.

3.2.3. The RSC is not responsible for carbonaceous contamination build-up on specimens.



#### 4. SEM settings

4.1. Choosing an optimal SEM voltage for EDS depends on both the X-rays to be detected along with the spatial resolution that is needed. Generally, the voltage should be ~2x higher than the highest energy X-ray of interest with lower voltages improving spatial resolution and providing higher surface sensitivity.

4.1.1. In general, higher voltages will be better for detecting high Z elements, but high Z elements will often generate characteristic X-rays over a wide energy range, so use of a high voltage may not be strictly necessary.

4.2. The beam current may be as low as 400 pA to as high as 6.4 nA; lower currents are necessary when high spatial resolution is needed.

4.2.1. NOTE: as probe size decreases with decreasing current, the tradeoff with improved spatial resolution will be reduced X-ray signal.

4.3. Please consult with RSC staff if unsure of beam settings to use for EDS.

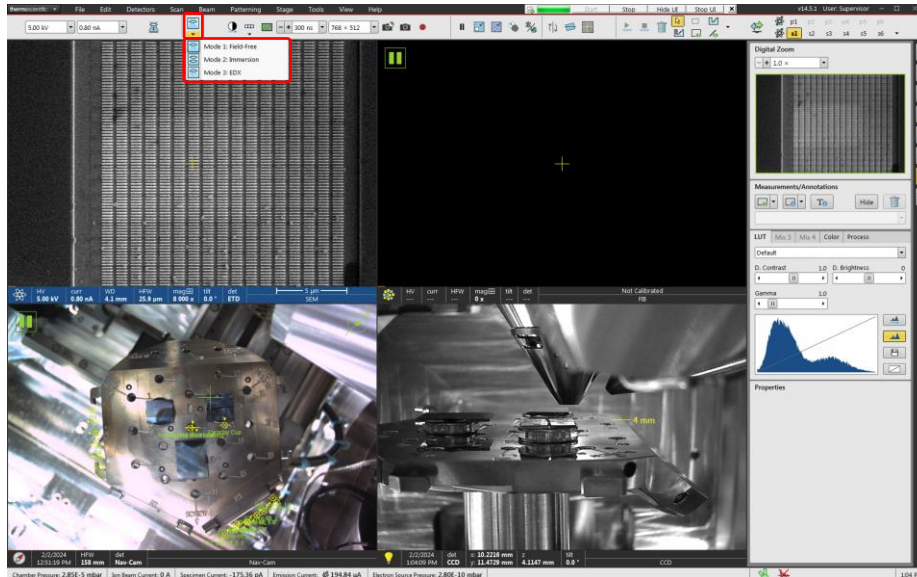
4.4. Navigate to the specimen, find an area of interest, and bring it to WD = 4 mm.

4.4.1. This is the WD for optimal EDS detector performance (most efficient X-ray collection).

4.4.2. NOTE: WD = 4 mm is the same WD approximately corresponding to eucentric height.

4.5. In the Microscope Control toolbar, select the SEM mode to be used; EDS may be performed in either “Field-free” or “Immersion” mode.

4.5.1. “Immersion” mode is recommended only when needing the highest possible spatial resolution (at or near the nanoscale); otherwise, “Field-free” mode is usually sufficient.



4.6. If using “Field-free” mode, perform the full SEM alignment now: source centering, lens alignment, and astigmatism correction (not shown).

4.6.1. If using “Immersion” mode, wait to perform alignment until the EDS detector is inserted as this will alter the SEM alignment.

## 5. Starting APEX EDS

5.1. Start APEX EDS; a window will pop up prompting for a project to be created and then a sample for the project.

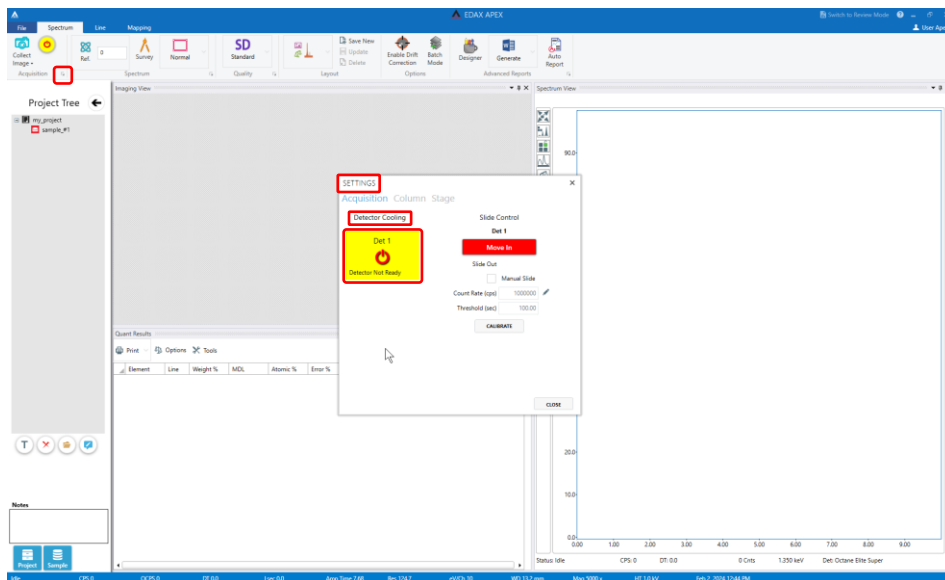
5.1.1. NOTE: there are three separate APEX programs: APEX EDS, APEX EBSD, and APEX Suites; make sure to select APEX EDS.

