Beamline 7.3.3 SAXS/WAXS Advanced training

Alexander Hexemer and Eric Schaible

Purpose: That you will be able to use the Beamline independently

WHAT NOT TO DO!!!

DO NOT EXPOSE DETECTOR TO DIRECT BEAM !!! (Double check, then double check AGAIN)

Before opening the shutter, THINK

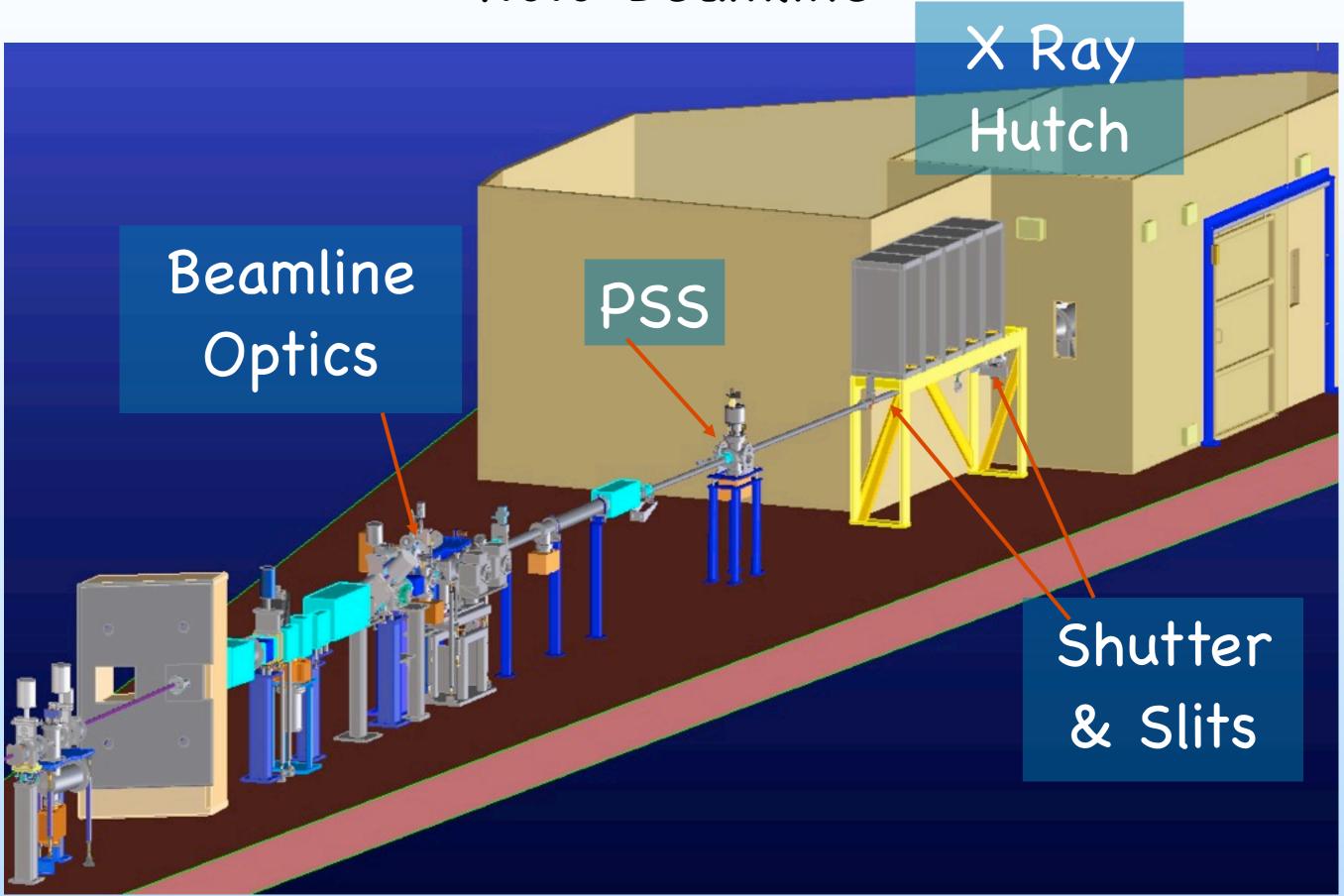
Info on Website

- www.SAXSWAXS.com
- Phone numbers
- Emails
- ESS webpage with David Malone info
- Ring Status webpage
- IgorPro webpage Nika

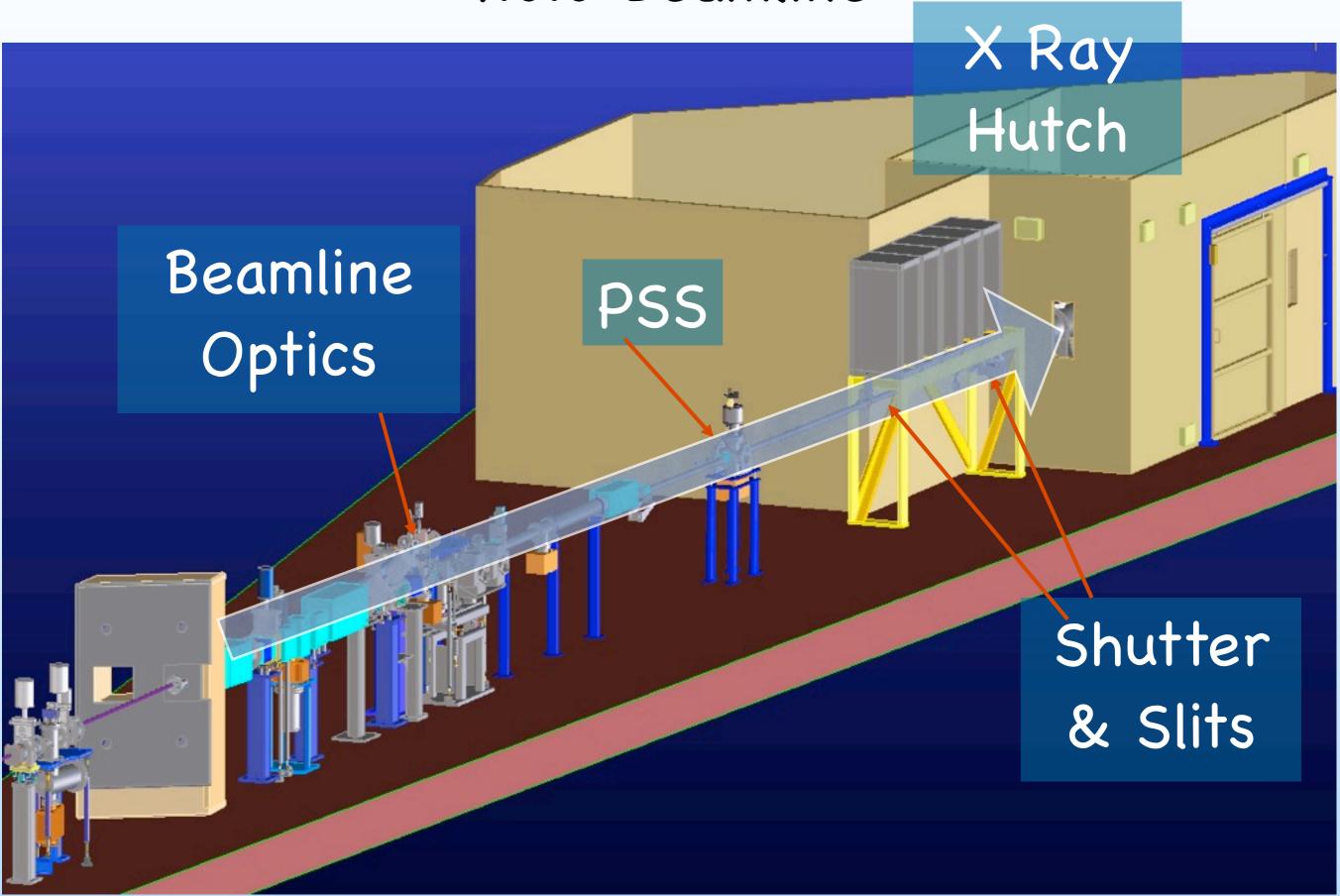
Outline

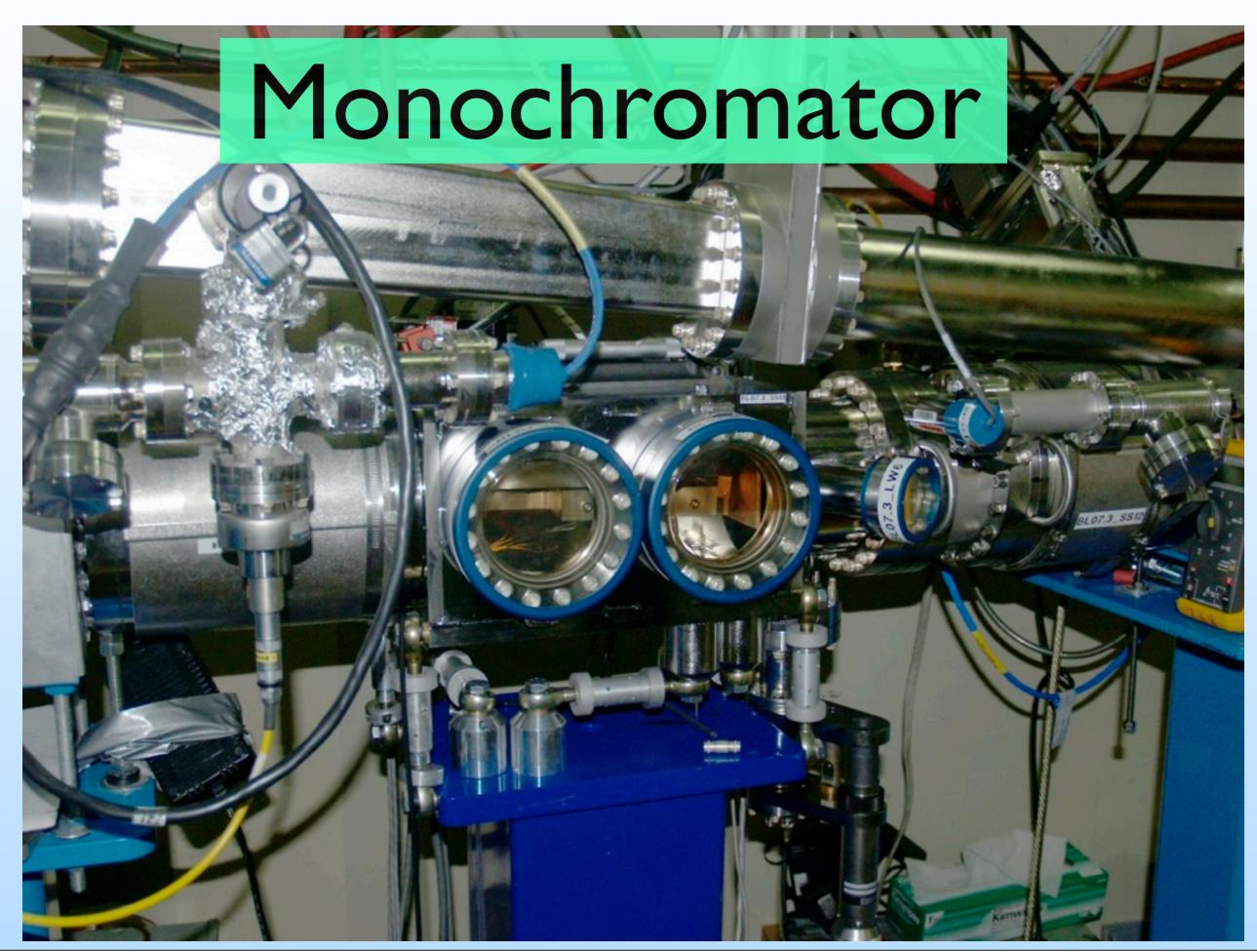
- Beamline Schematic
- Before you get to the BL
- When you get to the BL (Preparing BL)
- Performing your Experiment
- Analysis
- Leaving Beamline

7.3.3 Beamline

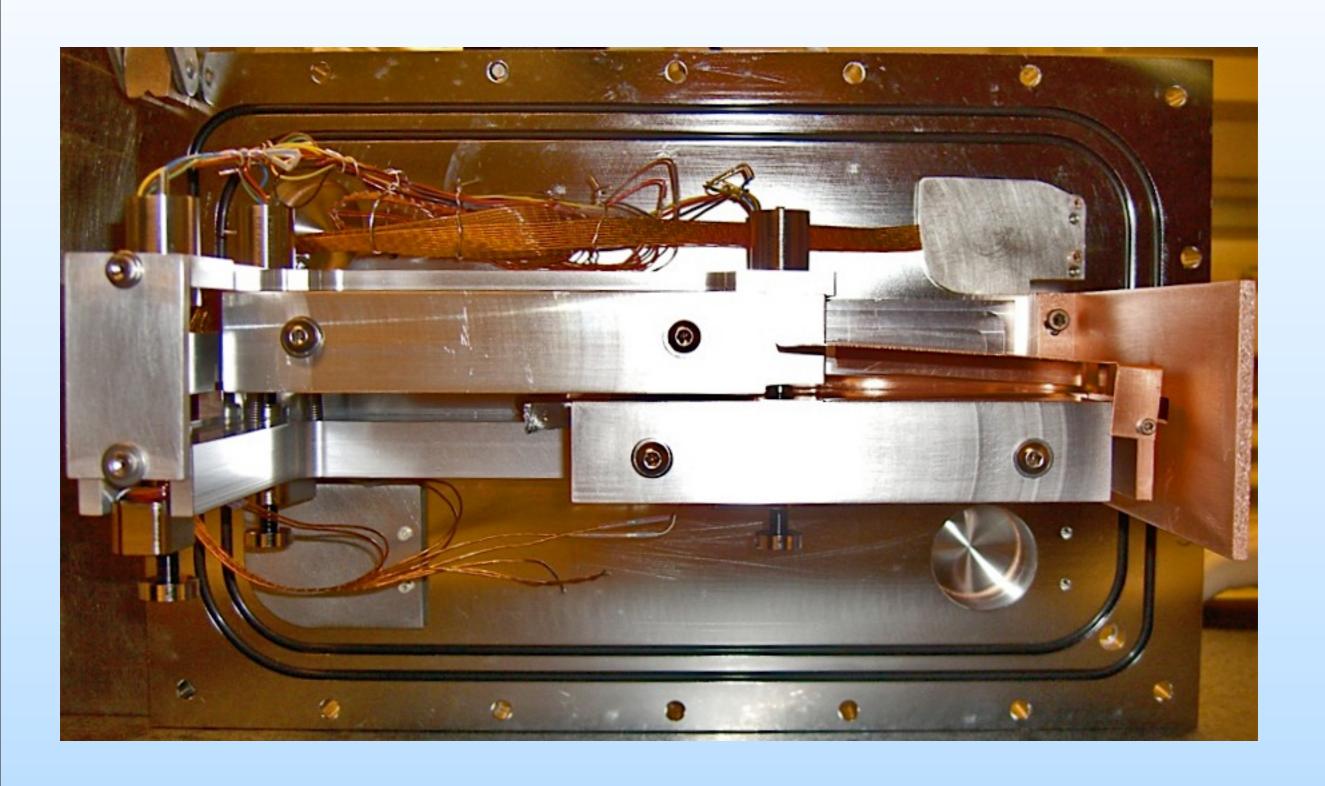


7.3.3 Beamline





Monochromator



Monochromator

Coating:

Material pair: Mo/B₄C

d-spacing: 2.0nm +/-0.1nm

N: 250

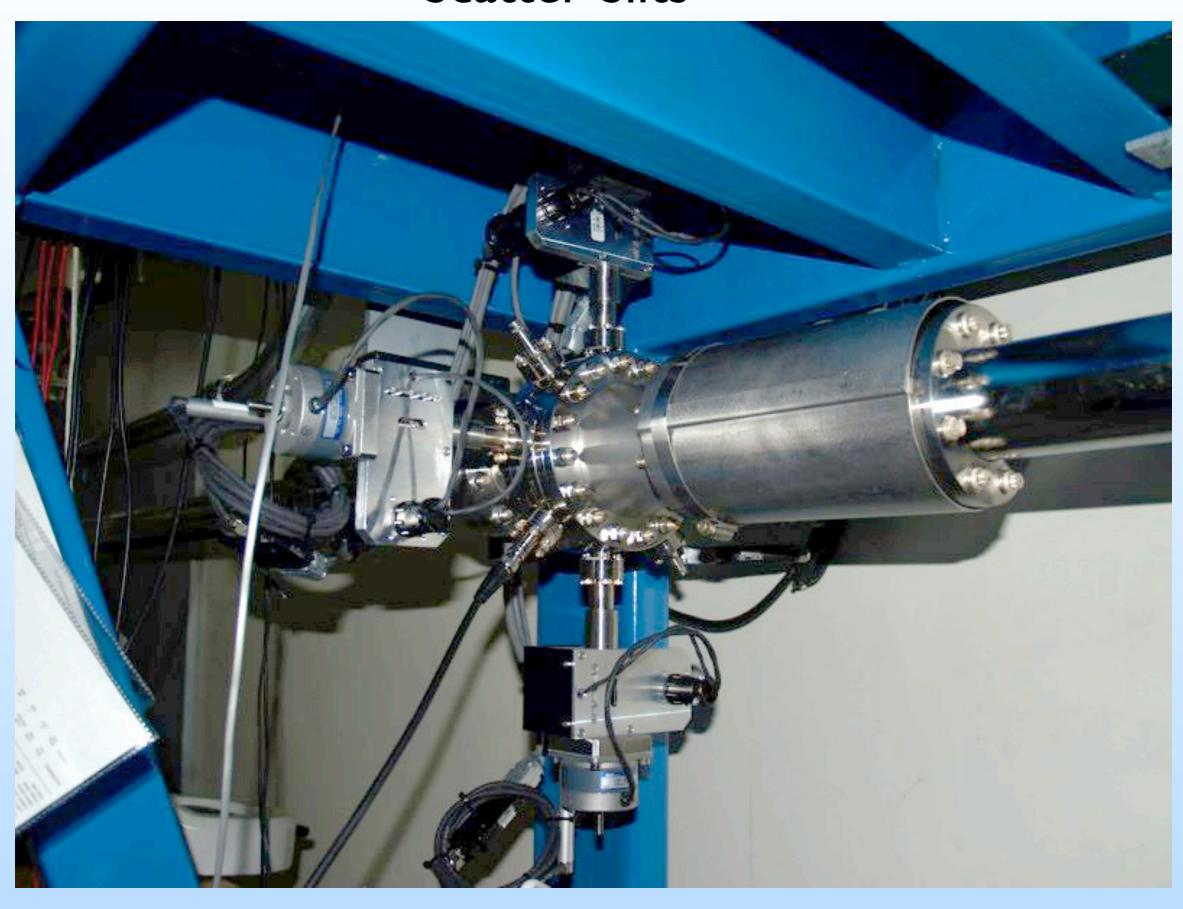
Substrates:

