

# ALS beamline 11.0.1.2 – Resonant Soft X-ray Scattering (15 Jun 2021)

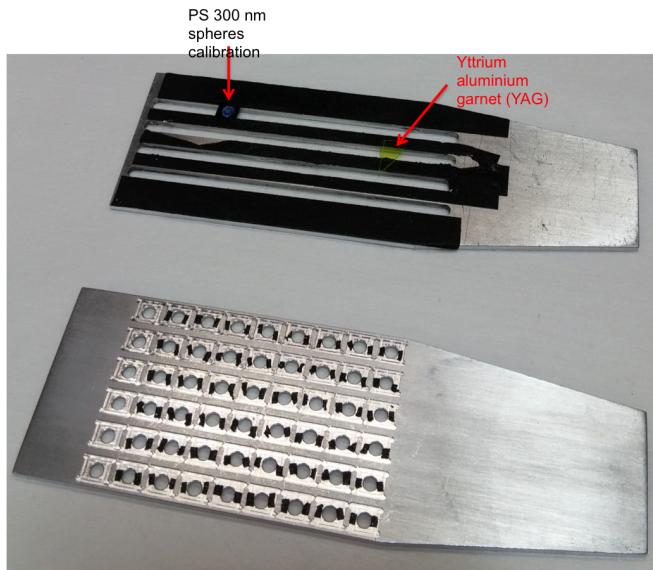
## Table of Contents

<b>Loading and Removing Samples</b>	<b>2</b>
Mounting samples onto sample holder	2
Loading sample holder and pumping down the chamber	2
Venting the chamber	3
<b>Sample and Detector Geometry</b>	<b>4</b>
Photodiode vs. CCD	4
Finding the beam spot	4
Collecting scattering data	5
Aligning beam stop to direct beam	5
Overview of Options in CCD Scan	6
Grazing Incidence RSoXS	7
Collecting NEXAFS data	7
Transmission NEXAFS	8
TEY NEXAFS	8
Fluorescence Yield NEXAFS	9
Making Scripts	9
Saving motor positions	10
Calibrating sample-detector distance	11
Computer and server access	11
Remote server access	11
Connecting to Server on Apple computer	12
Closing and restarting LabVIEW Program	12
Restarting Main Beamline Computer	12
Supplementary Notes	12
Alignment Procedures	12
Scattering Chamber settings - switching from PEEM (11.0.1.1) to RSoXS (11.0.1.2)	12
Extra notes: Scatering Chamber settings - M103 and M101 Mirrors and Exit Slits	13
Mirror Focusing	14
Alignment of Scatter Slits	14
Piezo vs. pneumatic shutter	15
Using the hot stage and temperature controller	15
Adjusting set point for temperature controller	15
Settings for different energy ranges	16
Normalizing Intensity of scattering patterns (need to update this)	17
Soft X-ray Reflectivity	17
Notes on Beamline Instrumentation	18

# Loading and Removing Samples

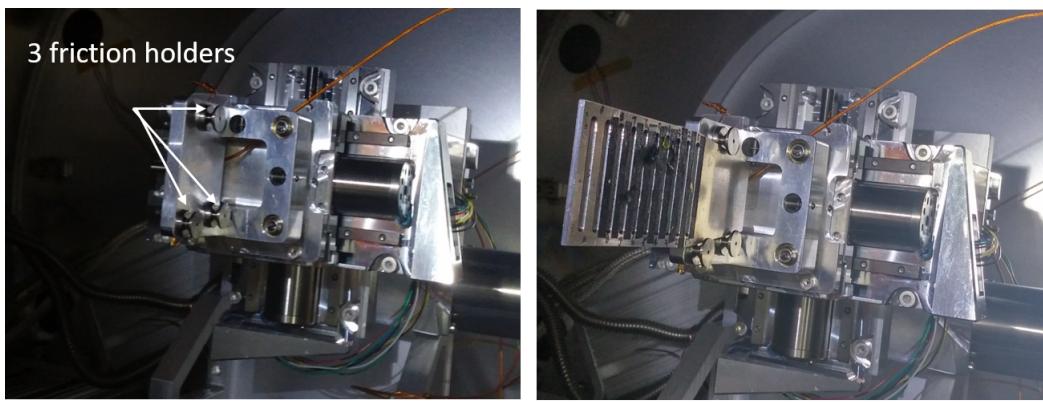
## Mounting samples onto sample holder

- Make sure they are within the tick mark boundaries so they are within the sample motor limits and can be reached by the X-ray beam
- Remember to include a reflective piece of YAG (to find the beam spot) and the 300 or 100 nm PS sphere calibration sample (or another calibration sample if you have your own)
- If you are planning to try any samples in grazing incidence geometry, try not to place other samples with thicker substrates in the same column (also along the beam path), this may make alignment more difficult



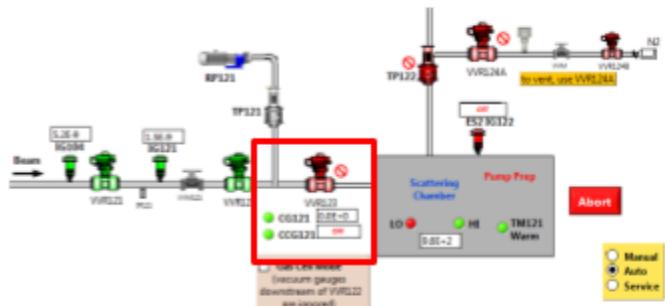
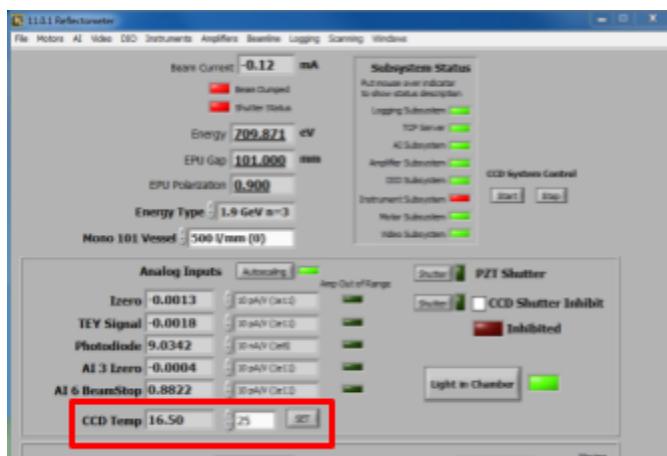
## Loading sample holder and pumping down the chamber

1. Best to put the photodiode into position first (in the Run Trajectories window, select the “Photodiode Far” position) before loading samples to prevent accidentally bumping the beam stop
2. Wear gloves when loading the sample holder. Load the sample holder with the tapered side in first and push the tapered end into the three friction fasteners. Make sure it is secure.



3. **Pump down**  In the BL1101\_New.vi LabVIEW window, click in the gray area labeled “Scattering Chamber,” and click pump from the pop-up menu.

4. Once you notice the pressure is starting to decrease, **set the ThermoCUBE chiller to 5°C**. (this makes the back side of the CCD detector less than 10°C).
    - a. Set temperature and press “enter”
    - b. Should see a “-” (or “+”) sign if things are working and the temperature is changing.
    - c. If there is an “\*”, may need to press “Start/Stop.” Might need to do this if ThermoCUBE was just restarted.
    - d. ThermoCUBE chiller will not keep decreasing temperature if the pressure is not going down
  
  5. When the chiller is at 5-6 °C, or the Peltier cooler (TMP 121, located on the left rack above computer screens) is less than 10 °C, and the vacuum is  $10^{-5}$  or less (the LabVIEW window only shows pressure down to  $1 \times 10^{-3}$  torr, the ion gauge will turn on and you can see the pressure on the controller window above the monitors), then **set the CCD temp in the 11.0.1 Reflectometer window to -45 °C** (If you are just doing X-ray absorption spectroscopy and not planning to use the CCD to collect scattering patterns, then you do not need to chill the CCD temp).
    - a. If the CCD System Control was turned off (the “Instrument subsystem” indicator will be gray), then it needs to be turned back on, i.e., 1) turn on the switch on the black control box, 2) then click start under “CCD System Control”
    - b. If notice that the CCD temps seems incorrect after turning this ON, one may need to cycle the power of the CCD System Control one more time
6. Open the valve (VVR123 in the BL1101\_New window) to the chamber when the pressure is below  $5 \times 10^{-6}$  torr.

