

FEI Themis Z S/TEM: monochromated STEM  
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**\*ANALYSIS OF RADIOACTIVE SPECIMENS IS STRICTLY PROHIBITED\***

This procedure assumes the user is proficient operating the Themis Z S/TEM in both TEM and STEM modes and proficient performing STEM-EELS.

1. General remarks

1.1. Monochromation reduces the energy spread of the electrons leaving the gun. At 60 kV, this will improve both STEM probe size (via reduction of chromatic aberration) as well as EELS resolution. However, at 200 kV (or higher), this will basically only improve EELS resolution.

1.1.1. The example shown here was performed at 200 kV.

2. Before exciting the monochromator

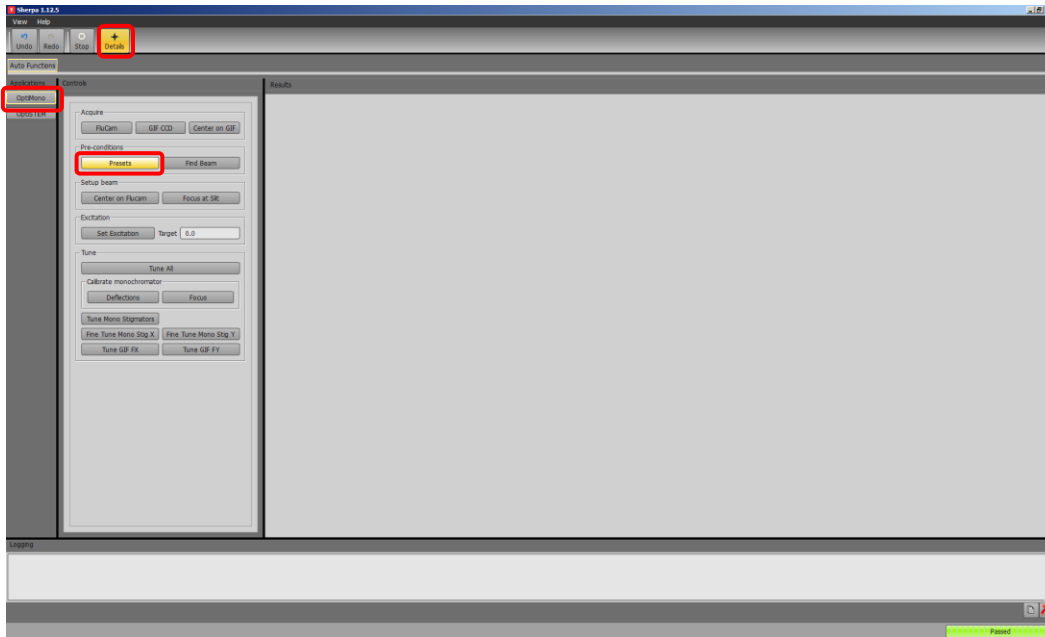
2.1. It is best to be located over vacuum when exciting and tuning the monochromator. If using a support film grid with particulates, it is recommended to store the stage position where analysis is intended and then to move to a grid opening where no support film is present; once monochromator tuning is complete, the stage may then be relocated back to the previously stored position.

2.2. Find a region of interest, perform monochromator centering, and bring the region of interest to eucentric height (not shown, described elsewhere).

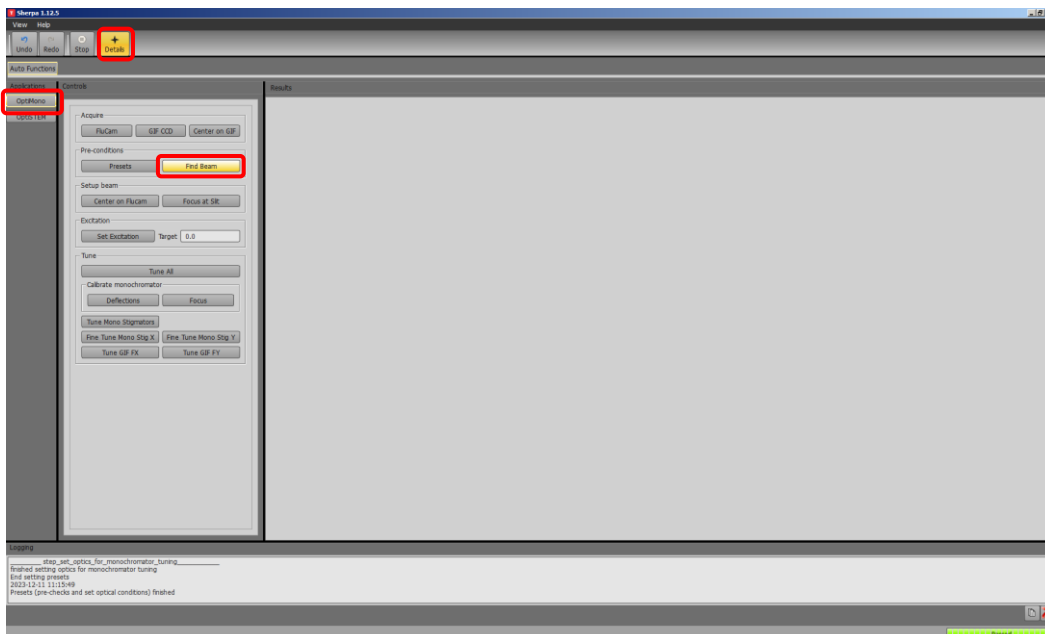
2.3. If intending on performing EELS, GIF tuning should be performed in EFTEM mode before energizing and exciting the monochromator (not shown, described elsewhere).

### 3. Monochromator excitation

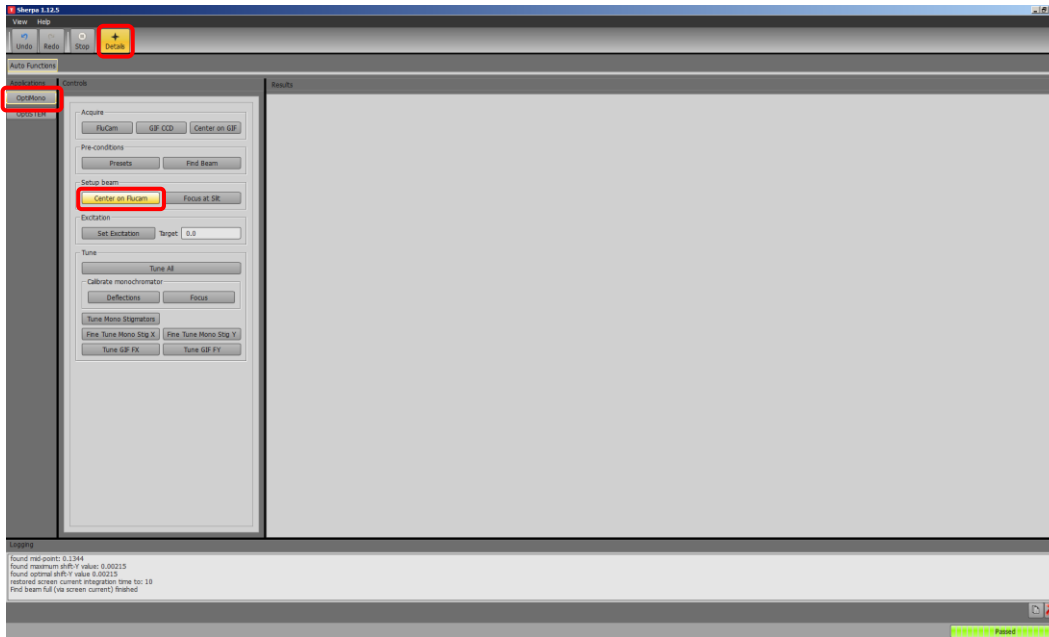
- 3.1. Maximize Sherpa and select the “OptiMono” application; activate “Details” (if not already activated) so the different steps may be observed.
- 3.2. Select “Presets” to set the instrument to appropriate settings (SA 2550x, spot size = 1, C3 lens off, largest C1, C2, and C3 apertures) for exciting (and tuning) the monochromator.



