Tuning the CEOS STEM Corrector

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Tuning Prerequisites.

- Use a standard specimen – Au nanoparticles on carbon.
- Microscope must be absolutely stable: ie no thermal drift, lens hysteresis, stage drift, HT instability etc. Typically, this means changing the microscope voltage on a Friday and tuning on the following Monday.
- Specimen must free from contamination. Ideally, the specimen should be left in the microscope overnight. It must be beam showered.
- Instabilities will make tuning difficult and invalidate the long-term stability of the tune.

Aberrations

Symbol	Order	Name	Target Value	How to Adjust
A1	1^{st}	2-fold Astigmatism	0-5nm	Manual: Coarse stigmators
C1	1^{st}	Focus	5-10nm	Manual: Focus
A2	2^{nd}	3-fold Astigmatism	<50nm	Corrector
B2	2^{nd}	Coma	<50nm	Manual: Beam Tilt (coarse) Corrector (fine)
C3	3^{rd}	Spherical Aberration Coefficient	0-3um	Corrector
A3	3^{rd}	4-fold Astigmatism	<1um	Corrector
S3	3^{rd}	Star Aberration	<1um	Corrector

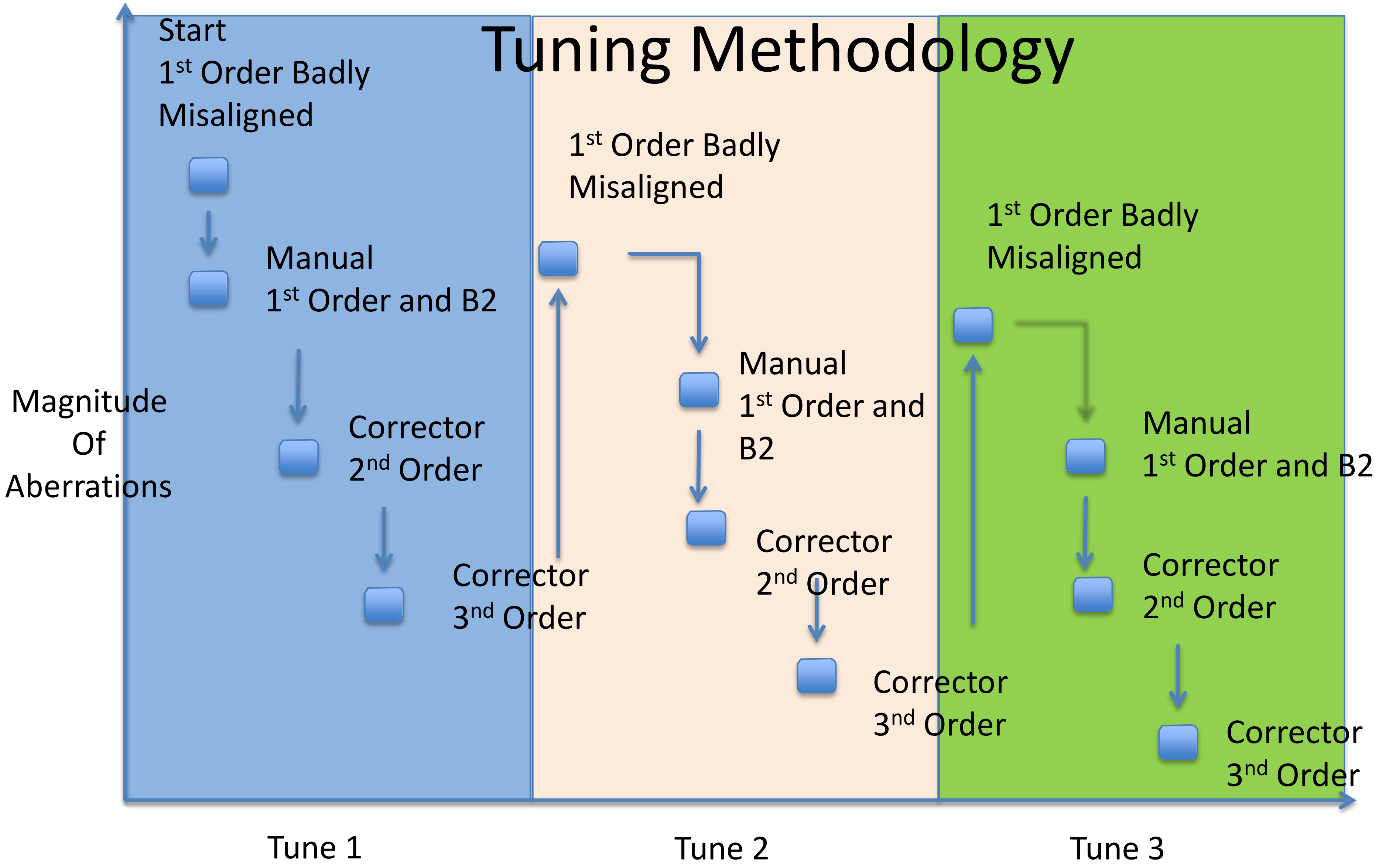
Precision vs Accuracy

- An appropriate specimen with enough density of heavy element particles on a low background support is needed in order to measure the aberrations.
- The CEOS software measures the sharpness of the edges of the particles (in all directions). Anything which affects that sharpness (drift, contamination etc) will invalidate the measurement.
- Magnification will affect the number of particles in the field of view (accuracy) as well as the detail in them (precision).
- A balance between accuracy and precision is needed for a good measurement of aberration coefficients.
- Mag of 400K-600K, 8C probe (30pA), 20um C2 aperture.