5.1.2. NOTE: when saving APEX projects, always save these to the “DATA E” drive on the EDAX PC.

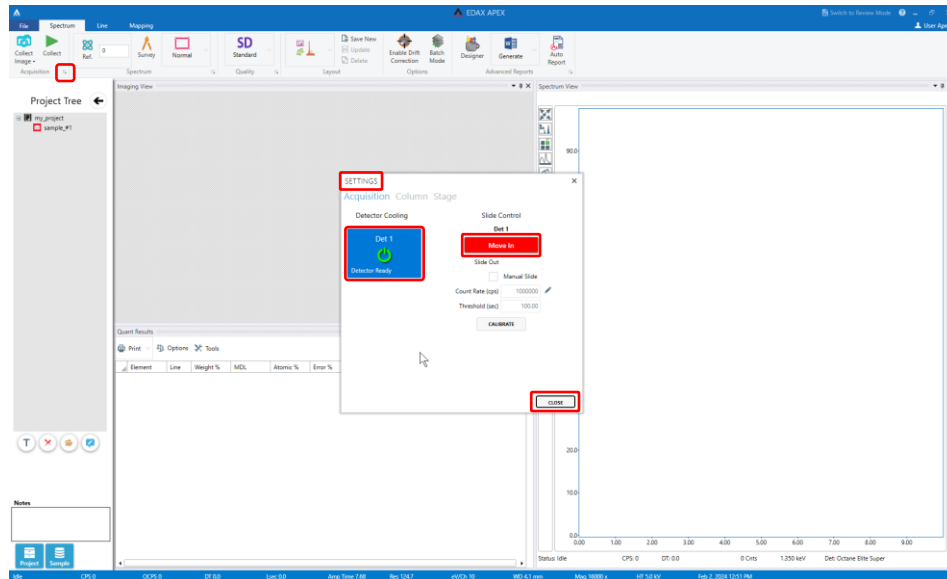
5.2. In the APEX toolbar in the “Acquisition” field, select the arrow to open the “Settings” window; under “Detector Cooling”, select “Det 1” to start cooling.

5.2.1. This will take several minutes to complete; leave the “Settings” window open to monitor the progress.

5.2.2. Cooling is complete when “Det 1” shows “Detector Ready”; continue to leave the “Settings” window open.

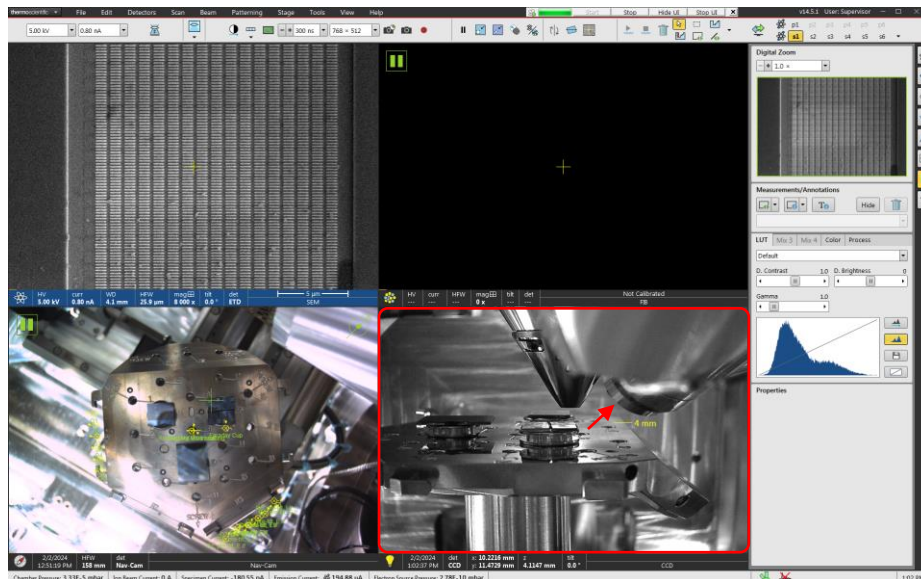


- 5.3. In the “Settings” window, select “Move In” to automatically move in the EDS detector; the “Settings” window may now be closed.



- 5.4. In Microscope Control, observe the CCD quad and verify the EDS detector is fully inserted.

- 5.4.1. NOTE: if using “Immersion” mode, now perform the full SEM alignment: source centering, lens alignment, and astigmatism correction (not shown).



## 6. Enabling Drift Correction

6.1. Even with a very stable specimen, it is best practice to use drift correction when performing linescan or map collection.

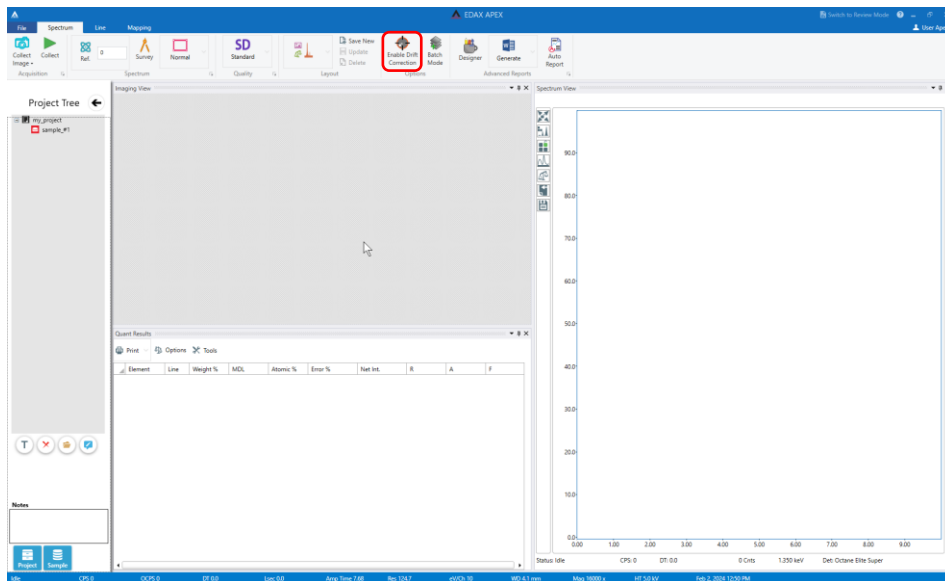
6.1.1. Specifically, use of drift correction is critical when performing linescan or map collection requiring high spatial resolution (at or near the nanoscale).

6.2. Select “Enable Drift Correction” from the toolbar to enable drift correction; it will now be highlighted.

6.2.1. A survey SEM image will automatically be collected in APEX and the FOV in the SEM image in Microscope Control will increase by a factor of 2.

