

# Basic 5.3.2.2 Operations

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## **I. Purpose**

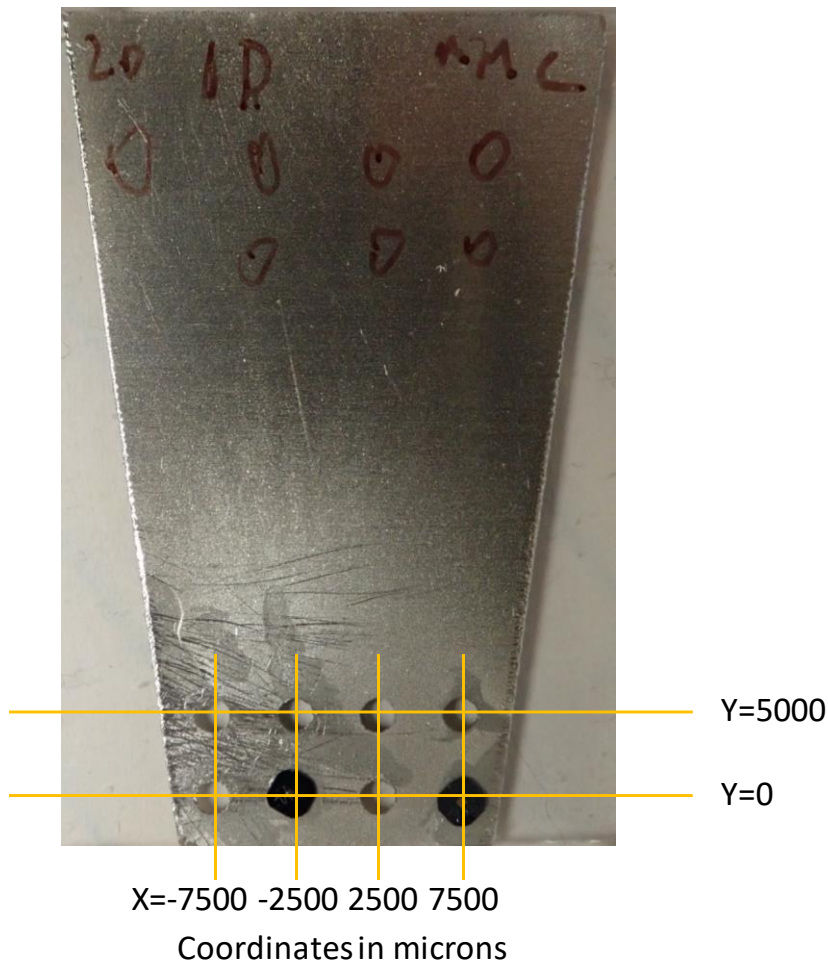
This manual is intended as a guide to the basic operations of inserting a sample, pumping it down, finding the desired region, focusing and taking stacks and linescans. It is not intended to be comprehensive or to cover operations, such as OSA focus scans, which are not part of normal operations. The sections below are organized roughly in the order in which operations are performed.

The typical sequence of operations for spectromicroscopy is:

1. Mount samples on a sample plate and find the approximate coordinates of where you want to image.
2. Introduce the sample plate into the microscope; pump and backfill with the chosen atmosphere.
3. Start imaging and find the feature(s) of interest; focus.
4. Do single-energy images, stacks, difference maps or linescans.

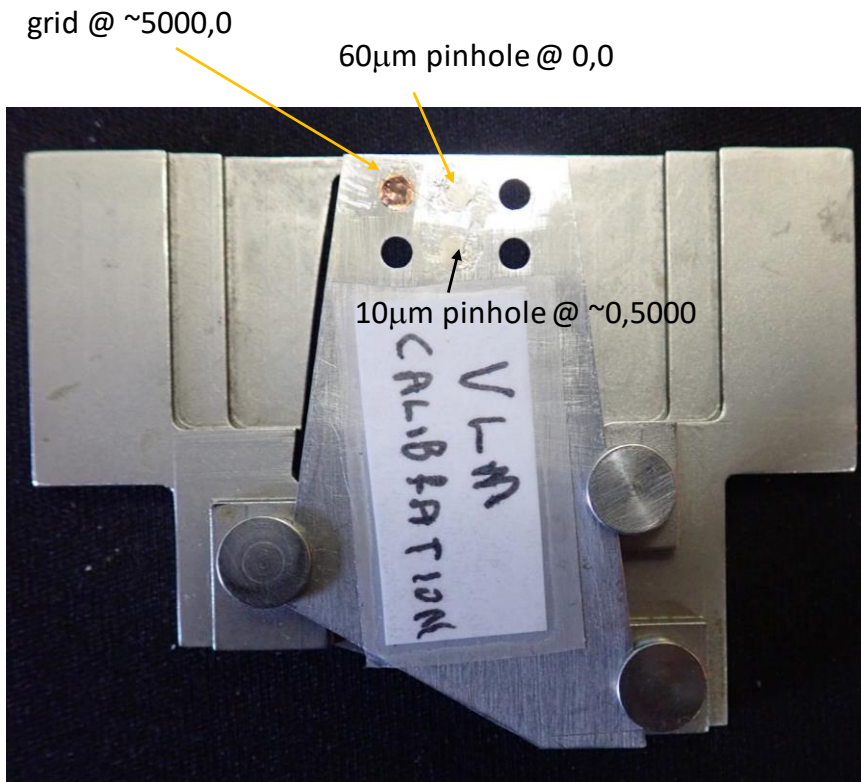
## **II. Sample mounting and location**

Here is a typical example of a sample plate with membrane samples, as seen looking downstream.

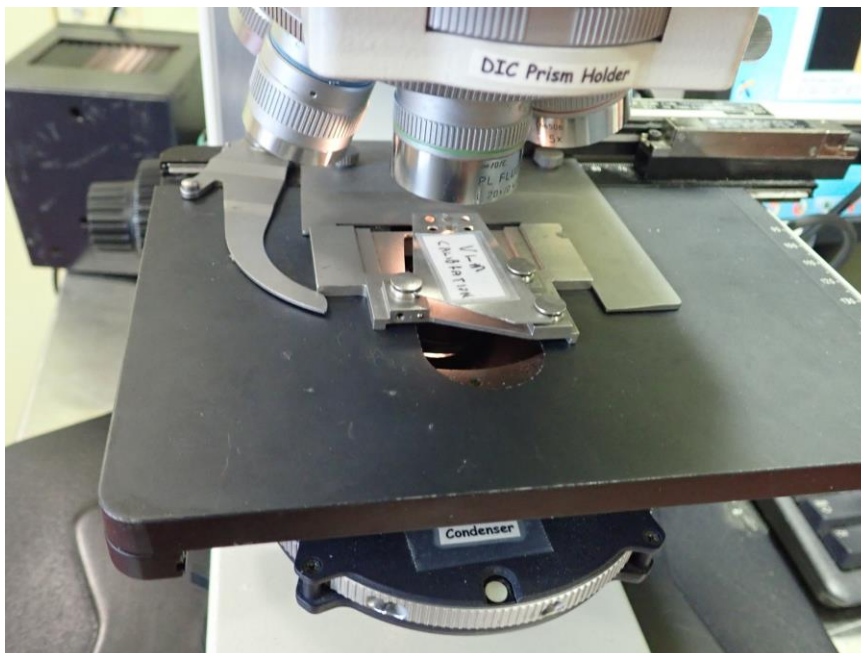


There are a few options available for mounting samples, which could be TEM grids or SiN windows. In the above, the windows are TEM-format (3mm dia.) frames with 0.5mm-square windows. To attach windows, users use nail polish, Elmer's glue or double-sticky tape. To use tapes, put the tape over the holes, then use a drill by hand to punch out the tape that's in the holes.

The holes are 2mm across and 5mm apart. The nominal Y positions of the two rows are +5000 $\mu$ m and 0 $\mu$ m (top and bottom), and the nominal X positions are -7500, -2500, +2500 and +7500 $\mu$ m for the horizontal positions, reading left to right. These positions could be used as starting points for imaging, but a 2mm-wide scan may then be needed, which takes time. Therefore, we have a visible-light microscope (VLM) outside the hutch with an encoded stage. Using this can get you to within 100 $\mu$ m or so of the actual position at which a feature will be found on the STXM. We have a calibrator sample which includes an aperture for which the STXM stages have been calibrated so that it lands at (0,0) when mounted. To use for calibrating the microscope, this is mounted in a model sample holder as shown here:

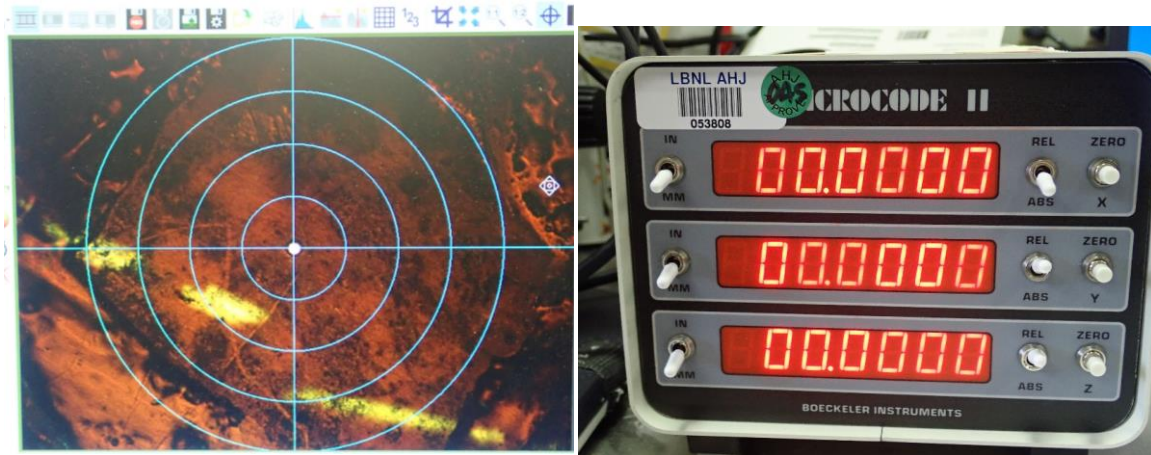


The one in the upper middle (overexposed) is the calibration hole. Make sure when mounting the calibration sample in the holder that it slides all the way forward on the three slotted pins; it tends to get stuck. Then, mount this package on the microscope stage and set the microscope to transmission illumination (the power supplies for the illuminators are long black boxes behind the VLM):

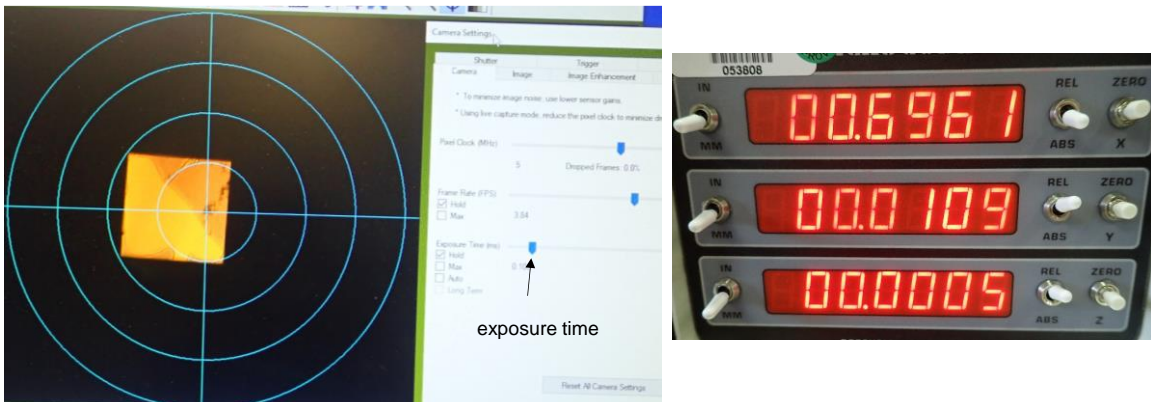


There is an encoder reader on the table next to the microscope. This reads on 3 axes, with the units being centimeters in all 3 axis. Thus, if you ignore the decimal point, you read in microns. The sign convention for X and Y is the same as for the STXM.

Next, fire up the Thorcam program on the VLM computer and start capture. Find the calibration hole. Make sure you're on that hole and not the smaller one in the top row. Hit the reset buttons on the encoder. You can activate the reticle as shown below for a position reference. The computer screen and encoder reader now look like this:



Carefully remove the calibration sample without dislodging the 3-pin holder from the stage and replace it with your sample. Navigate until you see the feature of interest. The screen and encoder reader now look like:



The sample here is a broken 500 $\mu$ m window with particles on the intact bits. Let's pretend that this is a region of interest. The red/yellow color is due to the color balance of the light source. The window shown to the right of the sample image is a Settings window from Thorcam, allowing the exposure to be shortened because the picture is bright. The encoder positions are now as shown on the encoder reader, (6961,109). That the Z encoder reads a small number shows that the sample happens to be at the same level as the hole in the VLC calibrator. This is

your starting point for finding the region in the STXM. Write down the coordinates for all samples on the plate so that you can quickly navigate to each in turn. You will find that the focus of each sample will be a bit different; this will also apply to STXM.