Tuning Methodology

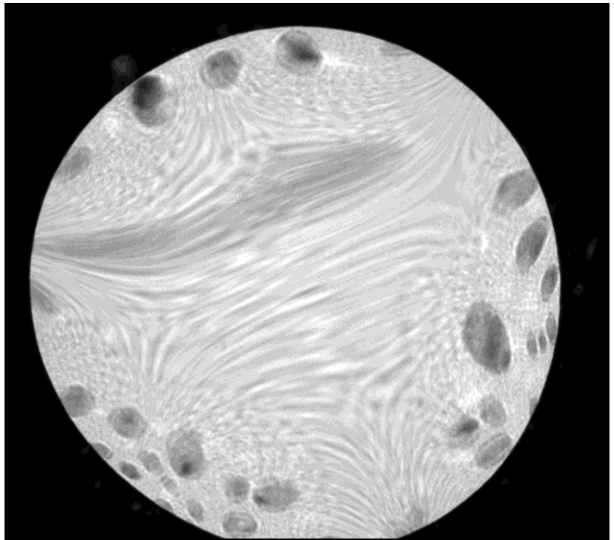
- You must correct all first order aberrations before tuning second order aberrations.
- You must correct all second order aberrations before tuning third order aberrations.
- After a 3rd order tune – first order aberrations will need correcting.
- Repeat the above tuning cycle (1st, 2nd, 3rd, 1st, 2nd . . . etc) until all aberrations are within target range.
- The Ronchigram will grow in size and uniformity as aberrations are tuned out.

Tuning Methodology

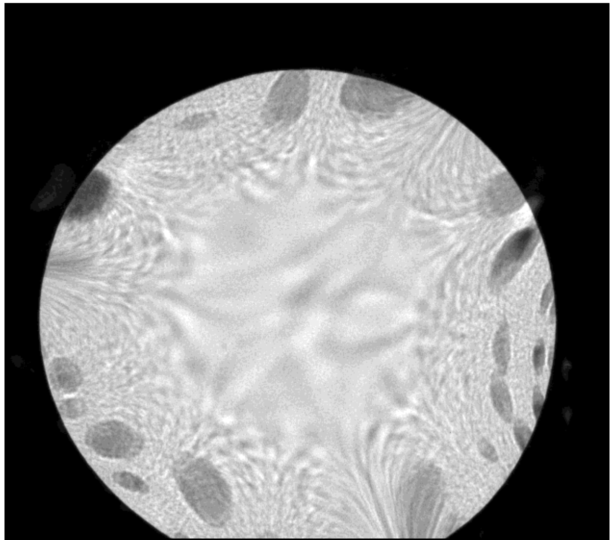


Manually Aligning Ronchigram

- Degauss twice then correct C1 and A1 and coarse alignment of B2 (coma) using the Ronchigram. Note you can manually correct B2 to perhaps 100nm. The software will improve this to ca 20nm.