Material: Silicon < 100>

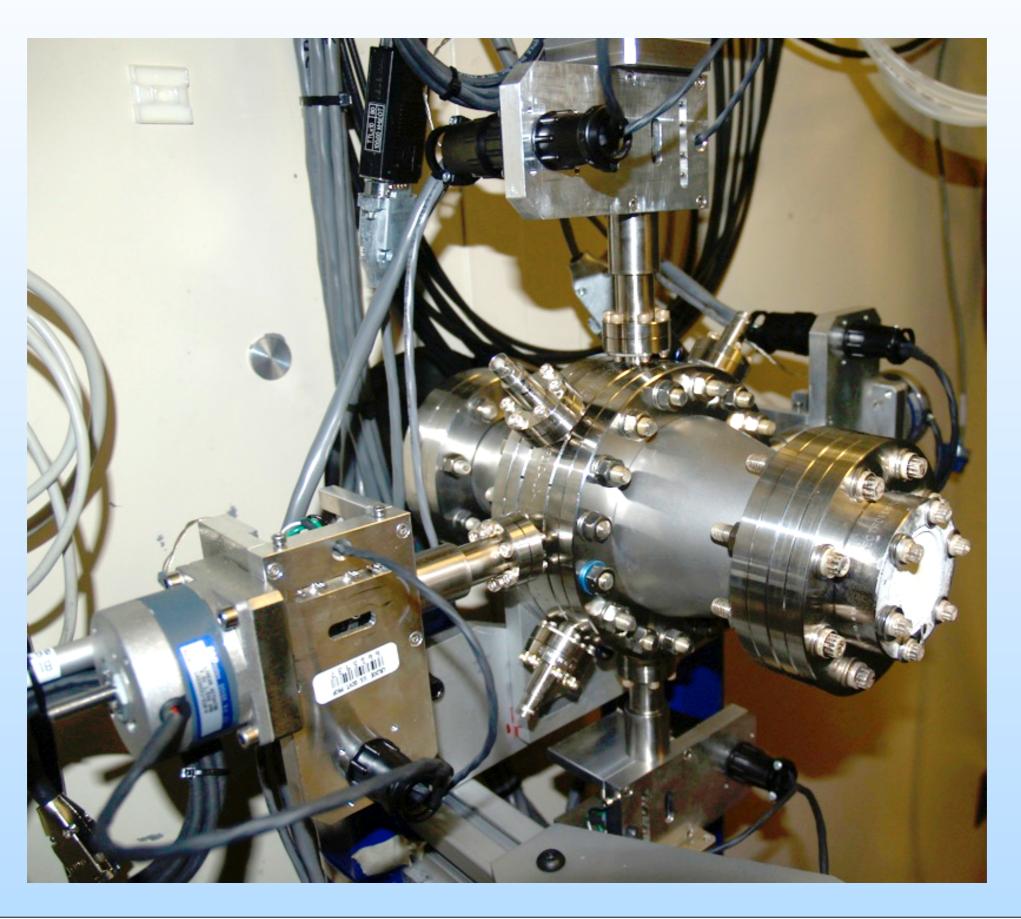
Target Energy: 10keV

 $E/\Delta E = 100$

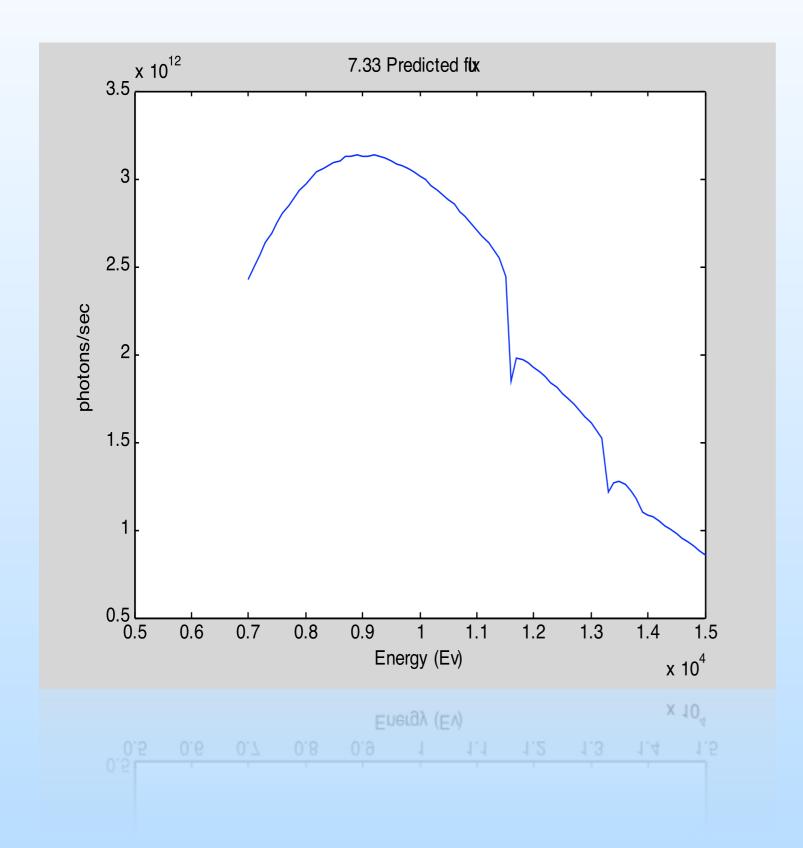
Scatter Slits



Exit Slits



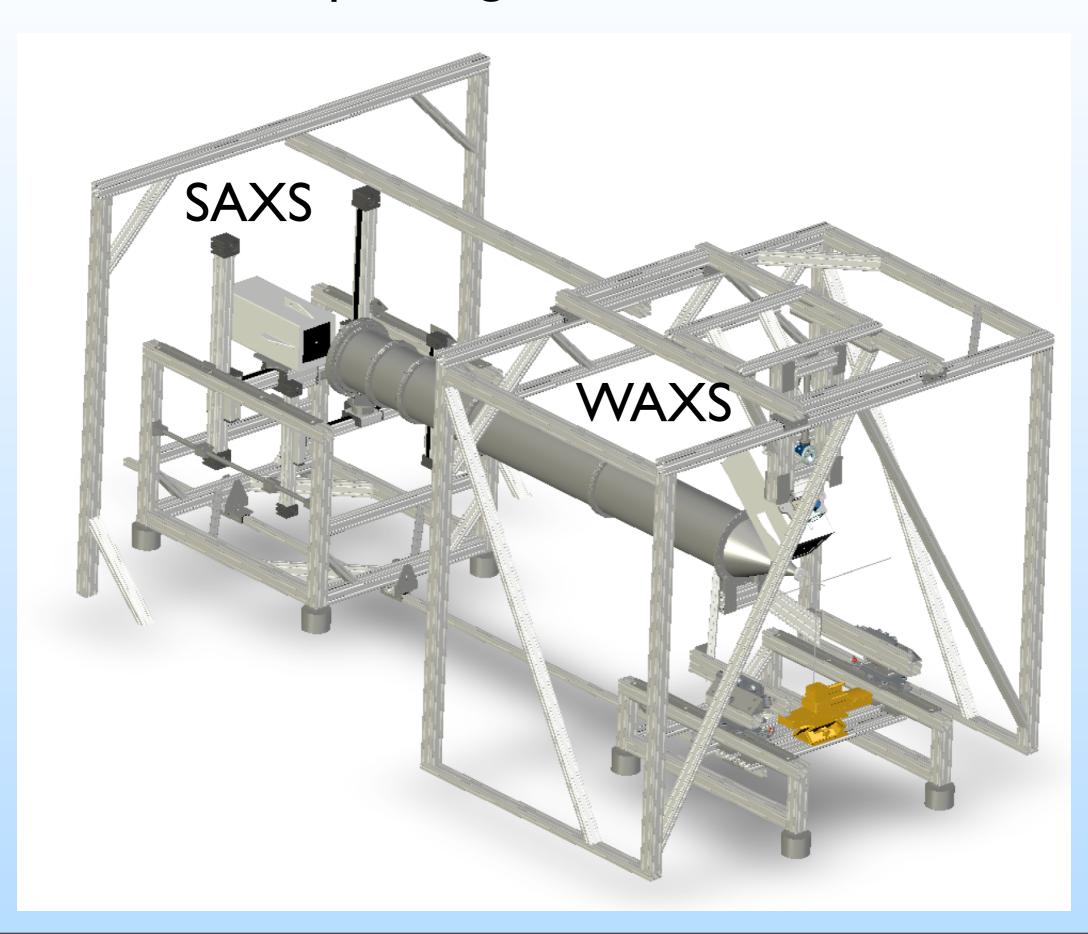
Predicted flux



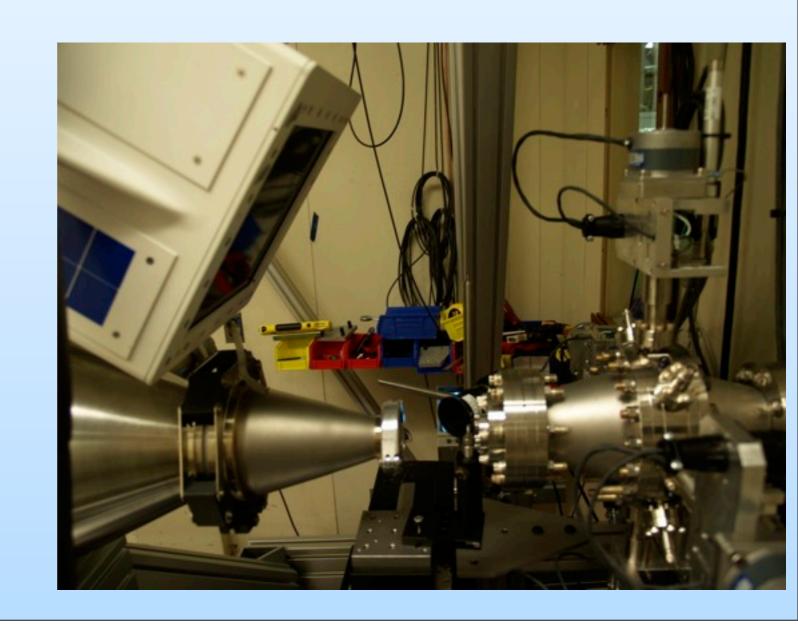
We measured 1.8 e12 ph/sec. This is 60% of predicted.

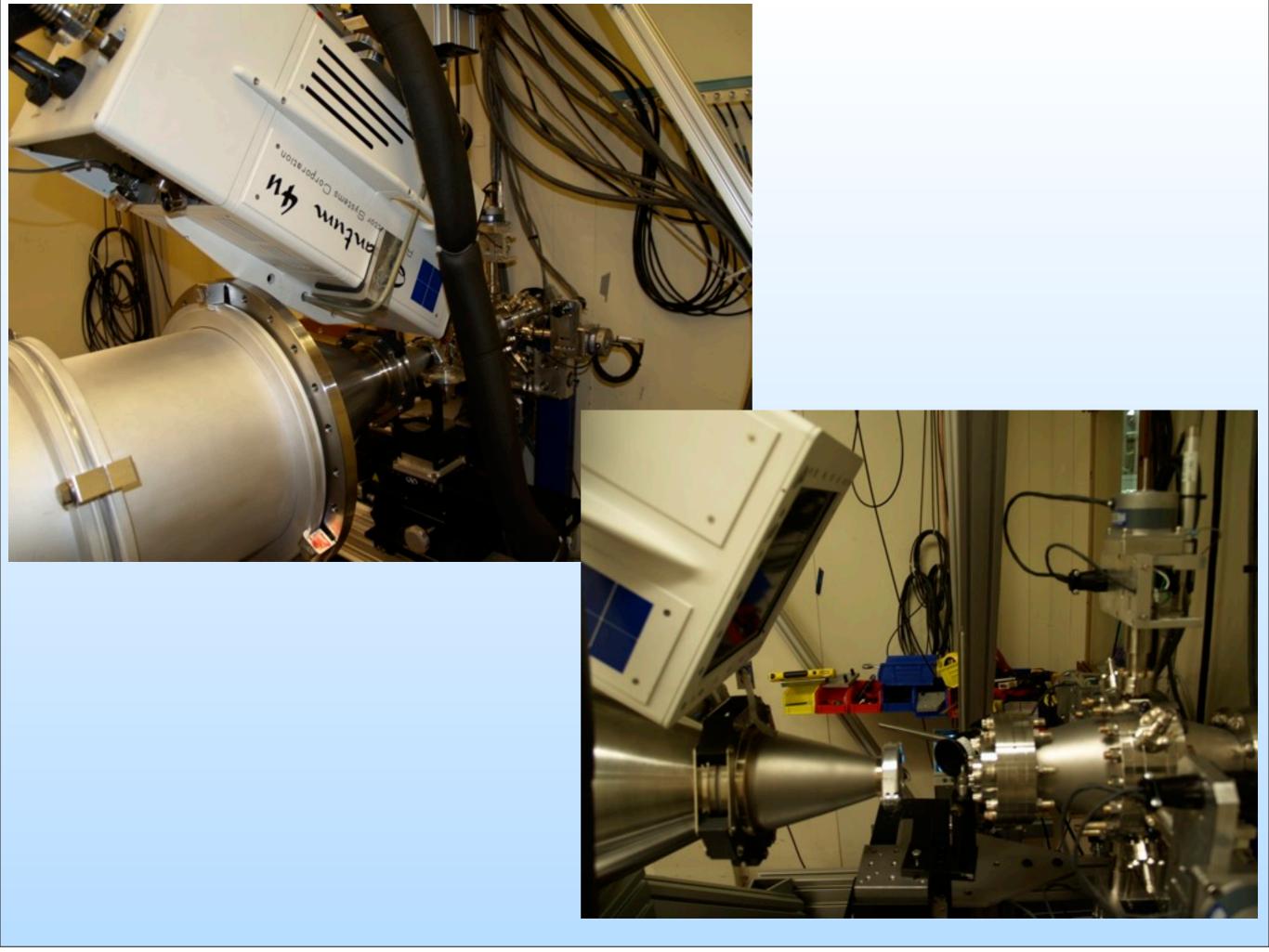
Spot size at sample position: 0.180 mm x 0.780 mm

Sample Stage and Detector





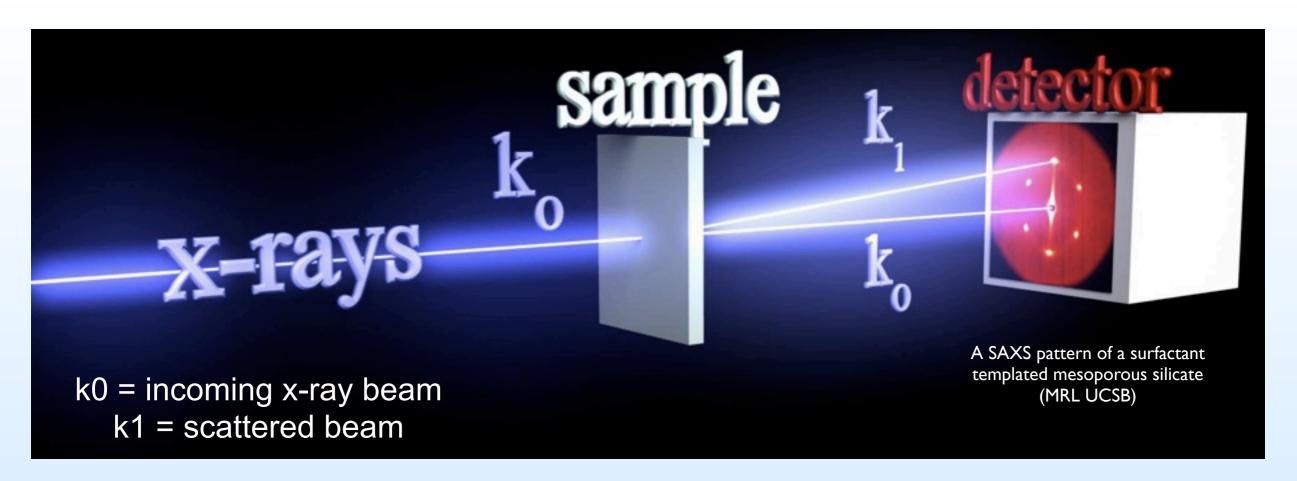




Detector Quantum 4



188mmx188mm	area
2304x2304 pixels	pixels
82um x 82um	pixels size
9 sec and 3 sec	read out time (full resolution)
I.I ADU/photon	Front End Gain
16	Bits



SAXS (CCD detector) Sample detector
distance = 0.7m - 4.3m

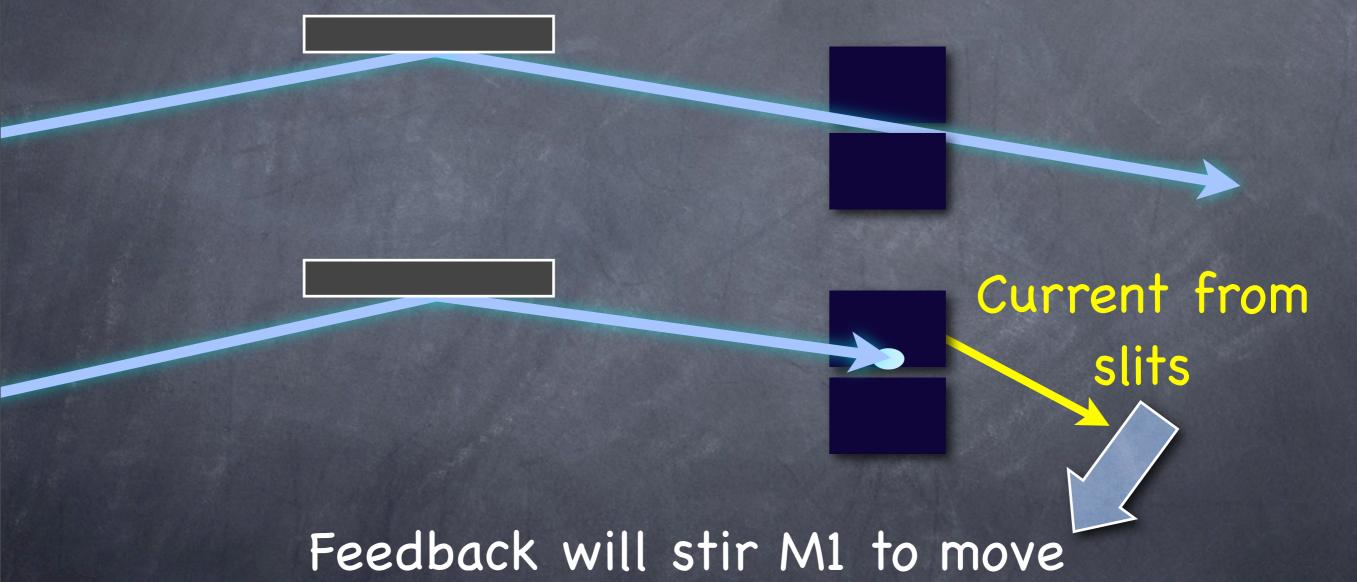
WAXS (CCD detector) Sample detector distance
= 0 m - 1m

Wavelength = 1.2389 Å (10 keV)
q-range = 0.004 Å-1 - 8.7 Å-1

d-range = 1500 Å - 0.8 Å

Feedback System

Movement of Beam due to Monochromator stirs beam into → slits to avoid this use Feedback M1 mirror Scatter slits



beam back between slits

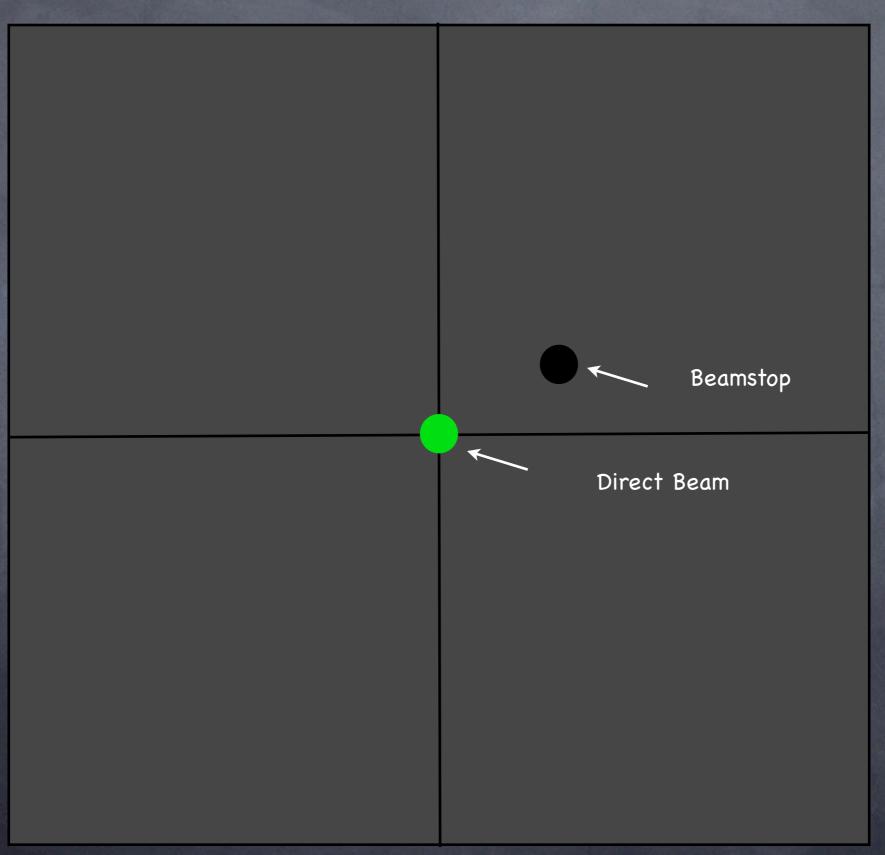
Screen blinking RED means feedback is not working