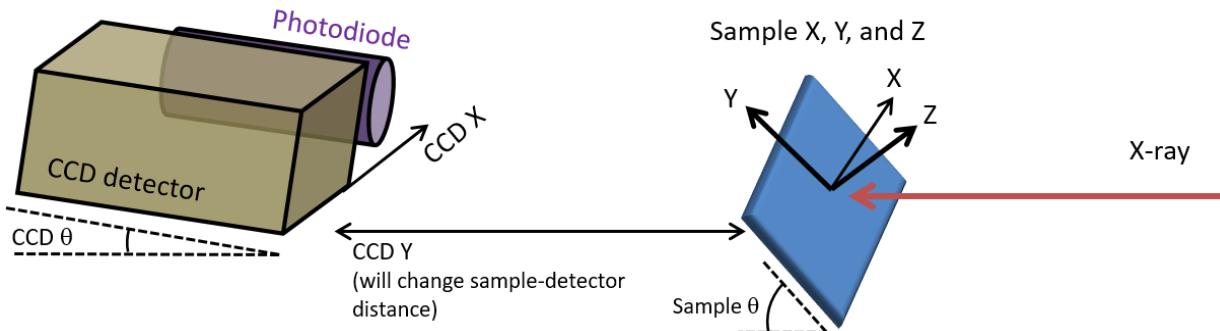


## Venting the chamber

1. In the LabVIEW program, set the CCD temp to 25°C and set the Thermo CUBE unit to 20°C.
2. Make sure the CCD temperature, chiller, and Peltier cooler indicator (TMP 121) are at 15 °C or above.
3. After the temperature has reached 15 °C, one can turn off the CCD system control (things seem to be fine if you don't do this): 1)click stop in the 11.0.1 Reflectometer window, 2) turn off the switch on the black control box .
4. In the BL1101.vi LabVIEW window, click in the scattering chamber and click pump □ “vent” (make sure light is green).

5. The chamber door can be opened when the pressure is  $10^3$  torr or higher (it should say “vented” in red text in the gray scattering chamber box when it is done).
6. Usually it’s a good idea to pump the chamber back down after samples are removed and if no one is coming soon after to run more experiments to keep the chamber under vacuum.

## Sample and Detector Geometry



- Sample  $\theta$  of  $90^\circ$  means the X-ray beam is normal to the sample plate surface
- Changing CCD Y will change the sample-detector distance. Changing sample Z will also change the sample detector distance (except when sample  $\theta$  is at  $0^\circ$ )

## Photodiode vs. CCD

There is a saved position to move the photodiode (PD) into the path of the beam, and the CCD out of the way. It is generally good to start out with the PD in position while finding the beam, etc. to avoid the direct beam unintentionally hitting the CCD.

- Photodiode position – There is a tab called “CCD Positions” in the Reflectometer LabVIEW window. Click the Photodiode Far button and the motors (CCD Y = 100, CCD X = 6, CCD θ close to 0) should automatically move the PD into place. If the light is green next to the photodiode button, then it should already be in the correct position.
- You can also go to this position using the run trajectories window.

## Finding the beam spot

It is always good to make sure the position of the beam spot is known. Then the cursor position on the video monitor can mark where the beam will hit on the sample plate. Note that if the LabVIEW is restarted, then the position of the cursor in the video display disappears. So make sure to put the cursor somewhere easy to remember (e.g. on the YAG) so that the beam spot can be easily found after restarting LabVIEW.

- Check that the Photodiode is in position to prevent direct beam on the CCD in case the beam is not close to where the cursor is.
- Set sample  $\theta$  to  $90^\circ$  so sample holder is perpendicular to the X-ray beam
- Assuming the beam spot is still not far from the cursor position, in the Video Center Display window, move the sample holder (Shift + click) so the cursor is over the YAG.

- Open the shutter (the lower Shutter button on the right of the Reflectometer window). If you see the beam reflecting off the YAG, center it and move the cursor so that it is in the center of the bright beam spot (ctrl + Alt +Shift +click).
- It might be a good idea to rotate sample theta while the beam spot is reflecting off the YAG to check that the beam spot is close to the center of rotation of the sample stage.
- Note that if sample Z is changed, then the cursor may no longer be where the beam spot is and this procedure may need to be repeated.

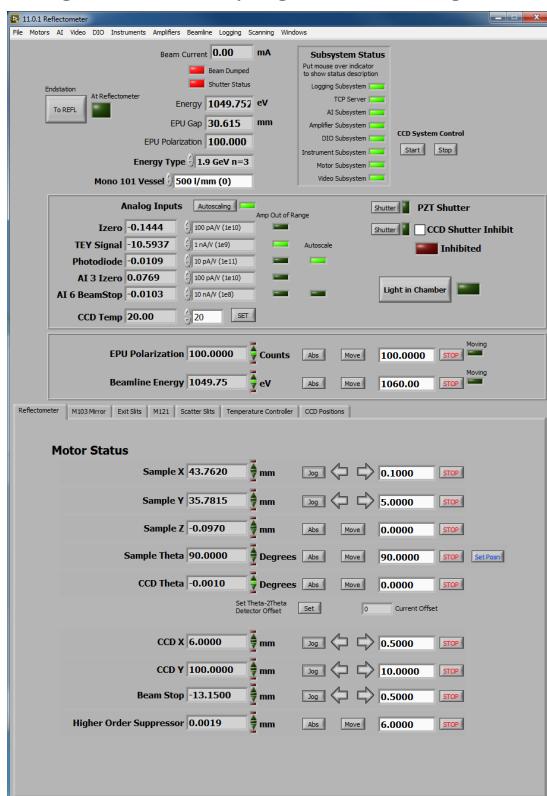
## Collecting scattering data

Scattering data is collected in the CCD Scan tab in the CCD and Single Motor SubPanel.vi window

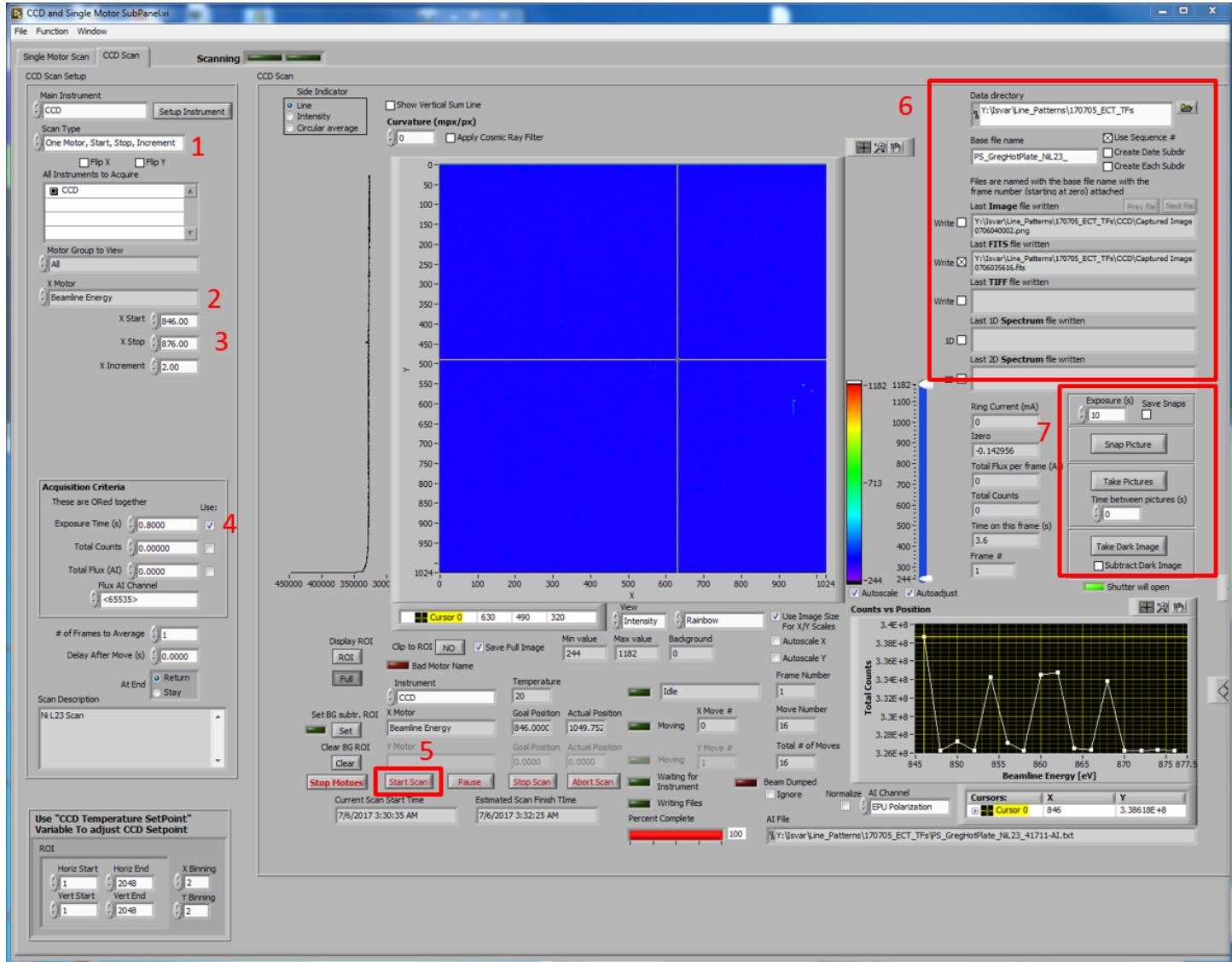
Move the CCD into position to collect 2D scattering data. There are usually a few preset positions saved in the Run Trajectories window (Motors>Run trajectories)