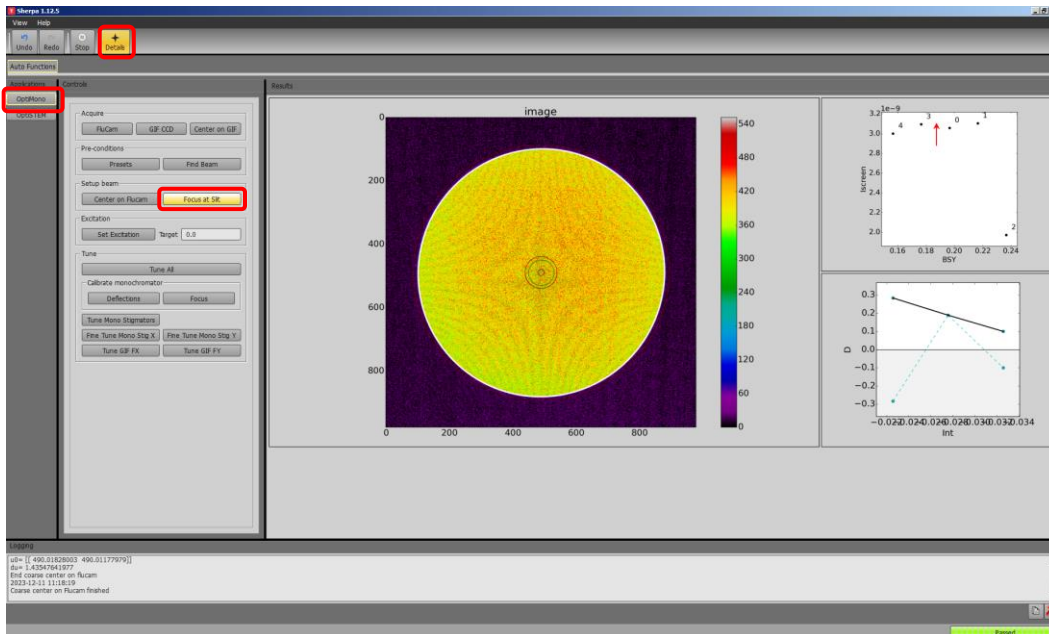
- 3.3. Select “Find Beam” to automatically locate the beam.



- 3.4. Select “Center on Flucam” to automatically center the beam (via maximizing screen current).



- 3.5. Select “Focus at slit” to automatically focus the C2 lens on the energy filtering slit (C1 aperture).

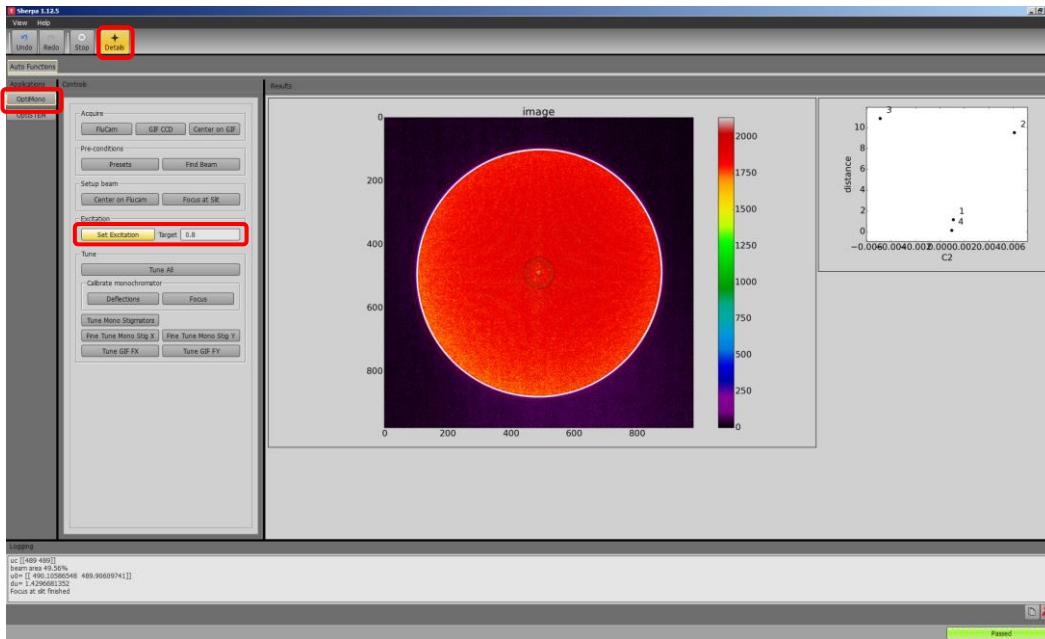


3.6. Input the desired value for “Target” and select “Set Excitation”; the system will automatically recenter the beam as the excitation is ramped up to the target value.

3.6.1. NOTE: larger excitations will improve energy resolution, but at the expense of reduced beam current; excitation = 1.2 is recommended for obtaining the highest possible energy resolution.

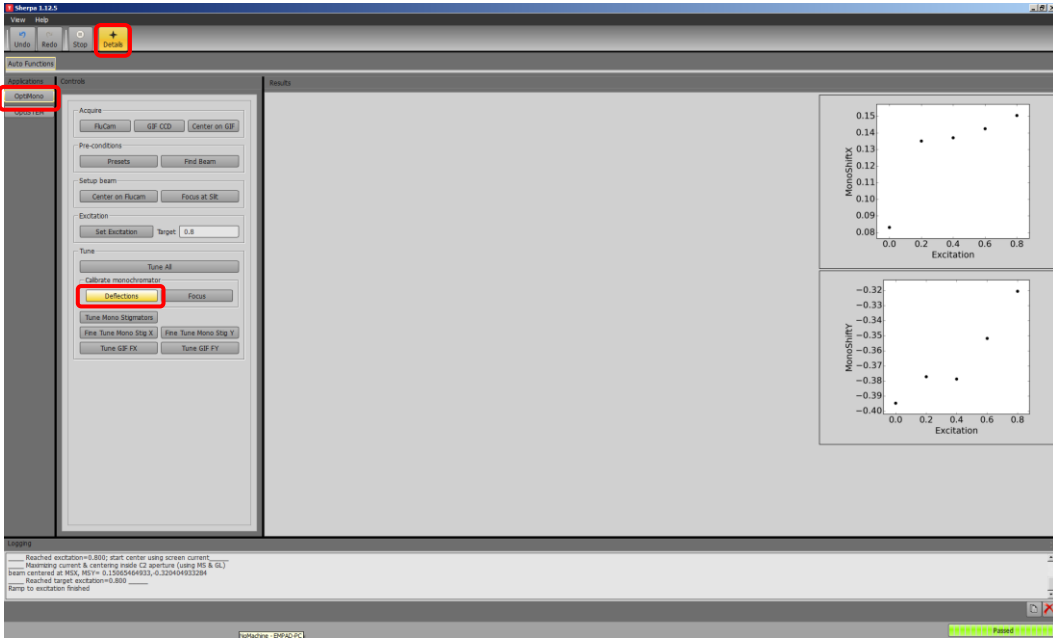
3.6.2. Once excitation is complete, it is recommended to allow at least 1 h for the monochromator to stabilize.

3.6.3. Afterwards, the beam will likely be off the Flucam; either manually adjust the monochromator shifts (not shown) or remain in Sherpa and select “Center on Flucam” to recenter the beam (previously shown).