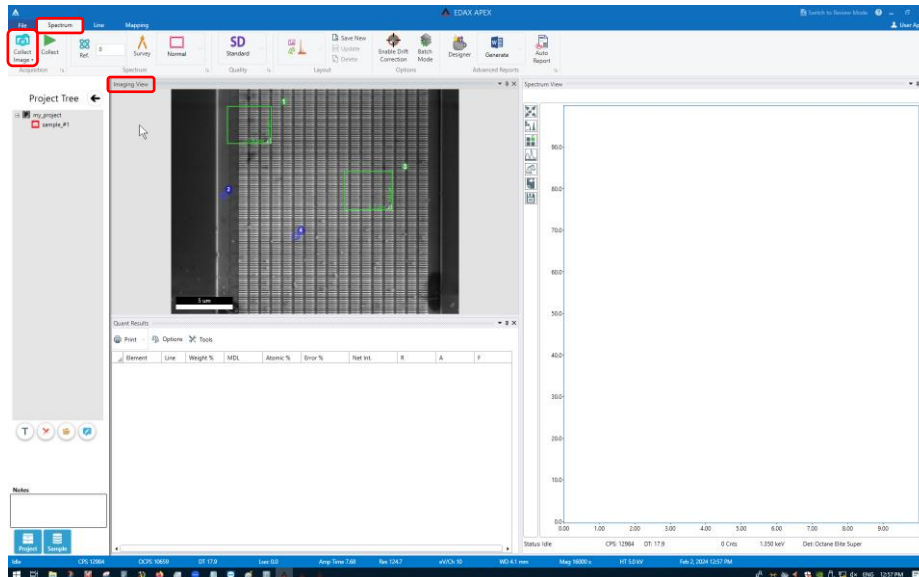
6.2.2. With drift correction enabled, any additional SEM images collected in APEX will possess half the FOV observed in the SEM images in Microscope Control; this is normal.

6.3. Once enabled, drift correction will remain active for all subsequent data collected (spectra, lines, or maps) until it is disabled, or APEX is closed.



## 7. Spectrum mode

- 7.1. In Microscope Control, center the ROI in the live SEM image and then optimize the image (set magnification, focus, etc.).
- 7.2. In APEX, select the “Spectrum” tab and then “Collect Image” from the toolbar to collect the SEM image in the “Imaging View” panel.
  - 7.2.1. Drift correction is not usually used for spectrum collection, but remember, if drift correction is enabled, the FOV in APEX will be smaller than the FOV in Microscope Control by a factor of 2.
  - 7.2.2. In Microscope Control, the CCD camera will automatically pause; this prevents interference of the CCD camera with the EDS detector.
- 7.3. Click on the SEM image to select points for spectrum collection and/or click and drag on the SEM image to create rectangular areas for spectrum collection.
  - 7.3.1. If no points or areas are selected/created in the SEM image, the whole ROI will be used for spectrum collection.

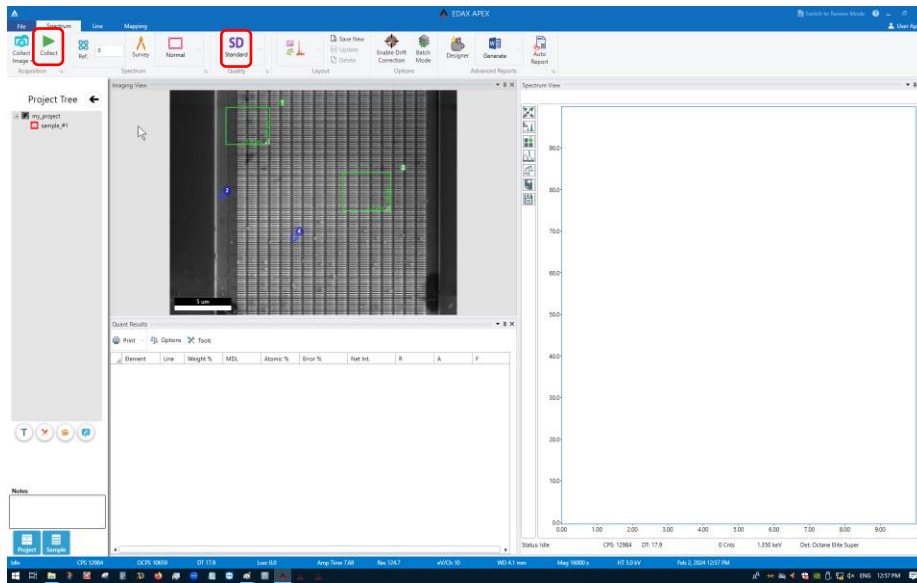




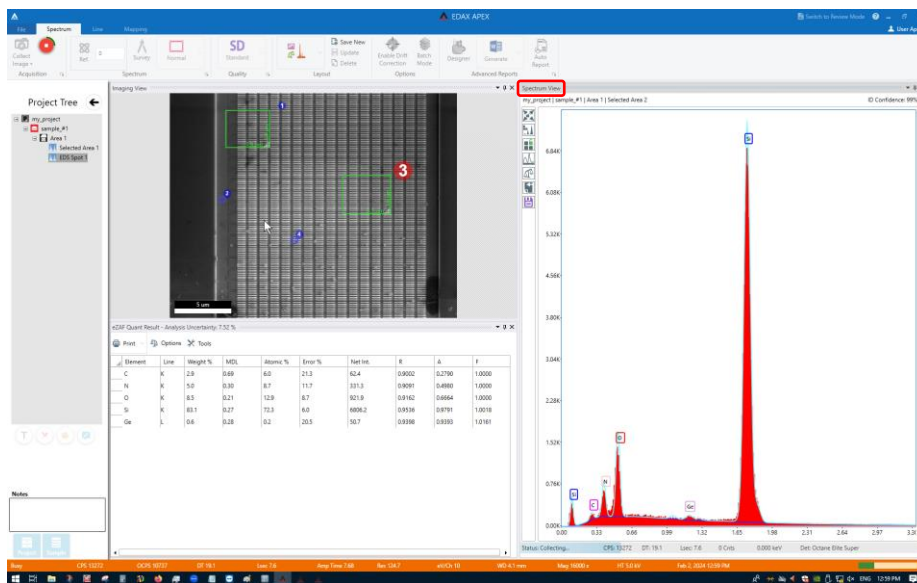
7.4. In the APEX toolbar, select either “Q”, “SD”, or “HD” quality for acquiring the spectra.

7.4.1. The only difference between quality settings is simply the time allotted to acquire each spectrum: 20, 40, or 60 s, respectively.