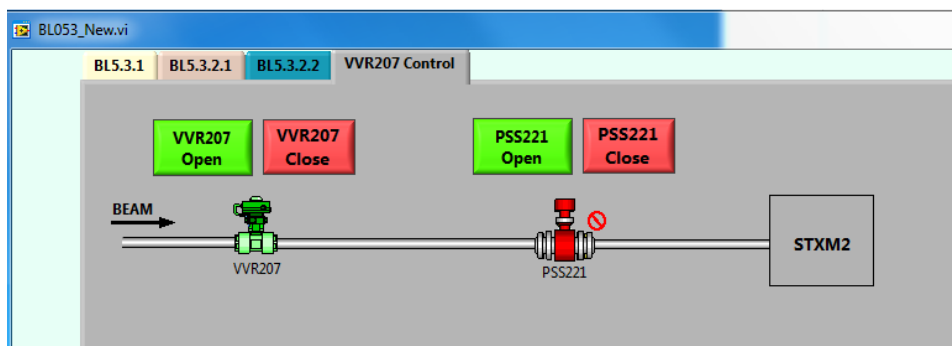
### III. Getting in and out of the microscope

There is a checklist for pumping and venting the microscope. It's accessible as a PowerPoint with a Desktop shortcut but it will be summarized here. The various parts referred to may be found on the following pictures and are explained below. To vent the chamber in order to introduce a sample, the steps are:

1. PMT HV off
2. VVR207 and shutter closed
3. If venting to 1 atmosphere, loosen door-closing bolt and top-lid knob
4. All valves closed
5. Fill gas flowing through pressure relief valve
6. Vent valve open until desired pressure reached
7. If changing/loading sample, Coarse Z to 9000

The reason for undoing the knob for the top lid is that it will act as a pressure relief valve and prevent any possibility of an overpressure condition which might pop the x-ray transmitting window.

The power supply for the PMT (detector) is in the NIM bin in the half-rack under the beamline. It's a brown, single-width module and the switch is a vertical toggle. VVR207 is the valve just upstream of the hutch and it's controlled through the EPS screen on the console computer:



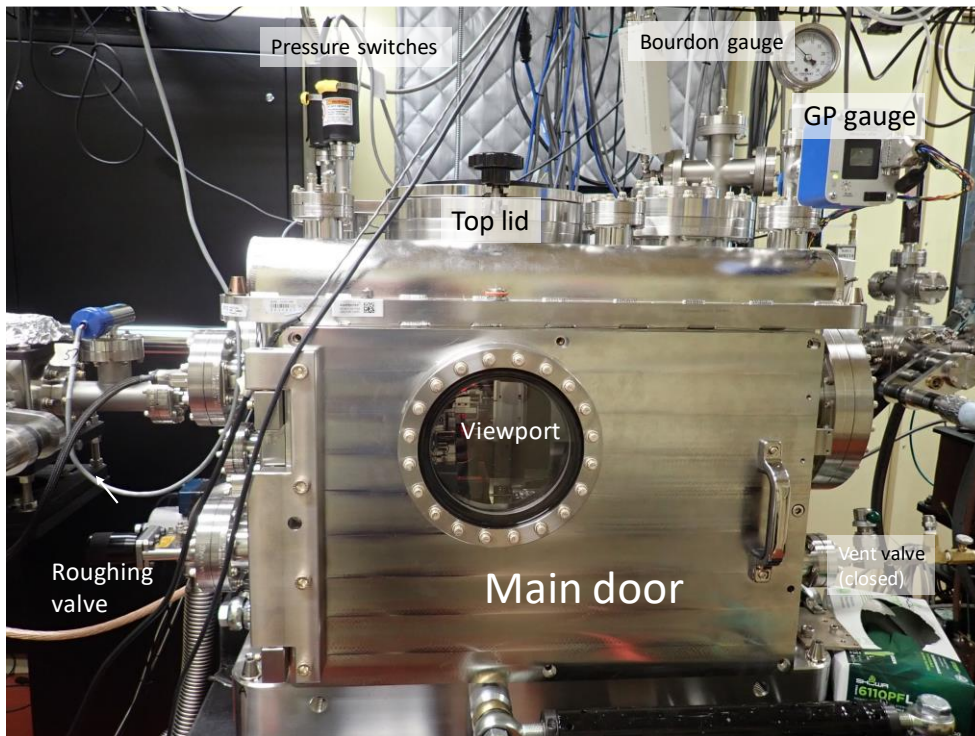
The Desktop shortcut is labeled 'EPS'. It's easier to find on the Taskbar at the bottom, where it looks like a LabVIEW icon.

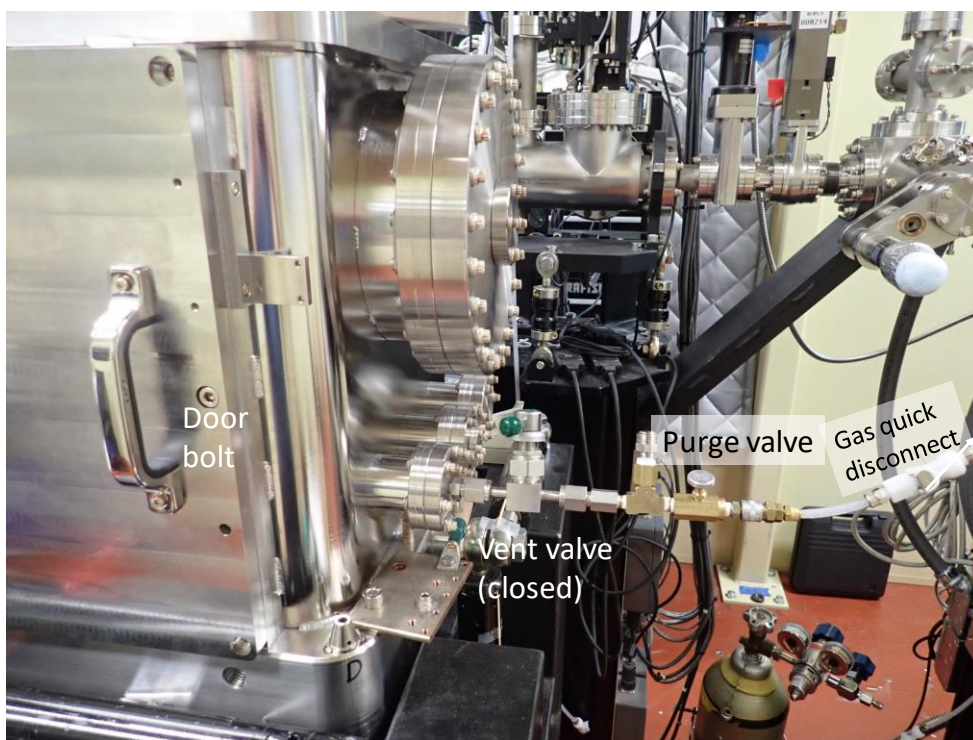
If there are yellow exclamation points on the valve icons, don't panic. Just dismiss and restart the EPS program. It's just that the program lost communication with the server.



The ‘Coarse Z’ referred to in step 7 is the longitudinal position of the sample, which is controlled through the STXM control program, which will be shown later. The purpose of this step is to move the sample away from the zone plate and OSA.

Here are the external parts of the microscope, showing the plumbing you’ll need to work with.

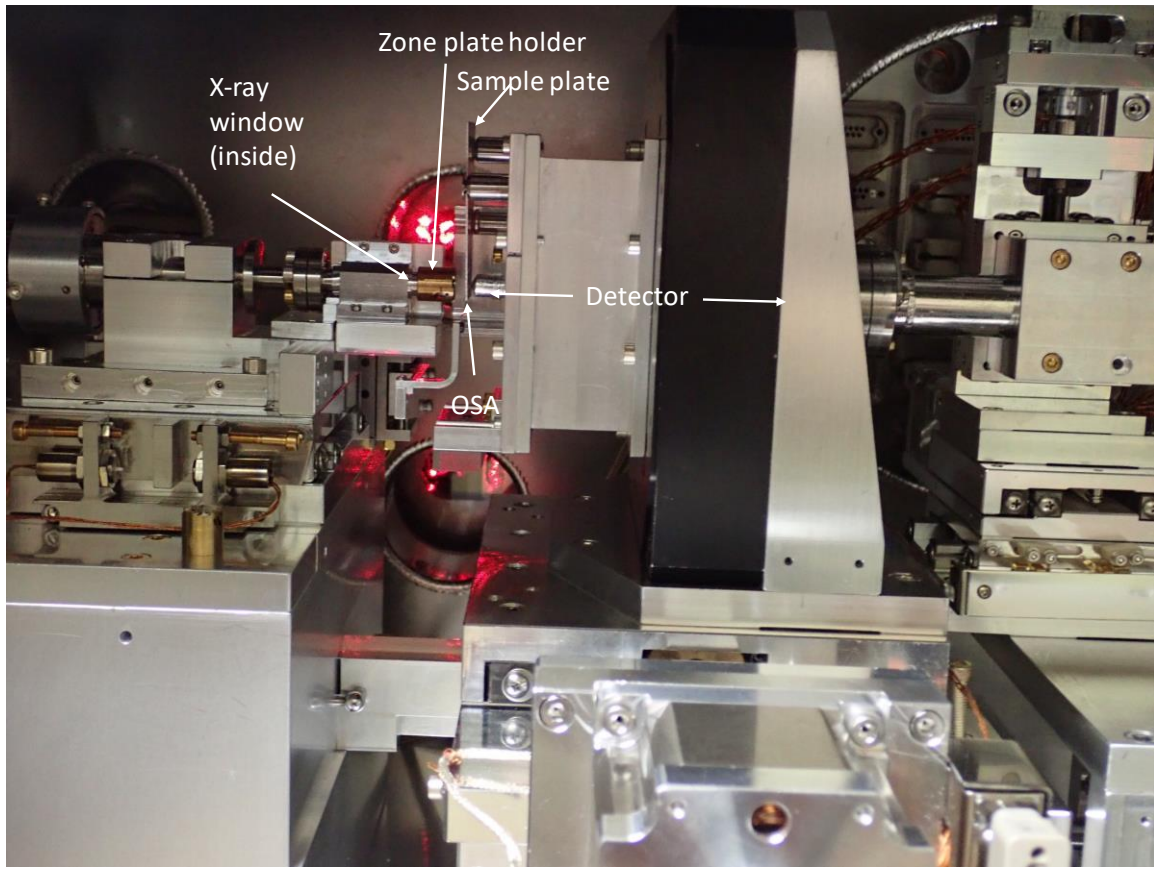




The microscope is pumped out through the roughing valve on the upstream side (black handle). The roughing pump is upstream of the hutch. There is a power switch for it in over the console to the left of the monitor, between two outlets. The vent/fill setup is on the downstream side of the microscope (lower picture). There is a choice of four fill gases, house nitrogen, helium, argon and CO<sub>2</sub> (for energy calibration), each of which comes in on a plastic hose terminated in a white plastic quick-disconnect fitting part of which is seen on the right-hand side of the above picture. The quick-disconnect fittings are labeled. To change gases, pop off the old fitting and snap on the new. The gas goes into a tee with a valve and a pressure relief on top (update: there is now an elbow in the line so the purge and pressure-relief valves are in a vertical line). This is the purge valve. Opening it purges the fill line of the previous gas and also limits its pressure, thus reducing the chance of overpressure and popping the X-ray window in the chamber.