Before you get to the BL

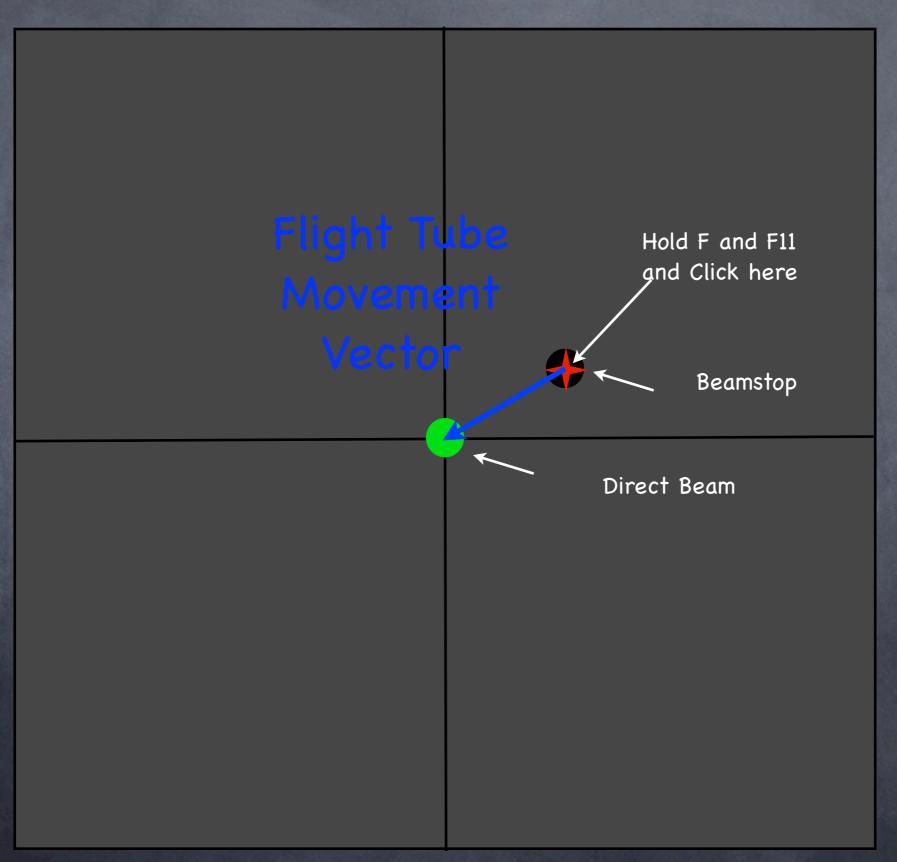
- Fill out ESS
- Make sure everyone is an ALS user
- Take the JHQ and all training you will need including Rad safety, and Hoist, as well as anything else you will use (ie laser, cryo etc)
- Calculate q-range and sample detector distance for 10keV
- Plan experiment ahead: GISAXS then SAXS etc. (call us before)

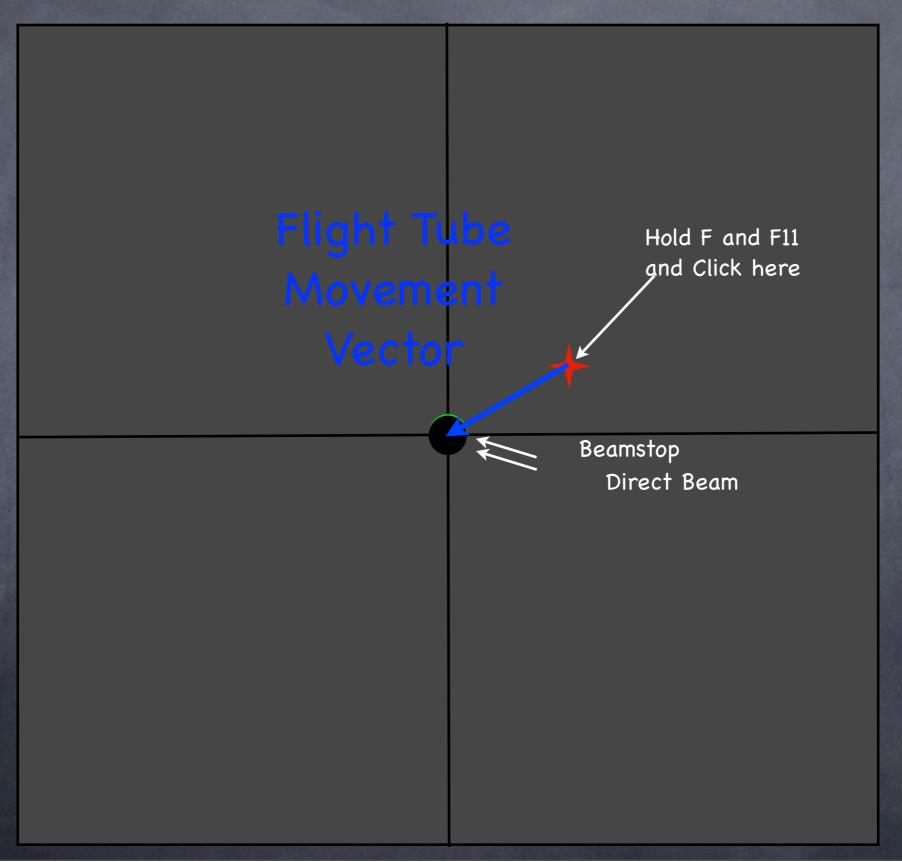
When you get to the BL

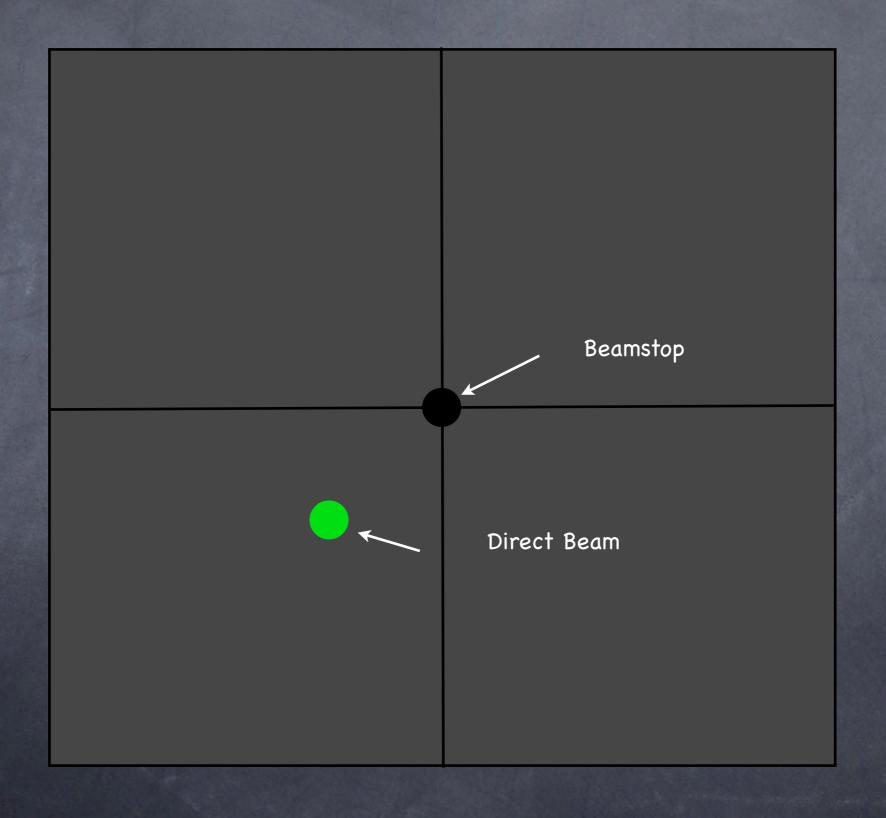
- @ Call or Email us, check beam status
- Check that previous user is done and cleaned up, if not call us
- Set Flight tube
 - Set Length, check beam path is unobstructed
 - Align beamstop (might have to change actual beamstop)
- Set up sample holder (DSC, SAXS, WAXS, GISAXS, Gas)