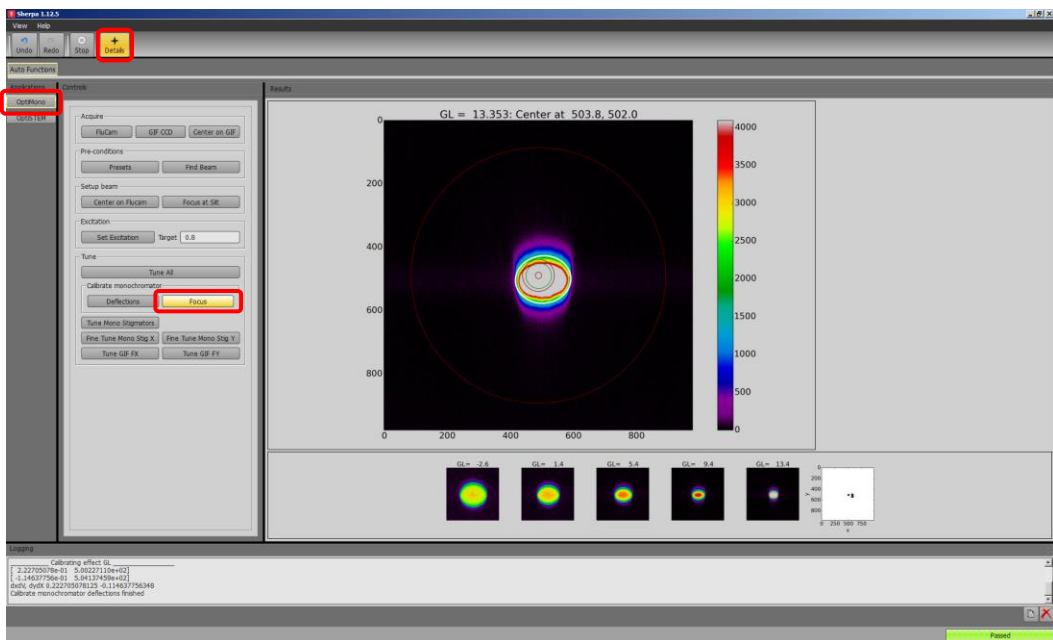


#### 4. Monochromator tuning

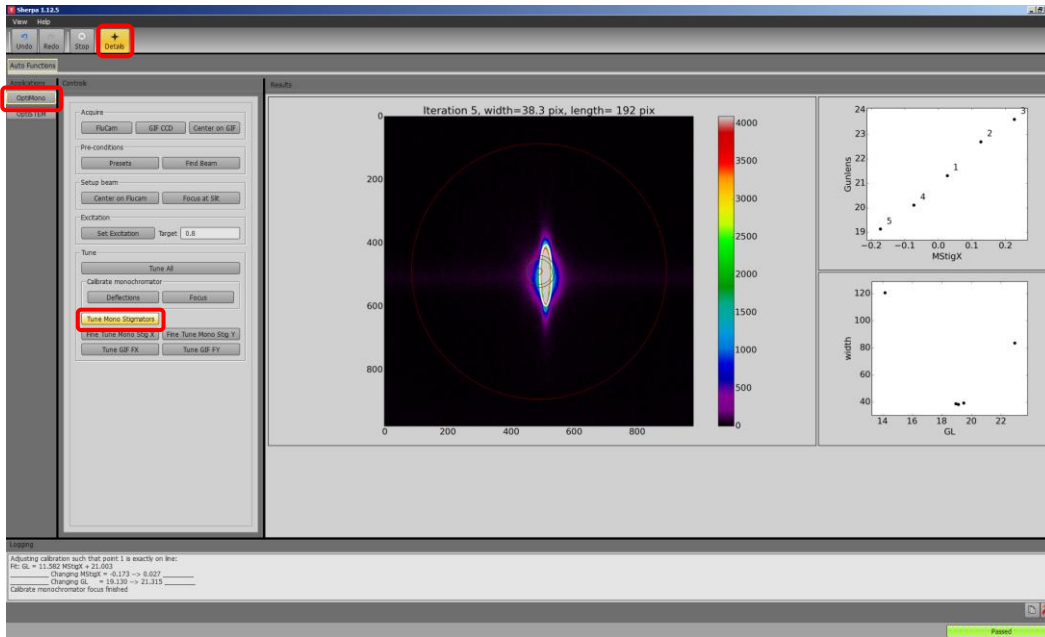
- 4.1. Select “Deflections” to calibrate the different monochromator deflectors; this helps to ensure the beam stays centered as the different deflectors are adjusted.



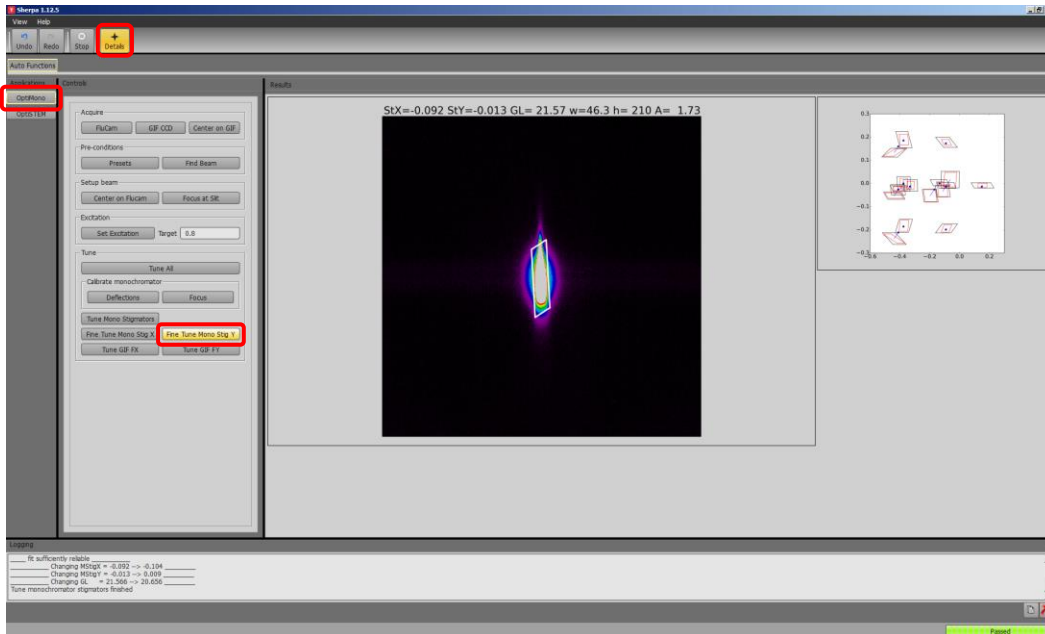
- 4.2. Select “Focus” to calibrate the optimal focus for different values of mono stigmator X; this helps to keep the beam focused while optimizing mono stigmator X.



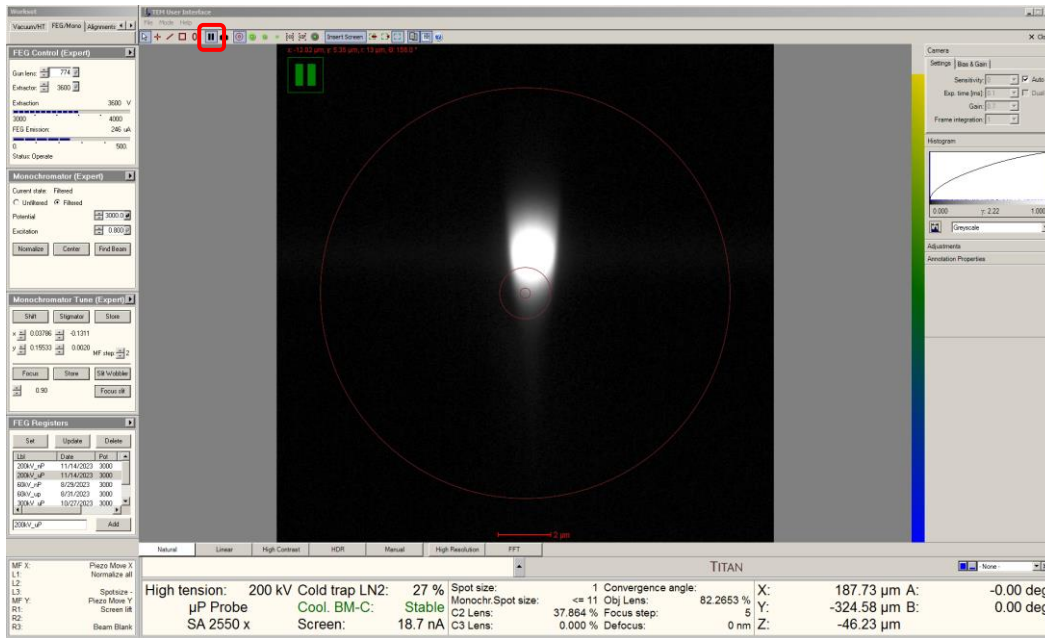
4.3. Select “Tune Mono Stigmators” to coarsely correct both X and Y monochromator astigmatism.