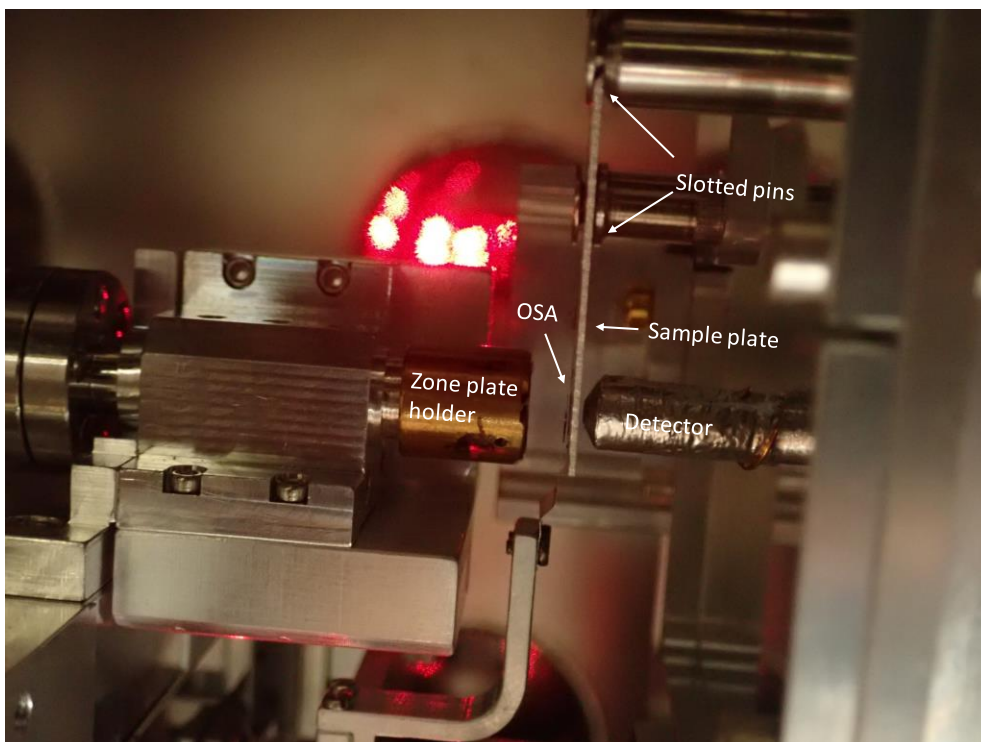
*Note: All valves need only light pressure to close properly. It is not necessary to put one's back into it. Please be gentle.*

Here's what it looks like inside the microscope, with the sample in position to be imaged at the Fe L-edges (710eV):



When the sample moves back, so does the whole sample plate/detector assembly. The OSA remains fixed and is the reference for  $Z=0$ . The zone plate moves longitudinally in order to focus at different energies. Zooming in on the sample area, we see:





You can see that the OSA is really close to the sample plate. The zone plate is several millimeters away from the OSA because the microscope was focused at a high energy; if it were doing C, the zone plate would be a lot closer. For scale, note that the ZP holder is 10.5mm in diameter. Note that the sample engages three slotted pins, just like on the VLM setup. When mounting a sample, make sure it engages all three pins.

To remove a sample, put on a glove, grasp the sample plate at the top and pull straight up. There is a wire which can be swung around to engage with the top of the sample; make sure that's off before pulling up. To put in the new sample, insert it straight down, feeling with a gloved finger to make sure that it got into the slots on all three pins, and isn't ahead of or behind or tilted. Push gently down on the top to make sure it's seated. Feel around for the wire and get it to hook on the top of the sample plate.

Once a sample is inserted, you need to pump out the chamber. Here are the steps:

1. PMT HV off
2. Vent valve closed
3. VVR207 closed
4. Doors closed
5. Chamber roughing valve open and rough pump on.
6. When pressure <1Torr, chamber roughing valve closed
7. If loading sample, Coarse Z to 1000

You can start imaging while the chamber is still being pumped, but for optimum stability, you want to have some He fill gas ( $1/3$  atmosphere is the canonical amount). Also, the shutter won't open until the pressure is less than about  $1/3$  atm. This is a safety feature which makes sure that the box is closed up before beam can be present. Pressures may be read on the GP gauge, but for high pressures like  $1/3$  atm., it's easier to use the Bourdon (aneroid) gauge. This gauge reads in inches of Hg vacuum for pressures below atmosphere, so  $1/3$  atmosphere absolute pressure = 20" on the gauge. For sensitive spectroscopy, you might want to pump and purge a couple of times.

In order to get beam into the microscope, the interlocks have to be satisfied and VVR207 open. The interlocks used to be switches on the doors, but now they're pressure switches which turn on only when the pressure in the chamber is  $1/3$  atmosphere (about 20" in the aneroid gauge) or less. The state of the interlocks is shown by the interlock box on the wall to the left of the console:



The upper picture shows the state of the interlocks when the shutter may be opened, and the bottom one shows their state when the pressure in the chamber is too high. As with other RSS systems around the ring, there are two independent switches which both have to be satisfied for beam to come on.

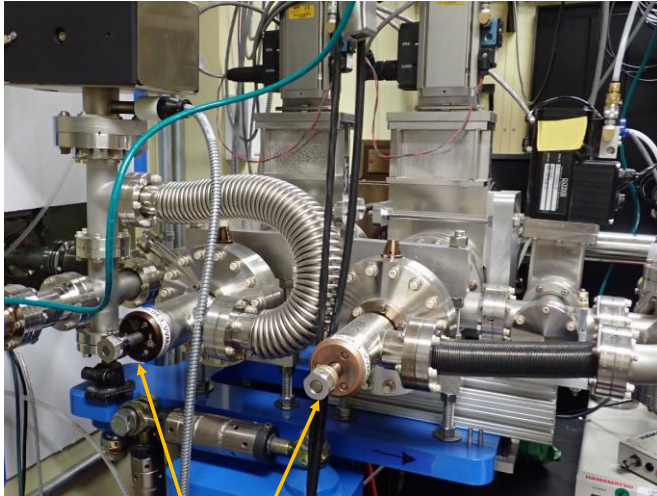
Here are the steps:

1. VVR207 open
2. Pressure low enough for interlocks to be satisfied
3. Shutter open. 'Door lock enabled' light goes off.
4. Doors covered and PMT HV on

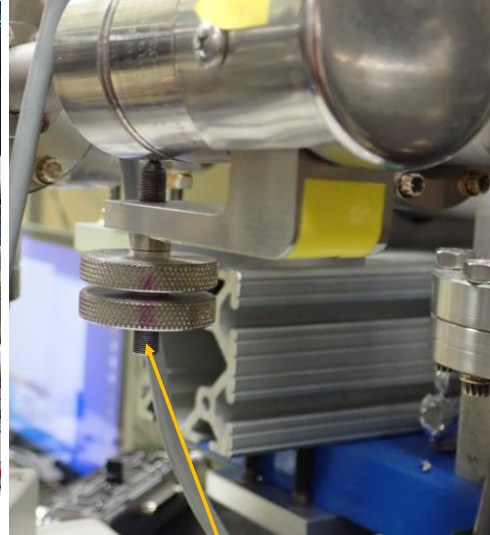
The door covers have to be on before the PMT is turned on because there is light leakage into the detector.

#### **IV. Nitrogen filter (skip this if not doing carbon)**

There is a considerable amount of second-order light coming out of the monochromator and getting into the microscope. This can produce a spurious feature at an energy equal to half the oxygen K-edge energy, which is within the range of a C-K scan. To reduce this effect, we add nitrogen, which absorbs most of the second-order light while passing the fundamental. The nitrogen goes in a section of beamline just upstream of the microscope. In order to allow a high pressure of nitrogen in this section without having a window, there is a differential-pump section consisting of four pinholes with turbo pumps in between and a leak valve downstream. The pinholes limit the amount of nitrogen which gets into the downstream-most ion pump. This arrangement is usually referred to as the nitrogen filter, although the actual filtering is mostly done by the nitrogen between it and the microscope. The differential-pump section is shown here, as seen from the console, with the leak valve seen from the back (between the beamline and the tall rack):

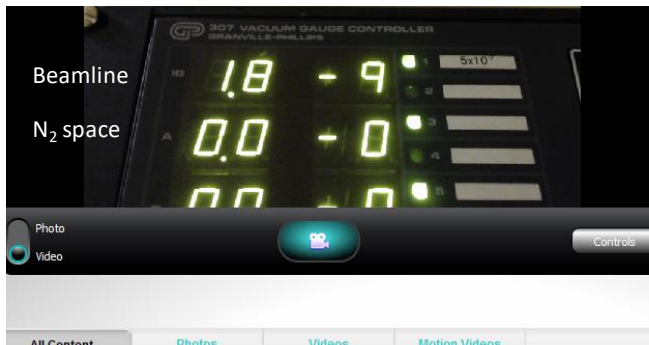


Bypass valves



Leak valve (open)

To engage the filter, close the downstream bypass valve (the upstream one is always closed) and open the leak valve slowly. Note that the leak valve is mounted pointing down, so to open it, you need to turn it CW as *viewed from above*, and to close it, CCW as *viewed from above*. At first, nothing will happen, then the pressures will increase. When they get to the -1's, stop turning and let it stabilize. Adjust from there, using plenty of patience as the rise is very slow. The pressure you want to aim for is between 0.6 and 1.0T. The pressures may be monitored using the Logitech camera program:



The top value is the pressure just upstream of the apertures and should be in the 9's. The lower two are the pressures in the gas-filtering sections. These should stabilize at 0.6-1.0T. To turn off the gas filter, close the leak valve and open only the downstream bypass valve; the upstream valve stays closed at all times.

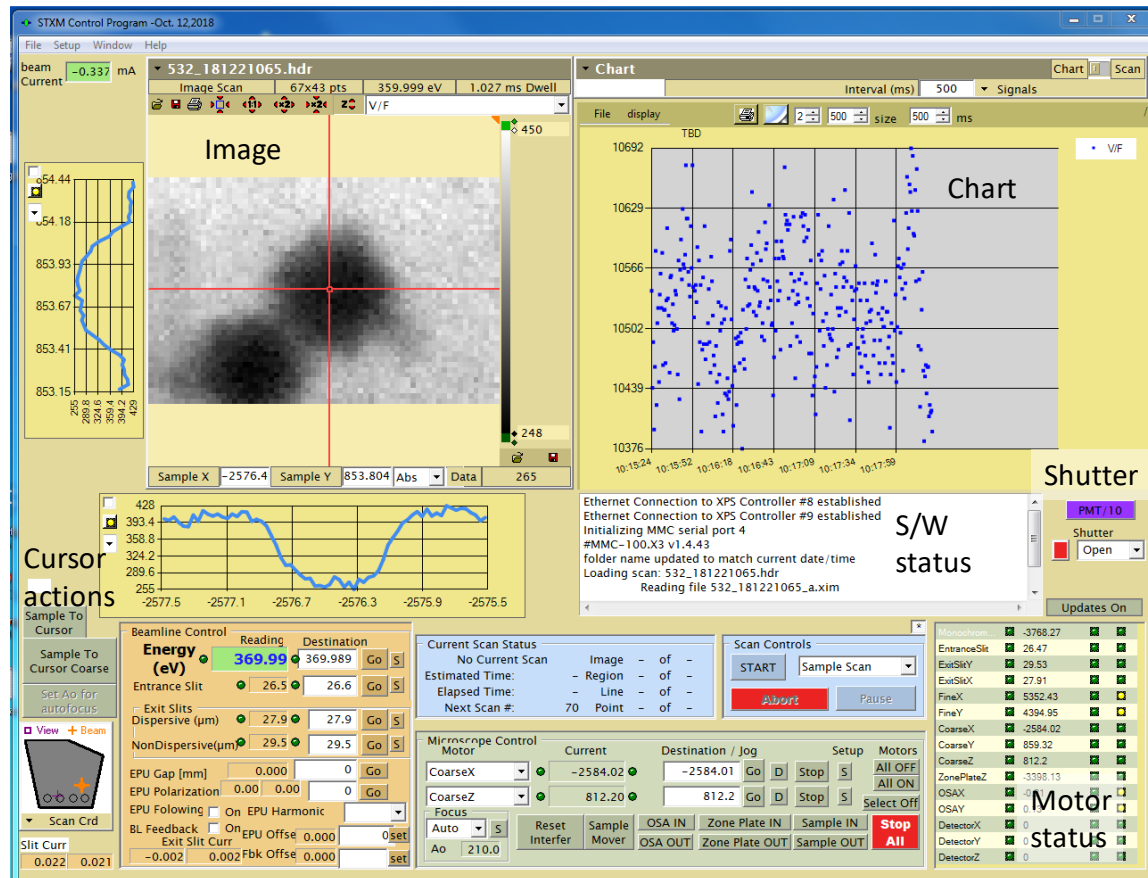
Note that the use of the nitrogen filter affects the operation of the beam feedback program for the vertical direction. This is because the exit slits are in the filter gas and so produce less



photoelectron signal for the feedback to work on than they would in vacuum. Thus, there may be a vertical shift between the beam position with and without gas under feedback control. At the Fe edge, the signal is so small that the feedback is ineffective, so the beam must be steered in manual mode.