7.5. In the APEX toolbar, select “Collect” to start collecting the spectra.

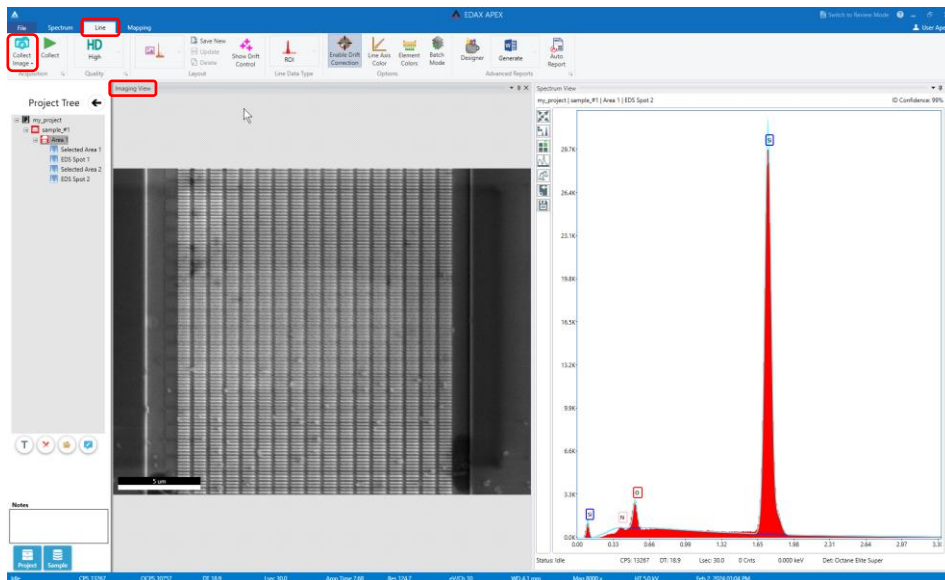


7.6. The spectra will be collected in the order of being selected and/or drawn and shown in the adjacent “Spectrum View” panel.



## 8. Line mode

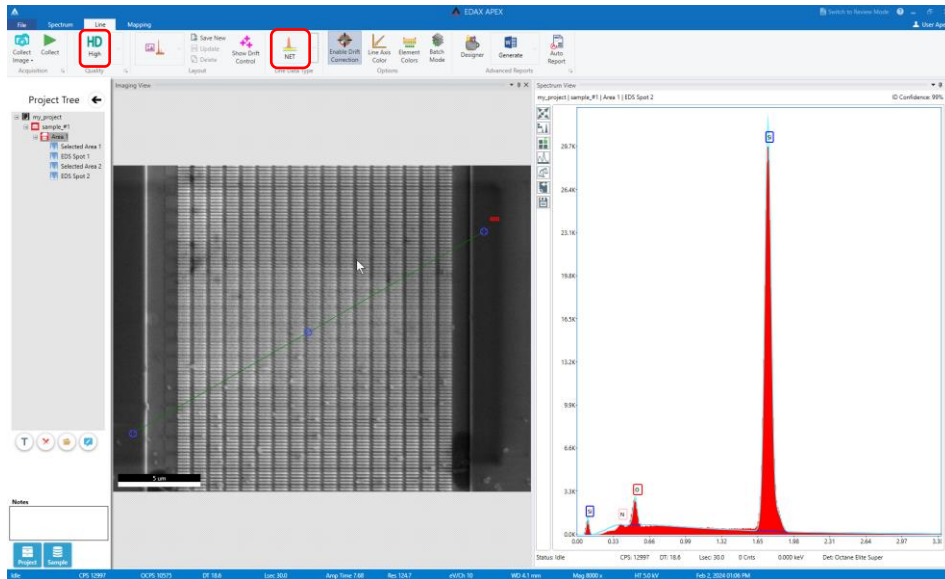
- 8.1. In Microscope Control, center the ROI in the live SEM image and then optimize the image (set magnification, focus, etc.).
- 8.2. In APEX, select the “Line” tab and then “Collect Image” from the toolbar to collect the SEM image in the “Imaging View” panel.
  - 8.2.1. Remember, if drift correction is enabled, the FOV in APEX will be smaller than the FOV in Microscope Control by a factor of 2.
  - 8.2.2. In Microscope Control, the CCD camera will automatically pause; this prevents interference of the CCD camera with the EDS detector.



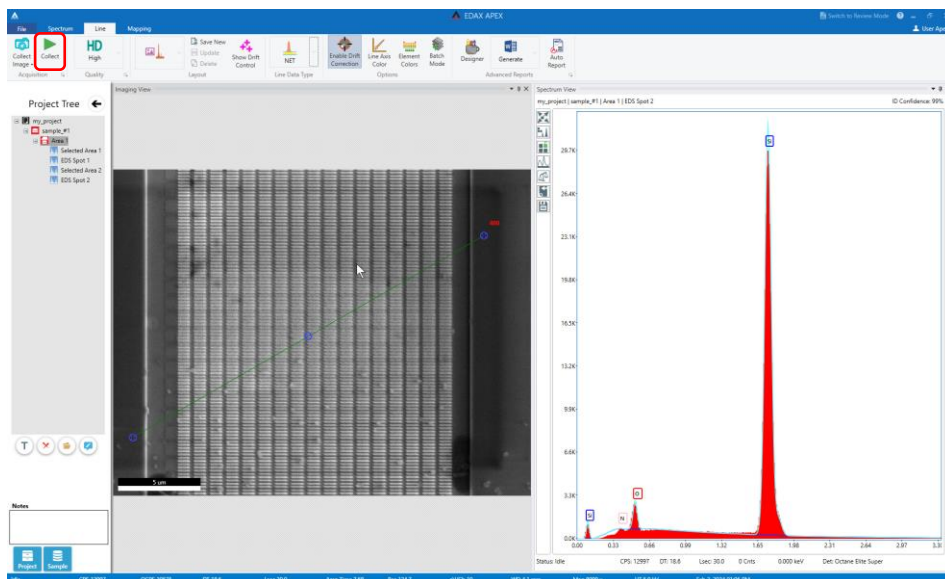
8.3. In the APEX toolbar, set the quality to “HD” and “Line Data Type” to “NET”, then click and drag on the SEM image to draw and position the line.

8.3.1. The “HD” setting uses 400 points independent of the line length; thus, changing line length only changes the spacing between points.

8.3.2. The “NET” data type most accurately displays the distribution of elements because background contributions are removed and overlapping X-ray peaks are deconvoluted (if applicable).