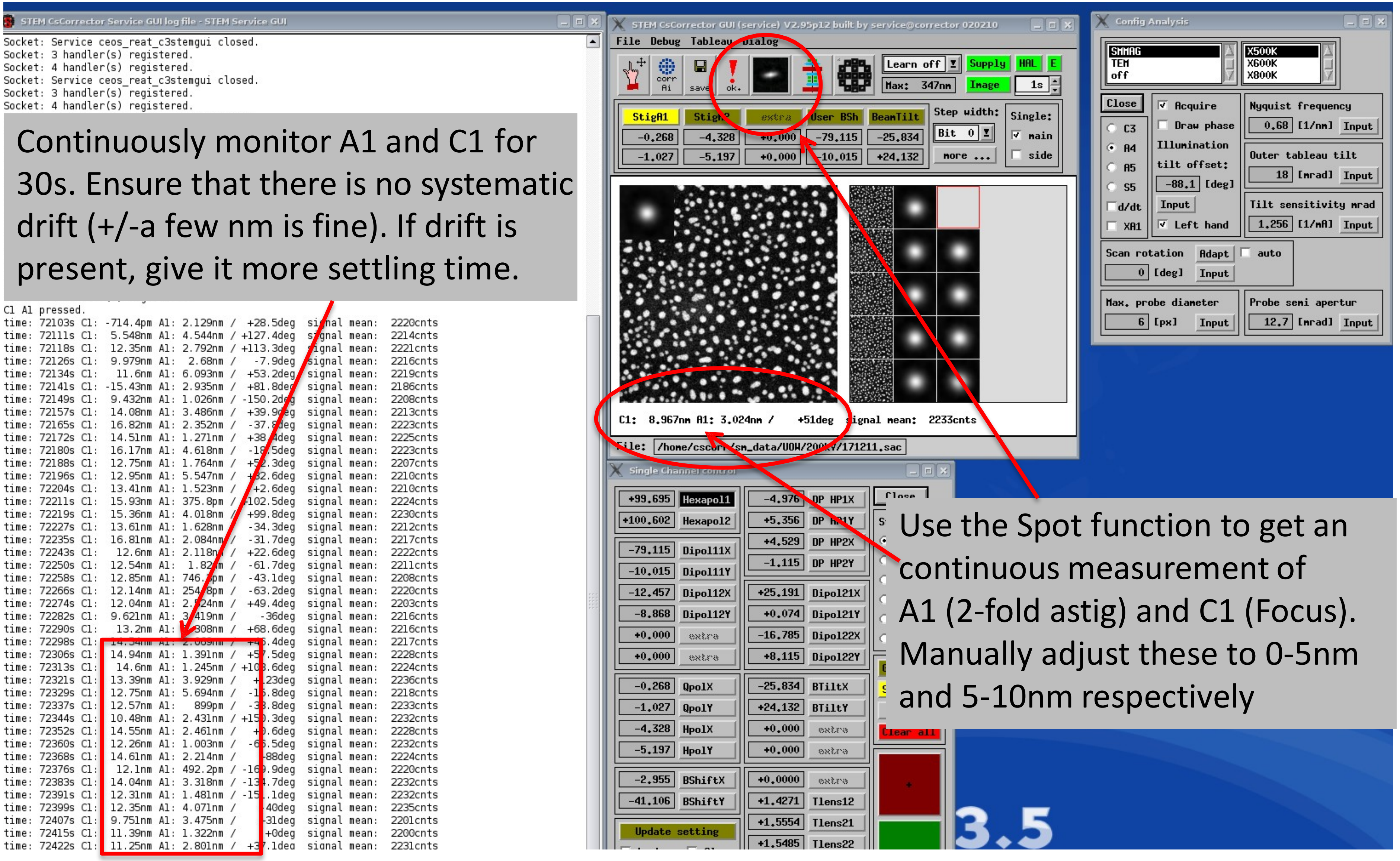
Start: bad A1, some B2



Correctly tuned Ronchigram

Manually Correcting 1st Order Aberrations

Ensure that the beam is centred in the HAADF detector (lower the magnification to 20kx and use projector lens shifts to centre the illumination, then return to higher mag. If off-centre, large tilts (24-27mrads) used for 3rd order correction (later), may run the beam into the HAADF detector.



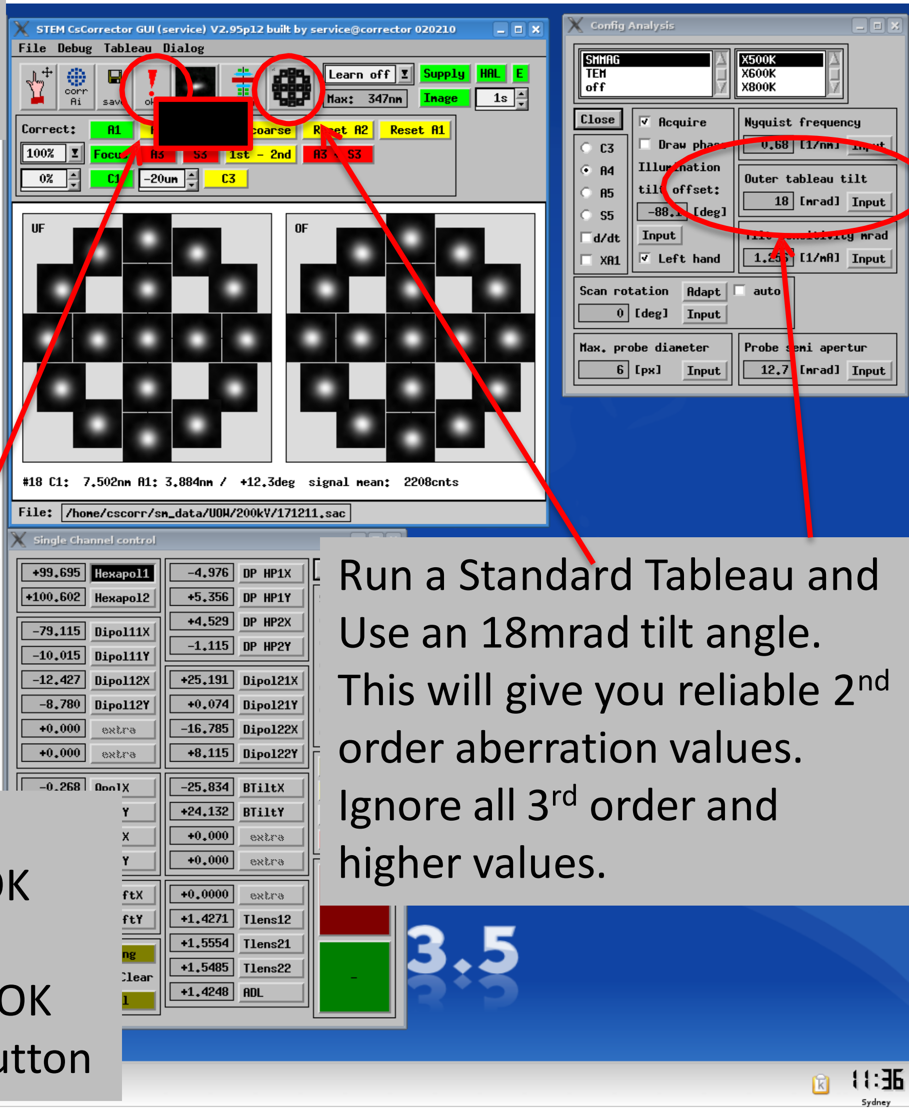
Continuously monitor A1 and C1 for 30s. Ensure that there is no systematic drift (+/-a few nm is fine). If drift is present, give it more settling time.