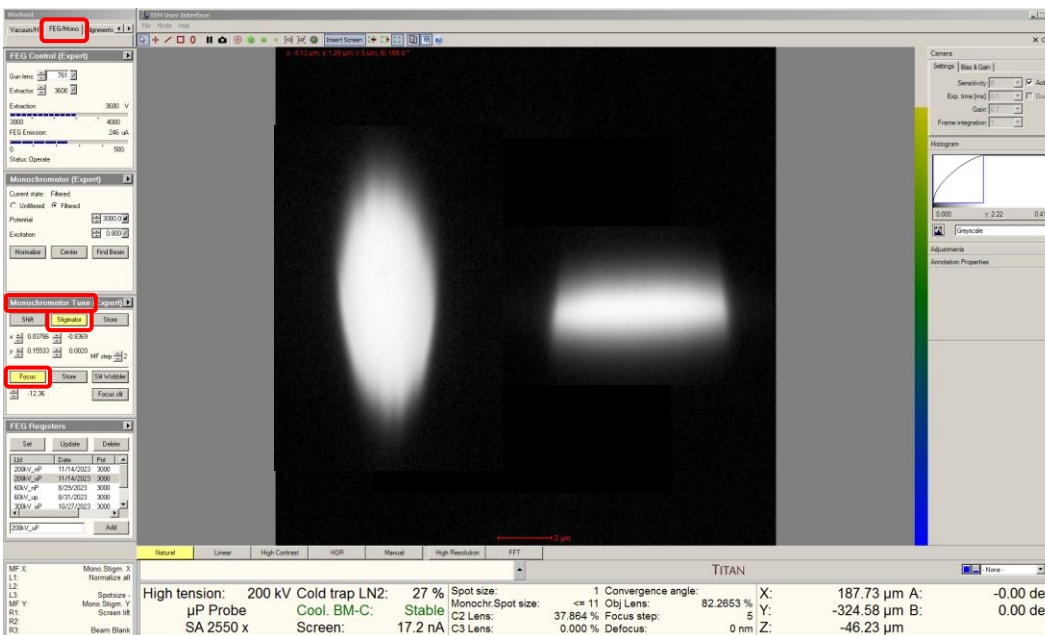
4.4. Select “Fine Tune Mono Stig Y” to finely correct Y monochromator astigmatism.



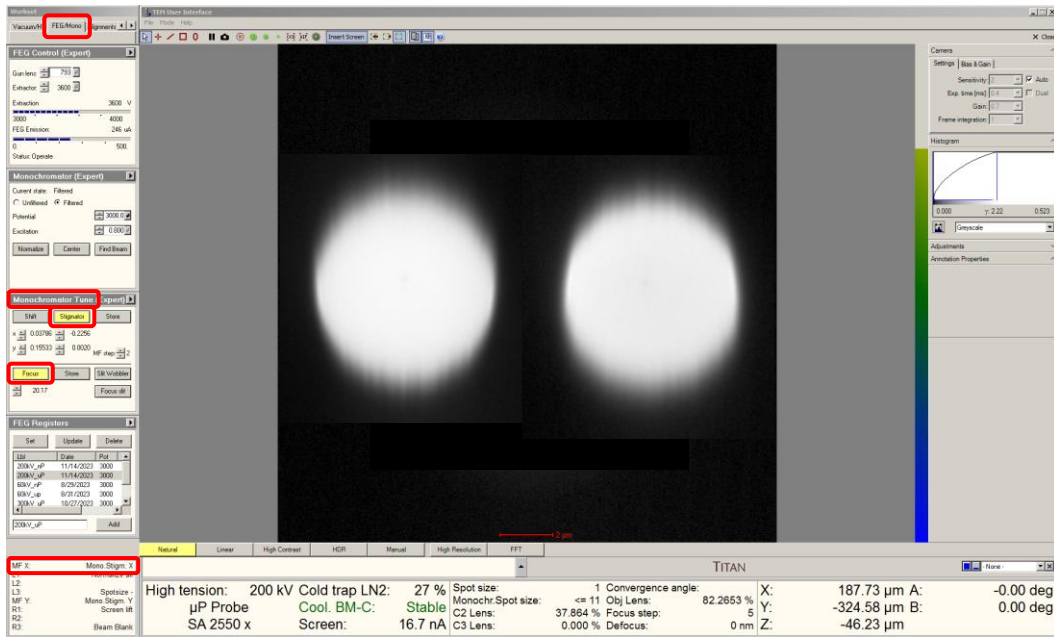
- 4.5. Fine correction of the X monochromator astigmatism must be performed manually; return to Microscope Control and un-pause the Flucam to resume live imaging.



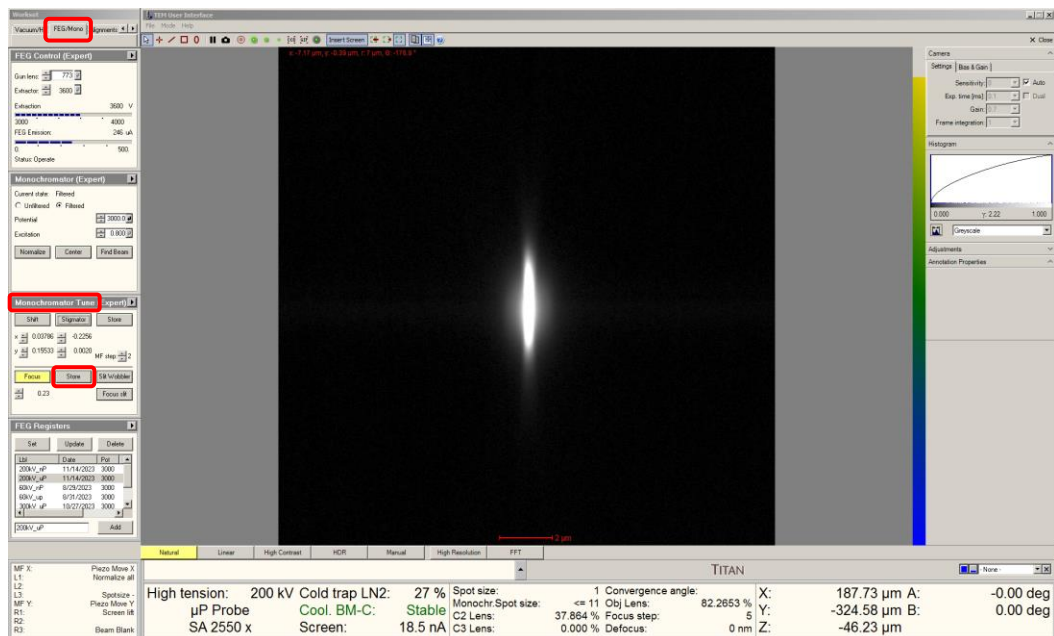
- 4.6. Select to the “FEG/Mono” tab and navigate to the “Monochromator Tune” panel; activate “Stigmator” and “Focus”. Monochromator astigmatism will present as asymmetry in the dispersed beam between under (left) and overfocus (right) conditions.



- 4.7. Adjust the X “Multifunction” knob (mono stigmator X) so the dispersed beam is symmetric between under (left) and overfocus (right) conditions.



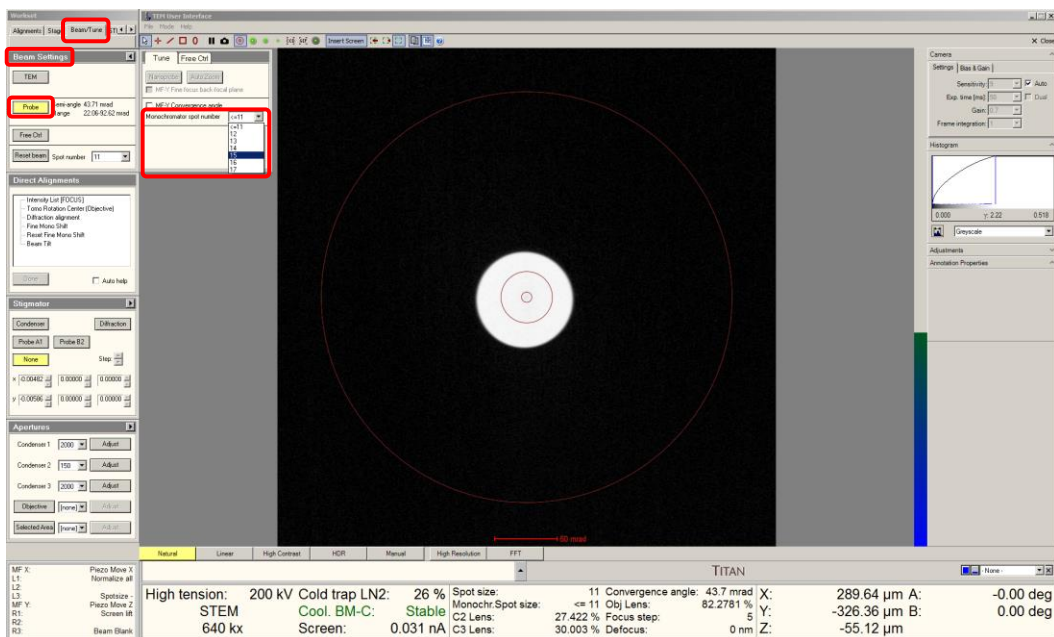
- 4.8. Use the “Intensity” knob to focus the dispersed beam as tightly as possible along the non-dispersive (horizontal) direction; In the “Monochromator Tune” panel, select “Store” to set this as the optimal monochromator focus.