## V. STXM control program and taking data

This is the control interface to the microscope and all the motors. While it is possible to talk to the motor controllers directly, this is not necessary unless there's a failure of some sort, which does happen occasionally. Not *all* the functions of STXM Control will be explained here because many are used for maintenance, diagnostic, calibration and adjustment purposes. The main screen is divided into several areas, most of which are labeled and set off from each other by different-colored backgrounds:



Now we'll go through a typical sequence. After inserting the sample and starting to pump down, we need to move the sample closer to the OSA than the 9000 $\mu\text{m}$  'safety' distance used when exchanging samples. If the sample plate is mounted only with grids, than a Z of 600 $\mu\text{m}$  is safe. If windows are involved, start with 800 $\mu\text{m}$ . To move this, select Coarse Z in one of the two

windows under Microscope Control Motor (bottom, just left of center). Type the desired Z value into the white window nearby and hit Go. The Current value should update. There is a camera which looks in from the top and shows the OSA and the sample plate, so you can see the relationship between the two. Do the same with Coarse X and Coarse Y to put the sample near the coordinates where you expect to find your features. Push the Reset Interfer. button. This is needed whenever a large move is done or when the sample has been far back. This action reconciles the Fine and Coarse motions. Now set up a coarse image scan. first, select the energy at which you want to do the scan. Typically, 390eV works because the flux is high there. Under Beamline Control, next to the Energy (eV) window, type the desired energy into the Destination window and hit Go. Next, set up to do a Sample Scan. Under Scan Controls, select Sample Scan and hit Start. A window will appear with parameters, such as the center position, range and pixel size, of the scan you're about to do.

To begin the process of finding the relevant area on the sample, start with a 200mm survey scan. The parameters for this have been pre-set in a scan definition which may be loaded by pushing the Load Scan Definition... button, then navigating to C:\STXM\_dev\StxmDir\Scan Configurations\Useful\200um\_survey.ims:

Image Scan - Estimated Time: 84s

Scan Type: Image (Line - unidirection)

Stage: Automatic

Dichroism: ☐ 2 En ☐ Pol ☐ EPU Offset ☐ 2 Reg

Defocus: Focus (nm) 30.5 Auto Defocus ☐

Accel. Dist. (µm) 60.452

Point Delay (ms) 1

Auto Parameters ☒

Line Delay (ms) 120

Energy point wait time EPWT (ms) 200

Wait Time Between Regions 500

Additional Wait Time 0

WT=EPWT+dE \* AWT

Scripted Scan ☐ TCP\_HOLDER auto ☐ FineX ☐ FineY ☐

Remote Scan Logging ☐

Sample/Scan Annotation (saved in .hdr file) MotorX MotorY

FPGA

Energies: Current Energy 345 Total Points 1 Single Energy ☒ Regions 1 Same dwell ☒ Dwell Time (ms) 3

Spatial Regions: ☐ Multiple Regions Regions 1 ☒ auto shutter ☒ UseGate

	Centre Pos (µm)	Range (µm)	# Points	Step (µm)
X	7786.0000	200.0000	100	2.0000
Y	6208.0000	200.0000	100	2.0000

Begin Scan Cancel Save Scan Definition... Load Scan Definition... Load Energy Definition... Recorded Channels... -1 Check Velocity Move To First Energy Point Move To First Position Record Position ☐ Change Magn Field ☐ Energy Move Test

There are a number of parameters which will be explained later. For now, the things to watch for are the Centre Pos (µm), and making sure the other parameters are as shown above.

Two ‘gotchas’: *you need to manually type in the coordinates you want for the Centre Pos*; the system does not automatically assume you want to center a scan where the microscope is parked, and the Scan Type (upper left), which should be as shown before loading the scan definition. The Centre Pos is very important because letting it have the wrong value could result in the sample moving thousands of microns while close to the OSA. Also, if you’re not paying attention, you could get seriously lost and not understand what you’re seeing – or not seeing. For navigation purposes it’s more efficient to uncheck the Use Gate box. When checked, this box causes the system to detect when there was a top-off injection and restart the line. Unchecked, it lets those go by, resulting in little black lines on your image. You’re not yet at the stage of producing publication-worthy images, so black lines are preferable to time wasted on redoing lines.

If you think you’re good to go, hit the Begin Scan button. Otherwise hit Cancel. Before pushing the red button, make sure the covers are on the ports, the PMT is on and the shutter open. Take one more look at the Centre Pos coordinates. Also, make sure there are no red lights like these on Motor Status areas at the lower right of the main screen:

Monochrom...	■	-3574.91	■	■
EntranceSlit	■	117.17	■	■
ExitSlitY	■	100.55	■	■
ExitSlitX	■	6.27	■	■
FineX	■	5222.29	■	■
FineY	■	4453.72	■	■
CoarseX	■	25.24	■	■
CoarseY	■	8404.62	■	■
CoarseZ	■	45775.01	■	■
ZonePlateZ	■	-6490.04	■	■
OSAX	■	0.18	■	■
OSAY	■	0.92	■	■
DetectorX	■	-350.29	■	■
DetectorY	■	72.19	■	■
DetectorZ	■	500	■	■

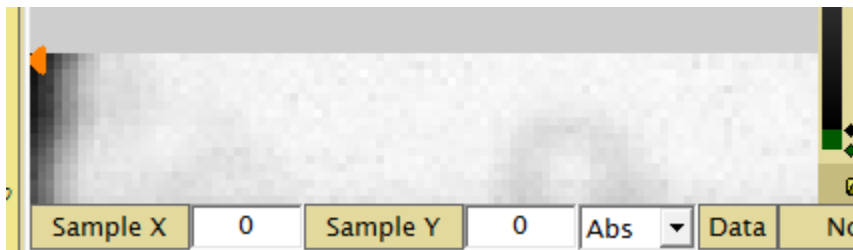
Such lights indicate a problem with the motion controller. Dealing with those is usually routine but outside the scope of this manual. To fix this:

1. Select the problem motor from one of the drop-down lists under the heading ‘Microscope Control Motor’ near the bottom of the STXM-UI screen.
2. Push the ‘S’ button. A long, narrow window will appear with the name of the motor. Make sure it’s the right motor.

3. If the problem motor is Coarse X or Coarse Y, back out the Coarse Z (higher numbers) so that it's safe for the sample to move.
4. Push the 'Home' button in the narrow window. The motor will move, which you will see from the numbers in the position display. Wait for it to stop moving.
5. If the relevant motor was Coarse X or Y, move it to where it was and do a Reset Interferometer, then move Coarse Z where it was. Relocate and refocus.
6. If it was Coarse Y, you may have to recalibrate it because it could be way off. To do this,
  - A. Move Coarse X to a position at which there should be no windows. For an 8-hole holder, X=0 will do, and for a 6-hole, X=2500.
  - B. Move Coarse Y down (-) in steps of 100um until you get light. This would be the bottom of the sample plate, which should be at Y=-3200.
  - C. Recalibrate it. In the narrow window that hosts the 'Home' button, there's an entry box under the word 'Calibrate'. Type in -3200 and hit the Set Pos button.
7. If it was Zone Plate Z, enter the energy you want to be at in the Energy window, then push the Go button, even if you're already at that energy. This moves the Zone Plate Z back where it belongs. Refocus.

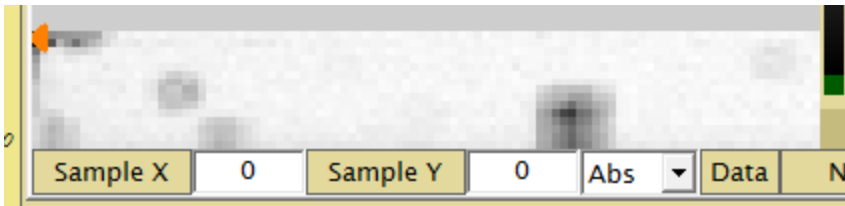
From here, I'll show a worked example of imaging. This will be done on a sample I've never seen before (provided by Jiaqi Li), so this is an authentic example of investigating a sample.

The image you get is likely to be very blurry. Particles may show up as vague smudges or low-contrast donuts and window edges or grid bars as fuzzy edges, something like this:

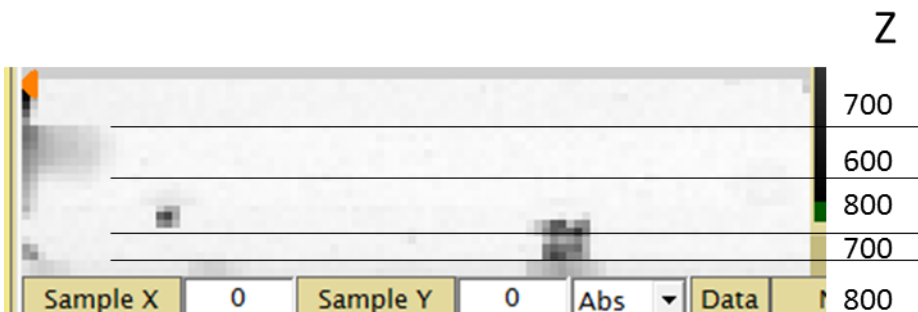


The scan always goes from bottom to top, unlike at 10.3.2. In this case, I didn't let it complete because there won't be anything useful to see. This is because we intentionally set up the sample out of focus so we could move it freely without fear of bumping into the OSA. The next thing to do is to focus it. There is a detailed manual on the theory and practice of focusing, so this manual won't dwell too long on it. For coarse focusing, we move Coarse Z in steps to get a sharp image, then zoom in and move by smaller steps. For this example I started at Coarse Z = 1000, then moved to 800µm:





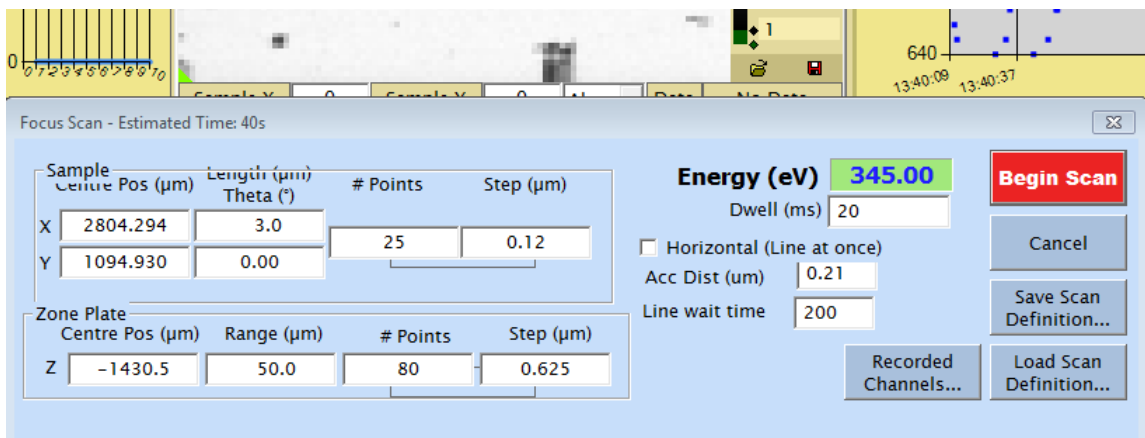
A useful trick at this stage: While a scan is going, you can move Coarse Z like this:



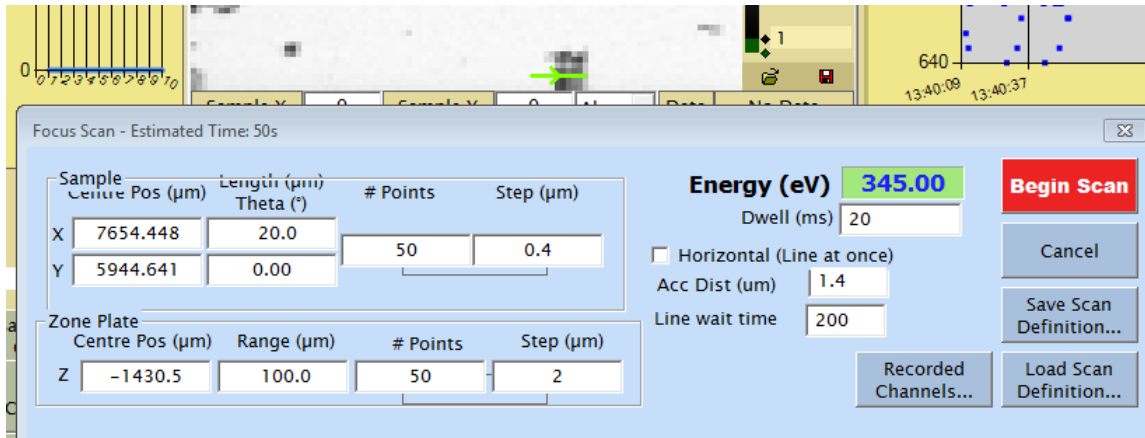
When you do that, the displayed “Current” position will not update. At this point, we’re close to focus, by the standards of a coarse survey scan ( $2\mu\text{m}$  pixels). This is good enough to make a navigation image, which I’ll save. Note the filename on top:



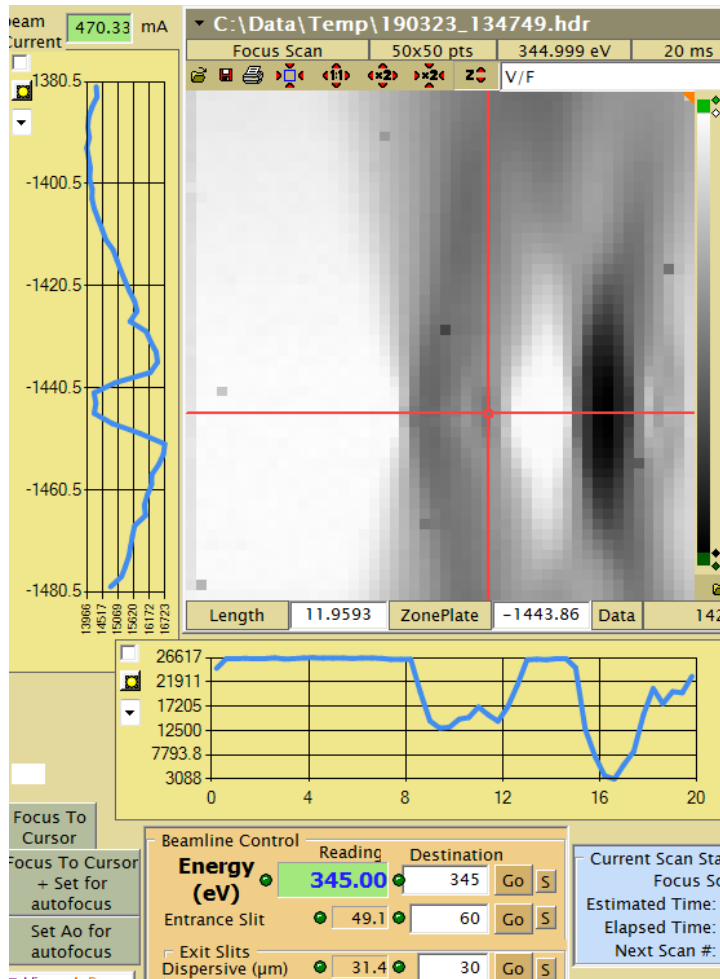
Let’s focus up more sharply. Selecting Focus Scan brings up this window:



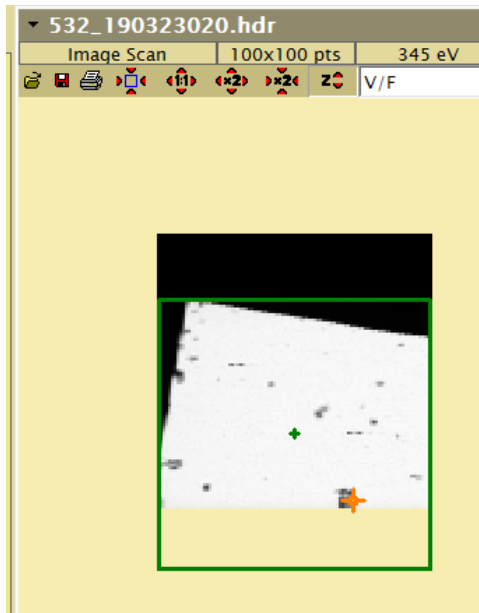
Note the little green triangle at the lower left of the image. This tells us that the selected starting point of the focus scan is off-screen. Also, we will want a longer scan that goes over a particle or an edge: Drag that triangle onto the imaged area:



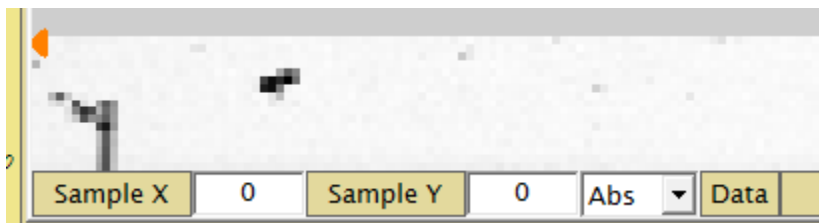
The scan will go over the line represented by the green arrow over and over again, moving the Zone Plate Z each time. We know that the Sample Z is within ~100µm of focus, so we make the Range 100µm. The Length is chosen to go over the desired edge. The result:



I've put the cursor where the interfaces seem the sharpest. An edge will show up as an hourglass with the waist at the correct focus. Having put the cursor where I think focus is, I have two choices, represented by the two lower buttons on the lower left. The middle one, which is most often used, puts the zone plate back to its normal auto-focus position and moves Coarse Z to put the sample in focus. The lower one changes the zone-plate auto-focus position, changing A0, so that the sample doesn't move. A0 is the system's idea of how far back from focus the OSA is, or put another way, how far the sample surface is from the OSA at focus. There is an energy-dependent maximum value which can get read off by pushing the 'S' button next to the Focus mode selector. Do the middle button (Focus to Cursor + Set for autofocus). Let's scan the navigation image again, but this time, move the center so as not to leave so much black space at the top. Pull up the previous image (file-open icon) and select Sample Scan. The previous scan parameters reappear. Also, on the image, a green square with a + in the middle shows the coverage area of the scan. Grab the + and move it. I hit the x2 zoom-out button (second from the right in the row below the filename) to get an overall view:

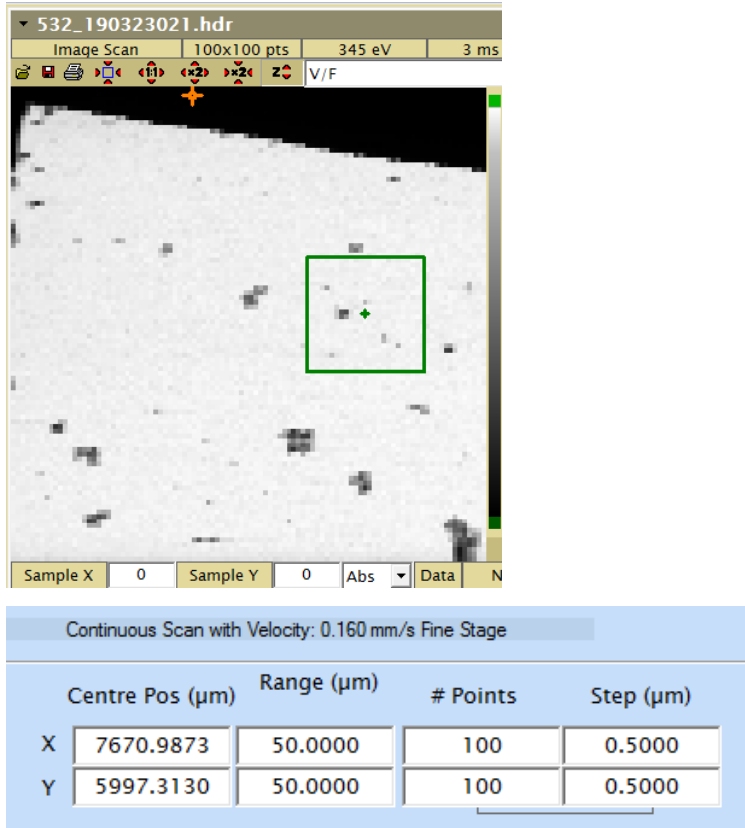


The scan doesn't have to be square; you can grab any edge of the box and pull it. The opposite edge will move in sync to keep the center fixed. The orange + is the system's idea of where the beam is now. I'll hit Move to First Position and Begin Scan. Whoops, a problem:

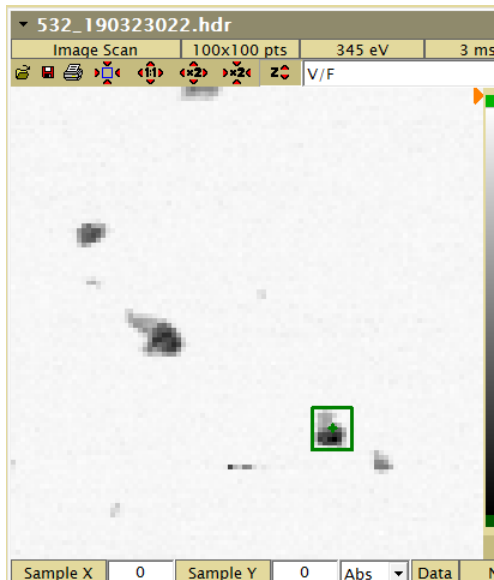


Notice how the image is vertically stretched at the bottom. This is because the fine Y stage got out of its scan range. Do Move to First Position and hit Reset Interf. before restarting the scan. Here's our new navigation image. Notice that you can see more particles, partly because the grayscale was adjusted, but more because it's more or less in focus. Also, the position has shifted tens of microns relative to the previous image. This is because of having hit Reset Interf. We want to find an area with small particles. Anything we see as dark in this image is going to be too thick to see through and get spectral data from. Thus, zoom in, starting at 50 $\mu$ m.





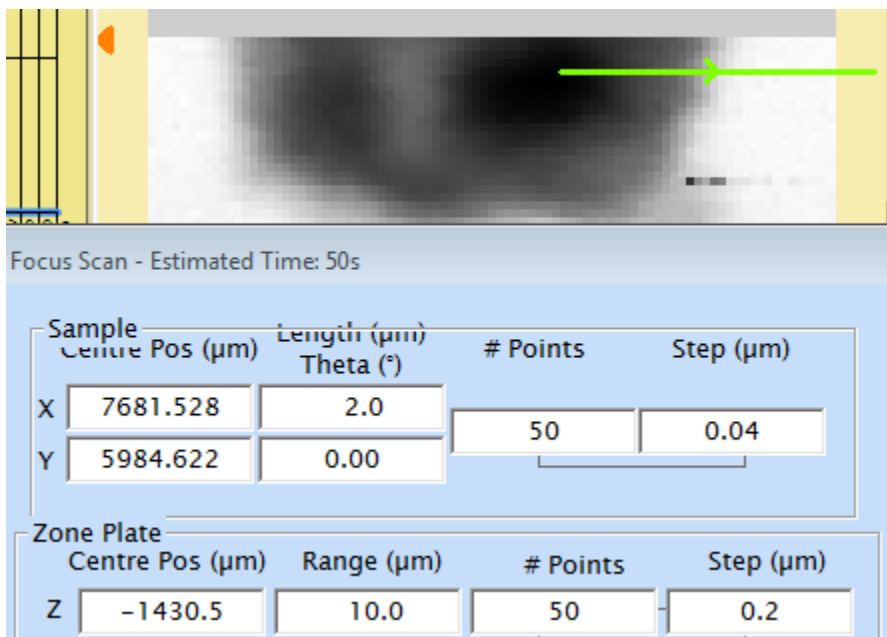
When we typed in the size, the step (pixel size) adjusted automatically to keep the same number of pixels. The selected type of scan is an Image (Line – Unidirectional), not to be confused with a Linescan. This means that the X stage scans continuously, with a speed given by the Velocity reported above the window where the scan range is shown. This has to be <2mm/sec and should be >0.01mm/sec. If it's too slow, go to Point-by-point mode. Also, use point-by-point mode for best resolution as there seems to be a tiny bit of line-to-line shifting in Line mode. OK, we have a couple of promising areas, mainly the gray blobs off the black ones plus the tiny gray ones.



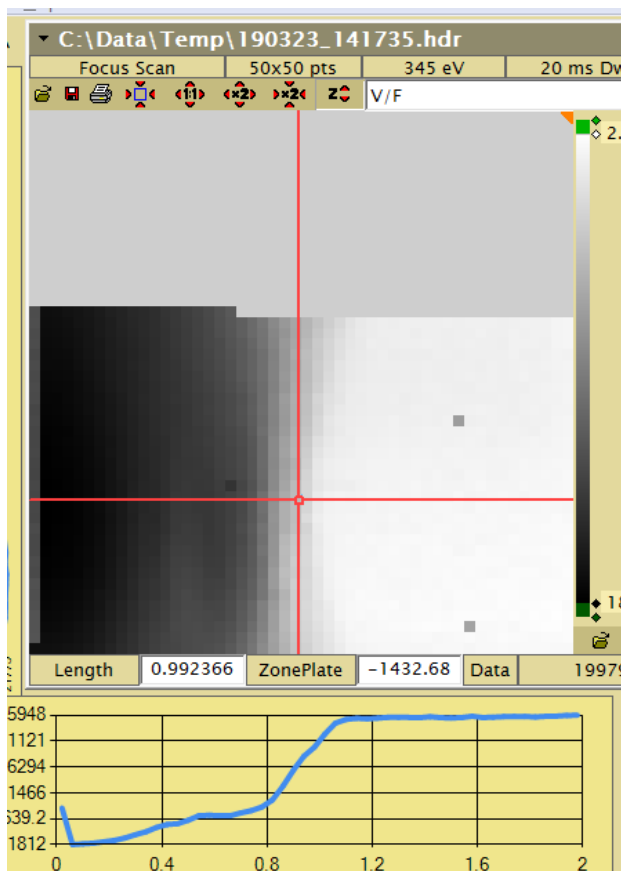
Continuous Scan with Velocity: 0.016 mm/s Fine Stage			
	Centre Pos ( $\mu\text{m}$ )	Range ( $\mu\text{m}$ )	# Points
X	7680.0840	4.3670	87
Y	5986.0535	5.0000	100
			Step ( $\mu\text{m}$ )
			0.0502
			0.0500

The little black line about 1/5 of the way up is an ALS fill. Let's zoom in some more on a promising object. The chosen object is marked by the green square. The pixel size has been adjusted to 50x50nm.