Use the Spot function to get an continuous measurement of A1 (2-fold astig) and C1 (Focus). Manually adjust these to 0-5nm and 5-10nm respectively

Corrector Tune of 2nd Order Aberrations

Ensure all (or nearly all) of the tableau measurements are used. If more than 3 or 4 are rejected – the result is unreliable. Discard and run again.

```
Tab pressed.
# 1 C1:  2.396nm A1: 5.258nm /  +26.5deg  signal mean:  2252cnts
# 2 C1:  5.329nm A1: 5.355nm /  -58.2deg  signal mean:  2515cnts
# 3 C1:  9.662nm A1: 10.09nm /  -87.4deg  signal mean:  2379cnts
# 4 C1:   7.85nm A1: 9.438nm / -102.7deg  signal mean:  2236cnts
# 5 C1:  5.553nm A1: 4.302nm /  -28.8deg  signal mean:  2120cnts
# 6 C1:   3.14nm A1:  2.84nm / -115.6deg  signal mean:  2100cnts
# 7 C1:  3.058nm A1: 2.797nm /  -54.2deg  signal mean:  2170cnts
# 8 C1:   -954pm A1: 3.174nm /  -11.7deg  signal mean:  2384cnts
# 9 C1: -3.159nm A1: 4.179nm /  -54.6deg  signal mean:  2653cnts
#10 C1:  1.528nm A1: 7.241nm /  -13.8deg  signal mean:  2822cnts
#11 C1: -482.1pm A1: 1.864nm /    +35deg  signal mean:  2895cnts
#12 C1:   3.34nm A1: 3.839nm /    -73deg  signal mean:  2811cnts
#13 C1:  7.517nm A1: 6.981nm /  -25.9deg  signal mean:  2677cnts
#14 C1:  2.867nm A1: 4.579nm /  -12.8deg  signal mean:  2331cnts
#15 C1:   4.92nm A1: 2.533nm /  -43.2deg  signal mean:  2136cnts
#16 C1:  1.896nm A1: 996.7pm /  +68.2deg  signal mean:  2266cnts
#17 C1:  2.246nm A1: 3.162nm /  -55.5deg  signal mean:  2475cnts
#18 C1:  2.204nm A1: 2.937nm /  -27.4deg  signal mean:  2251cnts
dTime: 73773s Date: Fri Jan  5 01:53:42 2018
1st order measured! (not used:)
Sall: 3.897nm Sused: 3.897nm (1.122%)
C1:    2.3nm               (95%:  4.9nm)
A1:   3.69nm /   +7.7deg  (95%: 6.06nm)
A2:  24.24nm /   -156deg  (95%: 45.7nm)
B2:  61.61nm /  +44.5deg  (95%: 29.2nm)
C3:  2.322um              (95%: 1.71um)
A3:  707.3nm / -152.2deg  (95%: 1.74um)
S3:  1.474um / +111.2deg  (95%:  753nm)
A4:  50.33um /  +74.8deg  (95%: 72.7um)
Btn 'Accept Aberr' pressed.
Btn 'B2' pressed.
```



A2=24 +/- 48nm OK
B2=62 +/- 29nm Not OK

Correct B2 by clicking OK and pressing the B2 button

Run a Standard Tableau and Use an 18mrad tilt angle. This will give you reliable 2nd order aberration values. Ignore all 3rd order and higher values.