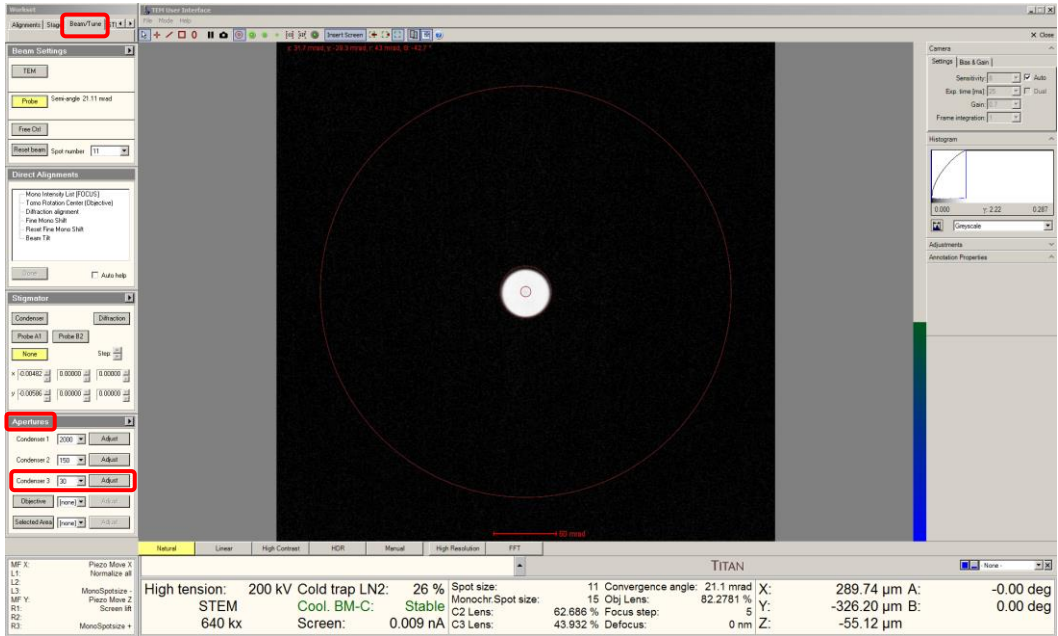
## 5. Monochromated STEM alignment

- 5.1. Before entering STEM mode, overfocus the monochromator to spread the dispersed beam out and facilitate easier STEM alignment (described elsewhere); it will be refocused once STEM alignment is complete.
- 5.2. Select the “STEM” tab and navigate to the “STEM Imaging” panel; select “STEM” to enter STEM mode, set the indicated magnification  $\geq 640$  kx, and press “Eucentric focus” (not shown)
- 5.3. Navigate to the “STEM AutoTuning” panel; select the flap-out arrow to expand the panel and select “Reset” (not shown).
- 5.4. Select the “Beam/Tune” tab and navigate to the “Beam Settings” panel; select “Probe” to switch the system into 3 condenser mode and the panel will automatically expand. In the expanded panel, select the desired “Monochromator spot number”.
  - 5.4.1. Monochromator spot number = 12 or 13 is recommended for performing core-loss edge EELS analysis.
  - 5.4.2. Monochromator spot number = 15 or 16 is recommended for atomic-resolution STEM imaging.
  - 5.4.3. NOTE: when using monochromation, it is no longer possible to arbitrarily specify a semi-angle of convergence; this will now be defined by the C3 aperture.

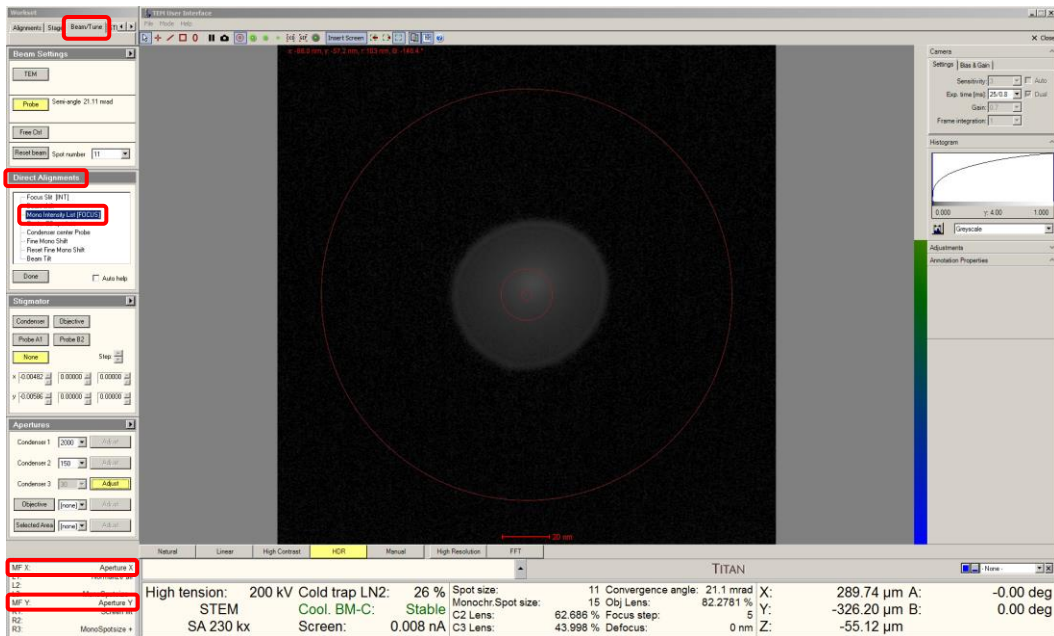


5.5. Remain in the “Beam/Tune” tab and navigate to the “Apertures” panel; select and insert the 30  $\mu\text{m}$  C3 aperture and select “Adjust”.

5.5.1. Selecting this C3 aperture will result in a probe semi-angle of convergence = 21 mrad.



- 5.6. On the right-hand panel, select “Diffraction” to observe an image of the probe and set the indicated magnification = SA 230 kx or higher (not shown).
- 5.7. In the “Direct Alignments” panel, select “Mono Intensity List [FOCUS]” to couple the C3 lens to the “Focus” knob.
- 5.8. Use the “Multifunction” knobs to center the C3 aperture by using the “Focus” knob to expand/contract the beam (using the C3 lens) and “UserBeamShift” in SCORR to shift/recenter the beam (described elsewhere).



5.9. Use the “Focus” knob (still coupled to C3) to focus the probe until a sharp, caustic spot is produced inside a diffuse halo; return to the “Direct Alignments” panel and select “Done” to save this setting for C3.

5.9.1. The “Focus” knob will then return to being coupled to the objective lens.



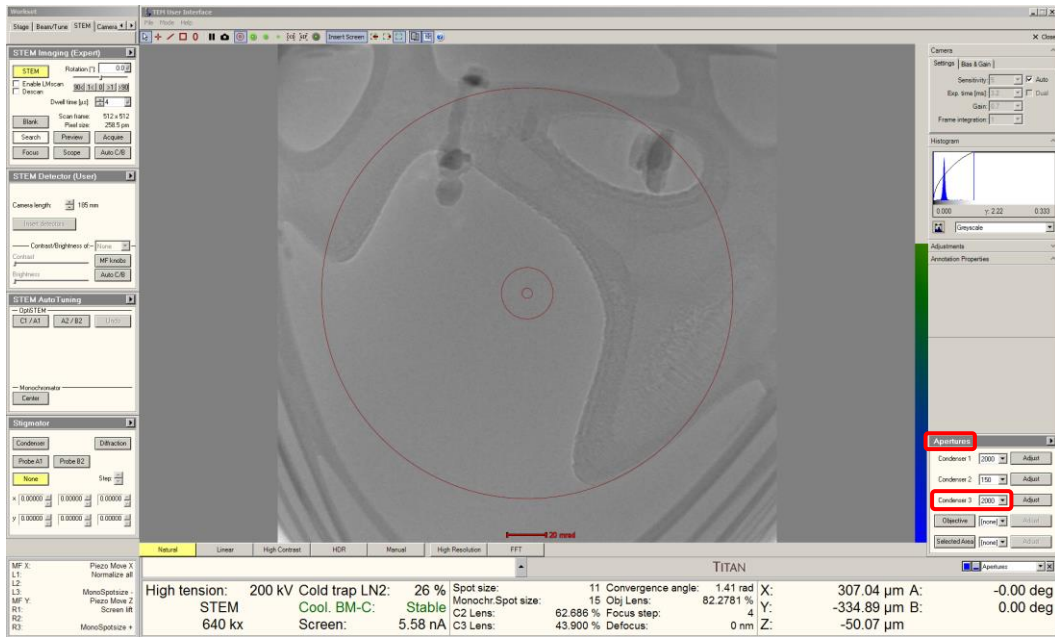
5.10. Center the caustic spot inside the diffuse halo using “BeamTilt” in SCORR (not shown).