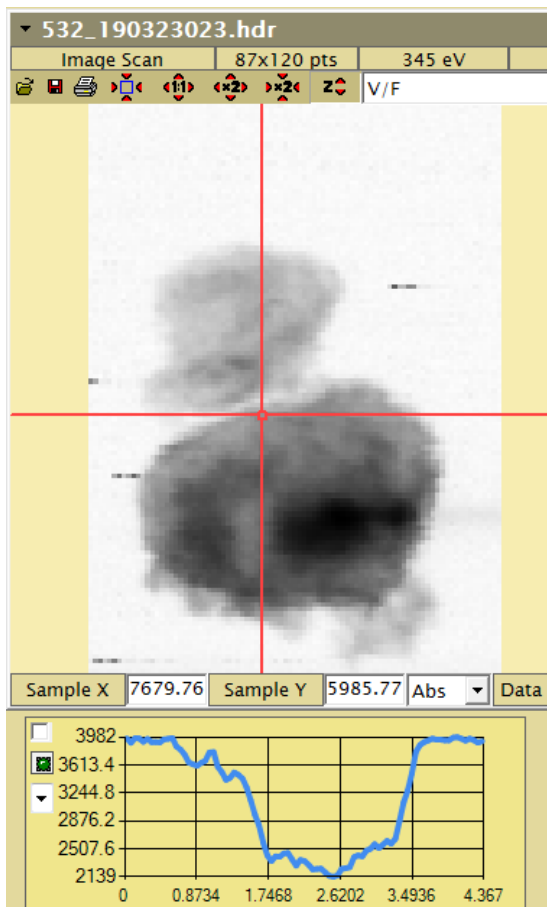
It's obviously out of focus – stop!



The focusing we did at 200 $\mu$ m was too rough for 5 $\mu$ m scan range, so refocus with a shorter scan and shorter range for the zone plate..



Notice that the profile isn't perfectly sharp. If you have a particle that's semi-transparent, the edge won't appear perfectly sharp. I put the cursor where there seemed to be symmetrical broadening above and below. You can use the profile graph (underneath) to look at different positions and decide where's the sharpest. Upon re-scanning, detail started to show up and it was clear that the bottom of the scan cut off some of the interesting stuff. Therefore, restarted with the Y range at 6 microns, still with 50nm pixel size. At this pixel size, it's important to make sure the roughing pump is turned off so that its vibrations don't jiggle the microscope.

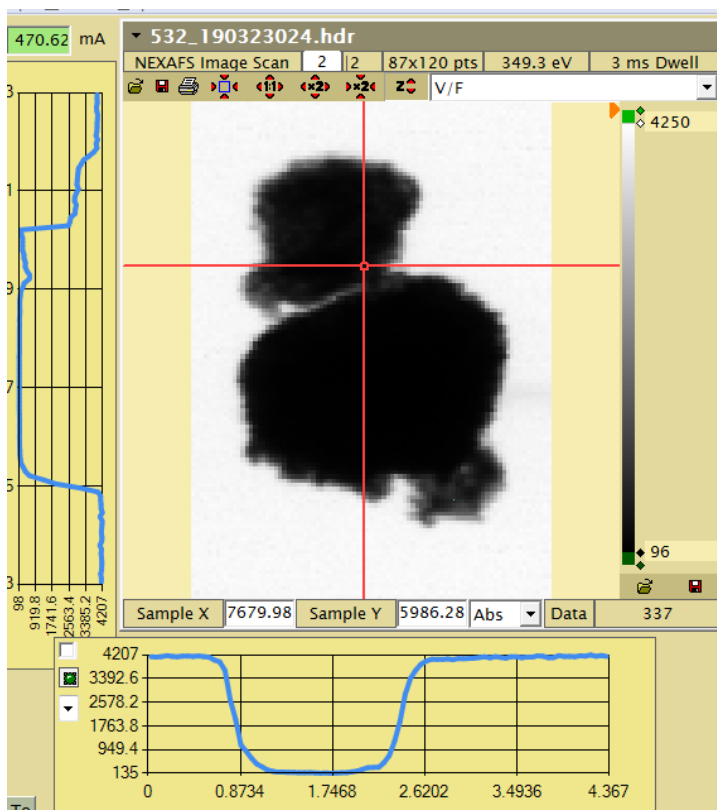


This looks like there's some interesting detail. The material is cementitious material (sample: Jiaqi Li) and the project involves spectromicroscopy at the Ca and O edges. Thus, we want to ask if there's any Ca here before embarking on a long stack. To do that, we do a "map", which is a stack containing two energies, one below and one above the edge of interest. To do a stack, uncheck the Single Energy checkbox and fill in this form:

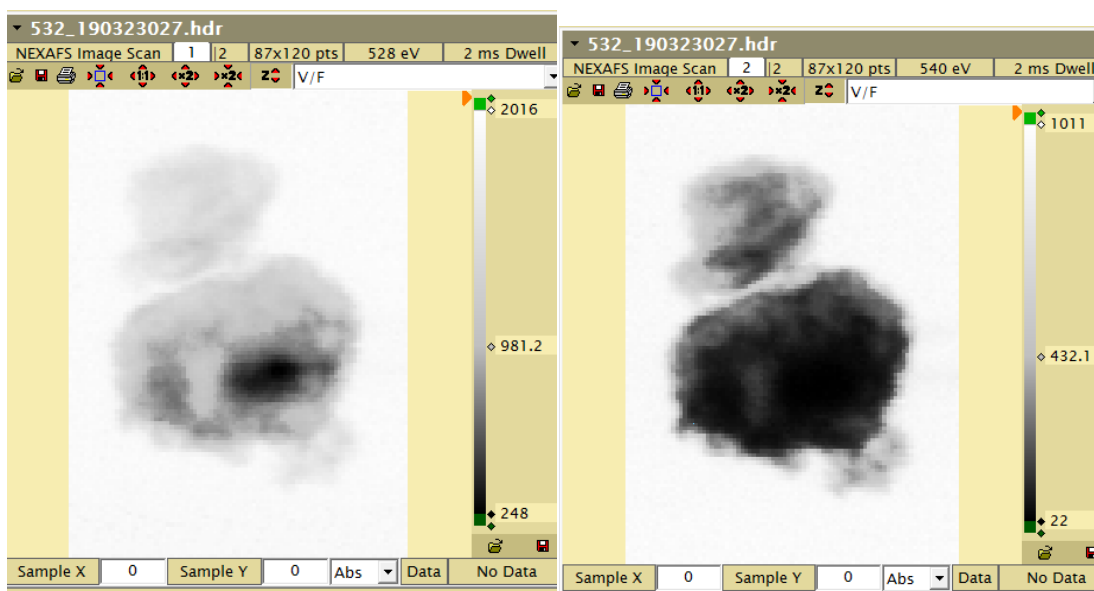
Region	Start Energy (eV)	End Energy (eV)	Range (eV)	# Points	Step (eV)	Dwell Time (ms)
1	345	349.3	4.3	2	4.3	3

You can also load in an energy definition from a saved scan definition. I've taken the opportunity to recenter the scan area and check 'Use Gate'. checking Auto Shutter ensures that the sample doesn't get exposed during the retrace. This option doesn't work in Point-by-Point mode. The post-edge image shows that there's certainly Ca there, when compared with the pre-edge image above. Too much Ca, in fact; there's no transmission. Spectroscopy on this area at the Ca edge will fail, though it may work at O.





Since the user wanted O data, and since O is at a higher energy, hence likely to transmit better than Ca, let's try an O map. This shows that there is transmission in spots, but the image is a bit blurry. Not surprising; the focus can shift slightly with energy despite best efforts at adjusting the tracking.

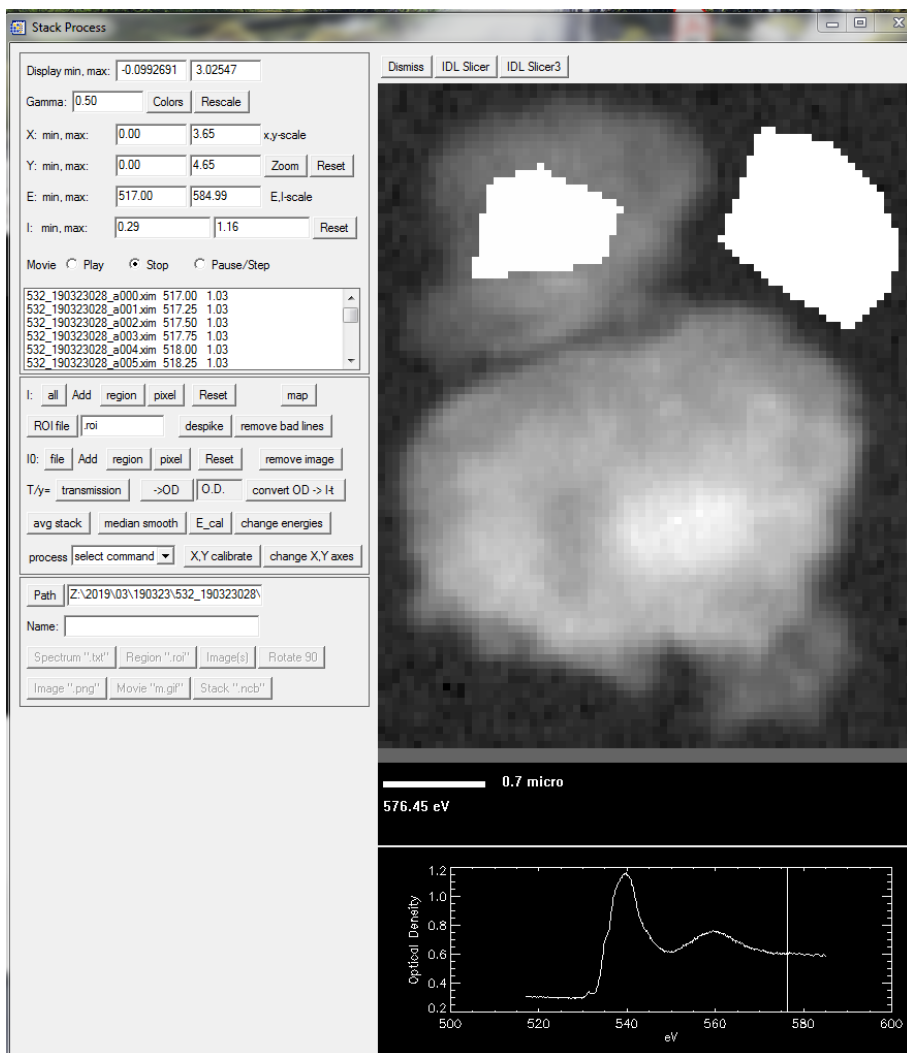


Look at the grayscale bars to the right of each image. I've added a marker, which defines a gray-level which maps into a specific color. The color chosen is medium gray, which gives an

approximation to a display gamma adjustment by dragging the marker up or down. Some users like to play with multiple markers in assorted colors to get some interesting color effects. This is promising enough to want to do a full stack. Import somebody's O stack energy definition that looks about right:

Region	Start Energy (eV)	End Energy (eV)	Range (eV)	# Points	Step (eV)	Dwell Time (ms)
1	517	524	7	29	0.25	1.027
2	524.1	577	52.9	354	0.15	1.027
3	577.25	585	7.75	23	0.352	1.027

and go. This will take 4 hours...Done! Here's a region spectrum and OD image from Axis2000. The white region on the right is the  $I_0$  region, and the one on the left is the set of pixels whose averaged spectrum is plotted below.