### Aligning beam stop to direct beam

- After CCD in place, do a quick test image. Make sure the Exposure time is short (0.1 s, in the box labeled “Exposure (s)” on the right side and the “Exposure Time (s)” box on the left under Acquisition Criteria) and click the “Snap Picture” button on the right side of the window.
- The beam stop should block as much of the direct beam (DB) as possible. If not, adjust the beam stop position (Beam Stop in the Reflectometer tab in the 11.0.1 Reflectometer window) and the CCD Theta as needed.
  - The beam stop only moves independently from the CCD in the horizontal ( $\pm x$ ) direction. That is what the Beam Stop position changes.
  - If the beam stop is above or below where the DB is hitting the CCD, then CCD Theta may need to be adjusted.
  - Keep taking quick 0.1 s Snap Picture images as the position(s) of Beam Stop and/or CCD Theta are changed to see the progress of the alignment between the DB and the beam stop.



## Overview of Options in CCD Scan



1. Scan Type – this drop down menu can be used to select different types of scans that move certain motors during the scan
  - a. One Motor, start, stop, increment – this will move one motor during the scan. You can set the position this motor will start at, where it will stop at, and at which incremental values it will take images at. To select which parameter to change during the scan, select that motor from the “X Motor” dropdown menu. For example, in the above image, the incident X-ray energy will be changed from 846 eV to 876 eV, and a scattering image will be taken every 2 eV
  - b. From File – this is what needs to be selected to run a script which can simultaneously move several motors at every step. See section on “Making Scripts.”
  - c. No Motor Movement – this will just take a scan and not move any motors. Be aware that this sometimes defaults to a very high number of scans, sometimes 100's (this option pops up underneath), so make sure you set this back down to 1 unless you want many scans of the same thing.
2. X Motor – this selects which motor you want to move during a scan
3. These values select the values or positions the motor will be moved through. Can also be the delta and increment for an around current positions scan

4. Acquisition Criteria – Important thing here is exposure time (s). When the Start Scan button is used (#5), then it will read the exposure time listed here (unless running a script which has a column for Exposure Time defined).
5. Start Scan – use this button to start a scan after you have setup all the motor movement limits, etc.
6. File saving options
  - a. Data Directory – browse to the folder to save data to. Click Current Folder. Note that a new subdirectory in that directory called “CCD” will be created where all the .fits files will go. The .txt file will be in the one specified.
  - b. Base Filename – what you want to name your file. It will add a set of number after the name. Check the Use Sequence # option
  - c. Make sure the Write FITS file option is checked. You can also write out a TIFF
7. Snap Picture can be used to take test images. These are saved in the directory you choose, but have the name Captured Image XXX. It will use the exposure time listed right above it, take just one image and won’t move any motors
  - a. Take Dark Image – This should take a dark image (no beam) using the exposure time listed above it. If this does not work, you can force a dark image by taking a normal single scan and checking the CCD Shutter Inhibit Button in the Reflectometer window, or closing the VVR123 valve.

## Grazing Incidence RSoXS

### Aligning the CCD

- set sample theta to zero and lower the sample stage down and out of the way
  - do a single motor scan of the CCD  $\theta$  around current position, should look like a step function. Move to the center of it, and that is the “0°” position
  - calibrate the sample height, which is sample z when sample  $\theta$  is zero
  - to find incident angle: can set the CCD  $\theta$  to 10 deg, (this is where the specular beam should hit for a 5 deg incident angle). Then adjust sample theta to maximize intensity
  
  - move sample theta to 0
  - single motor scan around current position, set the motor to sample Z (which is height when sample theta is 0)
  - should look like a typical sample z scan
  - set sample theta to 5 deg, and CCD Theta to 10, then do a single motor scan around CCD theta to check where the specular intensity is □ look at where the peak in CCD theta is and half of that value should be close to 5. If it is not, then reset the position (Set Posn button) of sample theta so that 5 is now that value (this is like a theta scan)
  - go back to sample theta 90 and check the location of the beam on the YAG in case it moved because changed the sample Z height
  - move to the calibration sample to check the position of the beam stop
    - can save these parameters as in the run trajectories presets [CCD far (BS center), CCD close, etc]
    - Also find beamstop etc. positions for CCDY=0 (close), and CCD=30 (middle)
  - May need to make hOS higher if over saturating
  - When moving to new GI sample
    - Set sample theta to 90, move, then move sample theta back to 5deg
    - Do a quick scan and maybe have to adjust sample theta a bit and beam stop a bit
-

# Collecting NEXAFS data

For NEXAFS, you just need to monitor transmitted intensity (or TEY signal), so be sure to first **move the photodiode into position** to avoid accidentally exposing the CCD to beam.

NEXAFS can be done in two main detection modes:

1. Transmission – In this case, the transmitted X-ray intensity through the sample is monitored as a function of energy, which provides information on absorbance. Sample must be on a transparent window such as Si<sub>3</sub>N<sub>4</sub>. If sample is too thick, a lot of the X-ray may be absorbed, reducing transmitted intensity. The photodiode is used to monitor the transmitted intensity.
2. Total Electron Yield (TEY) – As photoexcitations occur, electrons fly off the sample. This can be monitored by tracking the drain current needed to neutralize this charging effect. TEY is relatively surface sensitive (top few nm's or so). If just going to do TEY, a sample can be placed on a substrate such as a piece of Si wafer. Make sure the substrate is somewhat conductive.

## Transmission NEXAFS

- Do a single motor scan from a script, or start, stop increment through a range of energies
- Set the X motor to beamline energy
- Channel to view: Photodiode
- Have to do a scan of the direct beam too (sample moved out of the way) for normalization!!
- To plot transmission NEXAFS data: Plot  $-\ln(PD_{\text{sample}}/PD_{\text{directbeam}})$