Corrector Tune of 2nd Order Aberrations ctd

- Continue tuning 2nd order aberrations until the A2 and B2 values are within the target range (both <50nm).
- Only then move on to correcting 3rd order aberrations.

```
1st order measured! (not used:)
Sall: 3.141nm Sused: 3.141nm (0.9042%)
C1:   8.456nm                (95%: 2.68nm)
A1:   3.064nm /   -35.9deg (95%: 5.68nm)
A2:   29.99nm / -139.2deg (95%: 42.8nm)
B2:   25.86nm /   -50.9deg (95%: 19.9nm)
C3: -968.1nm                (95%: 1.08um)
A3:   507.6nm /     +33deg (95%: 1.63um)
S3:   350.6nm / +127.9deg (95%:  614nm)
A4:   44.91um / +121.3deg (95%: 68.1um)
```

Target Values

C1 5-10nm

A1 5nm

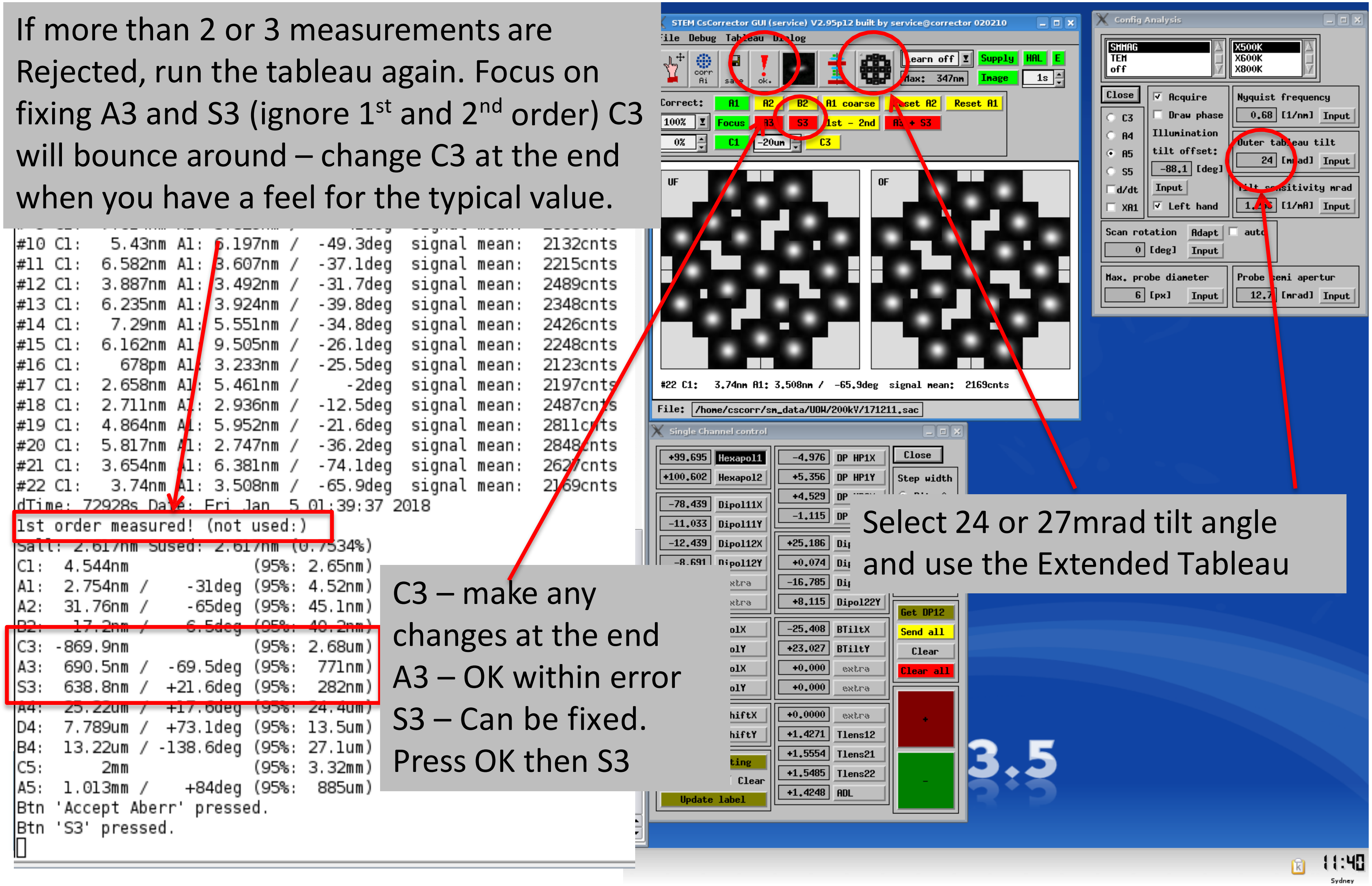
A2 <50nm

B2 <50nm

Ignore 3rd order

Corrector Tune of 3rd Order Aberrations

If more than 2 or 3 measurements are Rejected, run the tableau again. Focus on fixing A3 and S3 (ignore 1st and 2nd order) C3 will bounce around – change C3 at the end when you have a feel for the typical value.