5.11. On the right-hand panel, select “Diffraction” to view the Ronchigram again; set the indicated camera length = 185 mm (not shown).

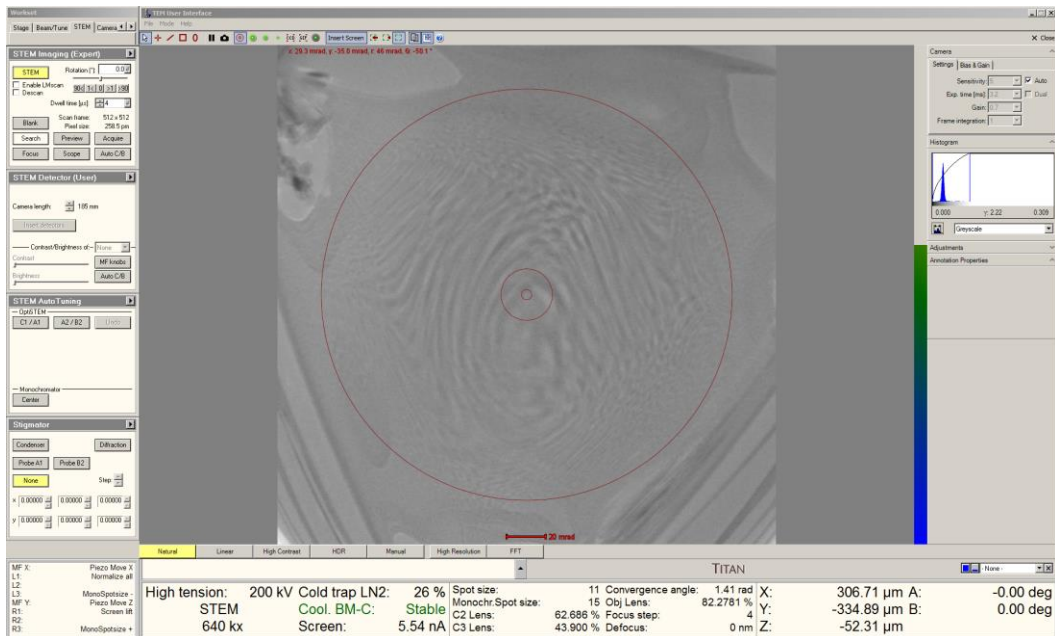
5.12. Precisely center the direct disk on the Flucam using “Diffraction alignment” (not shown).

5.12.1. NOTE: this is important as this will define the correct position for subsequent recentering of the C3 aperture.

- 5.13. Navigate to the “Apertures” panel and insert the 2000  $\mu\text{m}$  C3 aperture (it does not need to be centered).

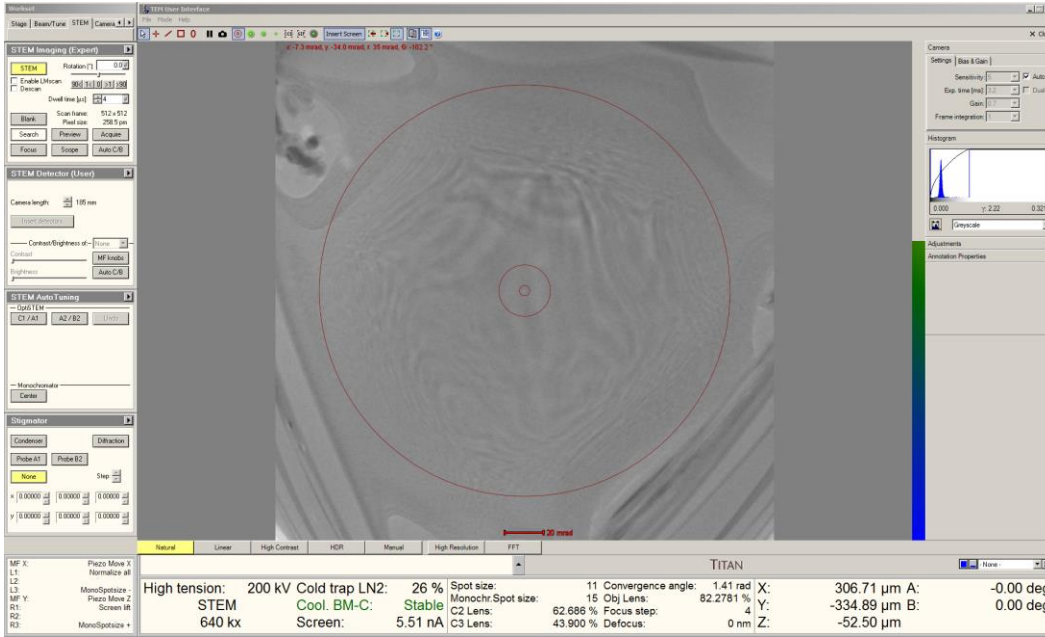


- 5.14. Move stage X and Y to center a suitable region for correcting A1 and B2 (amorphous C, electron-beam assisted Pt, etc.) on the Flucam; adjust stage Z to obtain a “blow-up” condition.



5.15. Coarsely correct A1 and B2 using “AT\_A1” and “AT\_B2” in SCORR (described elsewhere); the corrected Ronchigram should appear as show below.

5.15.1. Finer correction of A1, A2, and B2 may be performed later during STEM imaging.



5.16. Reinsert and precisely recenter the C3 aperture on the Flucam (not shown).

5.17. Set the indicated camera length = 115 mm (not shown).

5.18. Precisely recenter the direct disk on the Flucam using “Diffraction alignment” (not shown).

5.18.1. NOTE: once again, this is important as this will define the correct position for subsequent recentering of the C3 aperture.

## 6. Refocusing the dispersed beam

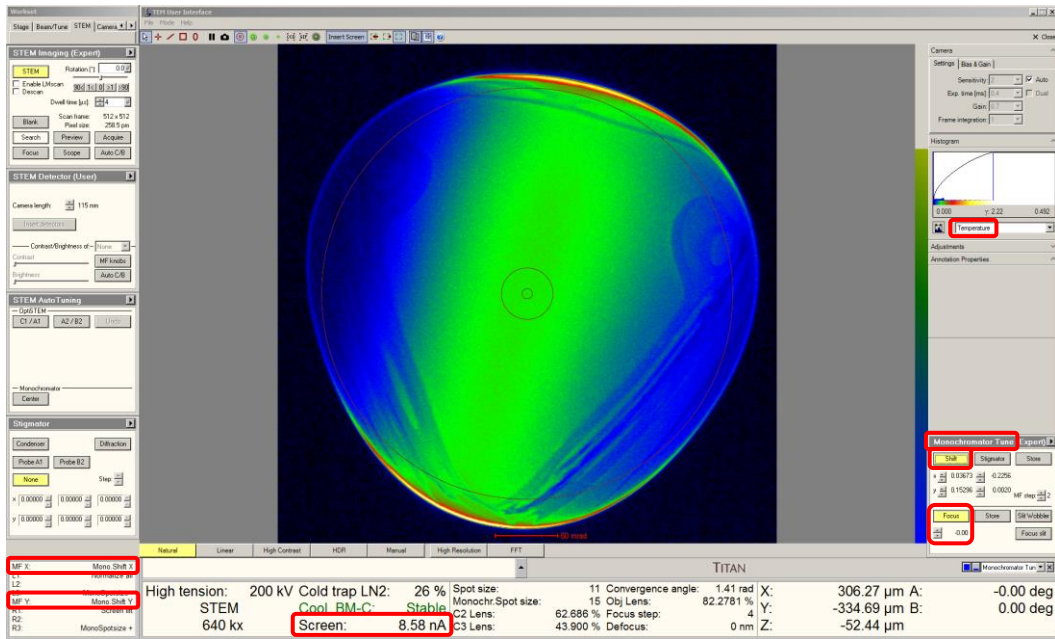
6.1. Navigate to the “Monochromator Tune” panel and select “Focus”; use the “Intensity” knob to set the monochromator focus = 0.0.

6.1.1. NOTE: this will produce an image of the dispersed beam that appears out of focus on the Flucam; this is normal.

6.2. Remain in the “Monochromator Tune” panel and select “Shift”; use the “Multifunction” knobs to center the dispersed beam on the Flucam.