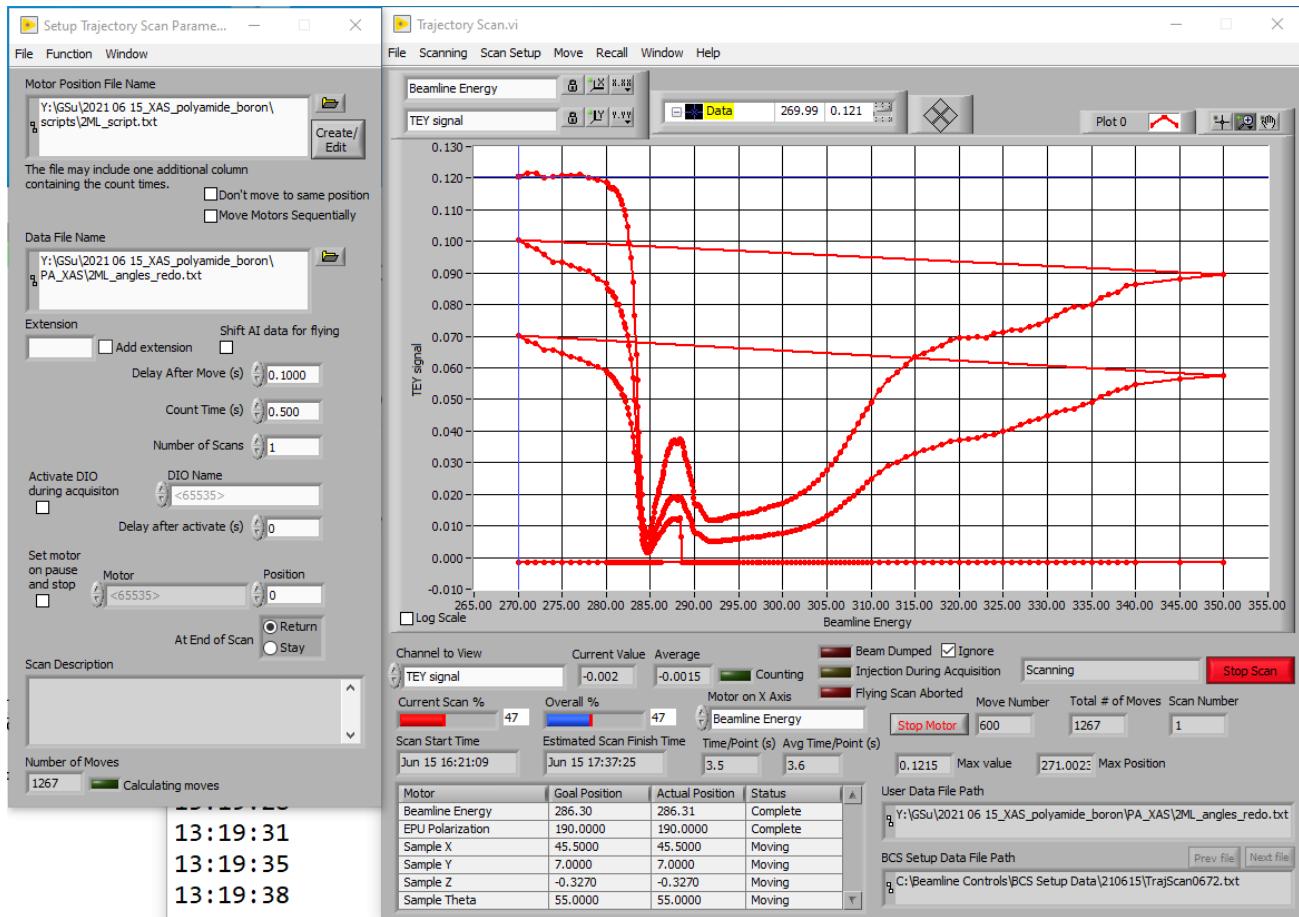
## TEY NEXAFS

- May need to have better electrical contact between the sample and the sample holder
  - Can use the little metal clips to fasten the sample that were screwed into the special sample holder that is fabricated with regularly spaced screw holds
  - However, TEY data on typical Si<sub>3</sub>N<sub>4</sub> membranes and the carbon tape usually works OK and allow for transmission and TEY to be collected on the same sample
- HOPG sample is used to calibrate energy (need to check where the main peak in the HOPG absorption is supposed to be)
- To improve the TEY signal, may want to use a shallower incident angle ( e.g. sample  $\theta \sim 30^\circ$ ). This spreads out the beam footprint on the sample allowing for more material to be impinged by the X-ray
- A reasonable “exposure time” for each energy is **1s**
- With the beam on the sample of interest, open the shutter and check that the “TEY signal” value goes up
- **Setup**
  - Single motor scan from script (or start, stop, increment); set X motor to beamline energy; view the TEY signal
  - The files are not automatically named so need to keep track of which file is being written (e.g. SigScan\_)
  - This .txt file will have columns of data for all of the parameters including energy, photodiode, TEY, etc
- For TEY seems like it may be necessary to turn off autoscaling and set the gain to **1E10 (have noticed that just changing from 1e11 to 1e10 made TEY signal seem more stable, not sure why)**.

- Probably turn on autoscaling for the PD when collect **TEY intensity of the sample by the photodiode intensity of the direct beam (DB) at the same energy**
  - Since the DB can fluctuate, should take a DB scan in between every sample or two using the same energy points that will be used for the sample's NEXAFS spectra
  - Further normalization such as setting a post edge value to 1, subtracting pre-edge etc. may have to be done later
  - Cheng's carbon K edge script (X:\Wang\script\carbon\_nexafs) is a good starting point

## Making scripts for NEXAFS

- Can't just make a normal script like scattering and do a "from file" run in the "Single Motor Scan" tab. Need to run the script from the "Trajectory Scan" windows
  - To get to Trajectory Scan, in the main 11.0.1 Reflectometer window, go to the top drop-down menu: **Scanning □ Trajectory Scan**



- In the Setup Trajectory Scan Parameters.vi window, you can load your script in the "Motor Position File Name" box.
- In the "Data File Name" field, browse to where you want the file to be stored and include the file name with extension, e.g. \*.txt
- Example form for a script that does energy at different incident angles:

Beamline Energy	Sample Theta
270	30
271	30
270	40
271	40

- Fill in the options like Delay After Move (s), Count Time, etc.

- Note, the shutter does not automatically stay open when the scan is started, and turned off when it's done. You can manually open the shutter before the scan starts and close it afterwards, or:
- To Open shutter: check the “Activate DIO during acquisition” box and in the DIO Name select “Shutter Output”
  - Note that this opens the shutter for the amount of time specified in count time, and closes it in between each energy value

## Fluorescence Yield NEXAFS

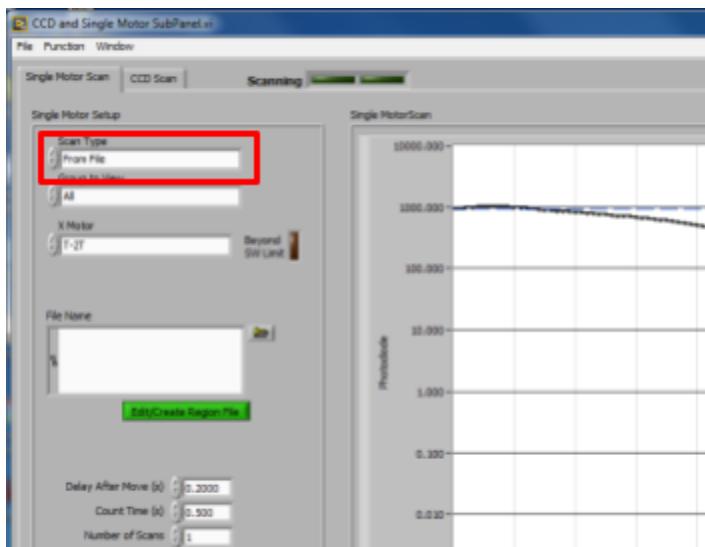
Coming soon

## Making Scripts

- Make a text file, can use one of the old files as a template
- Make sure there are no spaces, tab delimited
- First column is the X-ray energy, second column is the Polarization (100 for S; 190 for P), third column for exposure time in seconds (does not need a label)
- First col needs to be labeled “Beamline Energy” and polarization col labeled “Gap Polarization”
- Example:

Beamline Energy	EPU
280	100    0.5
284	100    1

- After saving the text file, go to the Windows PC and load it in Word Pad, should be in data(\bl11012data.lbl.gov)(Y:)
- Make sure there are **no extra tabs**, then re-save as text (MS-DOS file?)
- To run the macro: Under “Scan Type”, select “from file,” load the macro file, then can start



## Saving motor positions

In the 11.0.1.2 Reflectometer LabVIEW window, can go to Motors  $\square$  Run trajectories and can select preset positions to move the photodiode into position, the CCD into various positions, etc. These can be good starting points, but small adjustments may need to be made.

### Photodiode (Photodiode Far)

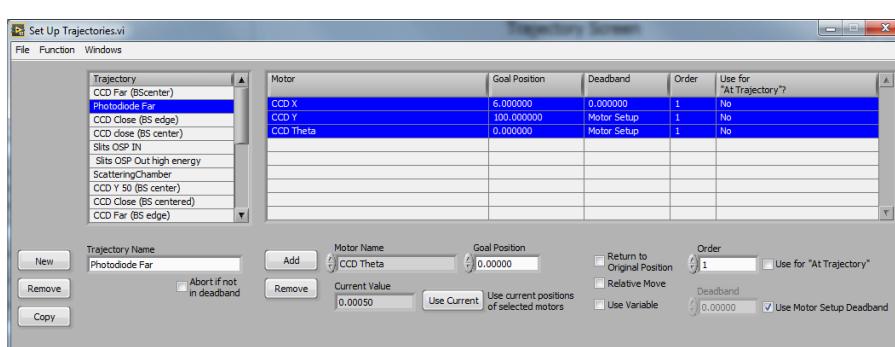
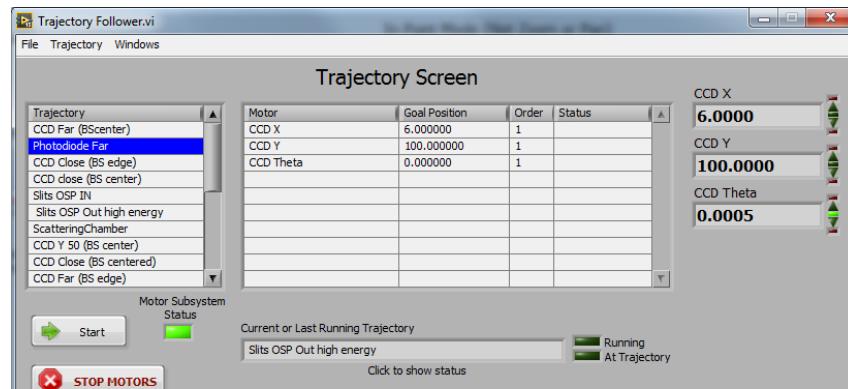
- CCD Y = 100
- CCD  $\theta$  = 0
- CCD X = 6.0