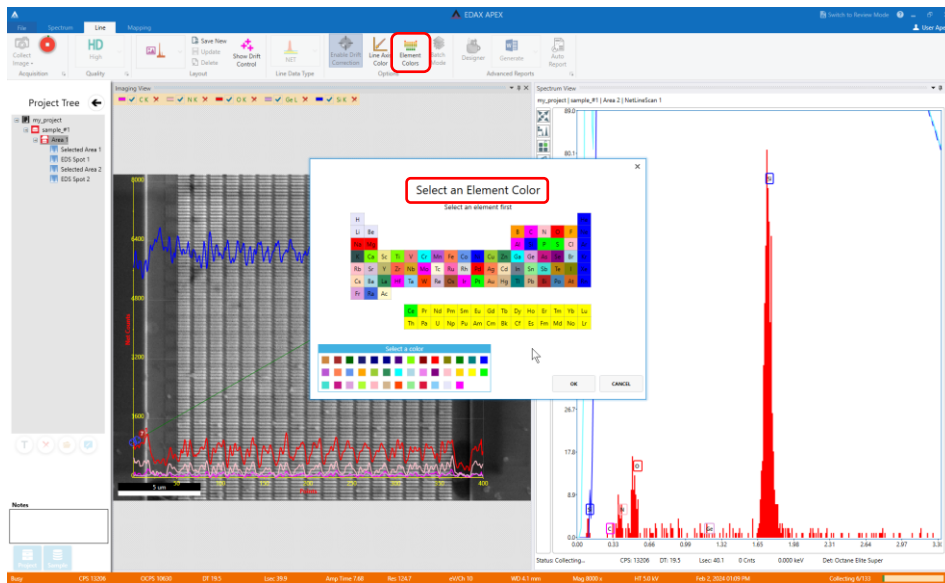
8.4. In the APEX toolbar, select “Collect” to start the linescan; the system will automatically determine the elements present and display all profiles in a single plot superimposed on the SEM image.



8.5. In the APEX toolbar, select “Element Colors” and use the periodic table window to assign the color for each element as desired.

8.5.1. NOTE: the assigned colors for each element will also apply to all data previously collected within the project.

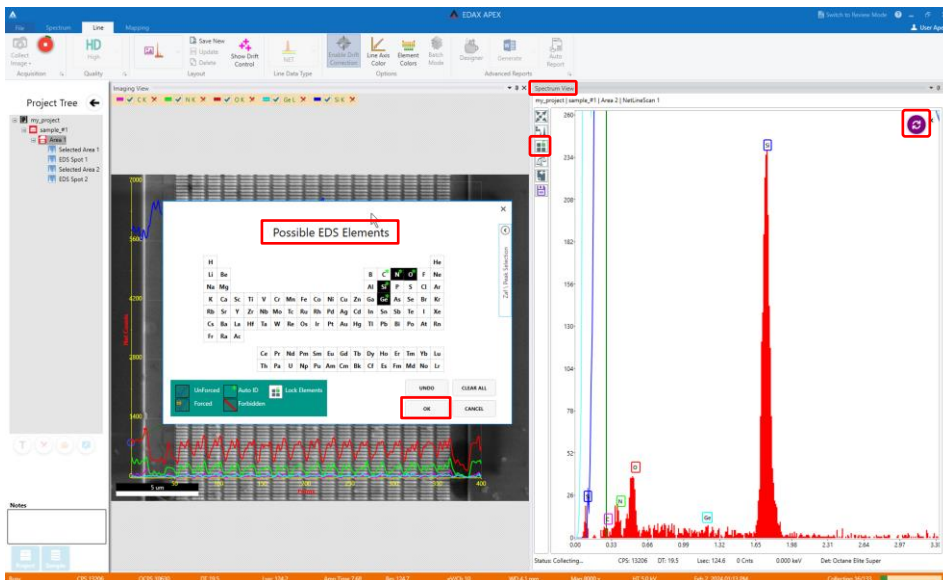
8.5.2. The assigned colors will also apply to all subsequently collected data.



8.6. To add or remove elements to the linescan, select “Modify Element List” from the left side of the “Spectrum View” panel.

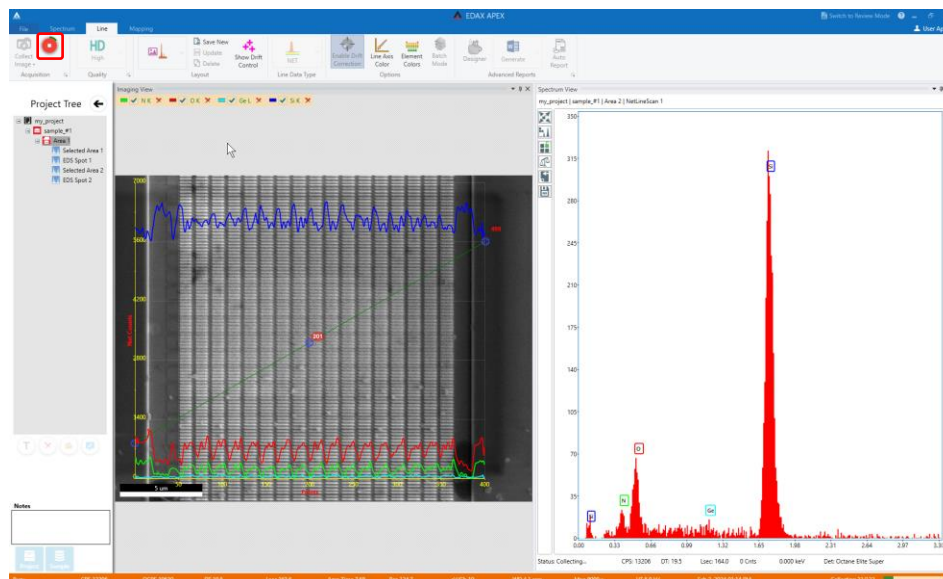
8.6.1. In the periodic table window, add or remove the desired elements and select OK.