6.2.1. Switching the Flucam display from “Grayscale” to “Temperature” may help with this.

6.2.2. The measured screen current will be at or near maximum when the dispersed beam is properly centered.



## 7. C1 (energy-filtering) aperture centering

7.1. Navigate to the “Apertures” panel, insert the desired C1 aperture, and select “Adjust”.

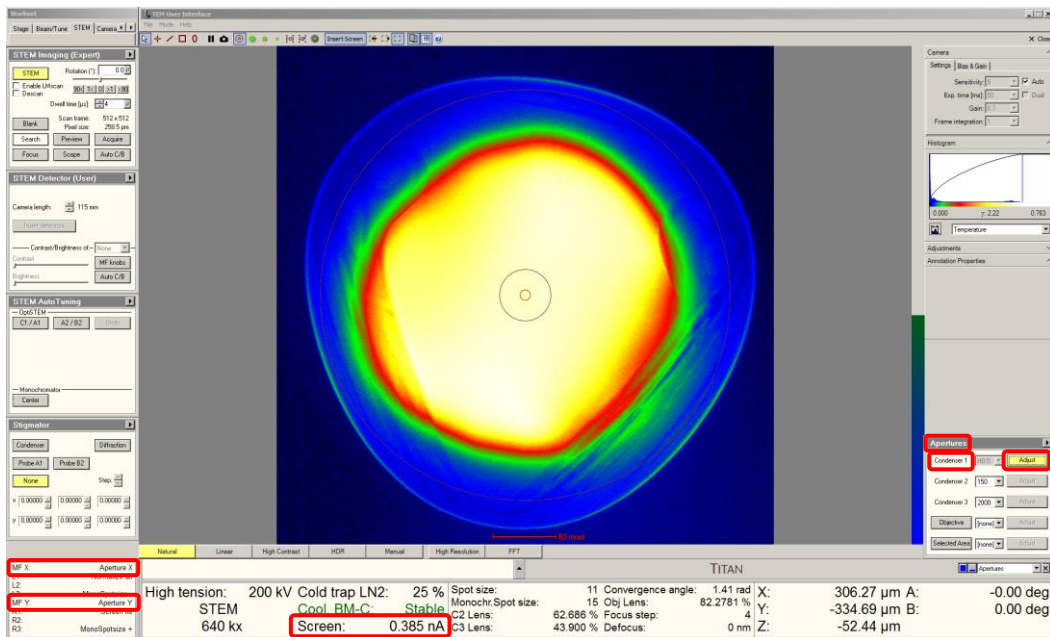
7.1.1. NOTE: energy resolution will improve as the aperture size decreases, but at the expense of reduced beam current.

7.1.2. NOTE: apertures are recommended over slits and wedges to produce the roundest probes.

7.1.3. The edge of the C1 aperture will appear out of focus; once again, this is normal.

7.2. Use the “Multifunction” knobs to center the C1 aperture on the Flucam.

7.2.1. Again, the measured screen current will be at or near maximum when the C1 aperture is properly centered.



7.3. After centering the dispersed beam and C1 aperture, re-insert and re-center the 30 μm C3 aperture on the Flucam (not shown).



## 8. STEM imaging

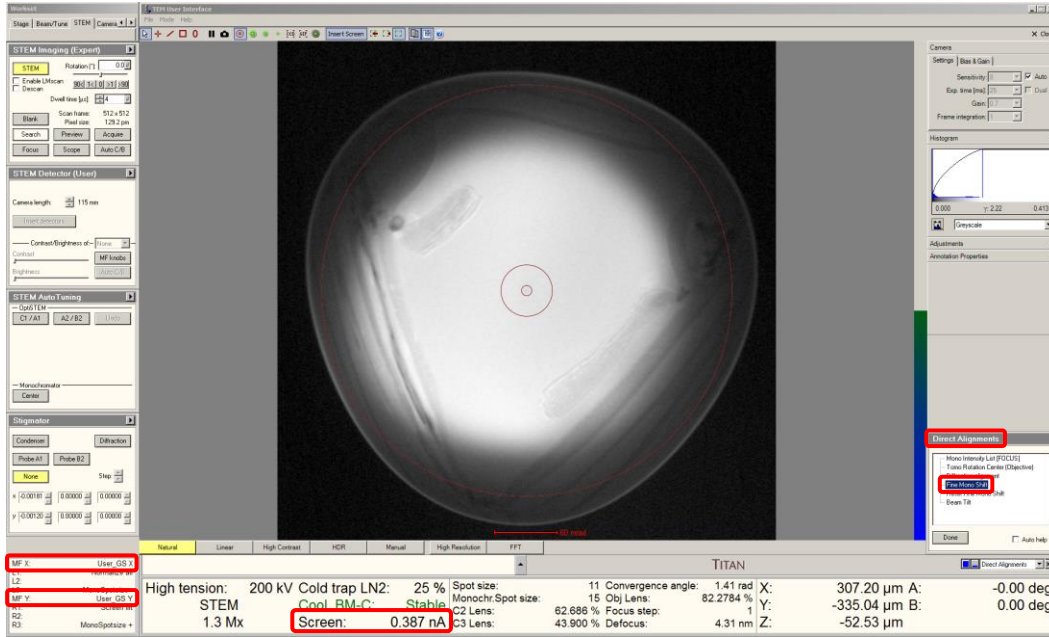
- 8.1. STEM imaging may now be performed as per usual; as is the case with unfiltered STEM imaging, roughly focus the image by adjusting stage Z and finely focus the image by using the “Focus” knob (described elsewhere).
- 8.2. Navigate to the “Stigmator” panel, select “Condenser”, and use the “Multifunction” knobs to finely correct A1 as needed (described elsewhere).
- 8.3. STEM autotuning to automatically correct C1, A1, A2, and B2 may be performed as needed (described elsewhere).

## 9. Additional monochromator adjustments

- 9.1. Once centered, the C1 aperture should remain stable and not need any additional adjusting during the session. However, even with sufficient time to stabilize, the dispersed beam will still drift and require periodically re-centering.
- 9.2. If using a low monochromator spot size, re-centering can be accomplished without changing the C3 aperture. However, if using a high monochromator spot size, it will be best to temporarily re-insert the 2000  $\mu\text{m}$  C3 aperture (shown here).
- 9.3. Before inserting the largest C3 aperture, precisely center the direct disk on the Flucam using “Diffraction alignment”; then insert the 2000  $\mu\text{m}$  C3 aperture (not shown).
- 9.4. Navigate to the “Monochromator Tune” panel, select “Shift”, and use the “Multifunction” knobs to maximize the screen current (described previously).

9.5. If needed, finer shifting of the dispersed beam may be accomplished by navigating to the “Direct Adjustments” panel, selecting “Fine Mono Shift” and using the “Multifunction” knobs.

9.5.1. NOTE: if “Fine Mono Shift” is used, it will need to be reset when finished using the monochromator.

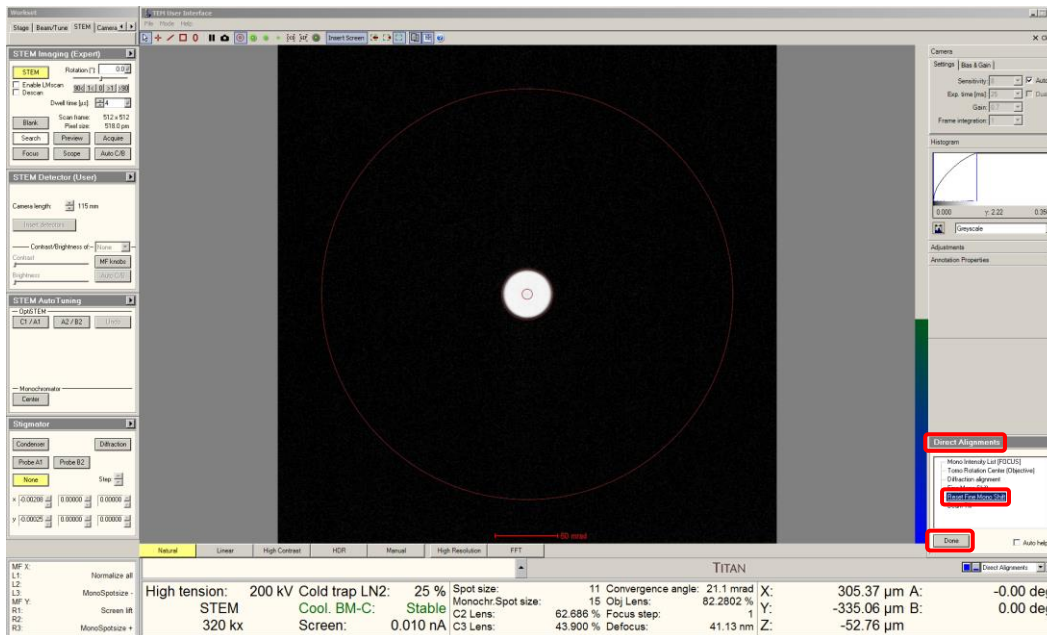


## 10. Considerations for STEM-EELS

- 10.1. When switching to EFSTEM mode for the purpose of performing monochromated STEM-EELS, there will likely be a shift in the dispersed beam resulting from the normalization process.
- 10.2. To address this, navigate to the “Monochromator Tune” panel, select “Shift”, and use the “Multifunction” knobs to maximize the screen current (described previously).
- 10.3. If finer control of the monochromator shift is needed, navigate to the “Direct Alignments” panel, select “Fine Mono Shift”, and use the “Multifunction” knobs to maximize the screen current (described previously).