### Approximate Settings for a SD distance of about 150 mm, [CCD Far (BScenter)]

- CCD Y = 100
- CCD X = 99.1
- CCD  $\theta$  = 0.273
- Beamstop: -13.5

Other commonly used trajectories:

- CCD Close (BS edge)
- Slits OSP IN – this is to save the scatter slit positions for energies where the higher order suppressor is used, such as carbon, nitrogen, and oxygen K edges
- Slits OSP Out high energy – for higher energies where HOS is moved out of the way



- Can save positions of CCDX, CCD Y, Beam Stop, etc. from the “Set Up Trajectories” window (Motors  $\square$  Setup Trajectories the 11.0.1 Reflectometer LabVIEW window)

- Select “Use Current” to copy the current positions, then do **Function**  **Save** to save it!
- 

## Calibrating sample-detector distance

Typically, the sample-detector (S-D) distance is very close to CCD Y + 50 mm (assuming sample Z is at 0 mm)

- Take a scan of the calibration sample
    - Note: We used to use 300 nm PS spheres, but this seems to not be the best material. We are planning to get a phase separated block copolymer sample soon
  - Do a few energies (270, 280, 285 eV if doing carbon edge)
  - Probably a good idea to open and do the beam centering/SD distance calibration in Igor to check that it looks OK
  - If the scattering rings from PS do not line up, it could be not the 300 nm sample
  - Make sure to take a new calibration if you change the CCD Y value, change sample Z, the beam dumps, etc.
- 

## Computer and server access

### Remote server access

- Address: bl11012data.lbl.gov
- Username: rsoxsuser
- Password: BL11012user
- Look under root/volumes/data/chabiny

### Connecting to Server on Apple computer

1. In the main Desktop go to Go  Connect to server
2. Server Address: smb://bl11012data.lbl.gov
3. Password: BL11012user

### Closing and restarting LabVIEW Program

- In the 11.0.1 Reflectometer window: Go to “File”  stop beamline controls” before quitting LabVIEW
- After exiting and restarting the LabVIEW program (“BL Control Main”, should be on Desktop), click the Run arrow in upper left
- Lights will start to turn green as things come back online
- Set the Energy Type and Mono 101 Vessel values (e.g. For lower energies like carbon edge use n = 1; Mono 101 vessel 250 l/mm)
- To bring up the main control window, go to Scanning  “Single Motor and CCD Scans”
- Turn autoscaling back on

### Restarting Main Beamline Computer

- Located behind the PEEM endstation

- If need to restart:
    - Password: xray\$1101
    - Open the “BL Control Main” LabVIEW program that should be on the desktop
    - Click the Run arrow on the window
- 

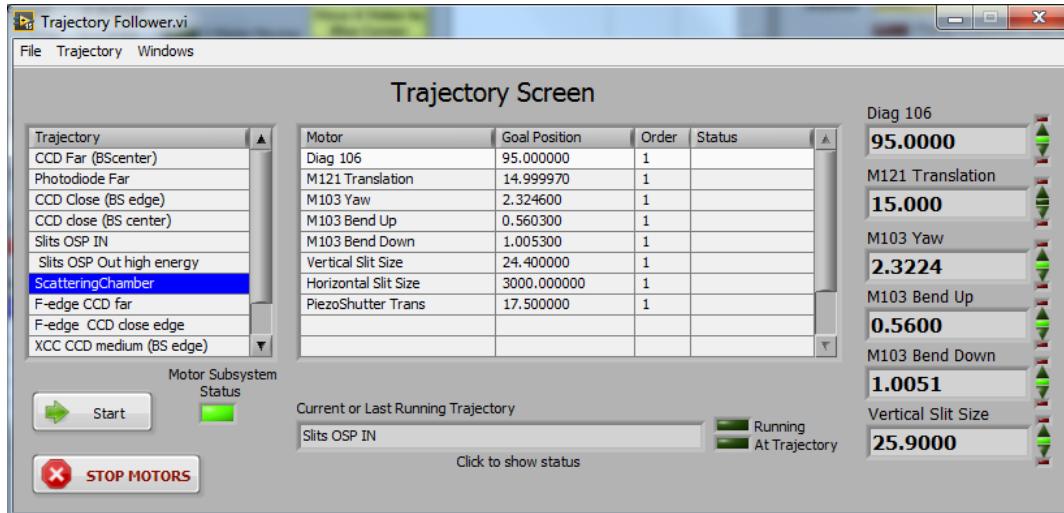
## Supplementary Notes

### Alignment Procedures

#### Scattering Chamber settings - switching from PEEM (11.0.1.1) to RSoXS (11.0.1.2)

Note: These steps typically only need to be done when redirecting the beam from the PEEM endstation (11.0.1.1) to the scattering endstation (11.0.1.2) and should normally already be done when users arrive

- Go to the preset settings for the M103, M101, etc that are under the “ScateringChamber” setting in the Trajectory Follower.vi window



- Make sure the photodiode is in position, not the CCD detector
- Open all the scatter slits (in he “Scatter Slits” Tab) to aperture values of 2.0 mm. These would be Upstream JJ Vert/Horz Aperture, Middle JJ Vert/Horz Aperture, and In-Chamber JJ Vert/Horz Aperture

#### Extra notes: Scatering Chamber settings - M103 and M101 Mirrors and Exit Slits

(These are now already set in the “ScatteringChamber” preset, so should not have to be done manually)

This is a Kirkpatrick-Baez focusing mirror

- Move the M103 mirror positions to the preset
- In the M103 Mirror tab, one can jog the positions of this mirror to help maximize beam position and intensity
- Make sure the photodiode is in position! Open the shutter and watch the Photodiode intensity as the mirror is moved around little by little

Exit Slits alignment

- Open the horizontal exit slit size to 4000 (or 3000)
- Vertical exit slit set first to -10 um, then to 25 um

- Horizontal exit slit position -700 um
- Vertical exit slit position to -0.25 um

Move the Peizo Shutter out of the way

- Go to beamline computer and set the “PiezoShutter Trans” value to 17.5 mm (in the Motor Display.vi tab)

Raising the M121 mirror up to deflect the beam into the 11.0.1.2 chamber

- Go to the Motor Display.vi tab
- Set M121 to 15 mm

If not using the gold mesh, then move diagnostics stick out of the way

- Make sure filterTi-Au motor (Motor Display.vi tab) is out of the way, at 94 mm

Aligning the M101 mirror (under the “M103 Mirror” tab)

- Yaw: 2.318, bend up: 0.56, bend down: around 1.0
  - These values should be fixed and not have to change, although M103 and M101 may need to be tuned

## Mirror Focusing

- Usually, the beam intensity can be pretty good just be adjusting the M101 mirror. This can be found in the “M103 Mirror” Tab, the bottom two rows are for the M101 Vertical and Horizontal Deflection. Can open all the scatter slits before focusing the mirrors
- Can do a single motor scan, start stop increment. Move to the maximum.
  - M101 Vertical Deflection: about -22 to -19.5 mm using 0.05 mm increments
  - M101 Horizontal: can scan from -2 to 2 in 0.05 mm steps
- Can also just jog the M101 vertical and horizontal Deflection in steps of 0.05 mm to maximize the photodiode reading Just open the shutter and watch the photodiode reading while jogging the M101
  - Keep track of the initial position, jog motor until photodiode is maximized (note this position), Then move back to initial position and move to the new position

## Alignment of Scatter Slits

- Again, this is typically already done for general users, but scatter slits may need to be re-aligned if switching to very different energies
- Slits are basically used to reduce parasitic scattering.
- When adjusting certain parameters, e.g. moving to higher energies such as Fluorine K edge where the HOS is removed, the beam position may change slightly. Therefore, may need to readjust the vertical (and horizontal?) translation position and Vert aperture sizes of the slits