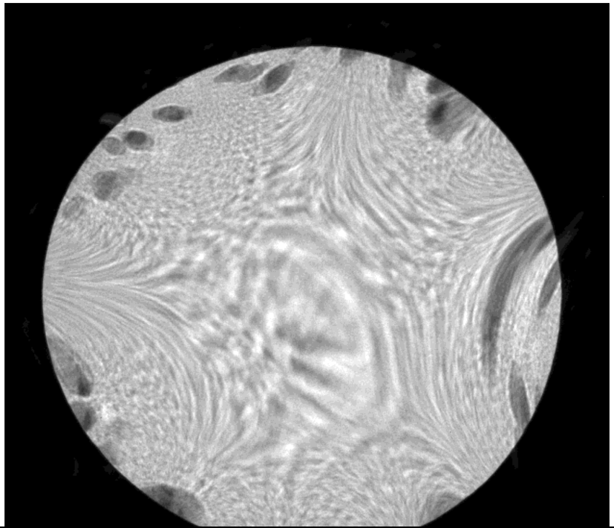


C3 – make any changes at the end
A3 – OK within error
S3 – Can be fixed.
Press OK then S3

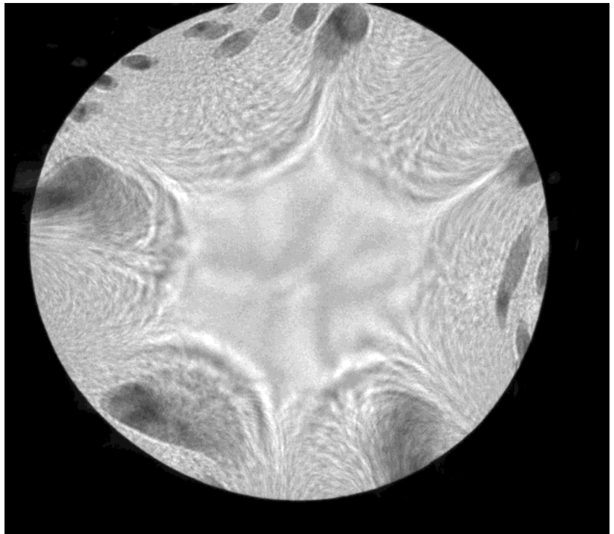
Select 24 or 27mrad tilt angle and use the Extended Tableau

After Correcting 3rd order Aberrations

- Correct whichever is the worst aberration (A3 or S3). Corrections will ruin 1st and 2nd order alignments.
- Correct these by generating a Ronchigram and manually correct C1 (focus), A1 (2-fold astig) and B2 (coma).