8.6.2. Select the “Refresh” icon in the upper-right corner of the “Spectrum View” panel to update the linescans.



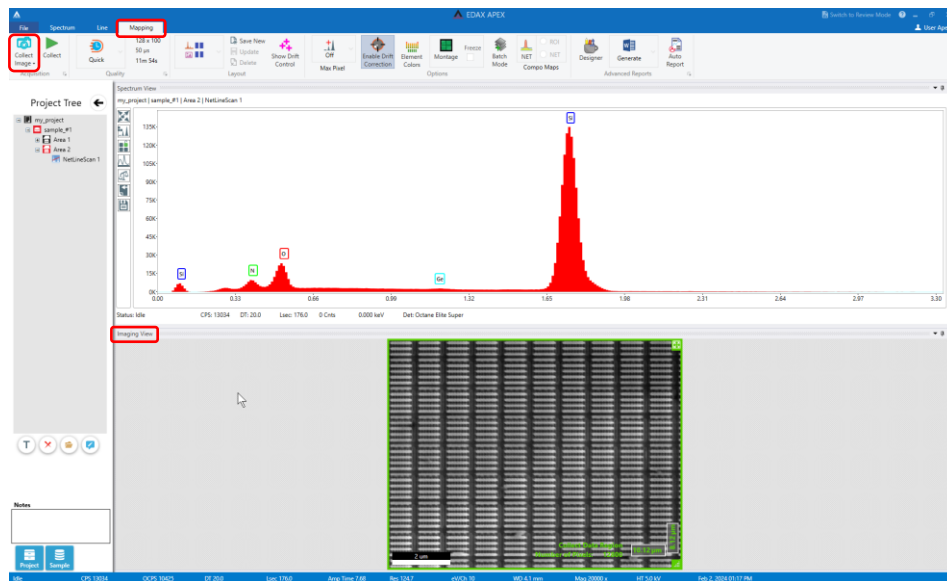
8.7. It usually takes only a few minutes for an “HD” quality linescan to complete, so it is usually not necessary to stop the linescan before completion.

8.7.1. Otherwise, selecting “Stop” from the toolbar will stop the linescan.



## 9. Mapping mode

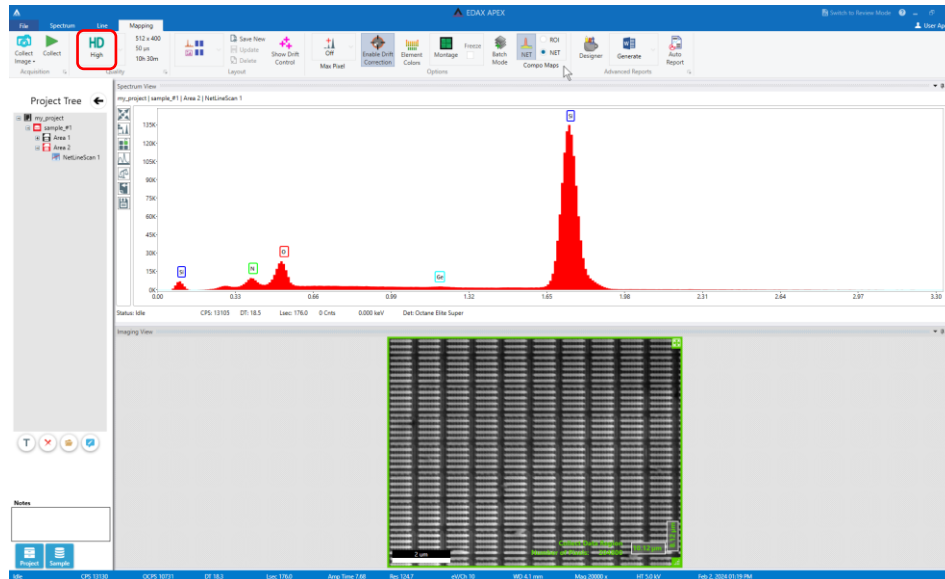
- 9.1. In Microscope Control, center the ROI in the live SEM image and then optimize the image (set magnification, focus, etc.).
- 9.2. In APEX, select the “Mapping” tab and then “Collect Image” from the toolbar to collect the SEM image in the “Imaging View” panel.
  - 9.2.1. Remember, if drift correction is enabled, the FOV in APEX will be smaller than the FOV in Microscope Control by a factor of 2.
  - 9.2.2. In Microscope Control, the CCD camera will automatically pause; this prevents interference of the CCD camera with the EDS detector.
- 9.3. In the SEM image, the area to be mapped is indicated by the green box.
  - 9.3.1. It is recommended to map the full FOV shown in the SEM image (this is the default setting) but if desired, the box may be resized and repositioned.
  - 9.3.2. APEX keeps the number of points in the map approximately constant regardless of the size of the green box; reducing the box size thus decreases map pixel size but does not otherwise result in faster mapping.





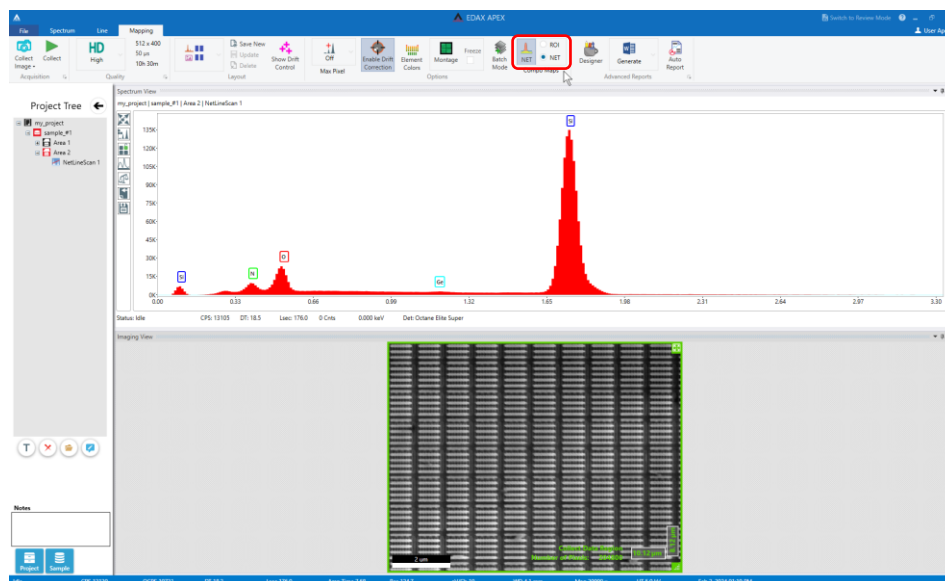
9.4. In the APEX toolbar, set the quality setting to either “Q” (126×110), “SD” (256×221), or “HD” (512×442).

9.4.1. NOTE: if using the “HD” quality setting and mapping the full FOV, the map pixel size will be equal to the SEM image pixel size.