## Basic procedure to align:

- There are some preset positions for doing slit adjustments: example Motors □ Run trajectories □ Slits OSP Out high energy (moves HOS out of the way)
  - Some of the values here such as the translational positions are saved from the previous alignment
- Set the main settings: Energy Type (n), Mono 101 Vessel, HOS, and go to an X-ray energy similar to where you will be working at

Open the slits

- Set all the aperture sizes to **2 mm** (opens them all up)

Narrow the slits one by one and find the center (better to go to position

- Set “Upstream JJ Vert Aperture” to **0.05 mm** – this makes just this slit very narrow allows you to find the center position

- Do a single motor scan, around current position □ set the X motor to the “Upstream JJ vert translation” – around current position
  - Range of 1 mm should be reasonable, step size 0.05 (need to have the slit of interest very narrow so can see the center)
  - Find peak and go to center of it
  - Make sure watching the PD signal
- After centering, open the aperture to **0.5** (should get about 2000 nA) (this is a temporary open position while center all the other slits)
- After upstream vertical repeat process for upstream horizontal
- After upstream do middle (after centering, open the slit to **0.4**)
- The In-chamber slits should be open up to **0.6**
- After all slits aligned, open apertures to following values, approximately (**this is for with HOS IN!!**)
  - Upstream: vertical and horizontal 0.2 mm (or 0.25 mm)
  - Middle: vertical and horizontal 0.35 mm (or 0.5 mm)
  - In Chamber: vertical: 0.65; horizontal: 0.75 mm
- After slits aligned for higher energies with HOS out of the way, open apertures to (these can be tweaked)
  - Upstream: 0.1-0.15 mm (maybe even as low as 0.05-0.1)
  - Middle: 0.35 mm
  - In chamber: 0.50 mm
- The final flux doesn't need to be over 2000 or 3000 (for 280 eV)
- Save positions of slits in the “Slits OSP IN” (or “Slits OSP Out high energy”) bookmark in the “Set Up Trajectories” Window

## Piezo vs. pneumatic shutter

If need shorter exposure times (less than 0.05 s), will need to switch from the normal pneumatic shutter to the piezo shutter. To do this:

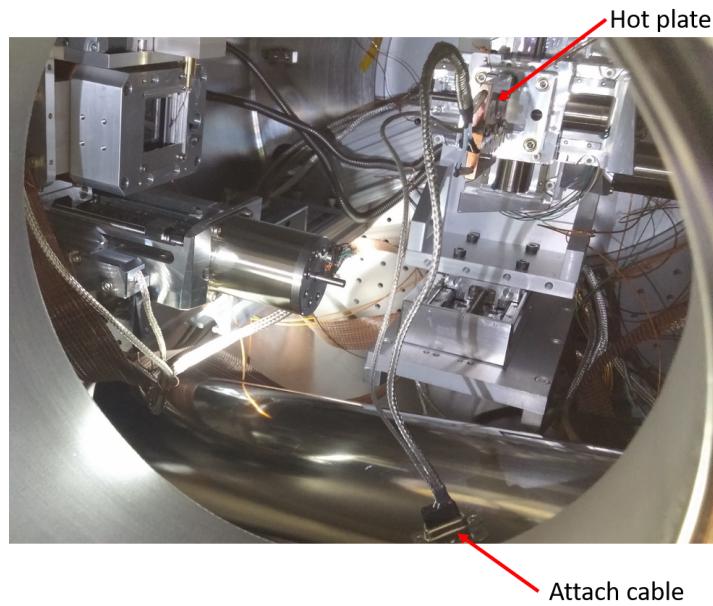
- Open the DIO Monitor.vi window (DIO □ Monitor) and check the Air Shutter box. This will keep the air shutter open so now the piezo shutter can do the opening/closing.
- In the 11.0.1 Reflectometer window, go to the M121 tab and there is a “Shutter In” button to put the Piezo Shutter Trans motor into position. Should be about 18.85
- In the CCD and Single Motor Window, under the Single Motor Scan tab, uncheck Shutter output option under “DIO to Actuate”
- Need to switch out some cables:
  - On the CCD Shutter Control box, on the “T” BNC connector, switch the Air Shutter cable with the Piezo Shutter cable (might be on the floor)
  - In the control rack behind PEEM endstation, replace the PEEM trigger BNC with the REFL trigger BNC





## Using the hot stage and temperature controller

- The hot plate has an angled fin that fits into the three friction holders like the normal sample holders do. Load it like a normal. There is a cable that should be sitting at the bottom of the chamber that plugs into the cable attached to the hot stage, as shown below.



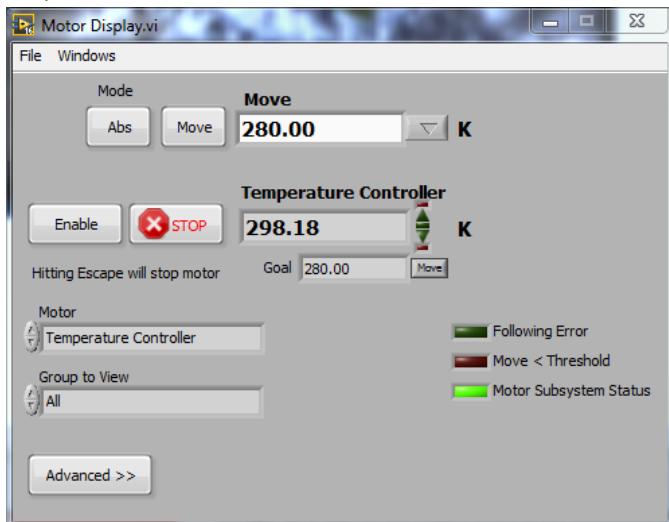
## Adjusting set point for temperature controller

*This is one way, there is likely an easier way to do this and will update the manual later*

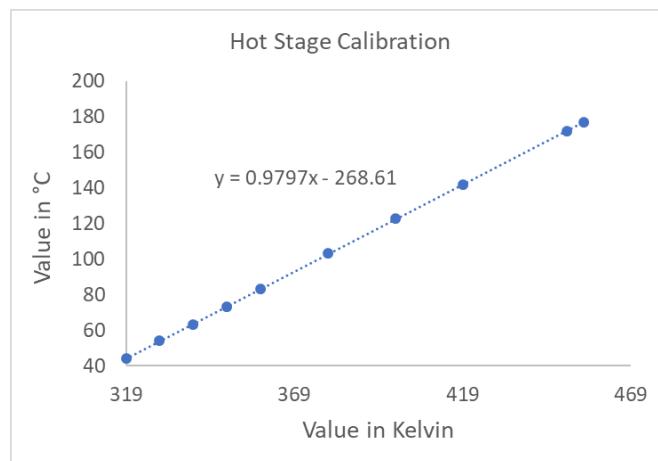
- In the 11.0.1 Reflectometer Window, there is a temperature controller tab that displays some set point and temperature reading values

Temperature Controller		Heater Range	HTR Pwr %
Temperature A	372.64 °K	Med	0
Temperature B	0 °K		Setpoint 400 °K

- The “Heater Range” value should be set to **medium**, and needs to be re-cycled between off and medium every time the cable is reattached to the hot plate
- To control the hot stage setpoint, go to Motors □ Display and change the Motor in the drop down menu to “Temperature Controller”