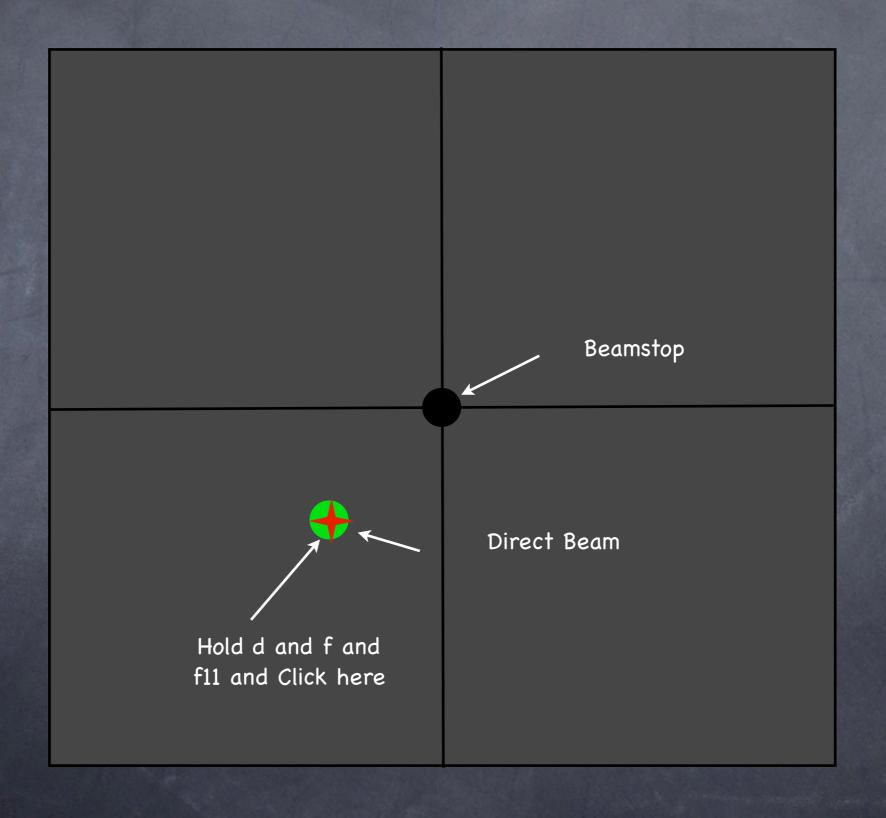


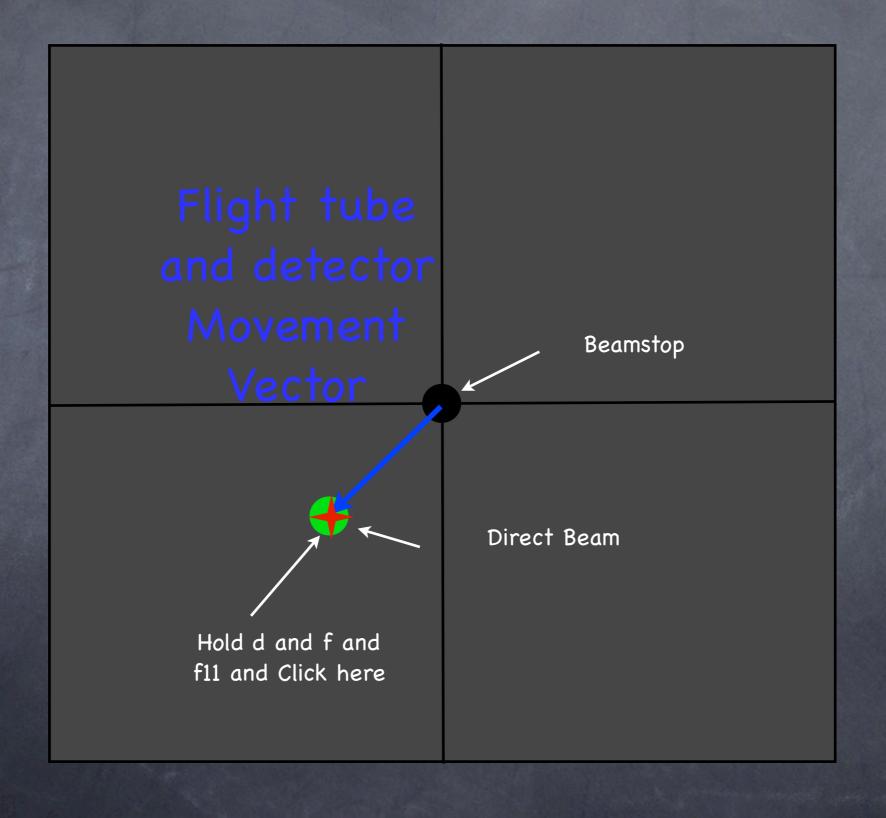


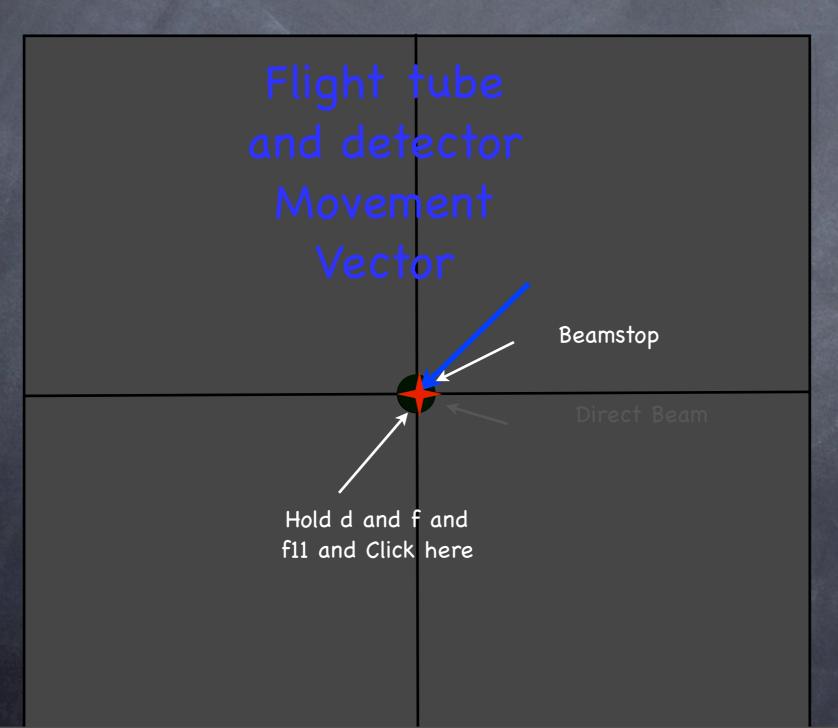


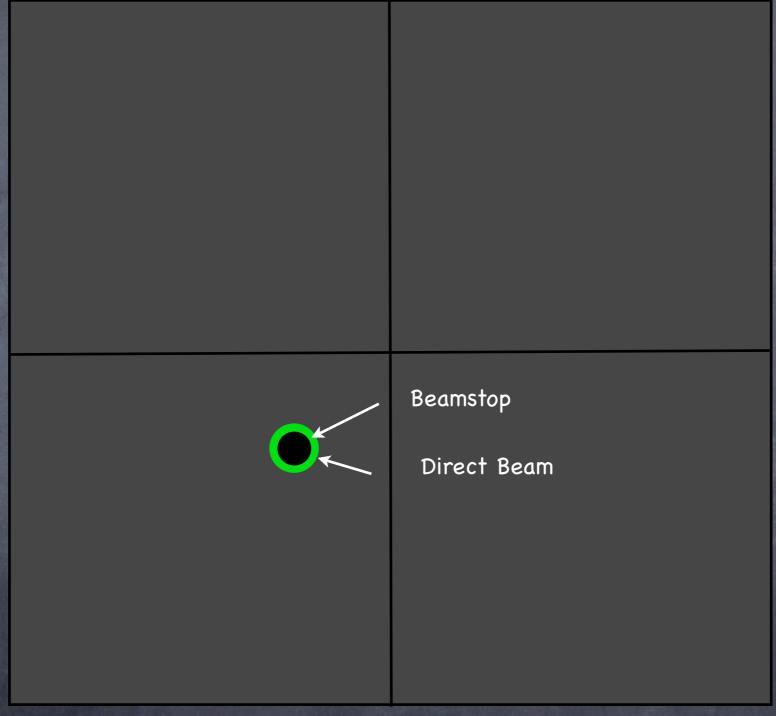


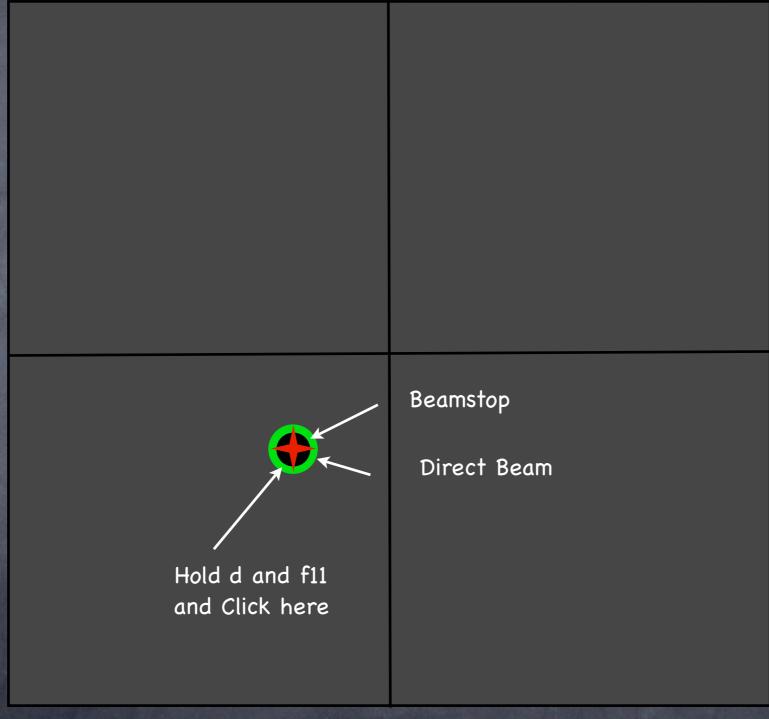


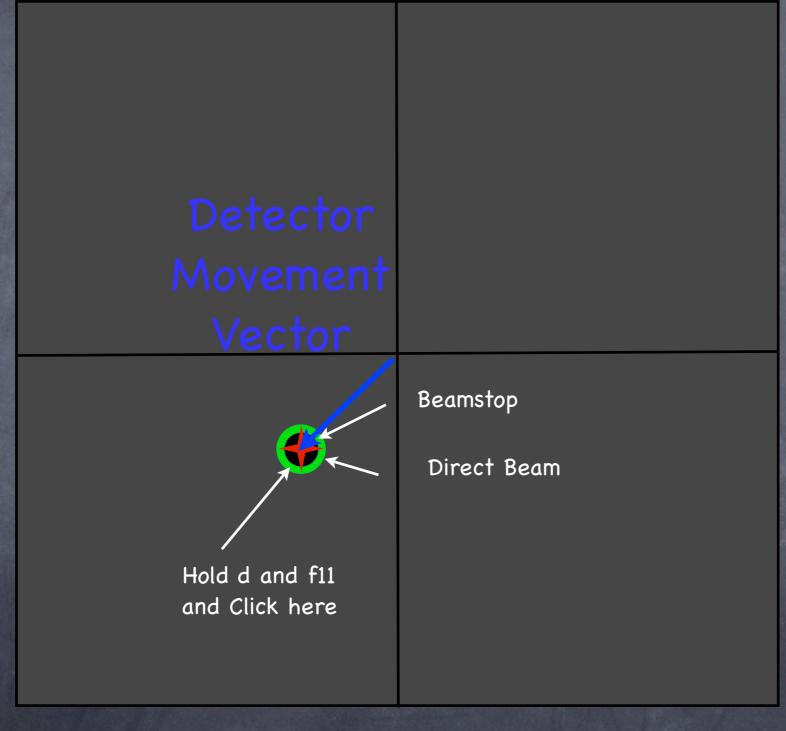


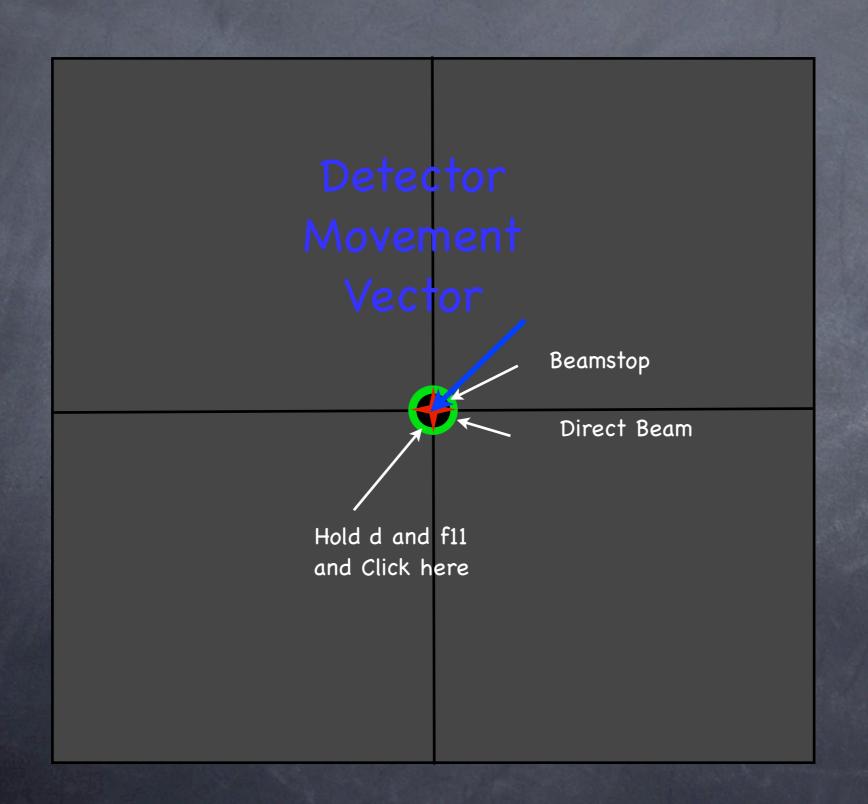










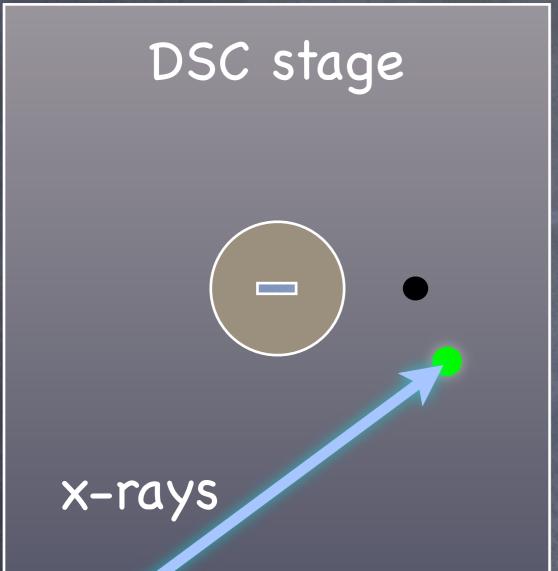




RISKS

- There is High Risk of messing this up and hurting the detectors!
- Don't trust that the program or the motors moved correctly
- Assume the worst and DOUBLE CHECK that detectors are safe
- ALWAYS check beamstop at least once (more if you are nervous) before taking any pictures

DSC stage alignment



alignment spot (black)

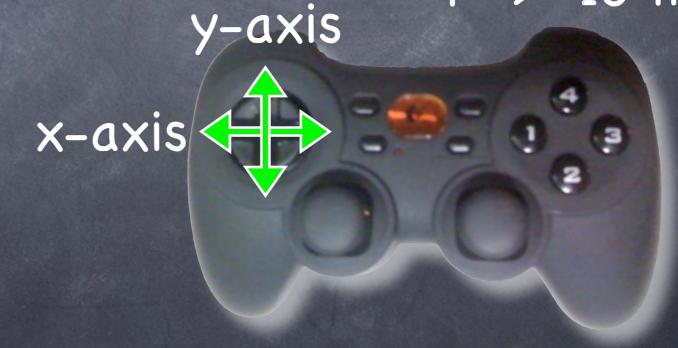
step size

1 -> 0.3 mm

2 -> 1 mm

3 -> 3 mm

4 -> 10 mm



DSC stage alignment

DSC stage x-rays

alignment spot (black)

step size

1 -> 0.3 mm

2 -> 1 mm

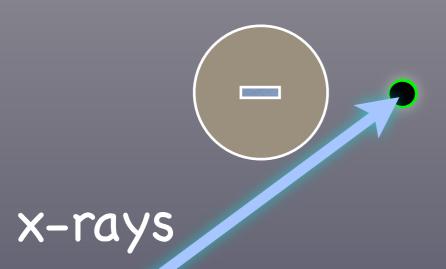
3 -> 3 mm

4 -> 10 mm



DSC stage alignment

DSC stage



alignment spot (black)

step size

1 -> 0.3 mm

2 -> 1 mm

3 -> 3 mm

4 -> 10 mm

y-axis



DSC stage alignment

DSC stage spot (black)