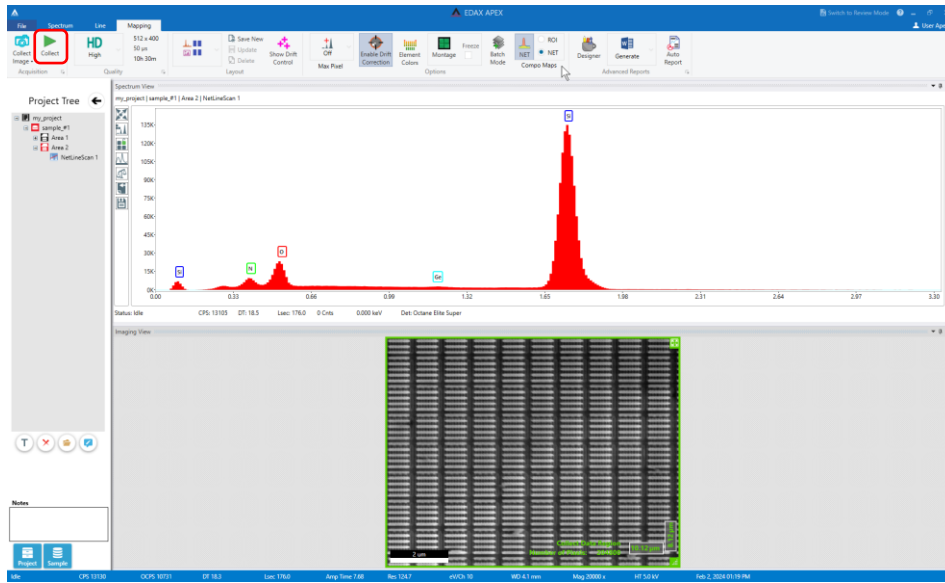


9.5. In the APEX toolbar, select “NET” and then select the “NET” radio button to enable live “NET” mapping.

9.5.1. “NET” maps most accurately display the distribution of elements as background contributions are removed and overlapping X-ray peaks are deconvoluted (if applicable).



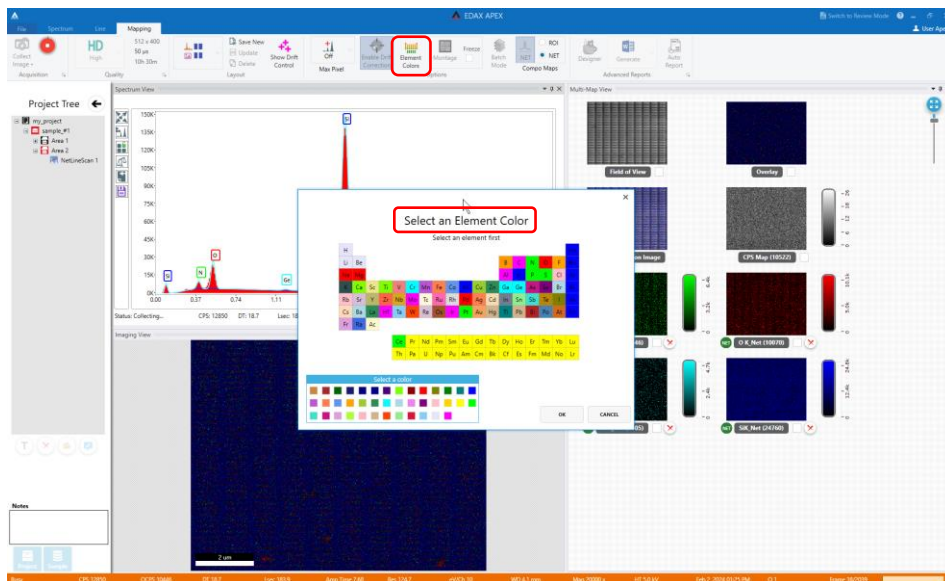
- 9.6. In the APEX toolbar, select “Collect” to start mapping; the system will automatically determine the elements present and display the individual and composite maps in a new “Multi-Map View” panel.



- 9.7. In the APEX toolbar, select “Element Colors” and use the periodic table to assign the color for each element map as desired.