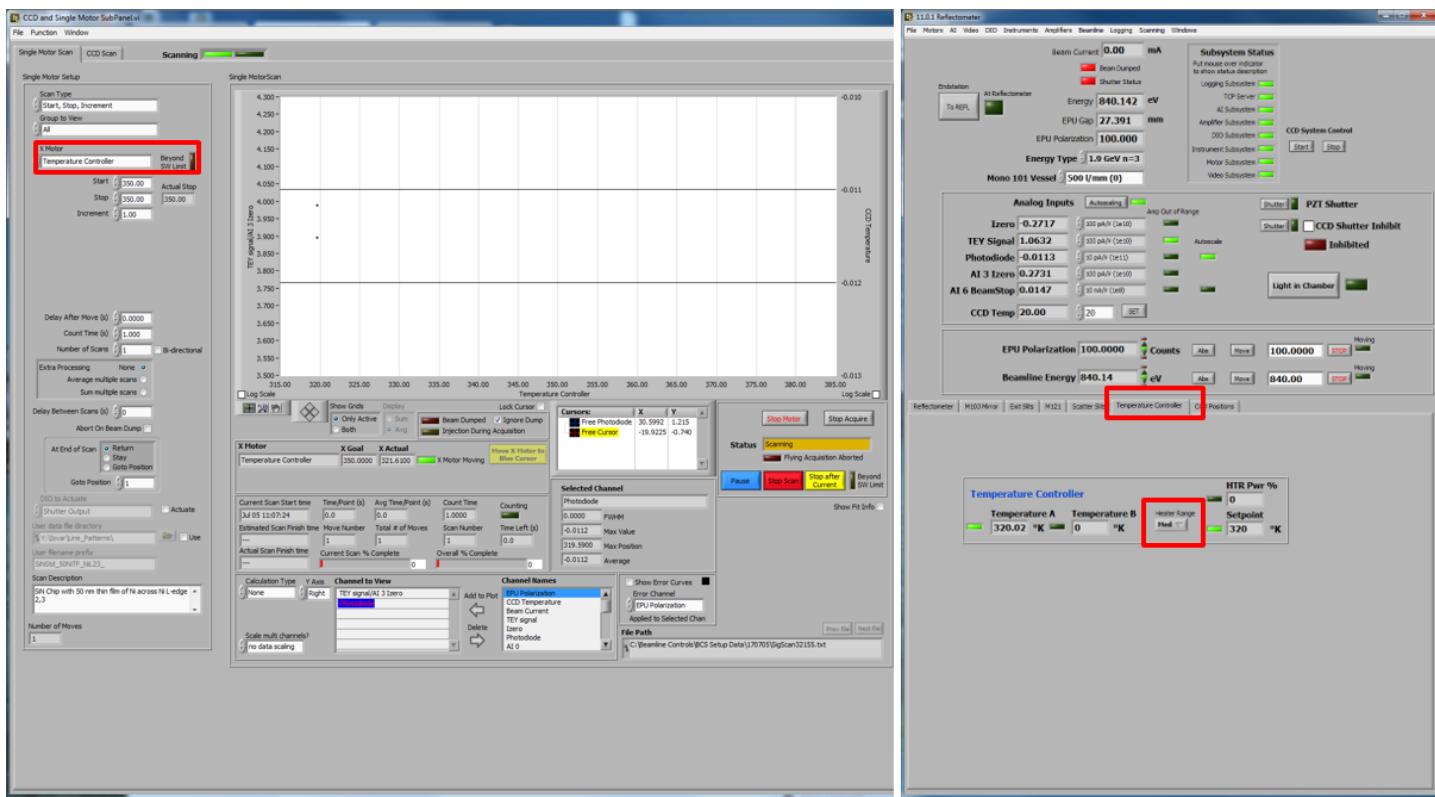


- Here, simply set the setpoint to desired value. A general calibration curve for the relationship between the setpoint value in K and the approximate temperature in C is below (note this is for an older version of the hot stage, new version should be coming soon).



### Old instructions?

- In the 11.0.1 Reflectometer Window, there is a temperature controller tab that displays some set point and temperature reading values
- In the single motor scan tab, you can do a start, stop, increment and set the X motor to “Temperature Controller.” Set the start and stop to the same value, whatever you want the set point to be.
- When click “Start Scan,” you should see the set point in the Temperature Controller tab in the Reflectometer window change to the corresponding value. Also the Temperature A value should start to go up. If the temperature A value does not reach the set point, you may need to change the Heater Range setting.
- The value in °C compared to the temperature reading in Kelvin is shown below.

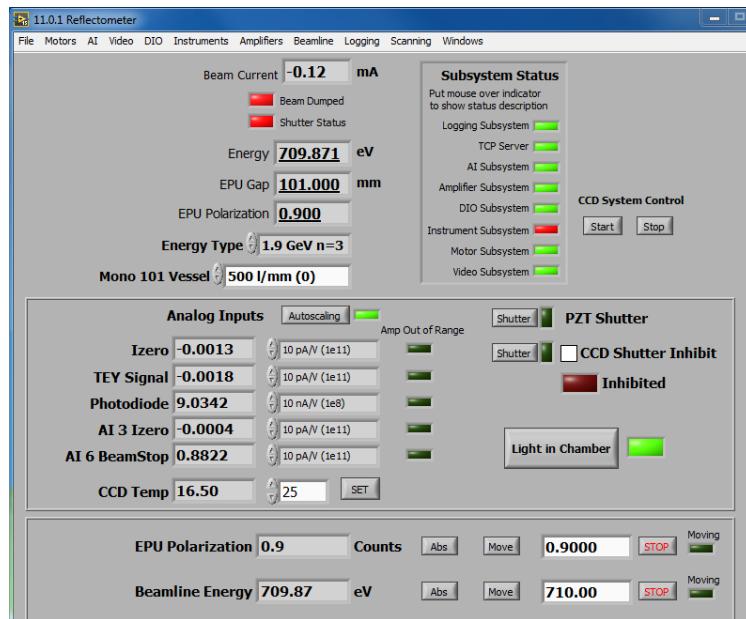


## Settings for different energy ranges

- For circularly polarized light use EPU=1 (or 0.9?); S polarized is EPU=100; P polarized is EPU=190 (see the chart at the end station)
- For 150 – 550 eV (carbon, nitrogen and oxygen K edge energies):
  - Energy Type: n=1
  - 250 lines/mm
  - Higher order suppressor (HOS) around 6.5 for lower energies, 6.0 should be OK for higher energies around 500 in this range
- For energies above 550 eV:
  - n=3
  - 500 lines/mm (use 500 for things above 500 eV)
  - hos=0 (-2 really moves the HOS completely out of the way)
- When changing these settings, the beam shift slightly so it is a good idea to align the M101 and the scatter slits separately for the lower energy range and the higher energy range. These are often saved as presets called “Slits OSP In Low Energy” and can be good starting points.

## Normalizing Intensity of scattering patterns (need to update this)

- As of Dec 2013, the gold meshes were removed. To normalize intensity, take the photodiode reading of the direct beam (move samples away)
  - Should do this every few samples since intensity fluctuates



- Divide the scattering intensity by this value
  - Also, take photodiode reading of the beam in transmission through the sample
  - Could also take an intensity through a bare window
- 

## Soft X-ray Reflectivity

Motors are not very well aligned at higher angles, so may be difficult to get good XRR data right now

- Z-alignment – aligns the height of the sample so it goes through the center of the beam
  - Single motor scan of the sample Z motor around current position. +- 0.5 mm or so, increment 0.05 mm
  - Should see the intensity drop off quickly at the point where the substrate begins to block the beam
  - Move the cursor to the midpoint of this drop off. To move to the position the cursor is in, click the yellow “Move X motor to blue cursor” button
- Theta alignment
  - Set sample theta to 5 deg and ccd theta to 10 deg
  - Do sample theta scan around current position
  - Peak doesn’t usually look so peaky here, but would move the sample theta to a point that maximized the intensity

Collect reflectivity curve

- Run a start, stop, increment with the X Motor set to T-2T (theta – 2 theta). Scan from 0 – 20 deg, or maybe 0 – 40 deg for extra range, resolution (increment) of 0.05 deg; 0.5 s exposure
  - use circularly polarized light (EPU 0.9) for reflectivity scans at high energies
- 