step size

1 -> 0.3 mm

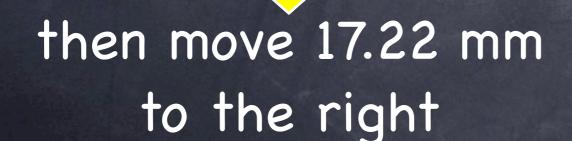
2 -> 1 mm

3 -> 3 mm

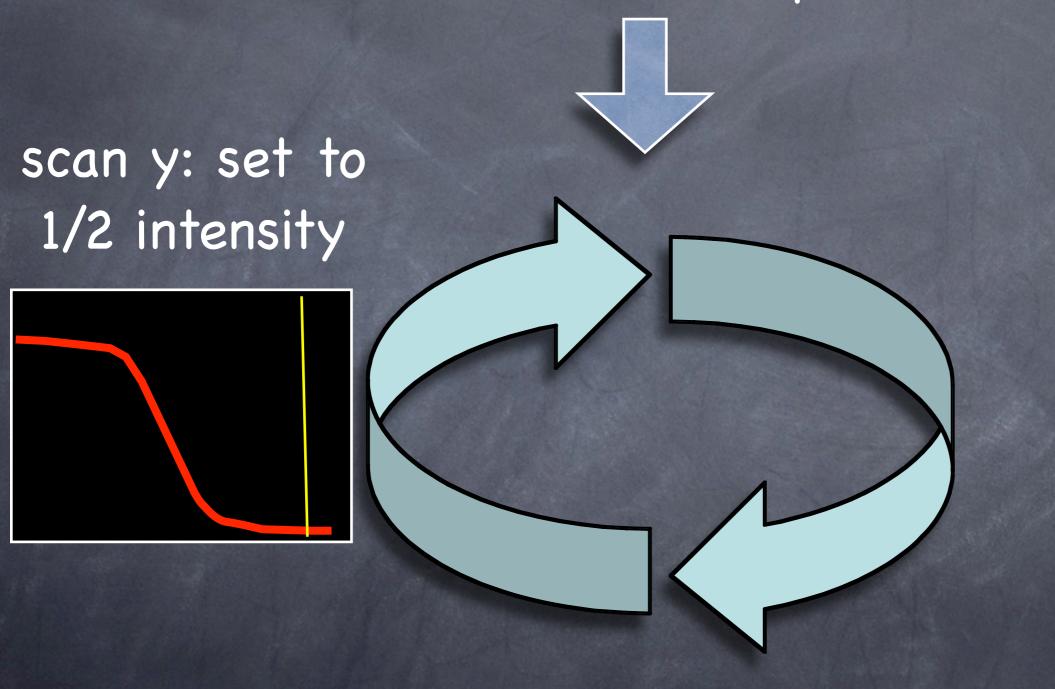
4 -> 10 mm

y-axis

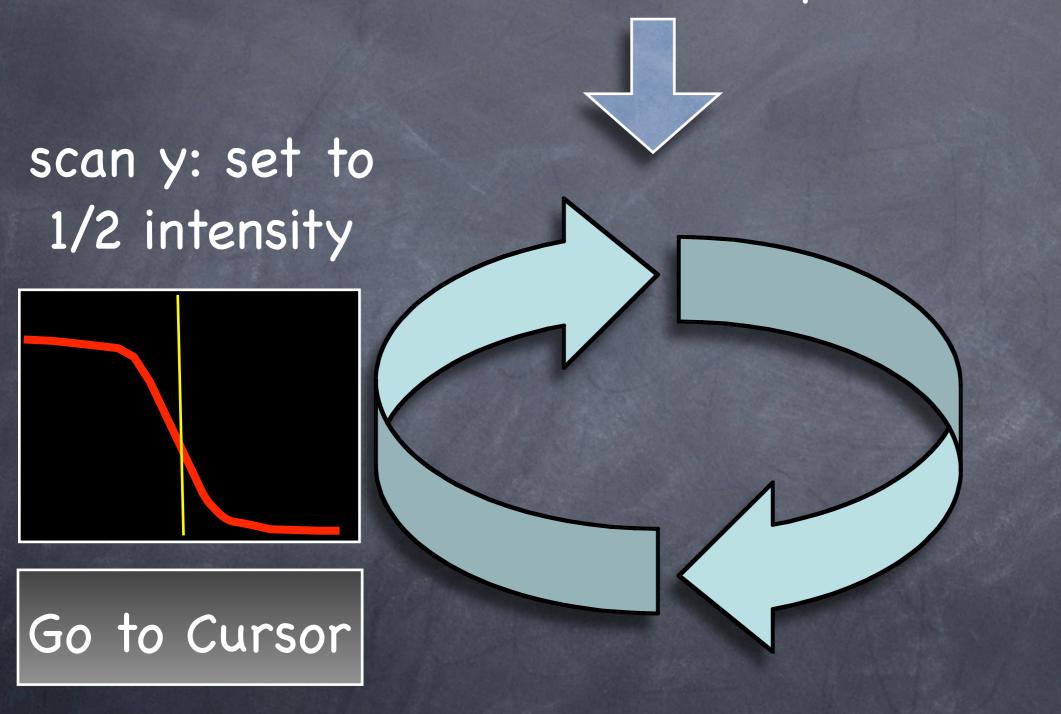
x-axis

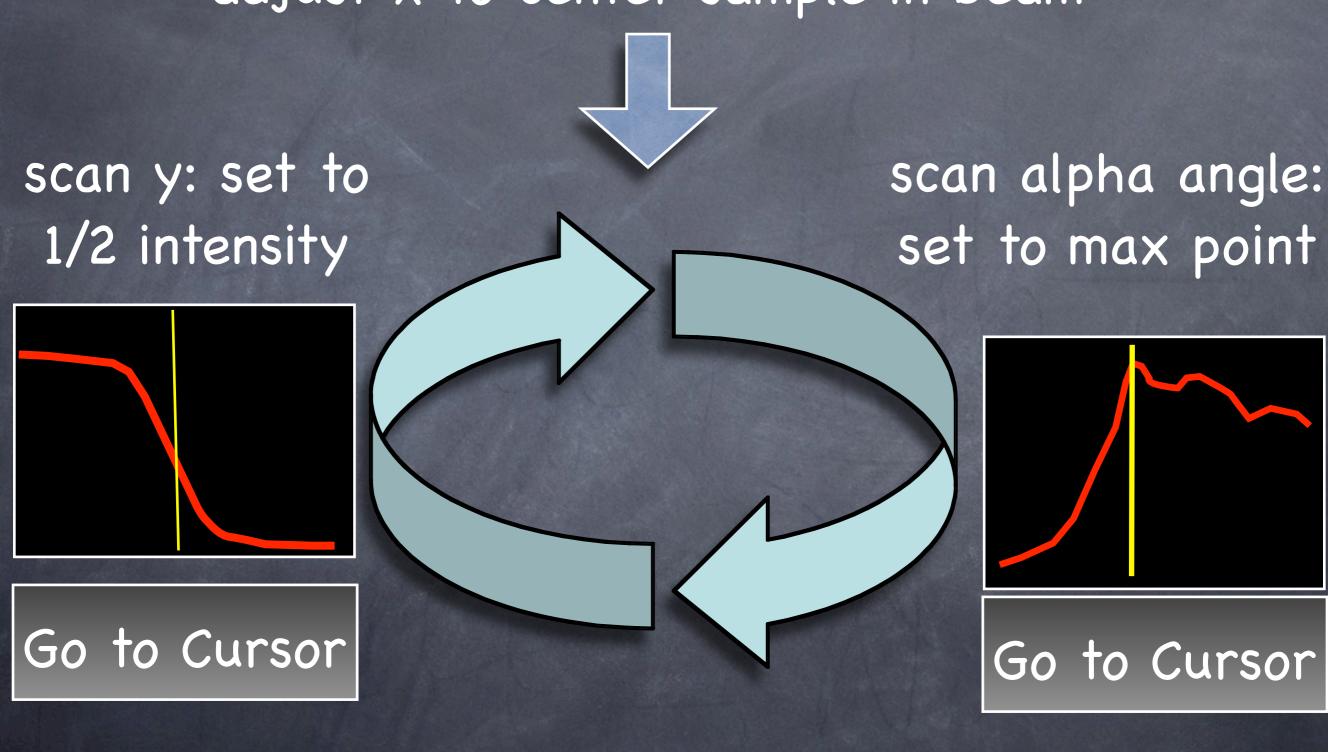


x-rays

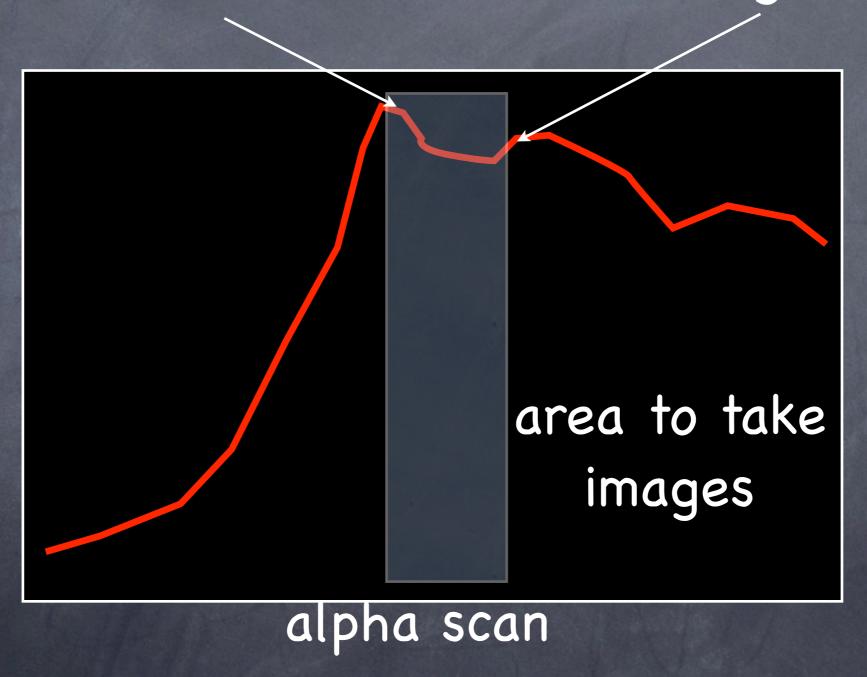




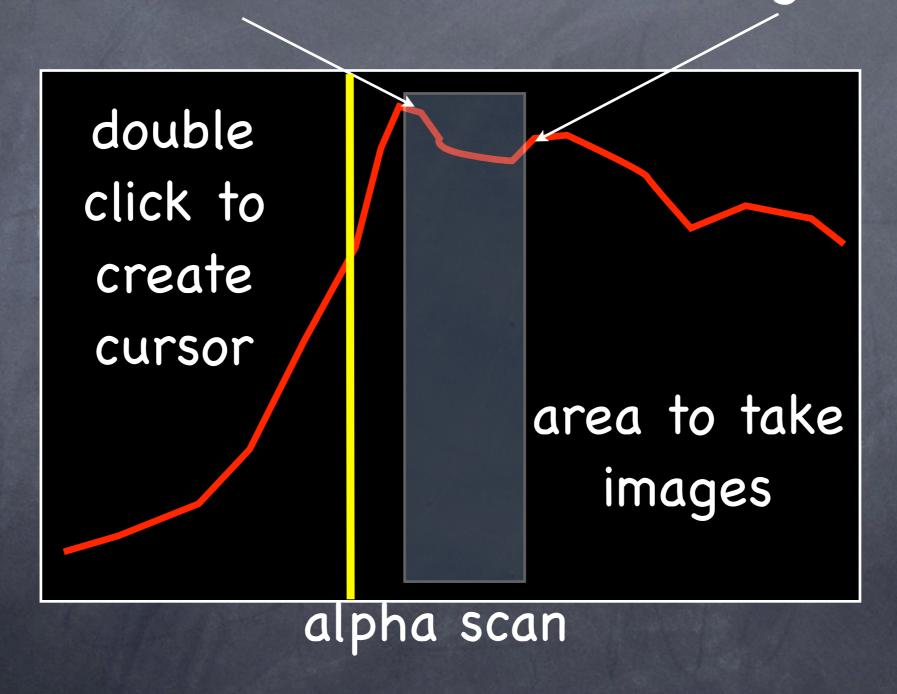




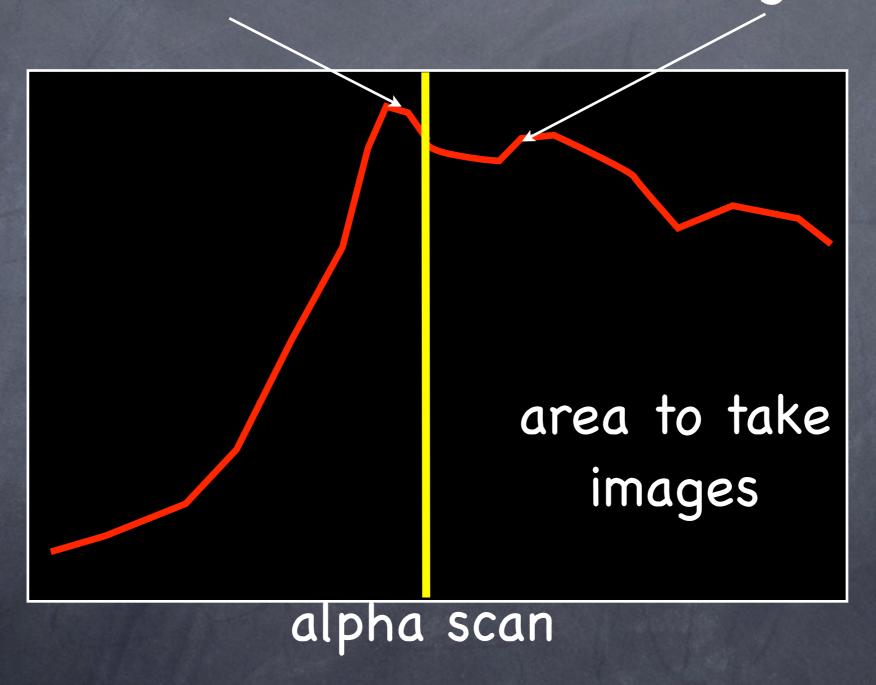
critical angle polymer



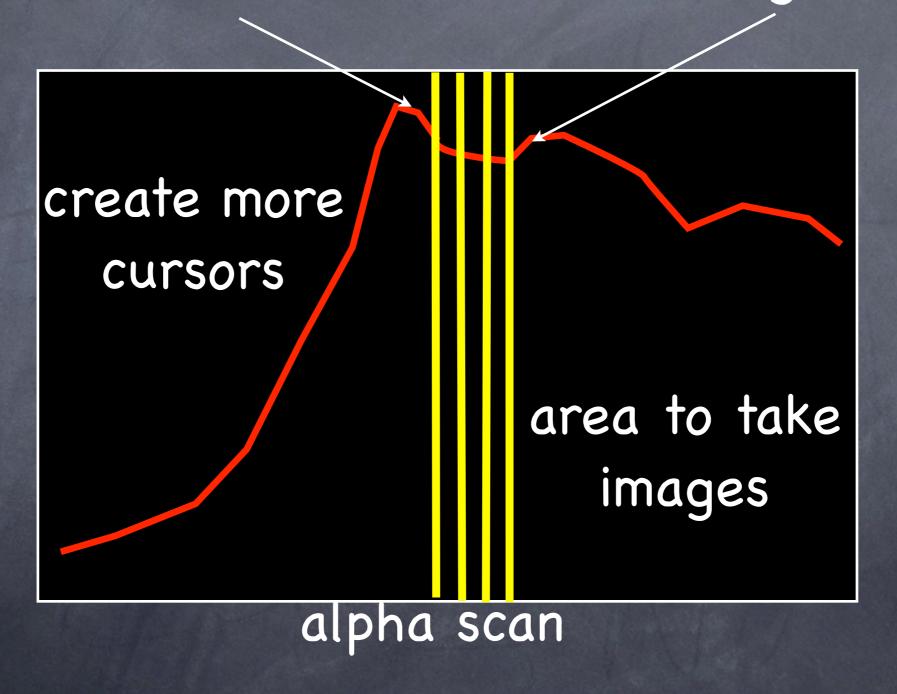
critical angle polymer



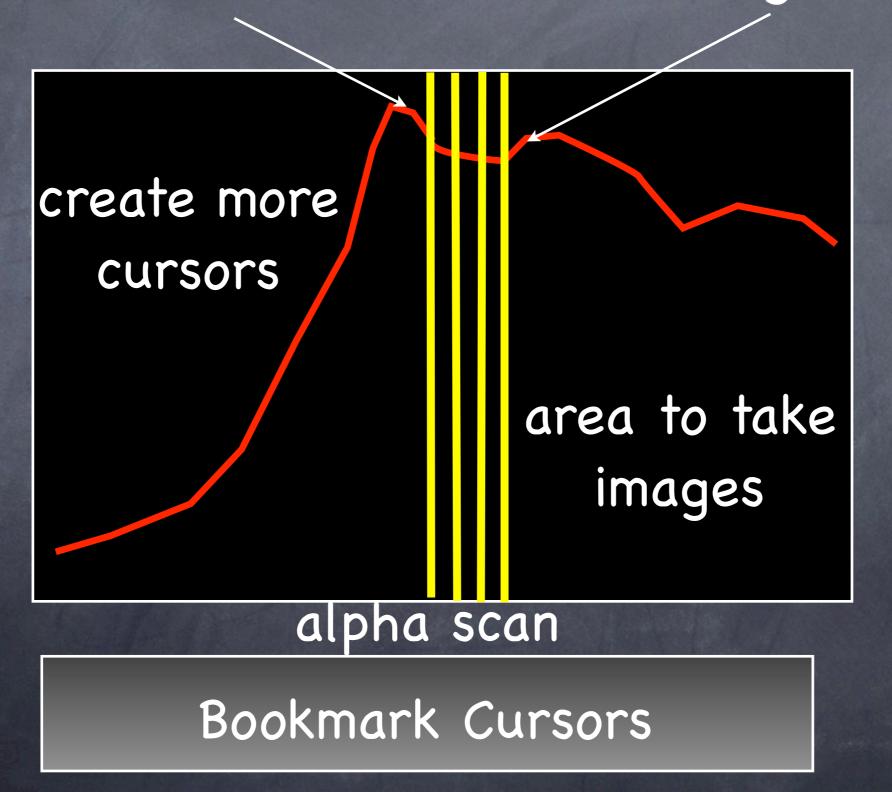
critical angle polymer

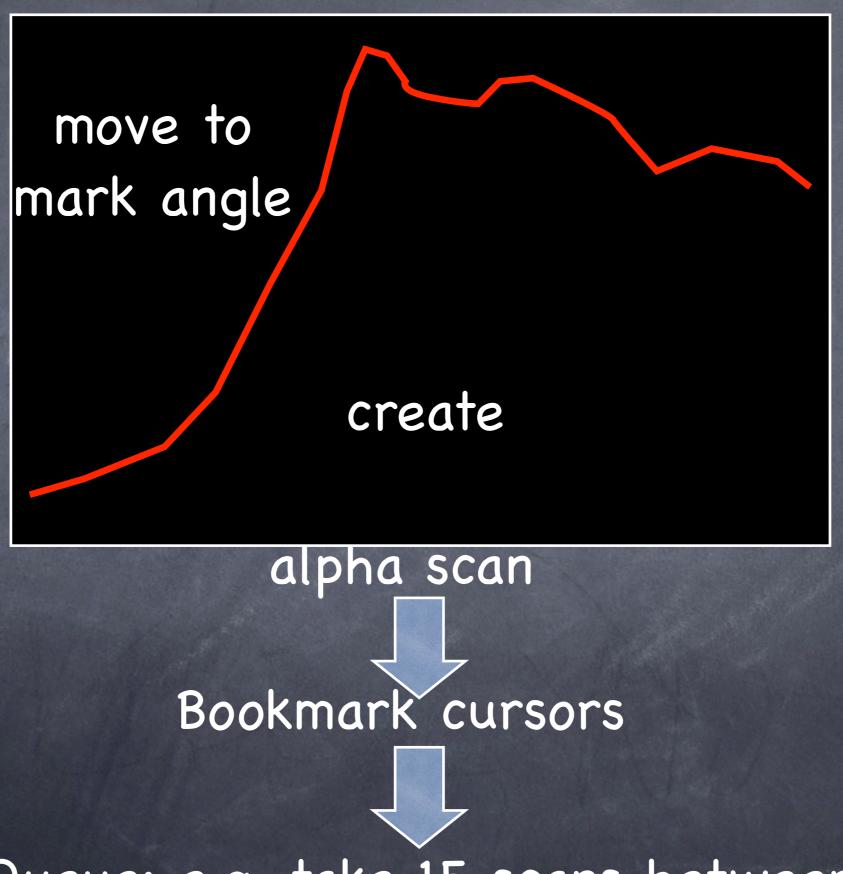


critical angle polymer

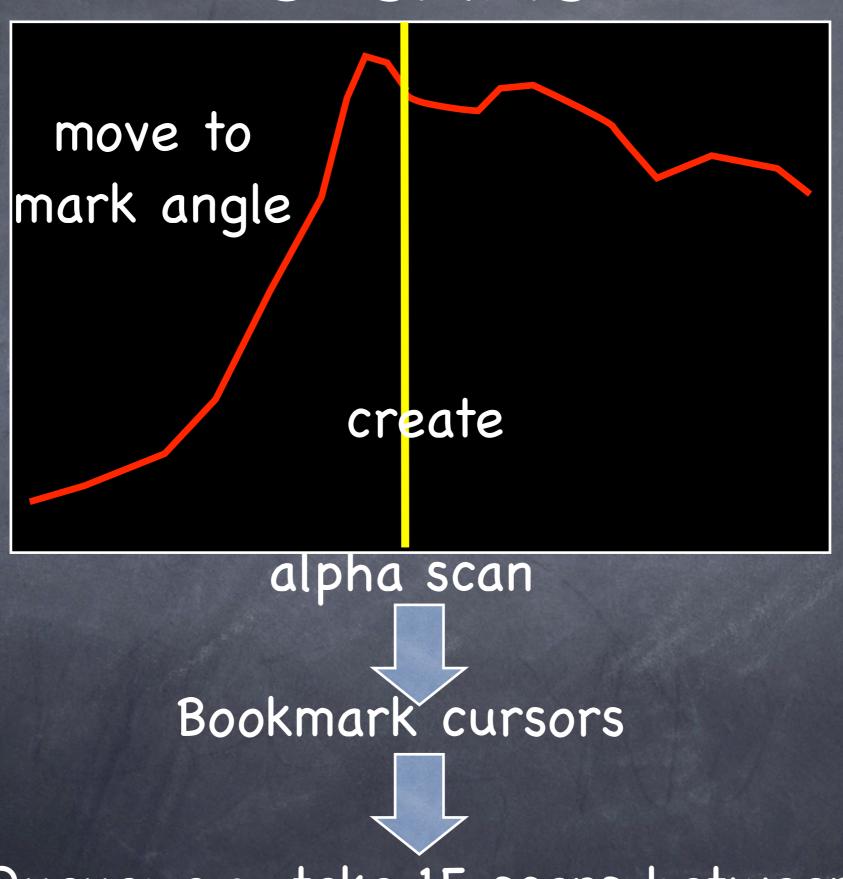


critical angle polymer

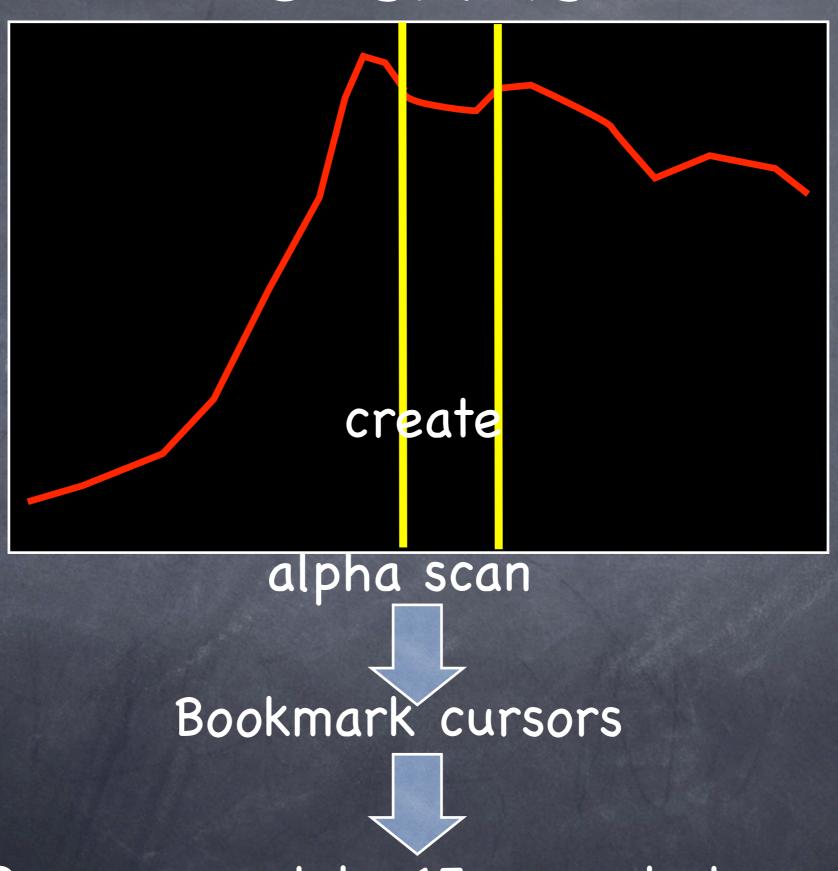




Create Queue: e.g. take 15 scans between cursors



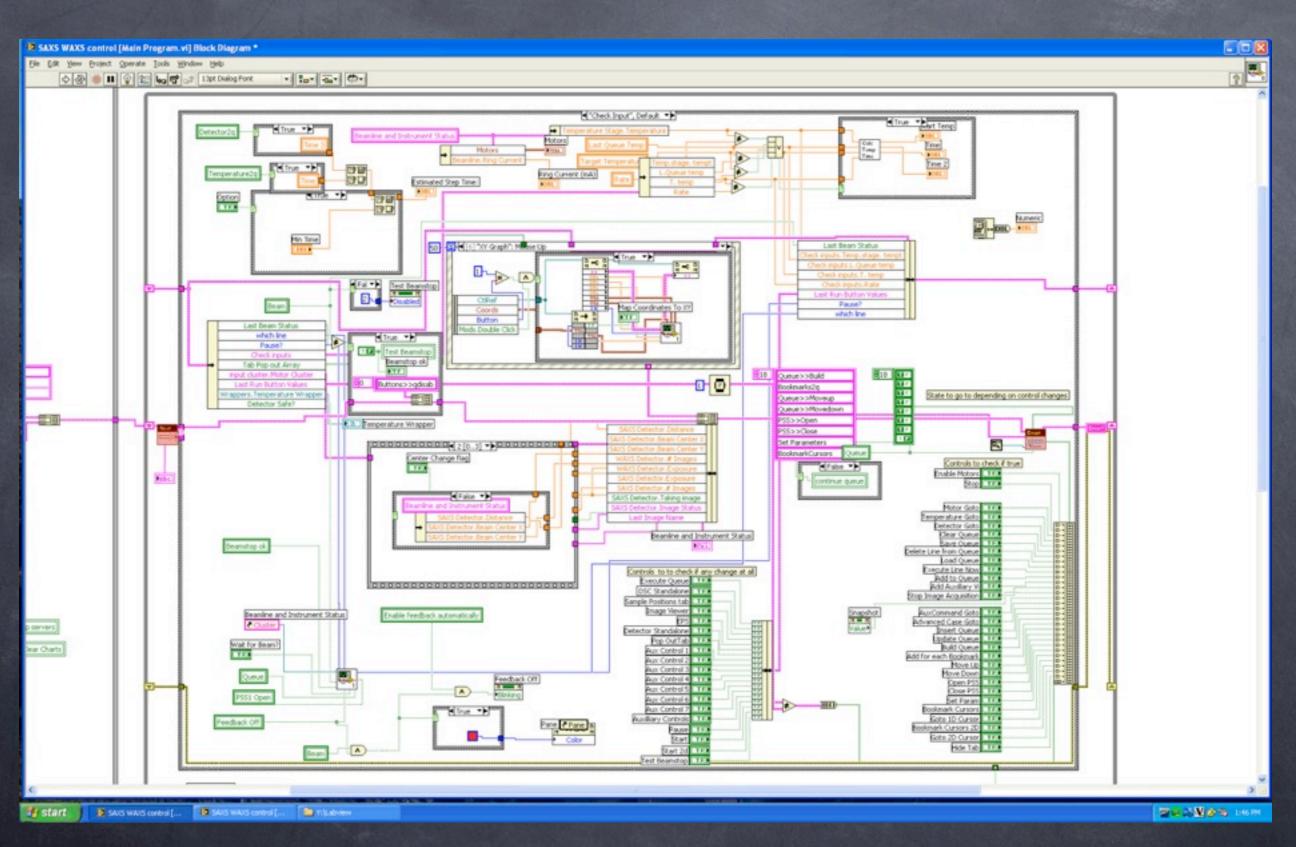
Create Queue: e.g. take 15 scans between cursors