9.7.1. NOTE: the assigned colors for each element will also apply to all data previously collected within the project.

9.7.2. The assigned colors will also apply to all subsequently collected data.

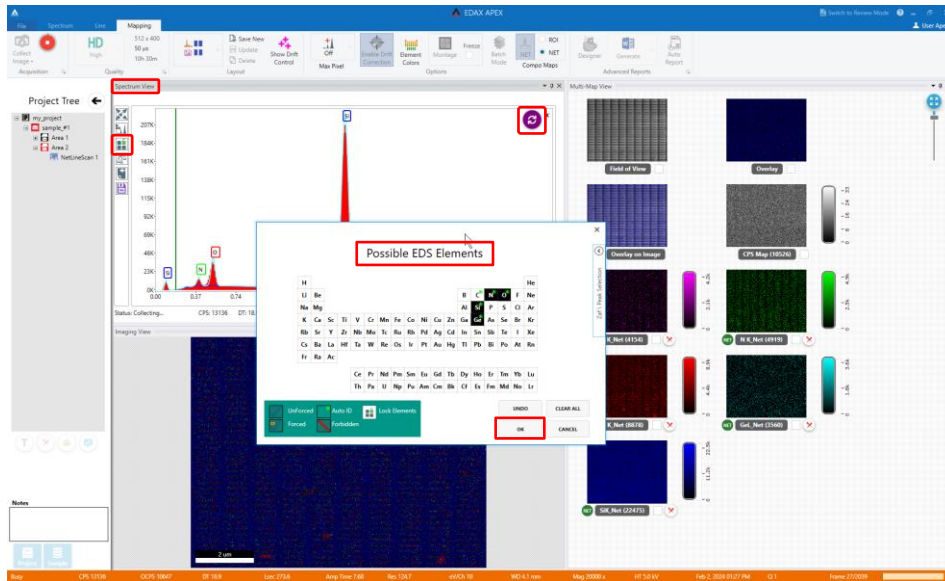




9.8. To add or remove element maps, select “Modify Element List” from the left side of the “Spectrum View” panel.

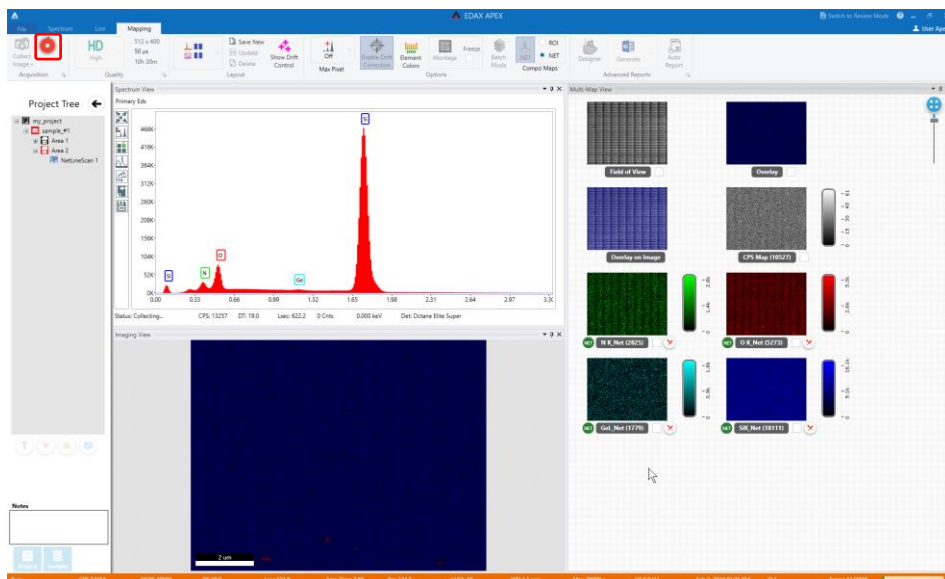
9.8.1. In the periodic table window, add or remove the desired elements and select OK.

9.8.2. Select the “Refresh” icon in the upper-right corner of the “Spectrum View” panel to update the element maps.



9.9. When the maps are satisfactory, select “stop” from the APEX toolbar.

9.9.1. Otherwise, mapping will continue for the automatically calculated length of time (which may be a very long time).



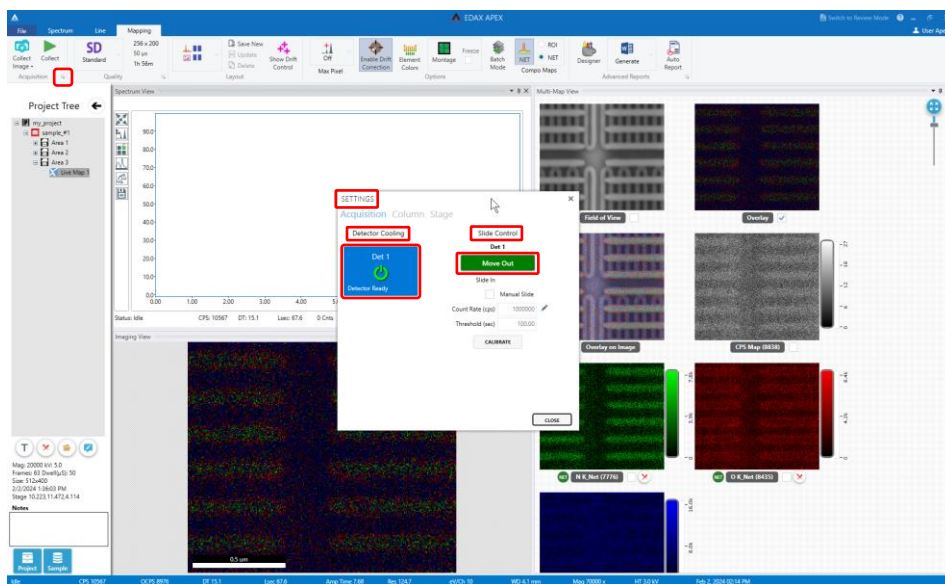
## 10. Finishing

10.1. In the APEX toolbar in the “Acquisition” field, select the arrow to open the “Settings” window.

10.1.1. Under “Detector Cooling”, select “Det 1” to turn off cooling to the EDS detector.

10.1.2. Under “Slide Control”, select “Move Out” to fully retract the EDS detector.

10.1.3. Close the “Settings” window and then close APEX.



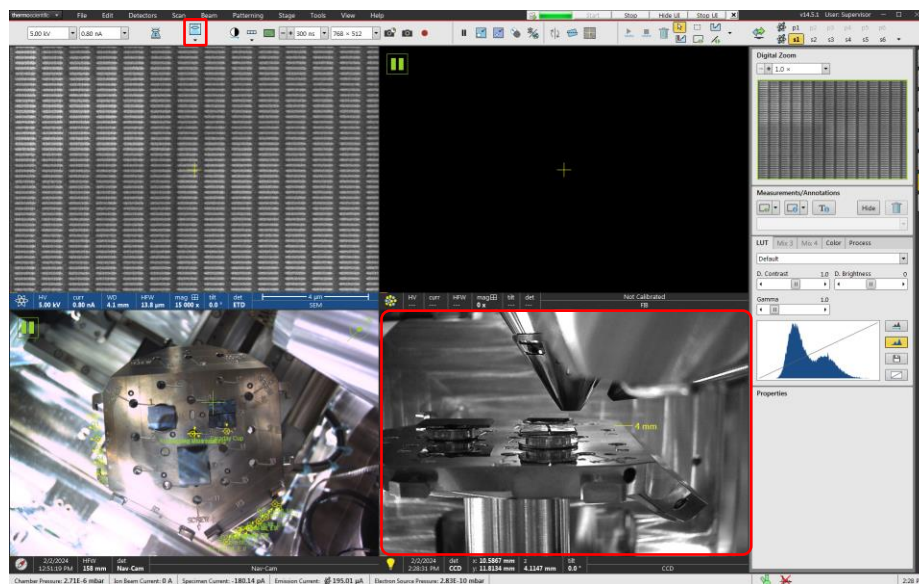
10.2. In Microscope Control, observe the CCD quad and verify that it is acquiring a live image (no pause icon is present in the upper left corner).

10.2.1. If not, select the CCD quad and un-pause it; this is critical to ensure subsequent safe movement of the stage.

10.3. In the CCD quad, verify that the EDS detector is fully retracted.

10.4. If using “Immersion” mode switch the SEM back to “Field-free” mode.

10.4.1. DO NOT leave the SEM in “Immersion” mode when finishing the session.



10.5. Finish the session as per usual: turn off beam(s), home stage, vent chamber, remove specimens, return chamber to vacuum (not shown).