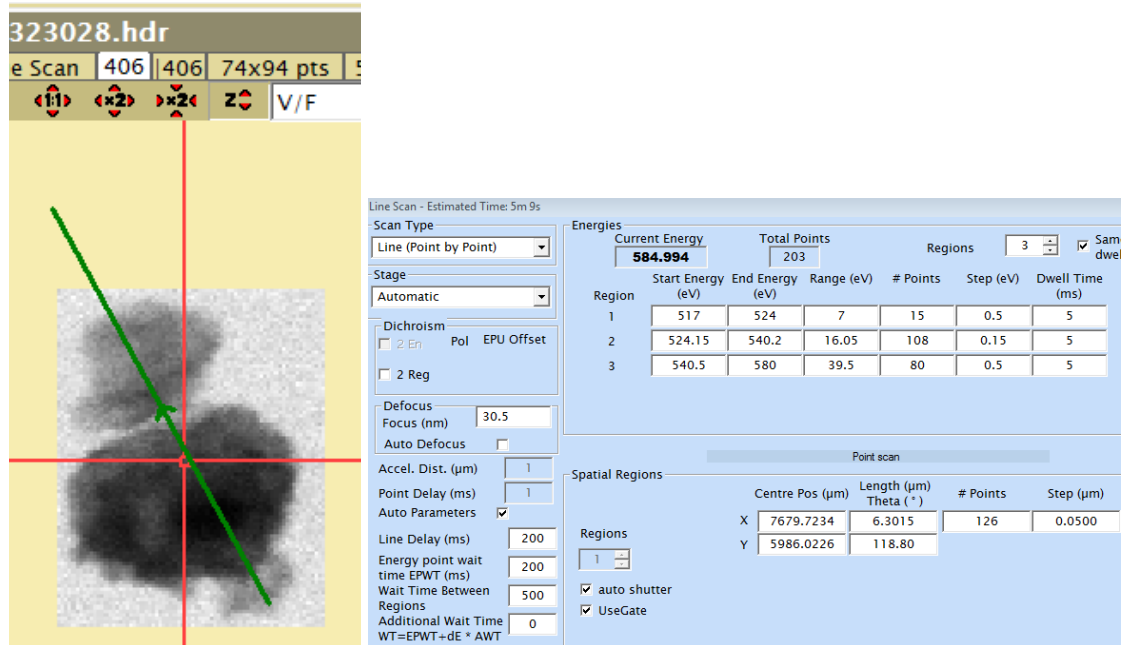


We see that the spectrum from 540eV on is tabulated more finely than it needs to be. Therefore, to prepare for the next O stack, let's edit the scan so that it doesn't take as long:

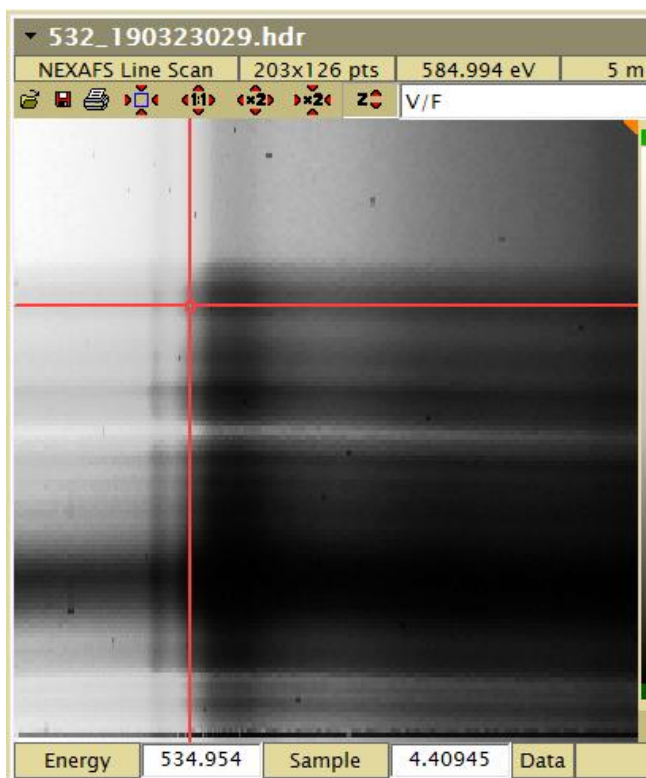
Region	Start Energy (eV)	End Energy (eV)	Range (eV)	# Points	Step (eV)	Dwell Time (ms)
1	517	524	7	15	0.5	1.027
2	524.15	540.2	16.05	108	0.15	1.027
3	540.5	580	39.5	80	0.5	1.027

A word about scan files: They have extension `.ims` and live in folders under `C:\STXM_dev\StxmDir\Scan Configurations`. They are simple ASCII and the computer has been set to open these with a text editor. It can be handy to look at a scan file before selecting it.

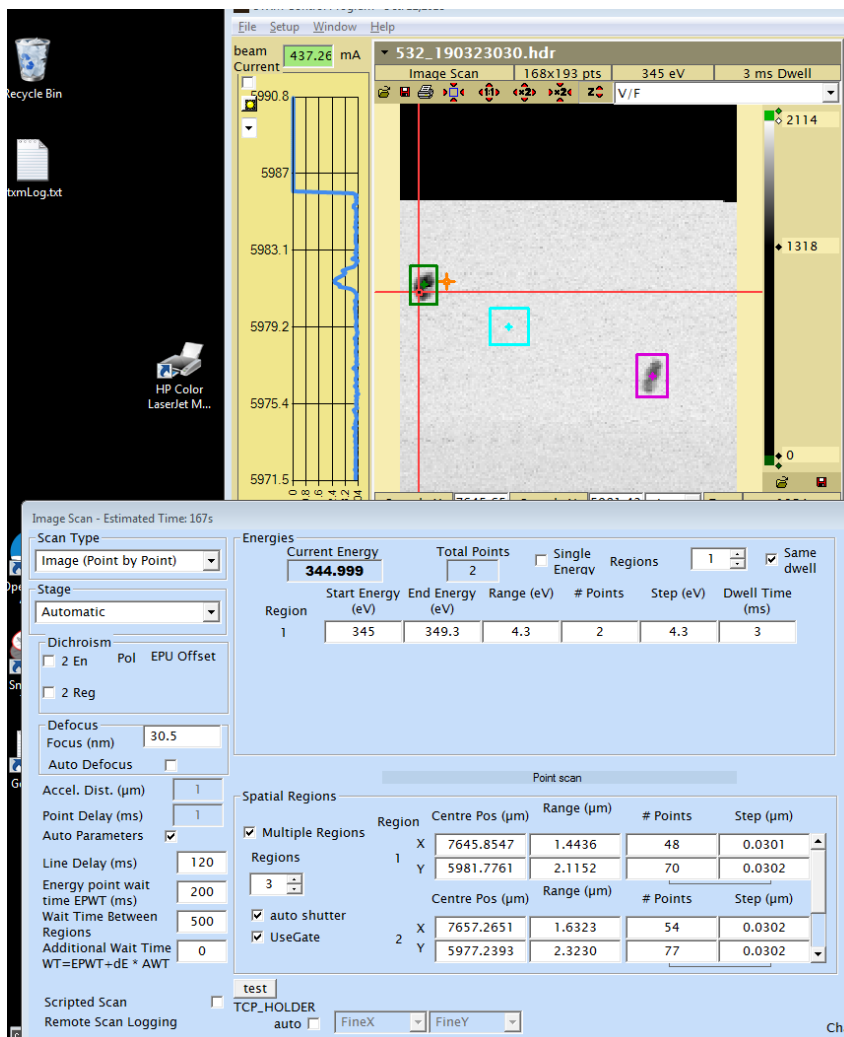
Another way to save some time is to do a line scan. This is like a focus scan except that it's energy that's varied, not zone-plate position. This only works if the sample is really stable so the scan line won't drift off the feature of interest. To illustrate the point, let's do a linescan on this same region, covering both lobes



A caveat: When you switch modes (Point scan, Line, Image), you lose the energy regions and have to re-load or re-type the values. This is the energy definition we just created. The scan line is chosen so that it includes an  $I_0$  region (at top) and all three distinct-looking regions of the particle. You can do a non-horizontal line in and only in Point by Point mode. You can specify the line by click-dragging each end. The same is true for focus scans. One difference between linescans and focus scans is that in a focus scan, the stack variable (zone-plate Z) is plotted on the Y-axis, while in linescans, energy is in X and distance along the green line is in Y. Here's the result, which took 8.5 minutes rather than 4 hours. A point to be aware of: the time estimation doesn't take into account the time required for the monochromator to move. This gets to be significant for linescans because one image's-worth of pixels requires moving the monochromator many times. Thus, this image was estimated at 5 minutes.



OK, we're done with the particle we've been working on for the last several hours. Now, referring back to the image on pg. 22 (file 022) we will work on the two particles which are barely visible to the left and down from center. Take an image around these, focus up at 345eV (Ca pre-edge) and now do Ca maps on both. There is now the opportunity to demonstrate the Regions feature. With this, one can define an image scan which does several areas in one stack. It takes the image of Region 1 at the first energy, then Region 2, ..., then goes to the second energy and does the Regions in order again, and so on. This feature is handy when there are separated areas of interest and one doesn't want to waste time scanning the boring material in between. It's also handy when the nearest blank area ( $I_0$ ) is relatively far away from the scanned area; one can define a region just for  $I_0$ .



The Regions feature is activated by checking the Multiple Regions checkbox. Here, we have three regions, shown as the green (1), magenta (2) and cyan (3) boxes. The coordinates for Region 3 don't show in this view; the vertical scrollbar would bring them in. Since Region 3 is just for  $I_0$ , the pixel size has been set to 100nm so that we don't spend a huge amount of time scanning it. The quick map scan (under three minutes) shows that the particles are a bit thick ( $OD \sim 2$ ) but doable, at least at the thin spots. Load in an energy definition for a Ca stack, adjust the centers of the scan regions, and press Begin Scan for a two-hour stack. A warning about Regions – they need to be within the scan range of the fine stage ( $\sim 200\mu m$ ) of each other.

On this particular sample, the next operation is to do an O stack on the two particles seen above to see if they're the same as the bigger one with the two lobes looked at earlier.

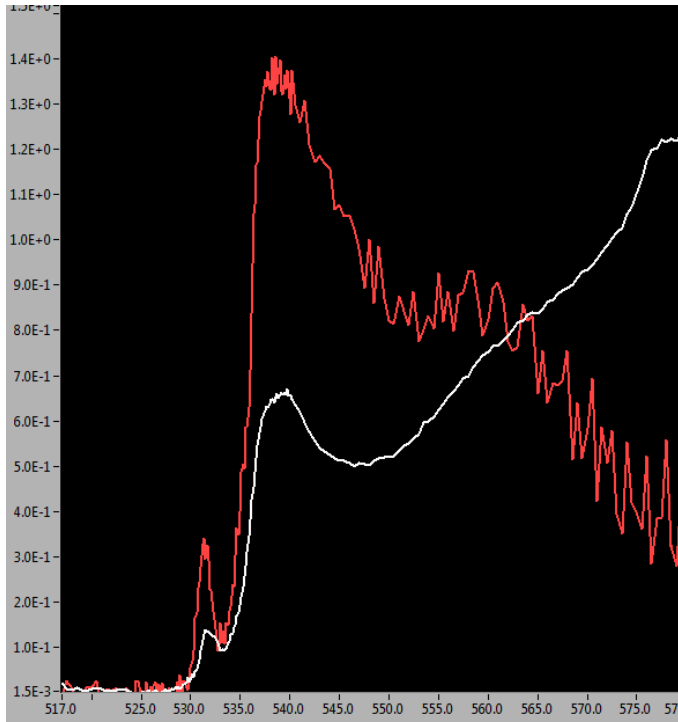
## VI. Afterward

To vent the chamber and remove the sample, follow the steps in the checklist:



1. Move Coarse Z to 9000
2. Turn off PMT
3. Close VVR207 and photon shutter
4. Loosen door-closing bolt and top-lid knob
5. Close roughing valve
6. With fill gas flowing through pressure-relief valve, open the vent valve until the box is vented.
7. Reach in with a gloved hand, unclip the wire from the top of the sample and pull it straight up.

At some point, you'll need to look at your data. One of the computers outside the hutch has Axis2000 and Mantis and has the data folder as a network-mapped drive. If you're keeping a running Word doc or PowerPoint, you can use the Snipping Tool to copy screenshots. On the STXM computer there are Desktop shortcuts leading to programs I wrote for analyzing images and linescans. Many users like these better than Axis. There are also some programs for the manipulation of 2-column data such as point spectra. When you take a point scan, you get a .xsp file which is simple 2-column data you could plot in Excel. We have Genplot (wants to be Origin when it grows up, which it won't) and a couple of executable LabVIEW programs from the Beamline 10.3.2 suite. Among these, Plot-Add-Multiply is handy for plotting spectra together. It also does arithmetic on data, interpolating the abscissa as needed, hence its name. 2-Column Editor is used for doing things like taking the  $\ln()$  of a spectrum or subtracting a linear background. This is an example of the use of both of these programs:



The white curve is the  $I_0$  from a linescan at the O edge, after taking the  $\ln()$  and subtracting a linear pre-edge. The red is the OD of a particle, again with pre-edge subtraction. Both spectra were extracted from the linescan by Axis2000, written out as .xas files, which are 2-column ASCII with headers, and processed further in 2-Column editor. That exercise was done before the Linescan Reader program was written, which would have done the same operation more easily.