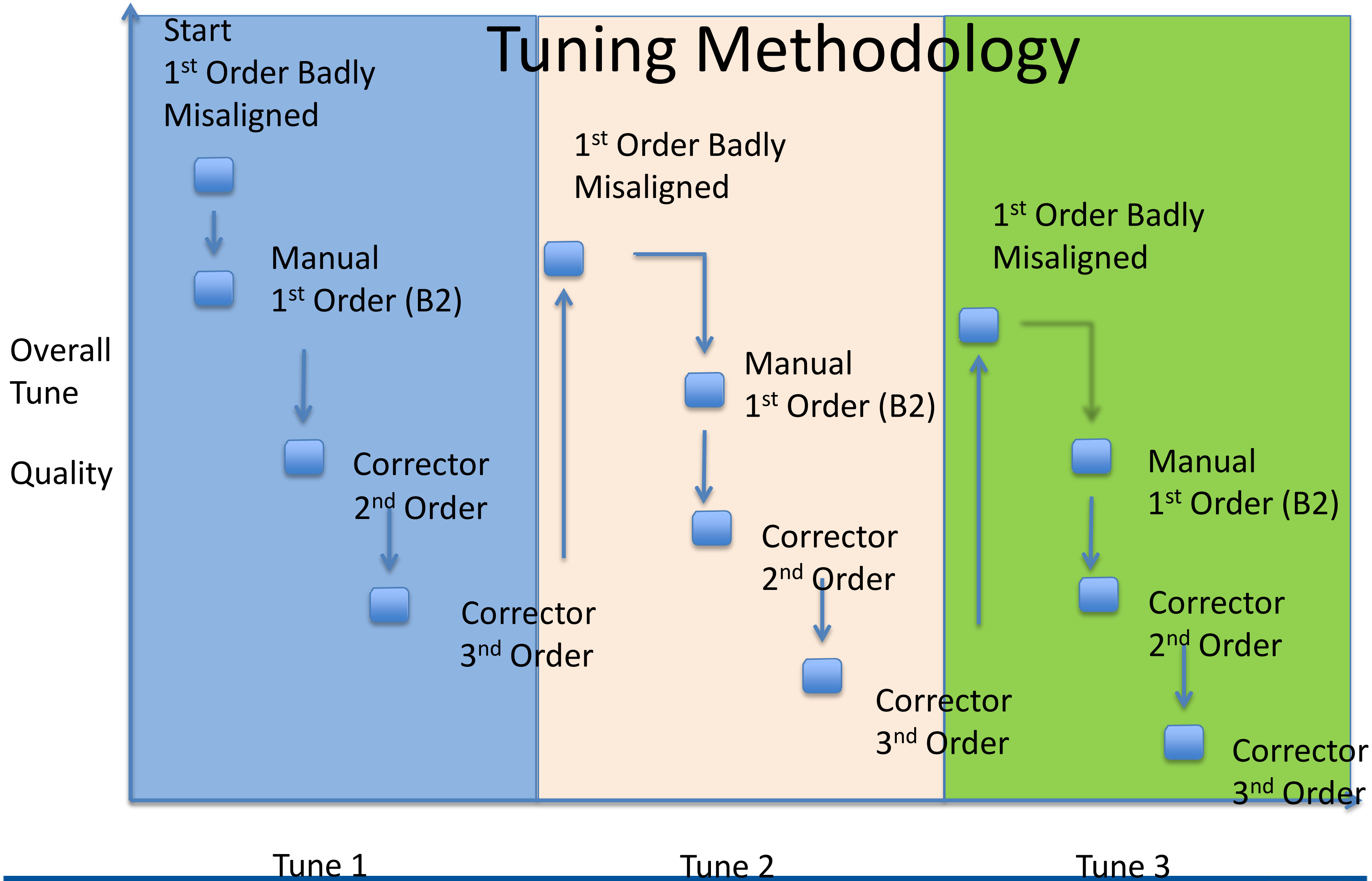


After correcting S3: bad coma



Correctly tuned Ronchigram

Tuning Methodology



After Completing the nth Tune

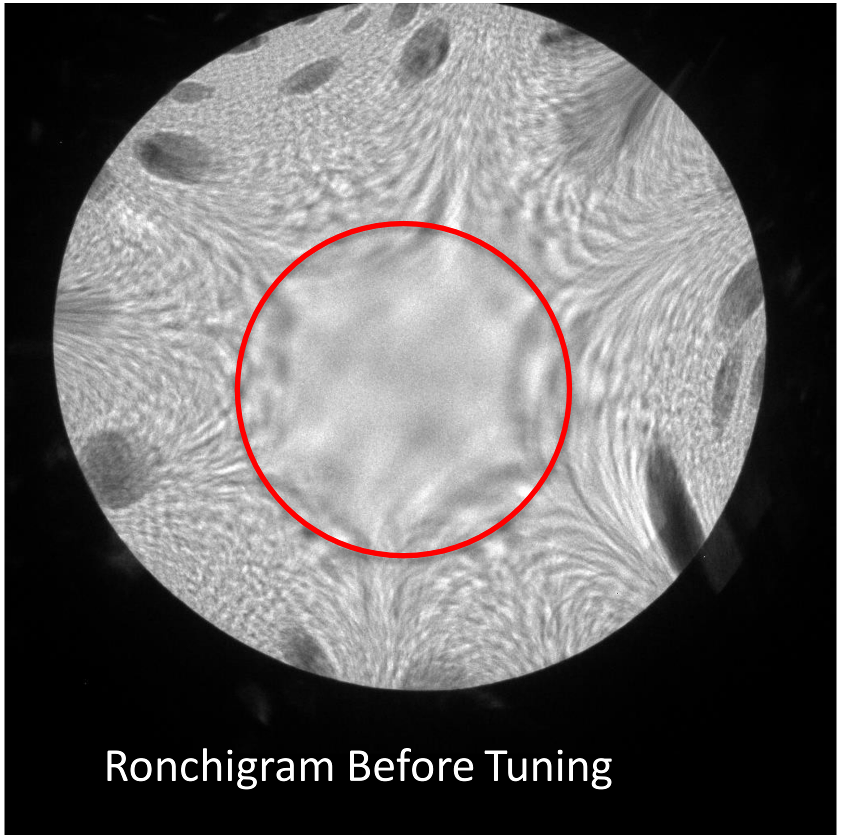
- After fixing 1st order aberrations, run one or more Standard Tableaux and fix 2nd order.
- Then run an Extended Tableaux and fix third order aberrations – Repeat the tuning cycle to converge on target values. An unstable microscope will not converge.