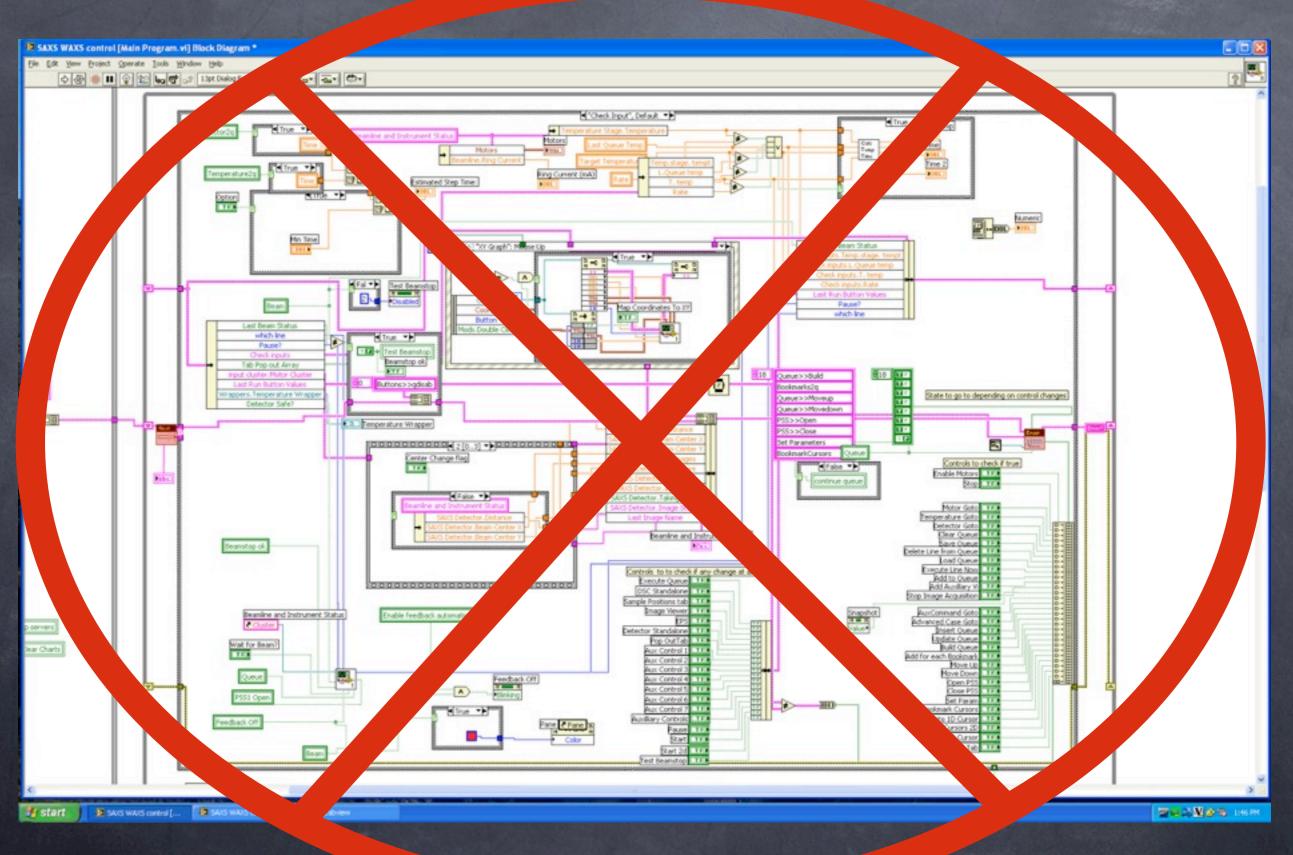


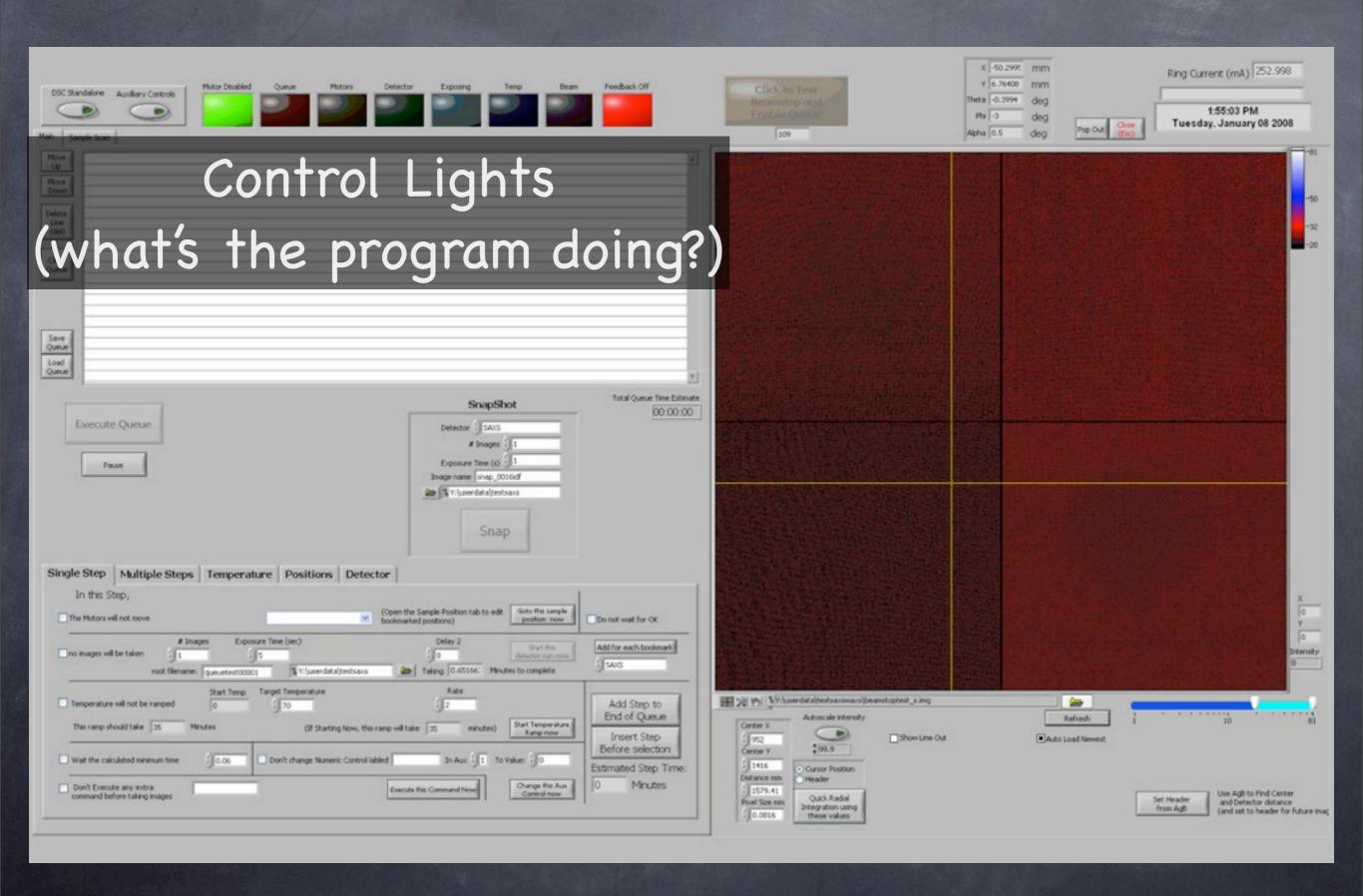
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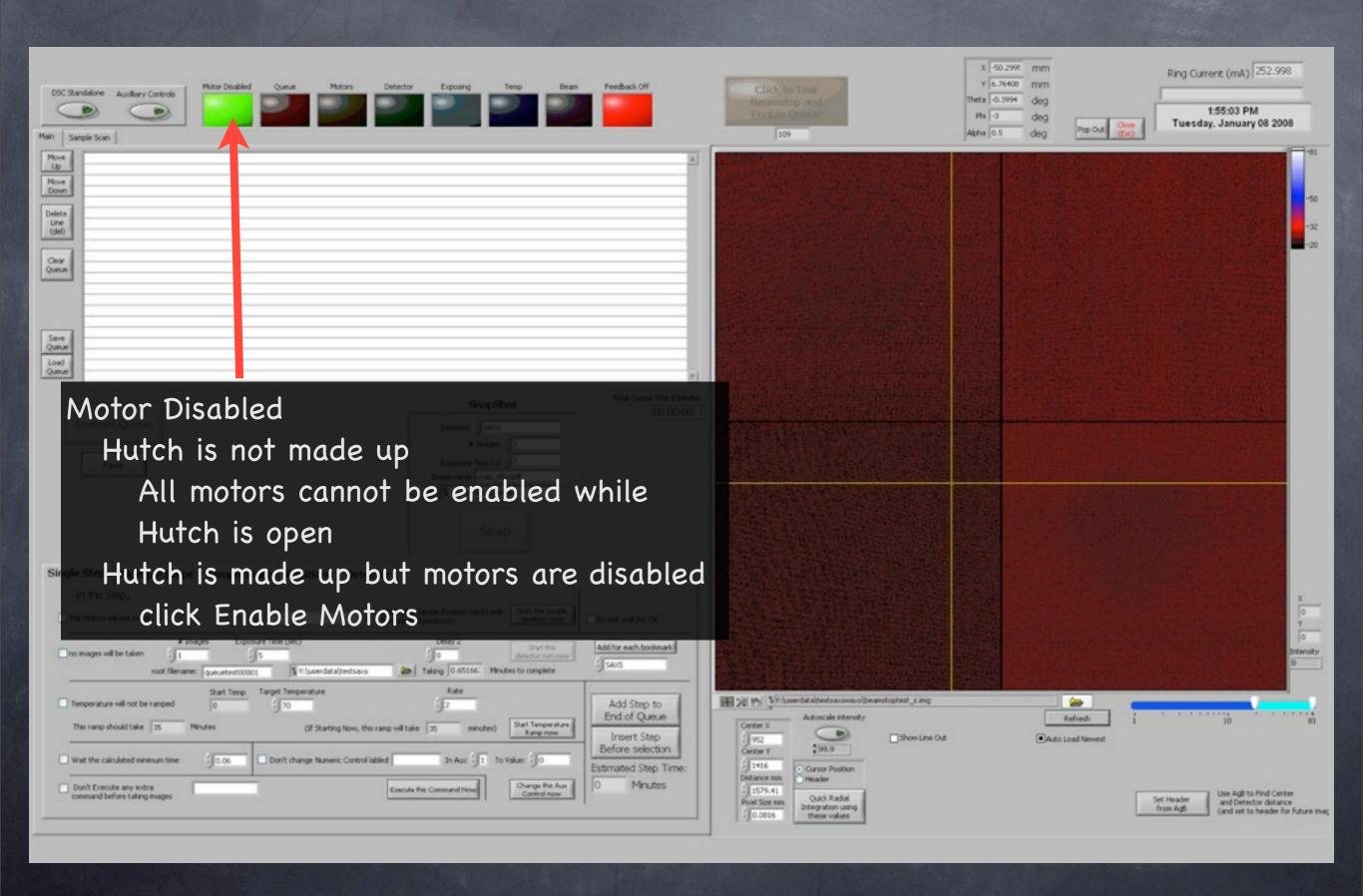
Performing Your Experiment

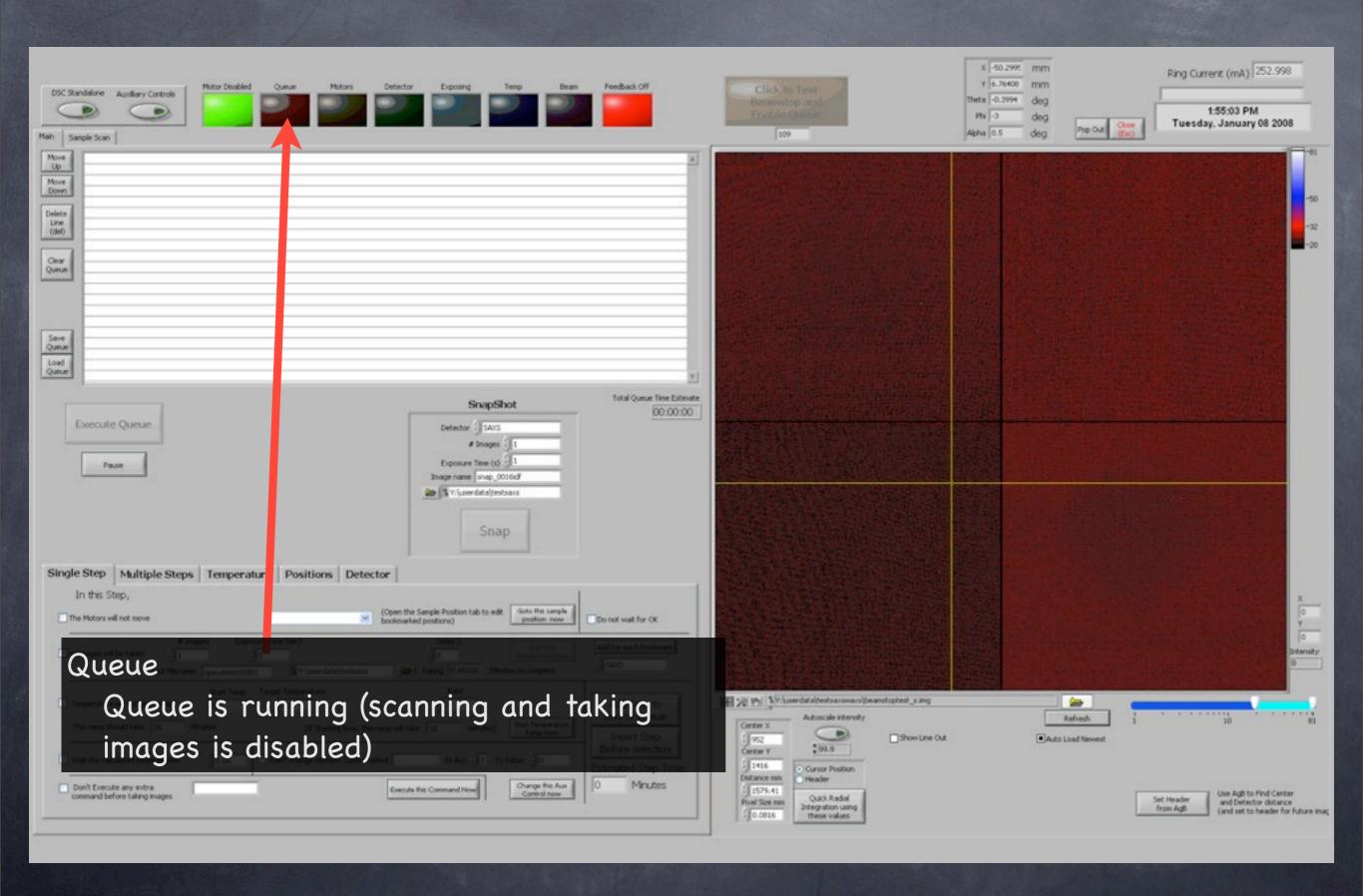
- Software
- When something unexpected happens
- Take Calibration!!! update header every time detector or flight tube might have moved
- Take Empty Cell
- Typical Experiments
 - SAXS
 - DSC SAXS
 - GISAXS
 - SAXS/WAXS