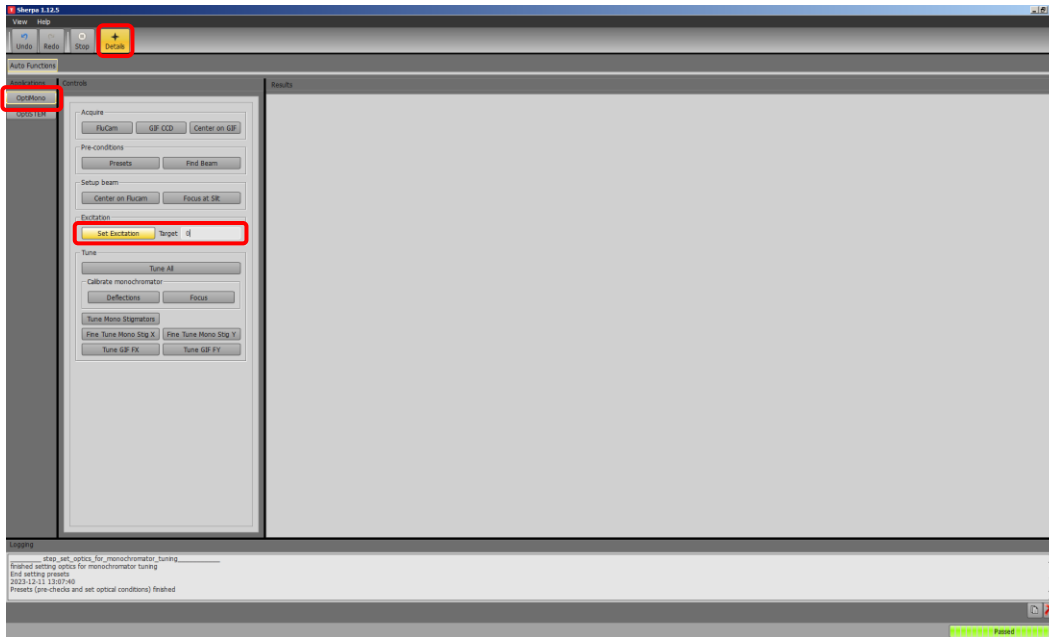
## 11. Finishing

- 11.1. If the projection system is still in EFSTEM mode, switch it back to normal mode (not shown).
- 11.2. If “Fine Mono Shift” was used, navigate to the “Direct Adjustments” panel, select “Reset Fine Mono Shift”, and then “Done”.

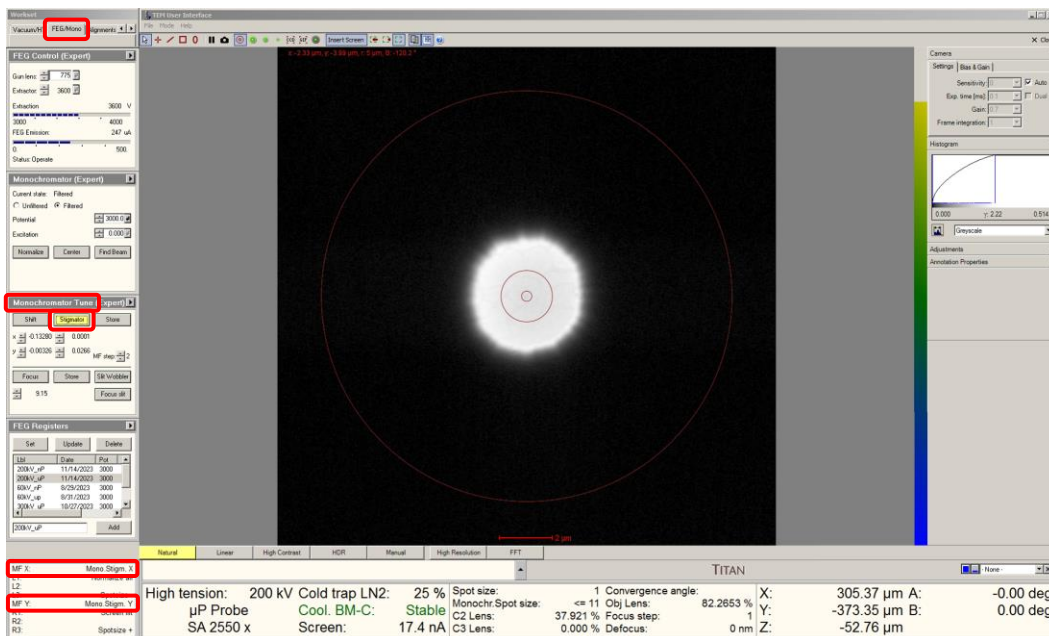


- 11.3. Switch from STEM mode back to TEM mode (not shown).
- 11.4. Navigate to the “Apertures” panel; re-insert the 2000  $\mu$ m C1 aperture and the 2000  $\mu$ m C3 aperture (not shown).

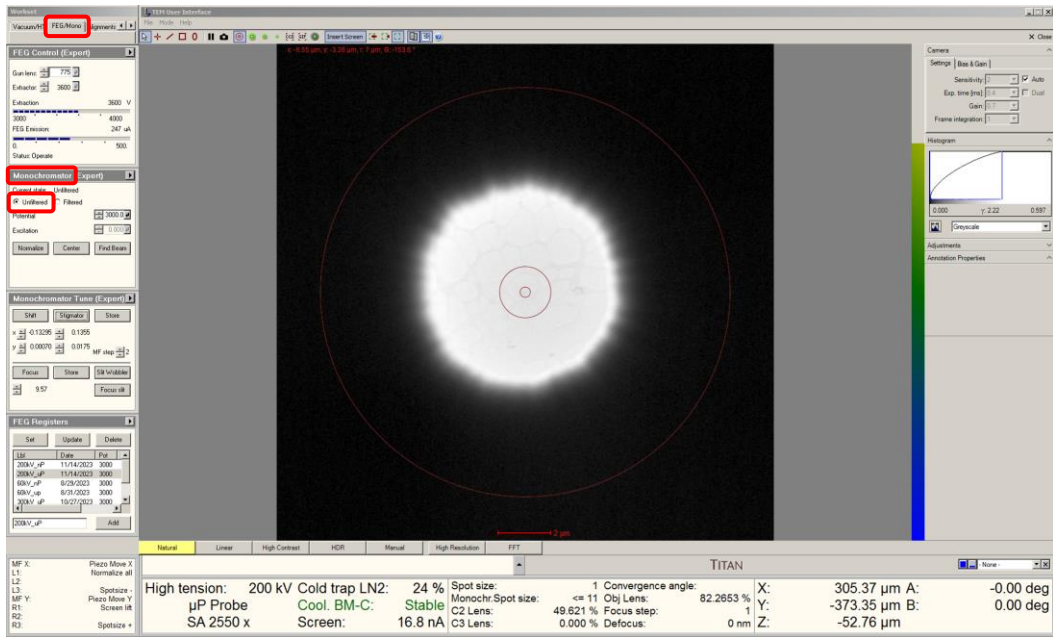
- 11.5. Return to Sherpa; set “Target” = 0 and select “Set Excitation”; the system will automatically recenter the beam as the excitation is ramped down.



- 11.6. Return to Microscope Control; navigate to the “Monochromator Tune” panel, select “Stigmator”, and use the “Multifunction” knobs to make the non-dispersed beam round.



- 11.7. Remaining in the “FEG/Mono” tab, navigate to the “Monochromator” panel and set the state to “Unfiltered”.



- 11.8. If necessary, turn the C3 lens back on (not shown).
- 11.9. Again, due to the change in excitation, it may take 1 – 2 h for the monochromator drift to fully stabilize.