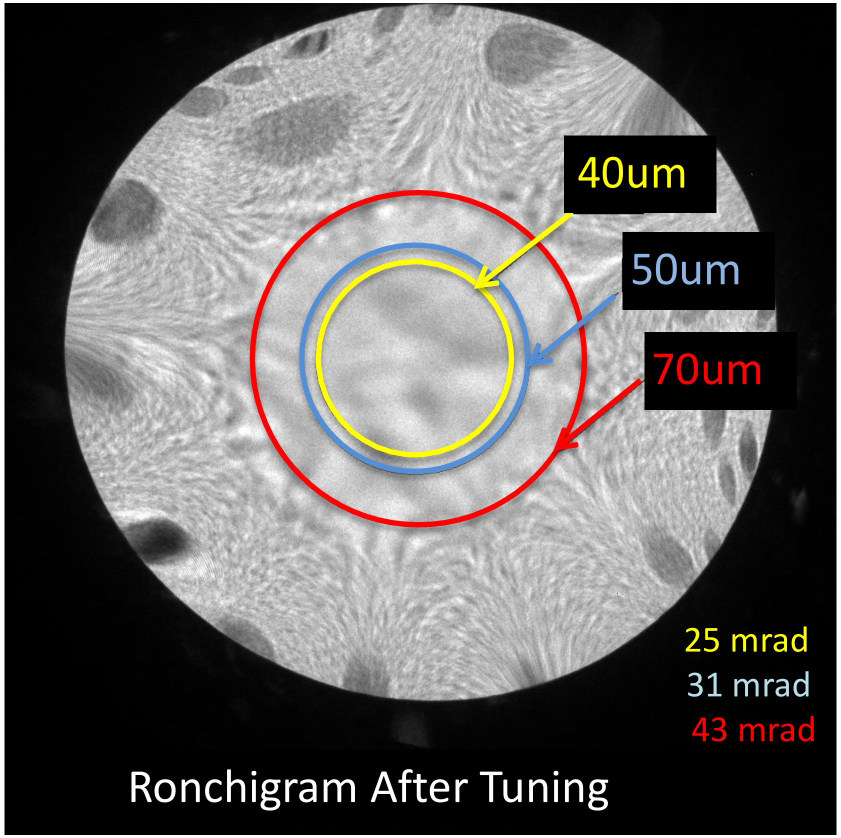
```
1st order measured! (not used:)
Sall: 3.913nm Sused: 3.913nm (1.126%)
C1:   2.614nm                (95%: 3.97nm)
A1:   1.813nm /  +45.5deg (95%: 6.74nm)
A2:   65.71nm / +122.6deg (95%: 67.5nm)
B2:   36.21nm /  +53.4deg (95%: 60.1nm)
C3:   3.034um                (95%: 4.02um)
A3:   461.2nm /  -64.9deg (95%: 1.15um)
S3:   552.1nm /  +72.6deg (95%:  422nm)
A4:   42.84um / +117.2deg (95%: 36.5um)
D4:   11.48um /  +62.6deg (95%: 20.2um)
B4:   20.74um /  -98.2deg (95%: 40.6um)
C5:   599.6um                (95%: 4.97mm)
A5:   1.602mm / +104.5deg (95%: 1.32mm)
```

- Only trust 1st and 2nd order values from a Standard Tableau.
- Only trust 3rd order values from an Extended Tableau – results shown left.
- Here A2 is higher than target (<50nm) – ignore it (note the large error). The value in the previous Standard Tableau was <50nm.
- C3 bounced around a bit during tuning, but the average was 3um – which is on target.
- A3 and S3 are <1um – which is fine.
- The Ronchigram appearance/size is more important than the numbers.

The Goal of Tuning

- The aim of tuning the corrector is to produce a very large aberration-free region within a symmetric Ronchigram.





Closing Remarks.

1. Minimise the amount of work the software has to do during tuning, by manually correcting focus (C1), 2-fold astigmatism (A1) and coma (B2) as well as possible, before asking the software to correct those values.
2. Monitor the hexapole values HpolX and HpolY, especially when correcting A2 (3-fold astitgmatism). Keep the values to within +/- 10um. If the values become very large, then heating/hysteresis may result in an unstable tune. If the values move slightly outside the +/-10um range during tuning eg +/-15um, then avoid changing them immediately, as they may move back towards zero during subsequent tunes. However, if they move outside that range significantly, reset them by choosing Reset A2. This may mess up the tune somewhat – so go back to stage 1 (Manual correction) and proceed from there.
3. Low voltage (60-80kV). Good tunes are much more difficult at low voltage, as minor variances have a much bigger effect. I routinely switch to 80kV on Friday pm. Allow the weekend to magnetically and thermally equilibrate, then tune the following Monday. However, I find this tune is not very stable and by Mon pm/Tues the Ronchigram has reduced in size. I reserve all the low resolution work, such as EDS mapping, for Mon and Tues. Then on Wed am I again retune the corrector. This results is a very stable tune for the remainder of the week. I reserve all high resolution work – eg graphene ring imaging - from Wed-Fri. In general, 200kV tunes are much easier. If I tune on a Monday (after switching to 200kV on Friday), the tune is very stable thereafter and will not need to be redone.
4. Microscope stability is critically important when tuning. Beam shower the specimen – there can be no contamination occurring during tuning. Always degauss (Lens Relax) the column twice before attempting a tune. Ensure the microscope is at standard focus and adjust the height to focus. Large changes in specimen height can result in subsequent focus drift. Ideally complete all major stage and height adjustments the night before tuning, to allow settling. At the start of tuning, monitor the focus and astigmatism for 30s or more to ensure that it is not changing. If it is, give it 30mins settling time and try again.
5. For high performance STEM, avoid using Low Mag mode if humanly possible. Low Mag mode turns off the objective lens (1kW of power). If you must use Low Mag – eg to find a FIB section, switch into Low Mag, find the region as quickly as possible (<20s), then switch back out immediately. In Low Mag mode the objective lens begins to cool, causing thermal drift. After using Low Mag, degauss the column (twice). DO NOT use Low Mag within 1hr of tuning. Even after degaussing, the objective lens may still be (thermally) unstable for 30 or more minutes.
6. When doing routine STEM, if you change spot size, the condenser lens crossover will move (relative to the corrector) and the Ronchigram will be degraded. Degauss the column twice then manually correct the A1, C1 and B2 in the Ronchigram. If you have a cold FEG microscope an alternative approach to changing spot size is to change the FEG extraction voltage, to change the probe current. Here the condenser lens crossover does not move (much) and the Ronchigram will not a retune – unless doing high res.