## VII. Less-routine operations and issues

### A. *Focus parameters (relevant when changing edges or if sample doesn't come to focus)*

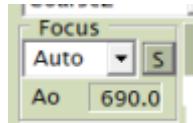
The focal length of the zone plate is proportional to the energy, with a proportionality constant denoted A1 (units, microns/eV). The nominal distance between the zone plate and sample is denoted A0. If this is set too big, the OSA will block out the rays from the outer zones of the zone plate, leading to a loss of flux and resolution. If too small, the sample will need to be unnecessarily close to the OSA, and the OSA may not block all the stray light, such as second harmonic. This condition usually happens at higher energies, where second harmonic is not an issue. The position of the zone plate (ZonePlate Z) is given by

$$\text{ZonePlateZ} = A1 * E + A0$$

and the focal length by

$$FL = |A1| * E.$$

The zone plate must move upstream of the OSA, so A1 is negative, since the convention is that  $-Z$  is upstream. These parameters are accessible through the 'S' menu on the 'focus' bank of



controls near the bottom: . Pushing this button yields this screen:

**Focus & Zone Plates Parameters**

**Focal Length Function**

A1: -4.7803    A0: 315

$f(E) = -4.78E + 315$

A0 is OSA to SAMPLE distance (μm)

**Calculator**

Energy (eV): 320

Expected positions for current energy

Focal Length (μm): 1529.7

Sample Z (add sample thickness) (μm): 315

Zone Plate Z (μm): -1214.7

**Zone Plates**

	Diameter	Central stop	Outer zone	A1
<input type="radio"/> #1	240	95	25	-4.7814
<input type="radio"/> #2	250	90	25	-5.0814
<input type="radio"/> #3	250	50	25	-10.1628
<input type="radio"/> #4	240	95	60	-11.7692
<input checked="" type="radio"/> #5	240	90	25	-4.7803

**OSA**

	Diameter
<input checked="" type="radio"/> #1	50
<input type="radio"/> #2	40
<input type="radio"/> #3	70

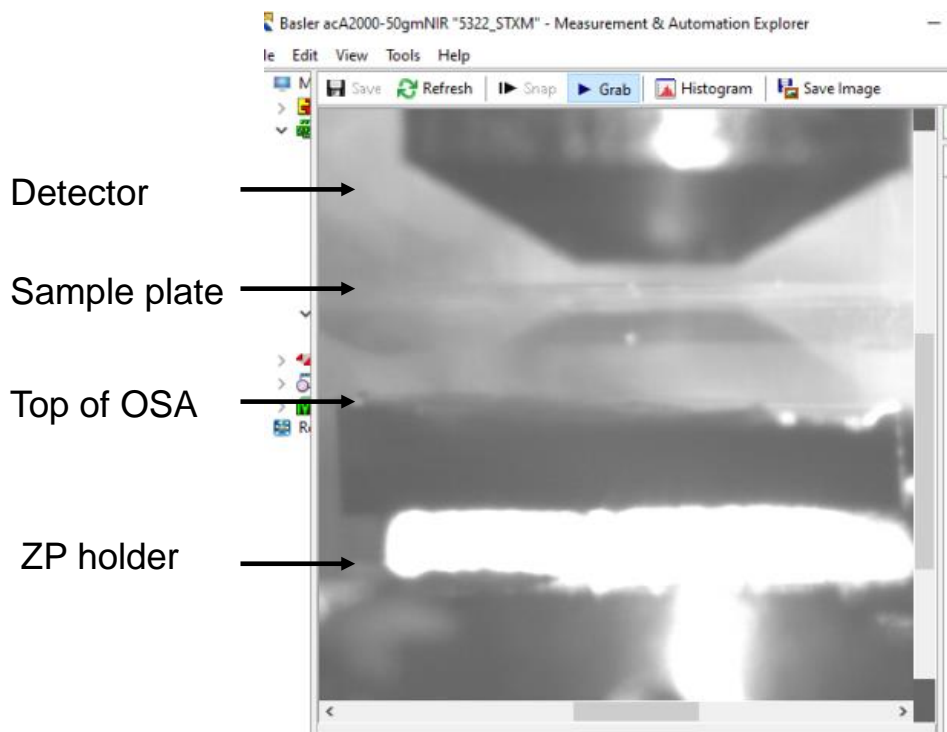
Max Ao for current energy estimation: Ao < 303

OK

The 'proper' A0 value is shown in the small box above and just to the right of the OK button.

What does this have to do with operations? If you change edges, then you will either want to set A0 to the value appropriate for the lowest energy *or* change it when changing energies. If you do the former, the sample will remain in focus. If you increase A0 by 100μm, then you have to move Coarse Z by +100μm to stay in focus. Most people do the former as it's much easier.

There is a circumstance in which you will need to operate at an A0 larger than what the Focus and Zone Plates Parameters screen calls for. This is when, for whatever reason, the sample bumps the OSA at larger Z than where focus would be. A symptom of this is that as you decrease Coarse Z, the image suddenly gets dimmer. You can also see the top of the OSA in the top-view camera:



If the sample (out of focus in this image) bumps the OSA, you can often see it move. If it didn't bump too much, it will spring back when you relieve the pressure by moving Z in the positive direction.

The bottom button of the three you see after doing a focus scan (Set A0 for autofocus) sets A0 to the appropriate amount for where Z is.

#### B. *Energy calibration*

The monochromator includes a grating which images the entrance slit onto the exit slit in the horizontal section. Tuning the energy is done by rotating the grating without changing the angle between the line joining entrance slit and grating and the one between grating and exit slit. This angle is called the included angle. Normally, the grating diffracts in first order to put beam onto the exit slit. It is possible to move the grating so that it reflects specularly (0 order), which is tantamount to asking for an infinite energy. There are two parameters which need to be calibrated; the system's idea of the included angle, and its idea of where the zero order is. In principle, both of these should be stable. In practice, there's a bit of drift. The BLS will normally do this calibration at the beginning of a run, but I will describe how to do it in case it needs to be done by the user.

If you're only doing low energies, say up to N, you can get away with a 1-point calibration in which you assume that the grating zero is correct and use a known feature to reset the system's idea of included angle. The procedure for calibrating the 0 order is complex and a

bit risky, so I won't include it here. Instead, for wide energy ranges, one could use a known standard for the edge of interest and measure the energy error and use that to correct the data in processing.

The calibrant for the 1-point procedure is CO<sub>2</sub> gas, which has sharp features at 292.74eV, 292.96eV and some other places. The procedure is as follows:

1. Remove the sample, unless you're sure it won't be affected by CO<sub>2</sub> exposure. If you leave it in, move CoarseY to -4500 to pull the sample up and out of the way. Move Coarse Z to 1000 or less.
2. Pump out the box and backfill with 6T of CO<sub>2</sub>. This is done just as with He, except using the line labeled CO<sub>2</sub>. The aneroid gauge isn't sensitive enough to read this pressure, so you have to use the GP gauge.
3. Set the entrance and exit slits to 7,5,10 um. This will reduce the flux a lot but give you high energy resolution.
4. In Sample Scan, select the Point scan mode. Load in a C calibration scan-def such as Useful\c\_calib. Check the box that says 'Use current beam position'.
5. Run the scan. This should plot out in the chart area on the upper right. You should see dips. The first prominent one should be at or near 292.74eV. If it's within 0.02eV of 292.74eV, you're done.
6. Use the crosshair to read the energy of that feature and move to that energy.
7. In the menus on the upper left of the STXM-UI screen, choose Setup->Control and ACQ devices. This pops up a screen:

Control and ACQ Devices

NI Devices  
 NI device # for multi IO card: 1  
 NI device # for PCI 6602 counters: 2  
 NI device # for DAC card: 0

Agilent Interferometer Controllers  

	X Axis	Y Axis	Z Axis
Address	576	608	0
Name	pmcX	pmcY	pmcZ

Position Update time [ms]: 1000

Piezo stage parameters  

	X Axis	Y Axis
Maximum range (µm)	200	200
Scan range (µm)	160	160
Volt to micron conversion (µm/V)	20	20

 Compensation number: 1  
 Comp # = 1.0

Angle scanning settings  
 Z piezo stage angle scan ON/OFF: ☐  
 Z stage angle voltage: 0  
 Z stage angle voltage offset: 0  
 Voltage to Angle coefficient: 0  
 Scan Angle (deg): 0  
 Angle = atan(coeff\*(Volt+Offset))  
 Coarse X stage orientation: 30 deg Normal

ESP Controller #2 (beamline)  
 Controller Address: 2  
 Buttons: ESP Write, ESP Write/Read, ESP Read, Check Errors

ESP Controller #3  
 Controller Address: 3  
 Buttons: ESP Write, ESP Write/Read, ESP Read, Check Errors

SGM Monochromator parameters  
 Monochromator Arm: 466900  
 Groove Density: 300  
 Included Angle: 172.8675  
 Angle Offset: 0

Sample Exclusion Zone  
 Sample X Min: -8000  
 Sample Y Max: 5500  
 Sample will not access position < Xmin AND > Ymax

UPDATE

If the energy at which the dip was found is less than the correct energy (292.74eV in this case) increase the value in the **Included Angle** box in steps of 0.001 or less and hit the **Update** button until the energy shown in the indicator labeled Energy (eV) in bold comes to the correct value. If the indicated energy is too high, decrease the included angle. Don't touch anything else. Close this window.

8. At this point, you should be done, but it wouldn't hurt to take another scan, for the record.

If you want to calibrate at the O edge, you can use CO<sub>2</sub> as well, but the energy for the peak is given different values by different authors, for instance 535.3eV (Yang, B. X., J. Kirz, and T. K. Sham. "Oxygen K-edge absorption spectra of O<sub>2</sub>, CO and CO<sub>2</sub>." *Physics Letters A* 110, no. 6 (1985): 301-304; McMaster gas-phase database <http://unicorn.mcmaster.ca/corex/name-list.html>), 535.0 (Sham, T. K., B. X. Yang, J. Kirz, and J. S. Tse. "K-edge near-edge x-ray-absorption fine structure of oxygen-and carbon-containing molecules in the gas phase." *Physical Review A* 40, no. 2 (1989): 652). Note that the peak is pretty broad, and to locate it accurately at 6T fill pressure, you need to take a blank spectrum with no CO<sub>2</sub> and divide. To do this division, recall that the \*\_0.xsp file is the spectrum in 2-column ASCII, so you can read it into any tool that handles this kind of data, such as **Genplot** (shortcut on Desktop) or **Plot-add-multiply**. If you don't do the division, the spectrum will be distorted because the system has O-bearing contamination.

#### C. *Manganese*

The Mn L-edge is a special case. The efficiency of the monochromator grating in first order has a deep dip at 620eV, which makes it impossible to get good data at the Mn L-edge unless a trick is used. The trick is to set up the zone plate to focus the second-order light from the grating. To do this, go into the Focus and Zone Plates Parameters screen:



Focus & Zone Plates Parameters

Focal Length Function

A1: -9.5606    A0: 315

$f(E) = -9.561E + 315$

A0 is OSA to SAMPLE distance ( $\mu\text{m}$ )

Calculator

Energy (eV): 320

Expected positions for current energy

Focal Length ( $\mu\text{m}$ ): 3059.4

Sample Z (add sample thickness) ( $\mu\text{m}$ ): 315

Zone Plate Z ( $\mu\text{m}$ ): -2744.4

Zone Plates

	Diameter	Central stop	Outer zone	A1
<input type="radio"/> # 1	240	95	25	-4.7814
<input type="radio"/> # 2	250	90	25	-5.0814
<input type="radio"/> # 3	250	50	25	-10.1628
<input checked="" type="radio"/> # 4	240	90	25	-9.5606
<input type="radio"/> # 5	240	90	25	-4.7803

OSA

	Diameter
<input checked="" type="radio"/> #1	50
<input type="radio"/> #2	40
<input type="radio"/> #3	70

Max Ao for current energy estimation: Ao < 606

OK

and see if there's a choice for which A1 is twice the current value. In the above example, choice #4 (selected) is a match for #5, current set. If there is no match, pick one of the parameter sets (say, #3) and copy the parameters from the current set (#5) except for A1, for which you should put the doubled value. Then, without clicking a radio button, hit the OK to make it 'take'. Once that's done, you can go back in and select either the normal, first-harmonic parameters (#5 in our example) or the second-harmonic set (#4). Then, for any energy, enter half the value you want into the scan definitions and the mono destination window. Thus, if you want 640eV, enter 320eV. A Mn map can be done between 318eV and 321eV, which is really 636eV and 642eV. There are a couple of 2<sup>nd</sup>-harmonic scan-defs in the Useful folder.