## Working up data

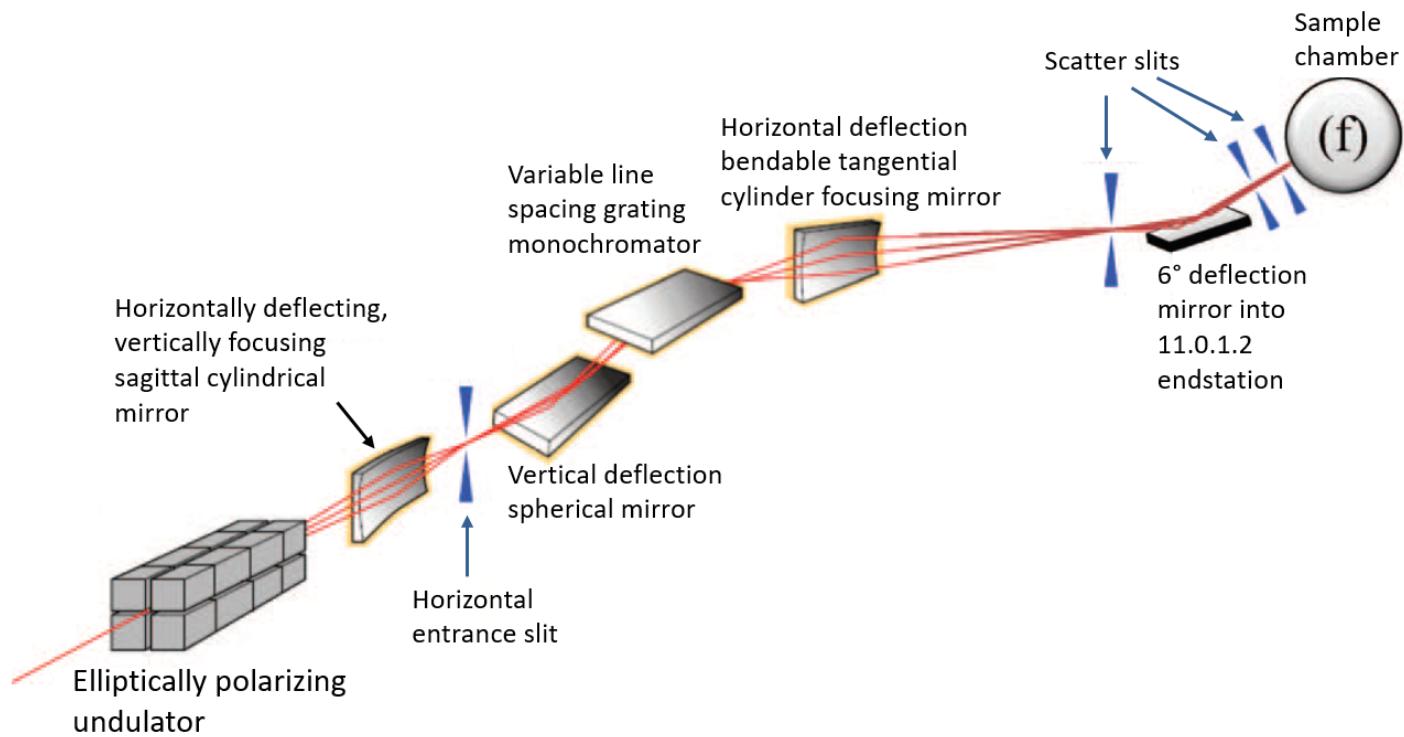
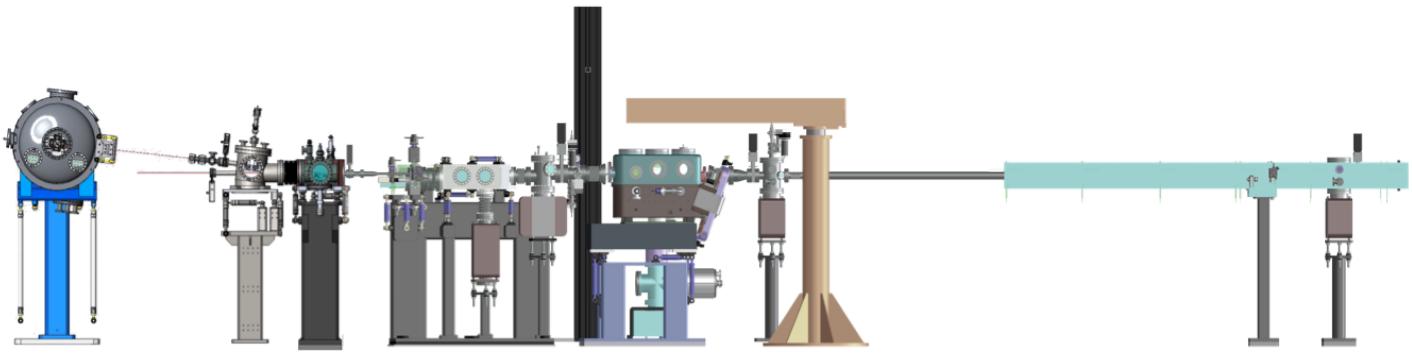
Flatten center lines: 980, and 50

Direct beam normalization: use 270 or 280 eV; the photodiode should be more than 400

Always go back to the same energy (270) make sure DB photodiode intensity not fluctuating much

- Need to take photodiode intensity for every energy used for scattering
    - Need to do this through the sample and also for the direct beam
  - To do a scan of the photodiode I vs E
    - Go to “single motor scan” tab
    - Scan type: start, stop, increment
    - X motor: beamline energynitrogen
    - Should be able to pick a fixed exposure time since using the photodiode, something like 0.5 s is probably ok
    - If already know what energies will do for scattering, can run that script just make sure adjust exposure times
- 

## Notes on Beamline Instrumentation



- **Elliptically polarizing undulator (EPU)** – 5 cm period; 165-1,800 eV [A. T. Young et al., *J. Synchrotron Rad.*, **2002**, 9, 270-274]
  - 4 rows of permanent magnets placed along axis of electron beam
  - 2 rows above the plane of the storage ring on either side of the beam; same for the 2 rows below
  - Linear polarization from 0° to 90° are available for energies above 160 eV
  - Using the fundamental output from the undulator, pure circularly polarized X-rays can be produced from 130-600 eV
    - For higher energies, 3<sup>rd</sup> and 5<sup>th</sup> harmonics must be used, but this leads to elliptical ( $P = 0.8$  to  $0.9$ ) rather than circular polarization

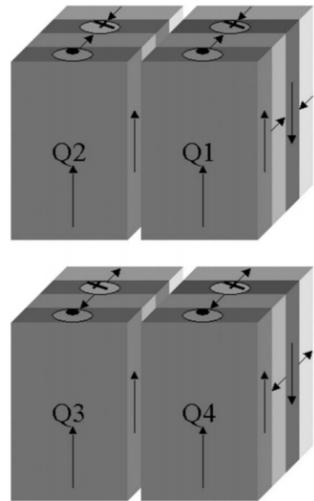
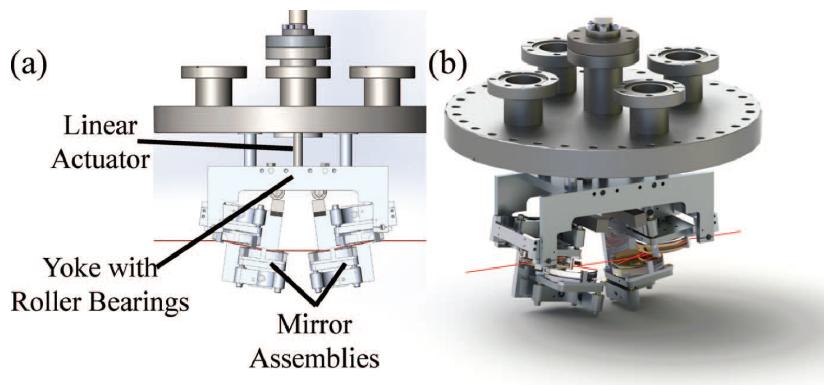


Figure 1. Quadrants 2 and 4 move longitudinally in and out of the plane of the figure

- **Higher Order Suppressor**

- Four-bounce mirror assembly
- Exiting beam is collinear with the entering beam
- Optical elements are standard 2" diameter uncoated mirror blanks coated with 20 nm of Ni for optimized performance in the 200-600 eV range
- Incident angle of the mirrors can be changed between 4°-8°, or moved out of the beam path to let unfiltered light into the chamber



- **Scatter Slits**

- Define beam on sample and minimize parasitic scattering
- Upstream: ~1.5 m upstream of sample chamber
- Middle: ~0.6 m upstream of the sample – remove most of remaining parasitic scattering from first set of slits
- In-chamber: ~0.2 m upstream of sample

- **Monitoring beam flux**

- After the 6° deflection mirror, there is a gold mesh assembly that can be lowered into the beam and connected to a picoammeter to measure photoelectric current
  - This gold mesh is upstream of the HOS, so the spectrum of X-rays measured here is not suitable when doing scattering experiments at lower energies (e.g. near carbon dip) that need to use the HOS

- Second gold mesh assembly directly downstream of the HOS, but before the first set of slits is used to monitor incident flux
- **Photodiodes**
  - Main photodiode separate from CCD: 5 mm x 5 mm GaAs from Hamamatsu – designed to be in the direct beam with CCD out of the way
  - Beamstop photodiode: 1 mm x 3 mm Si photodiode from Advanced photonics – flux monitoring and absorption measurements while scattering data is collected
- **CCD detector**
  - Princeton Instruments PI-MTE
  - Thermoelectrically cooled to -45°C
    - Heat produced by the thermoelectric cooler is removed by circulated chilled ethanol through the copper heatsink
  - 2048 x 2048 array of 13.5  $\mu\text{m}$  x 13.5  $\mu\text{m}$  pixels
  - Detector can collect scatter from -25° to +160°