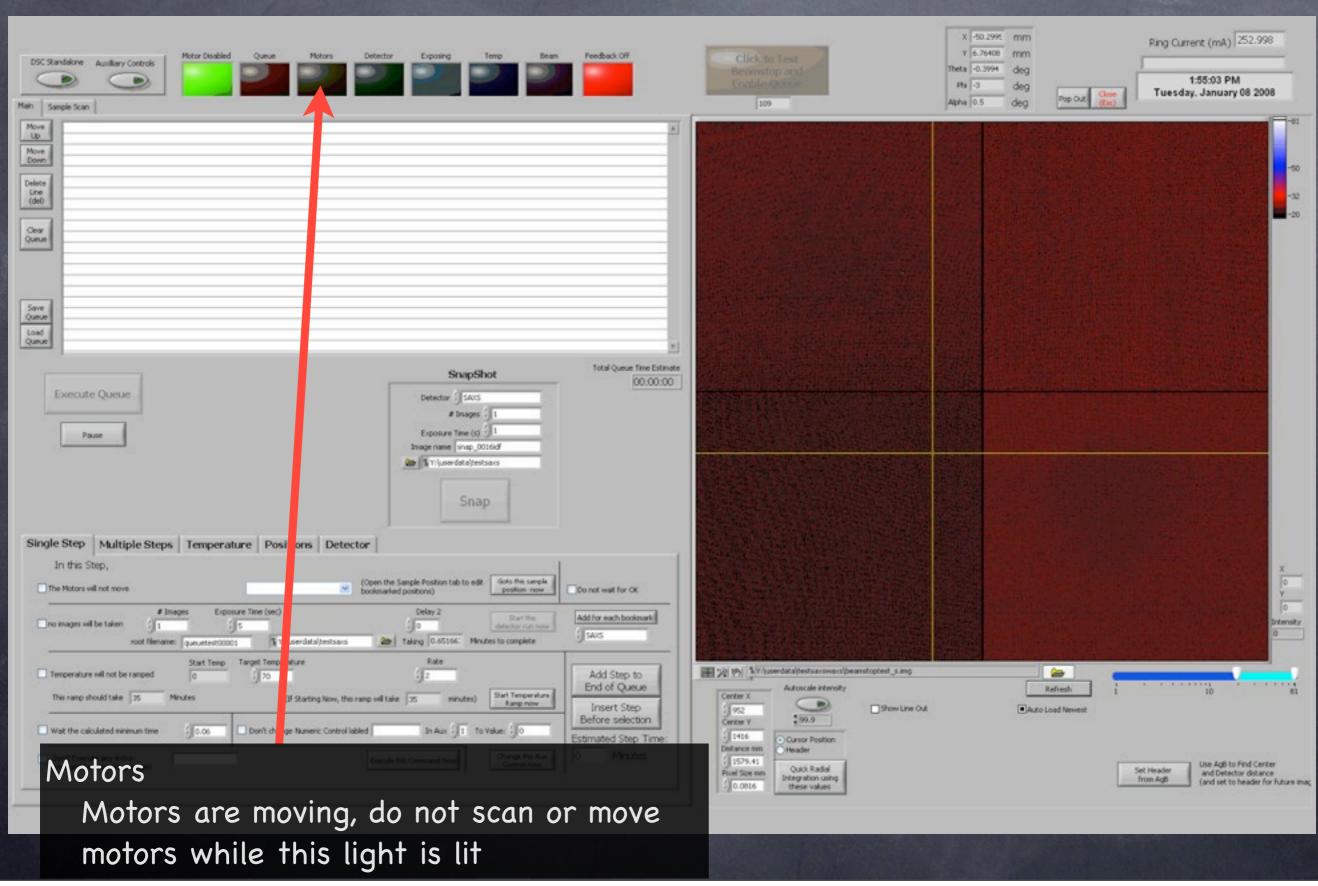


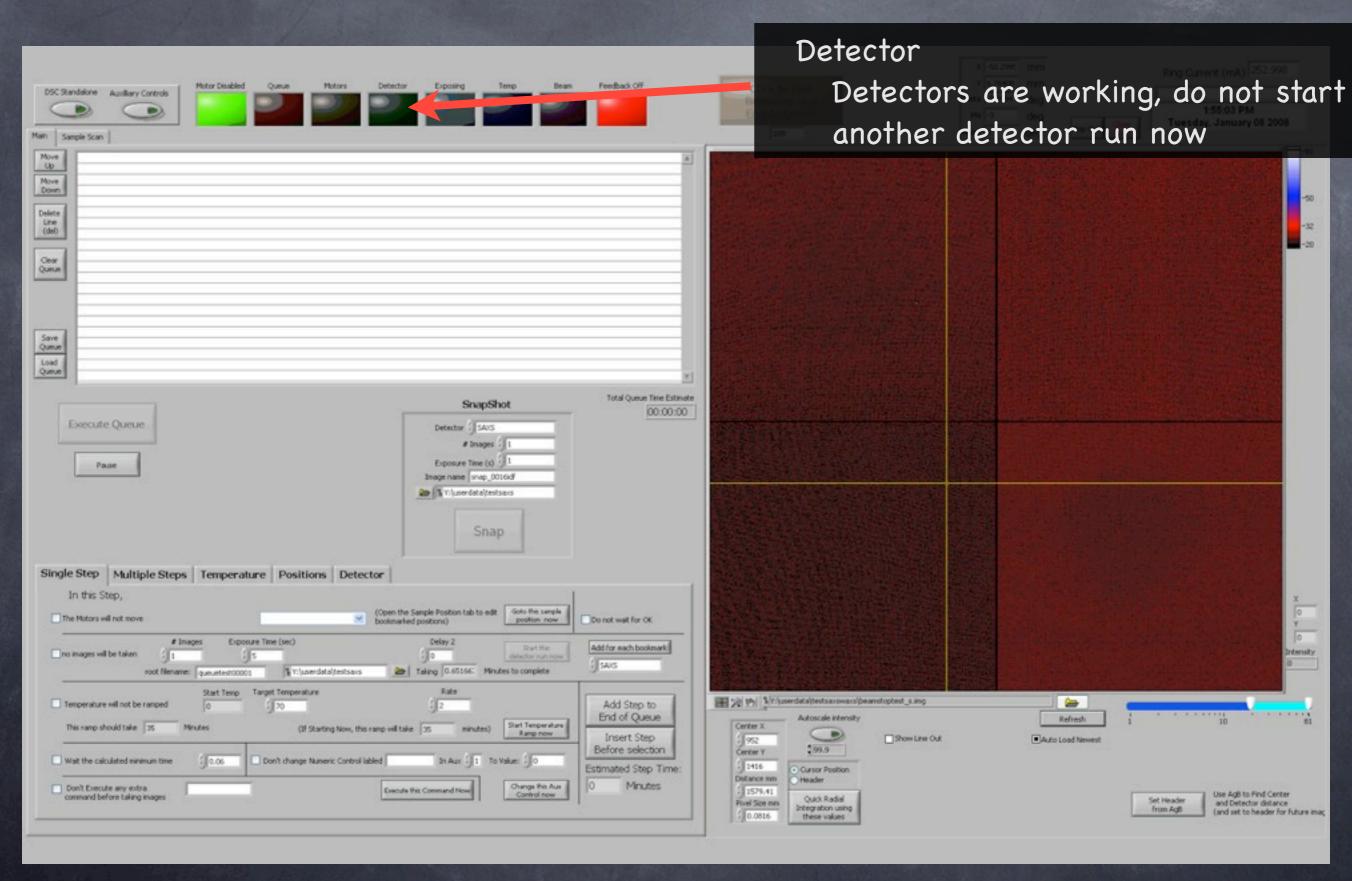


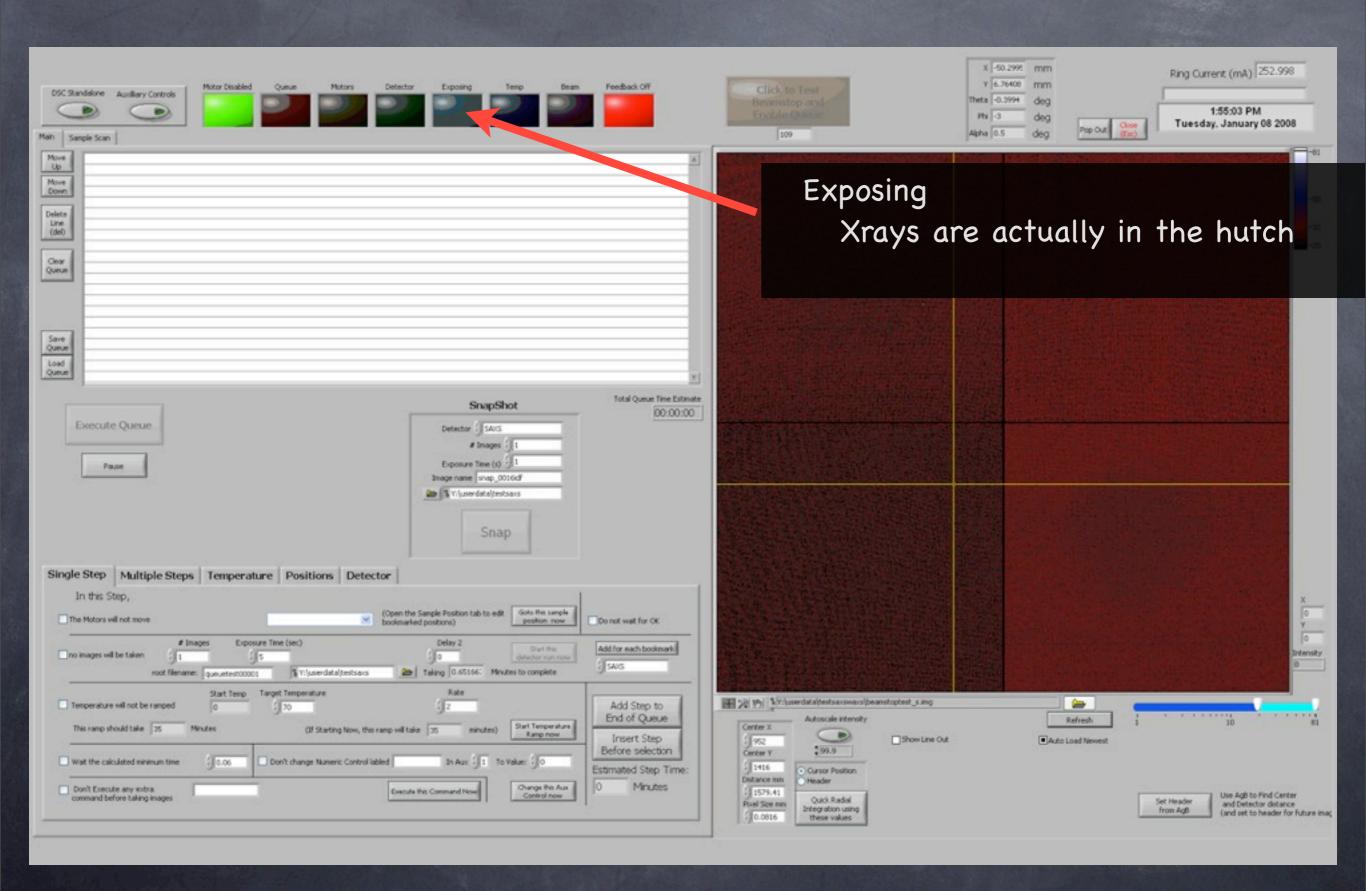


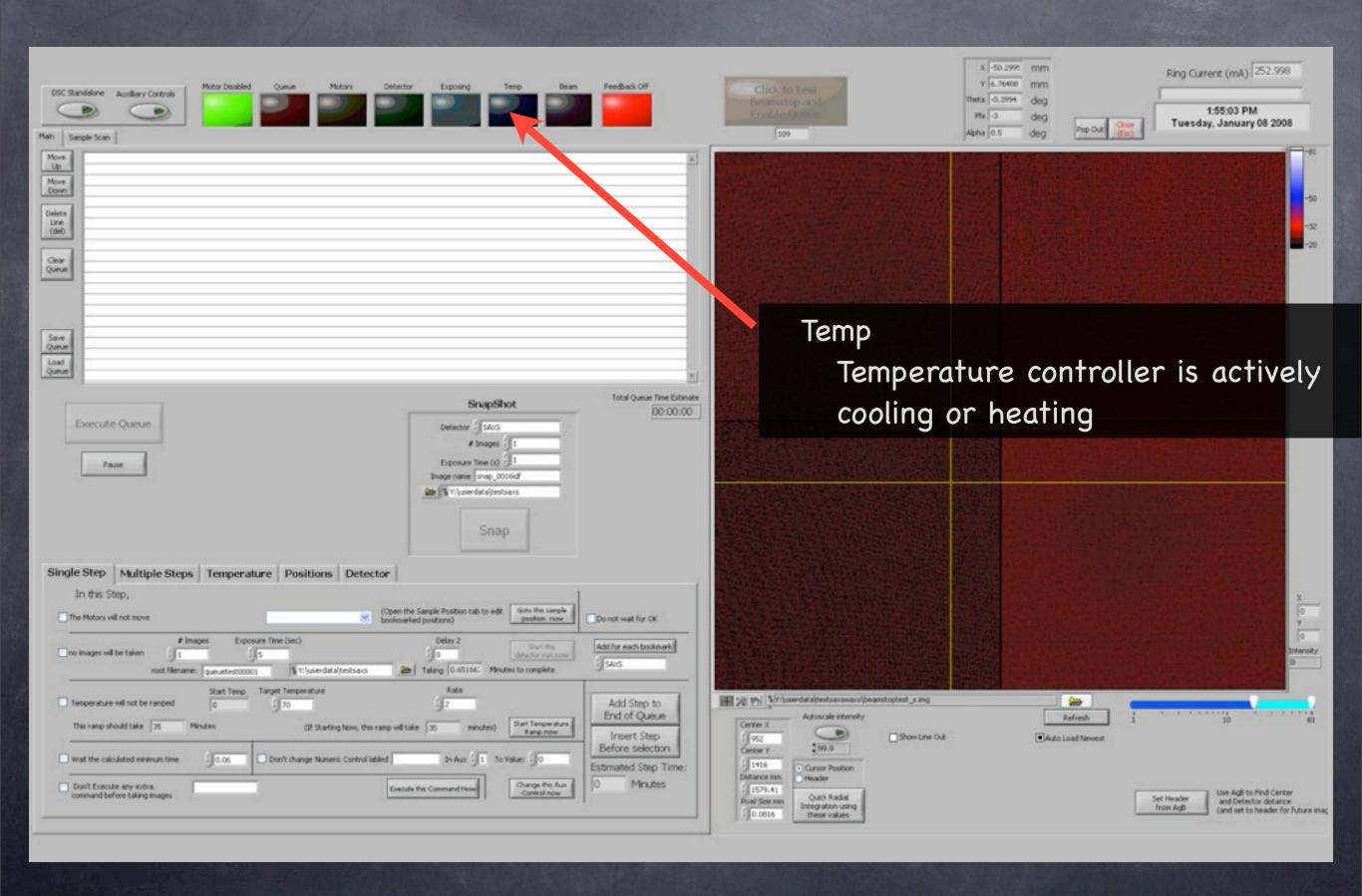


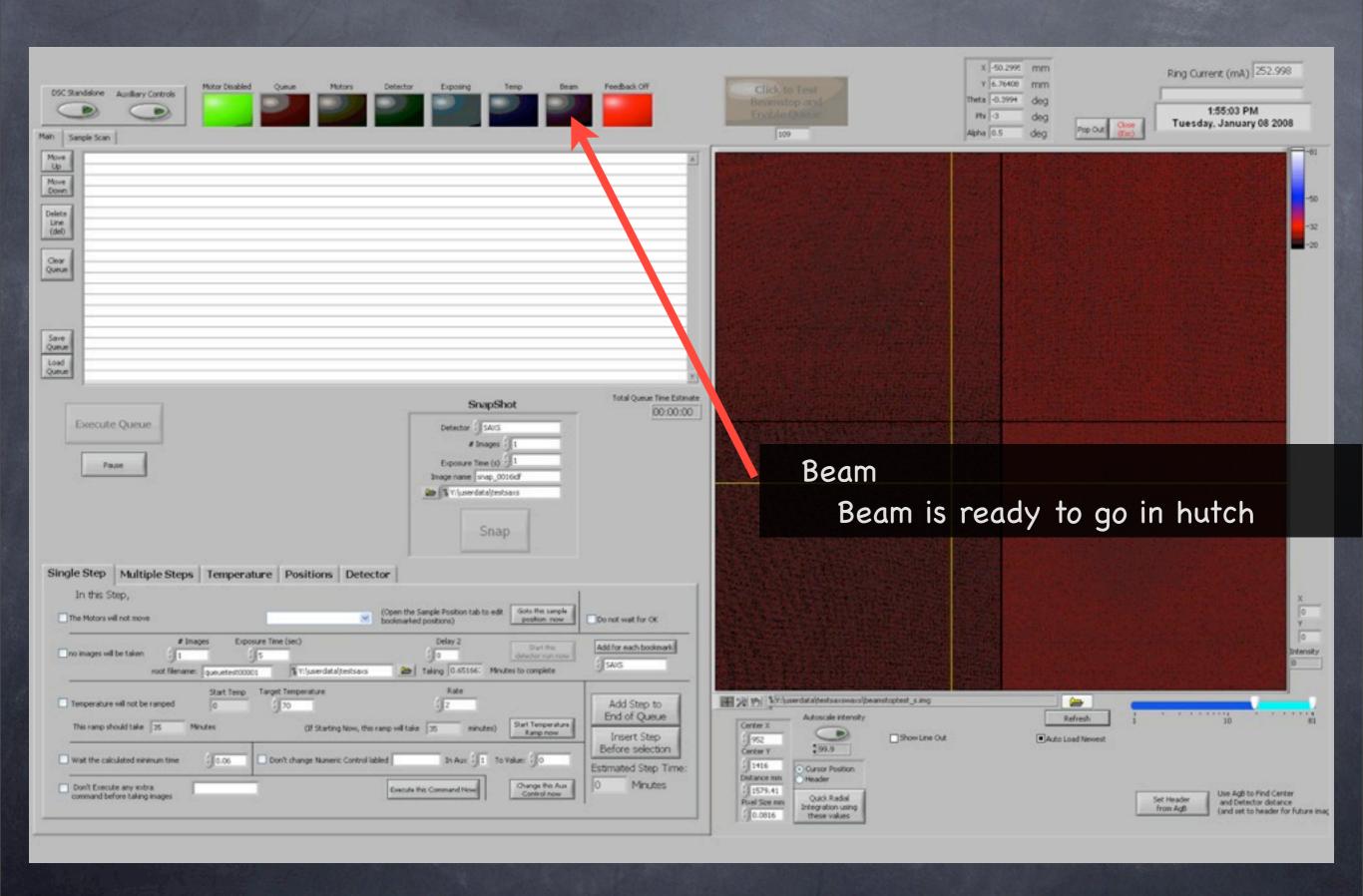


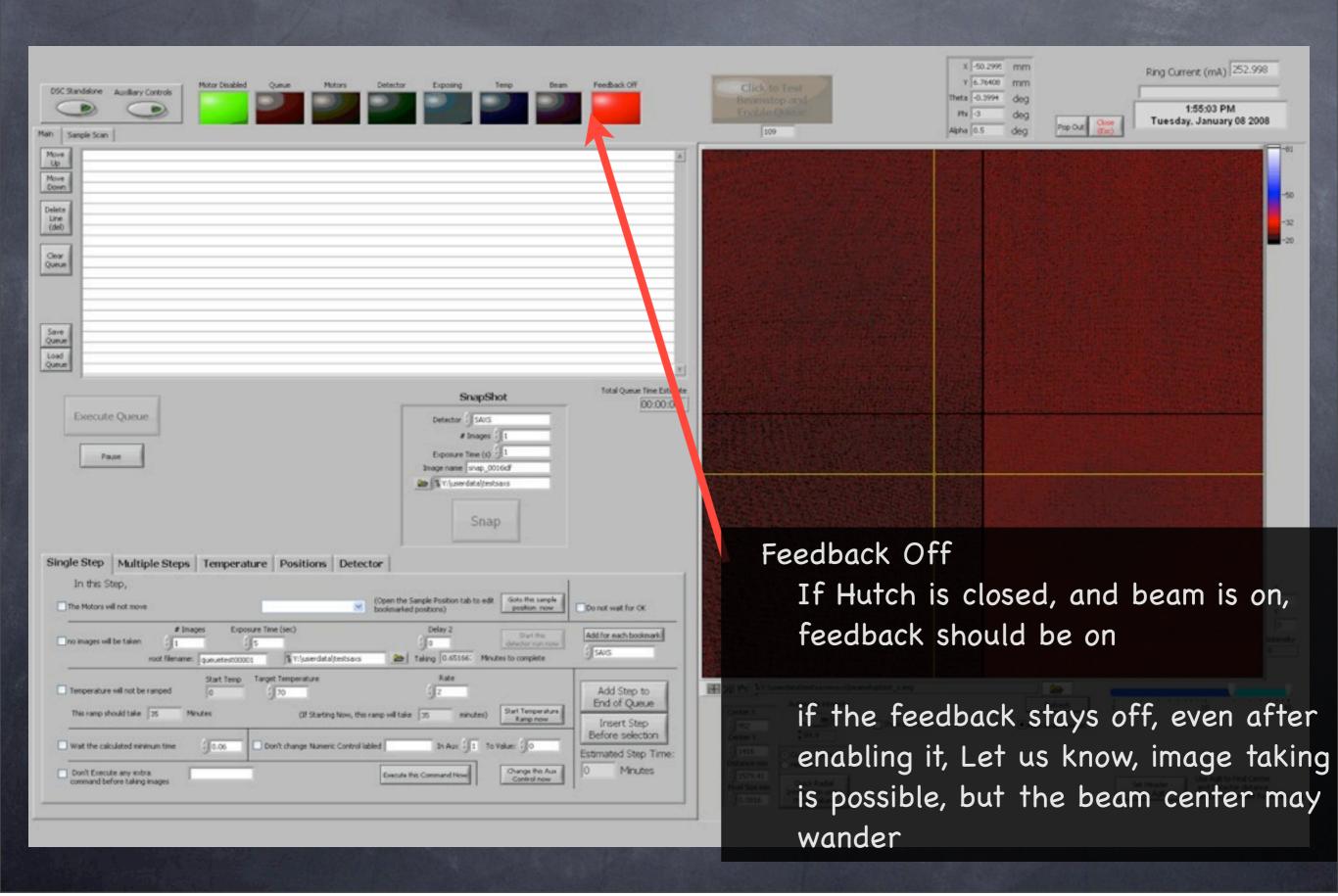


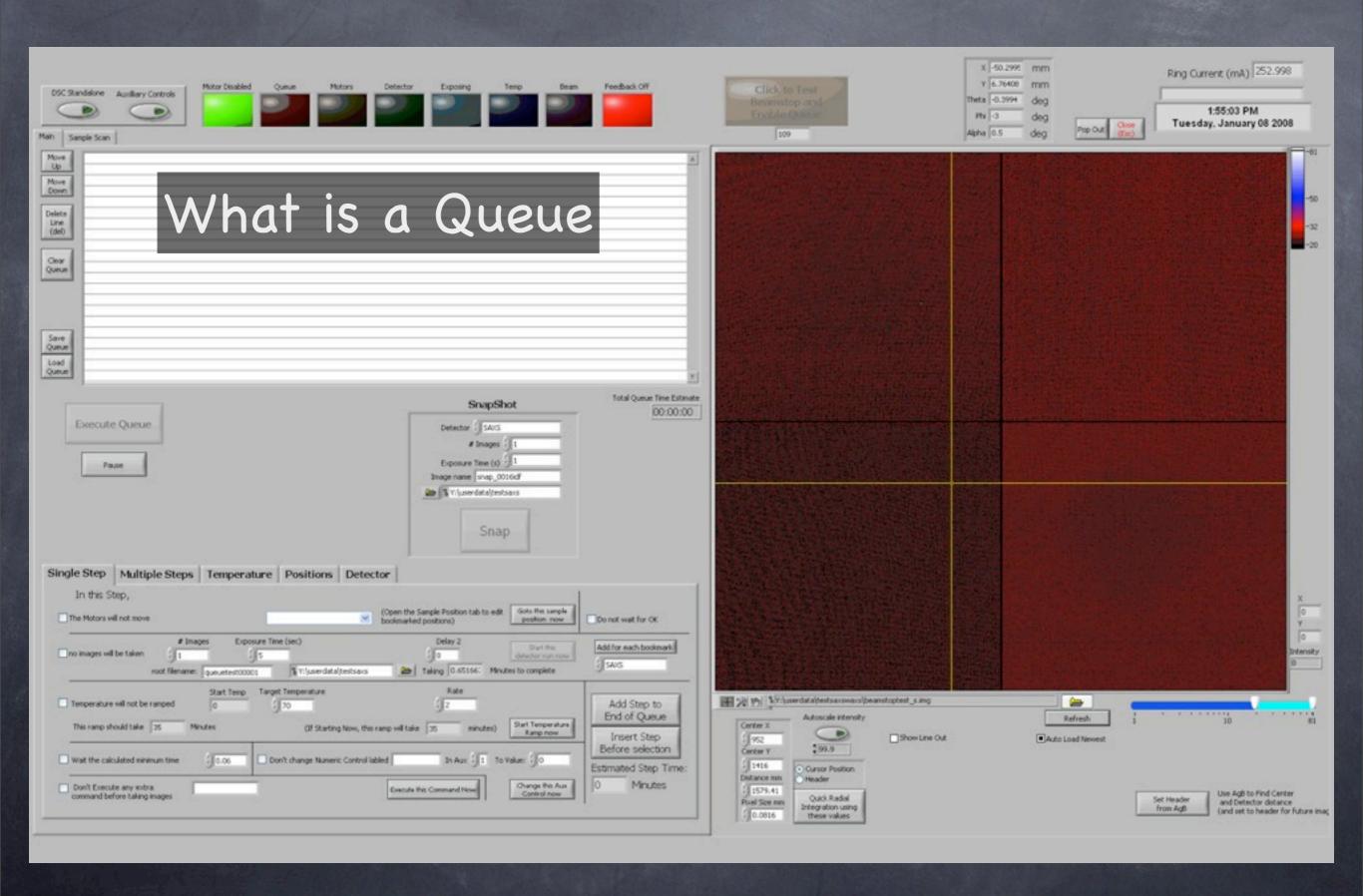


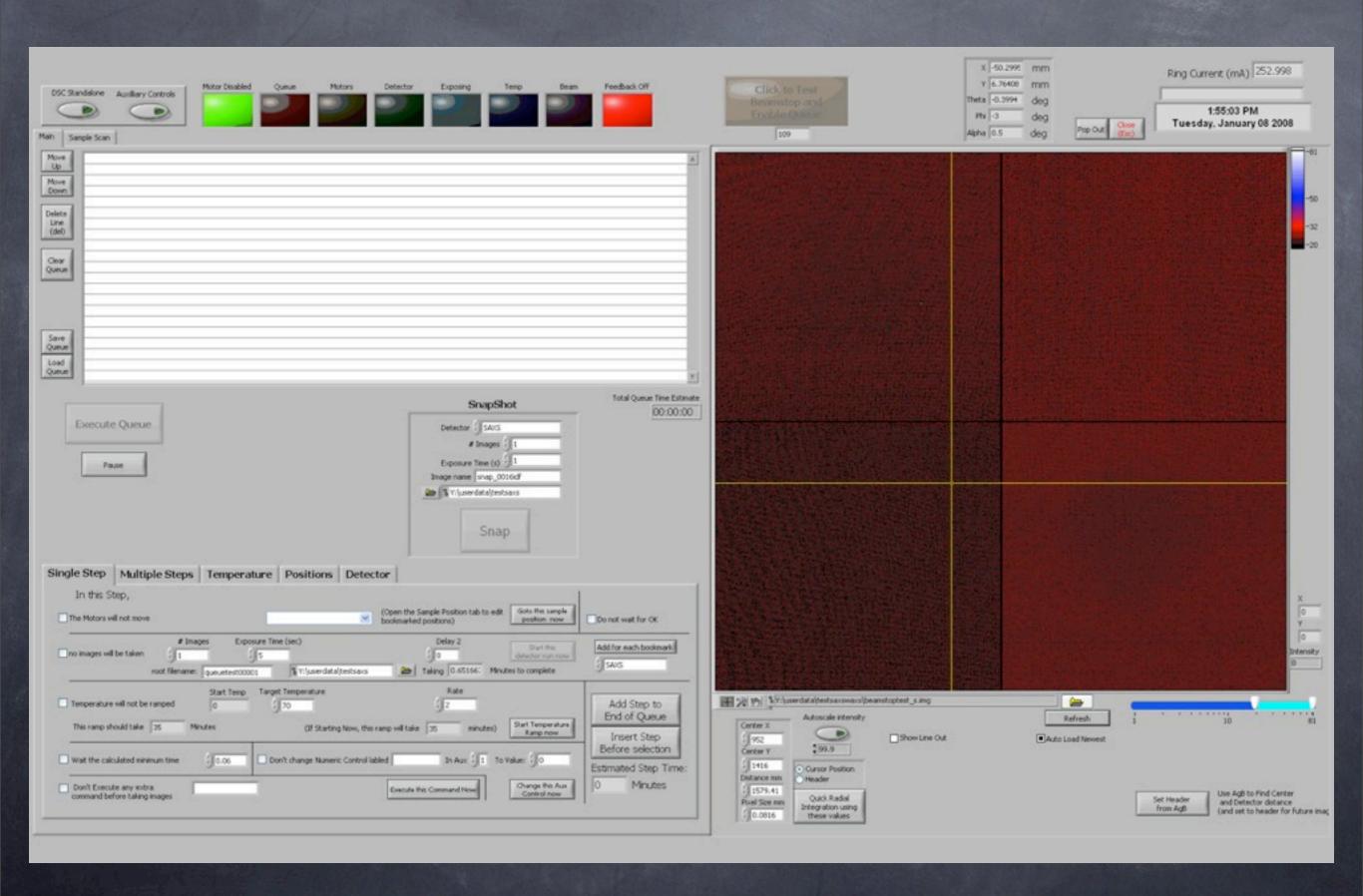


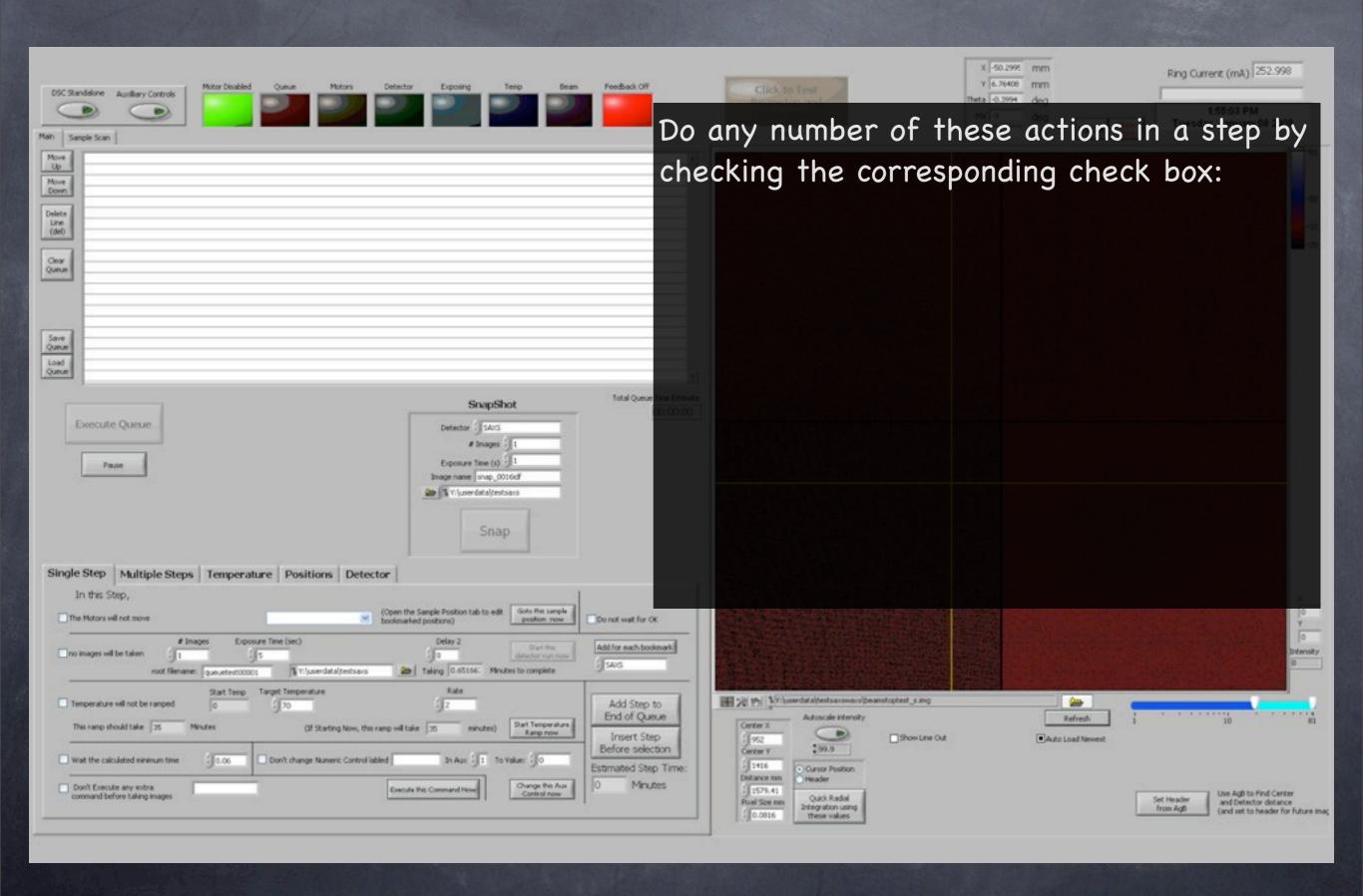


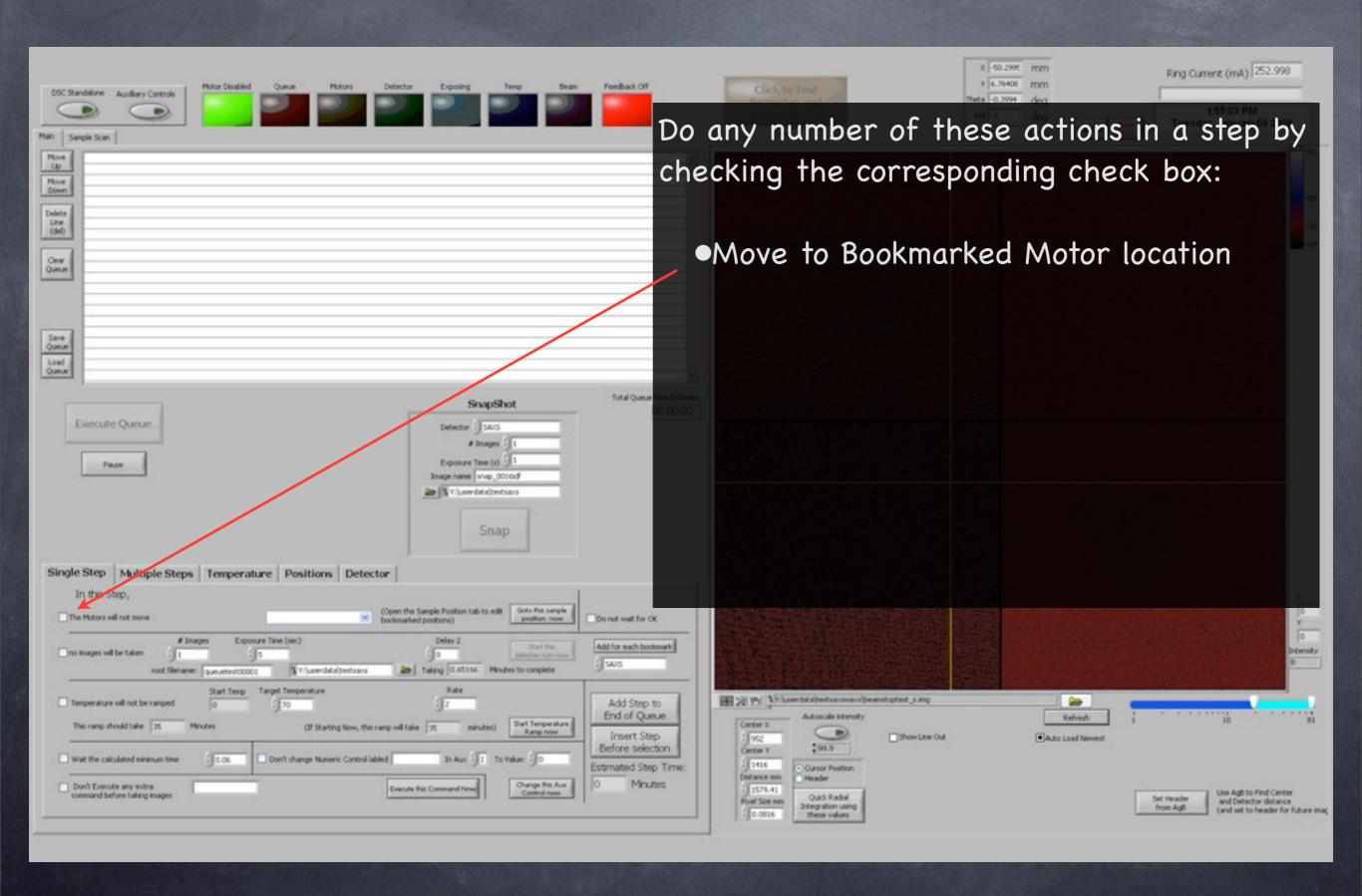


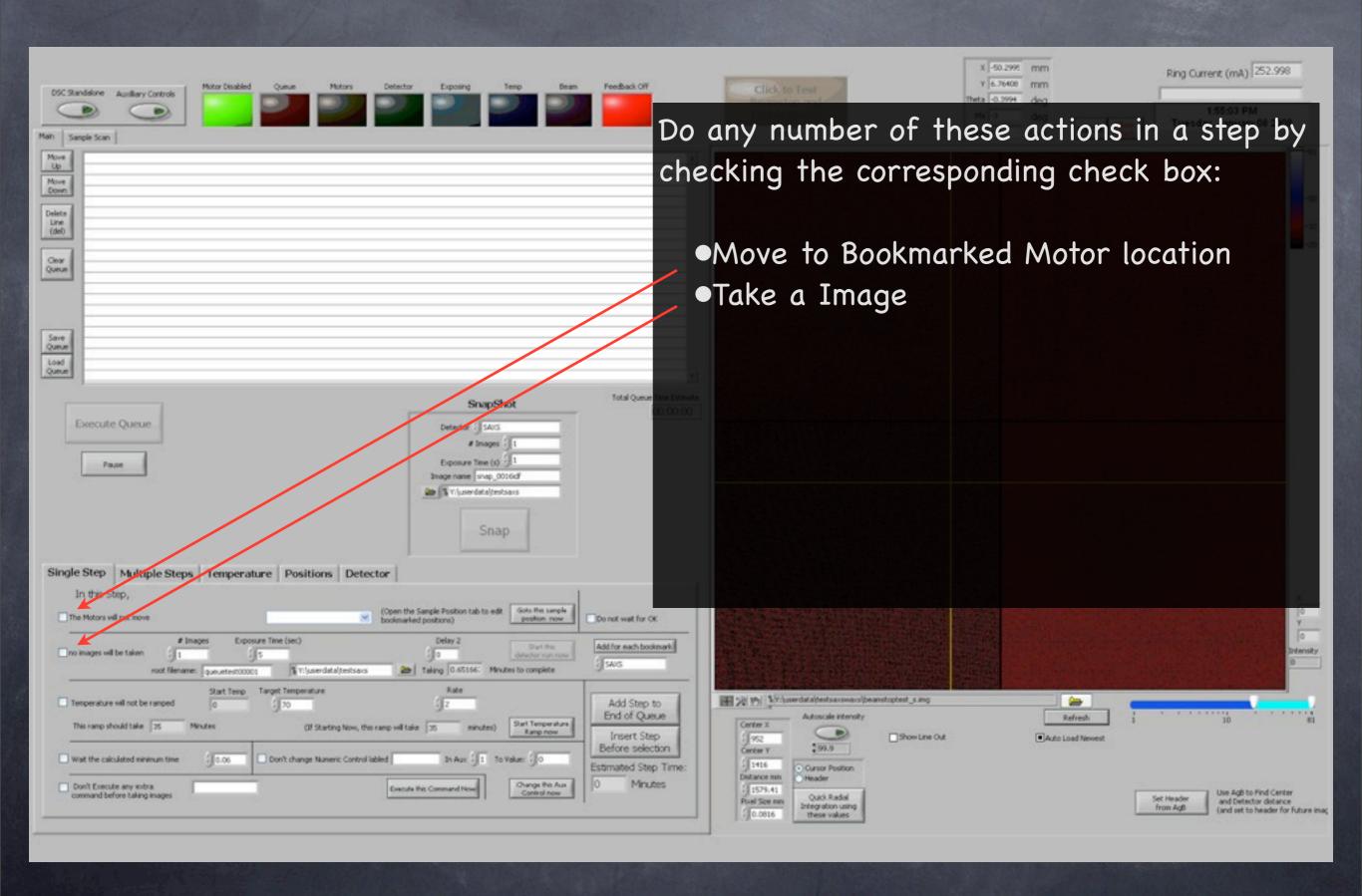


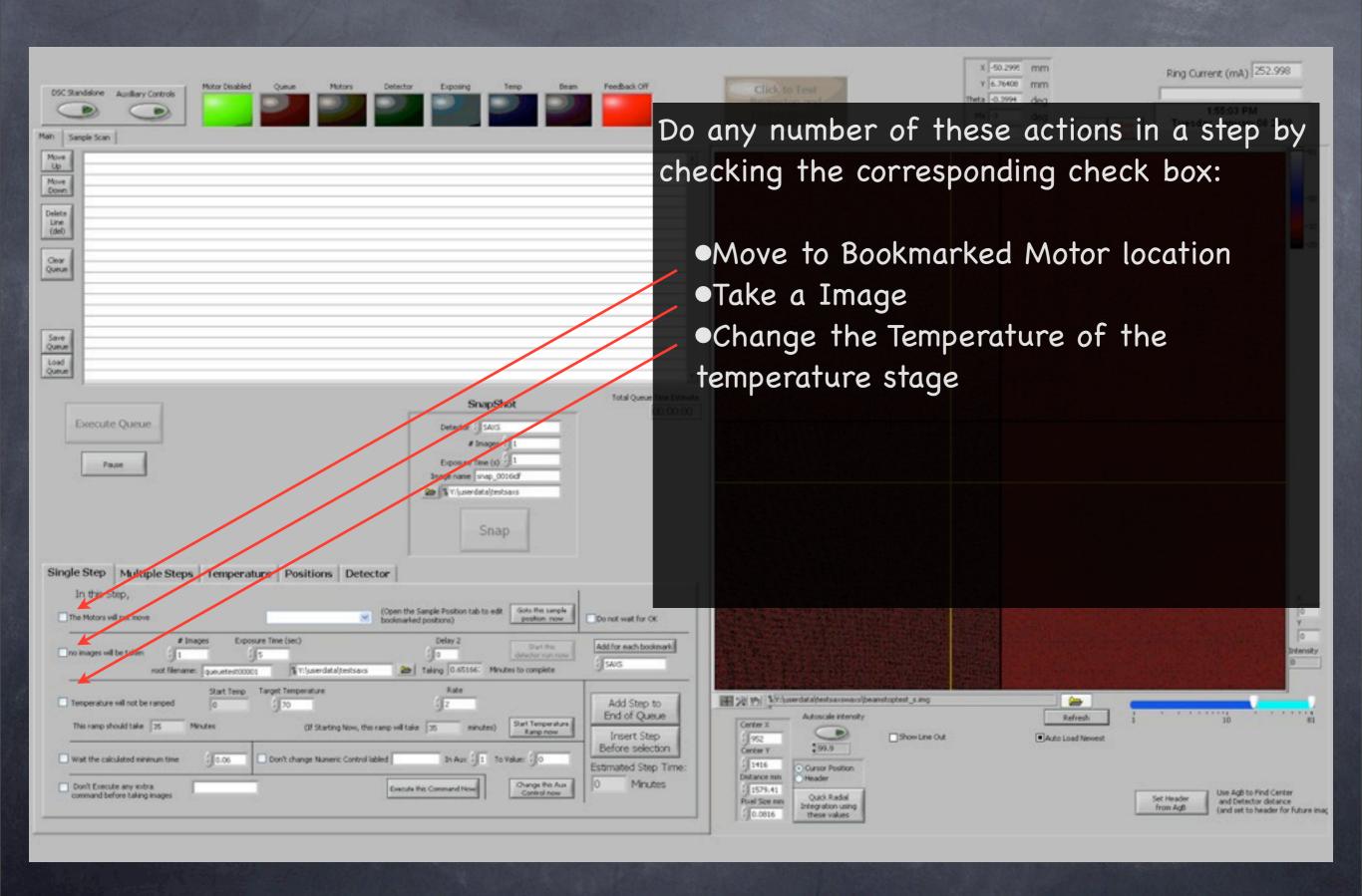


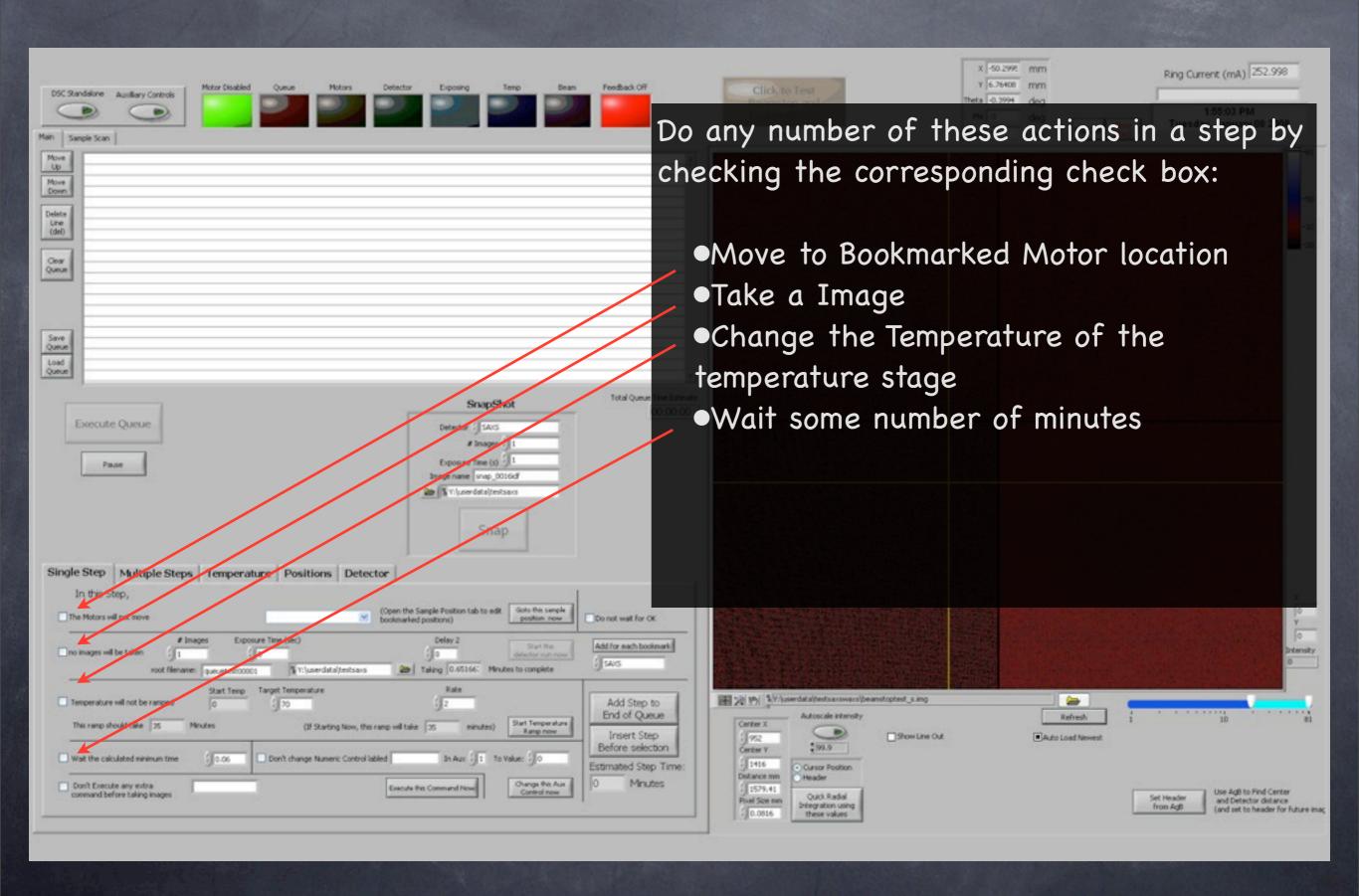


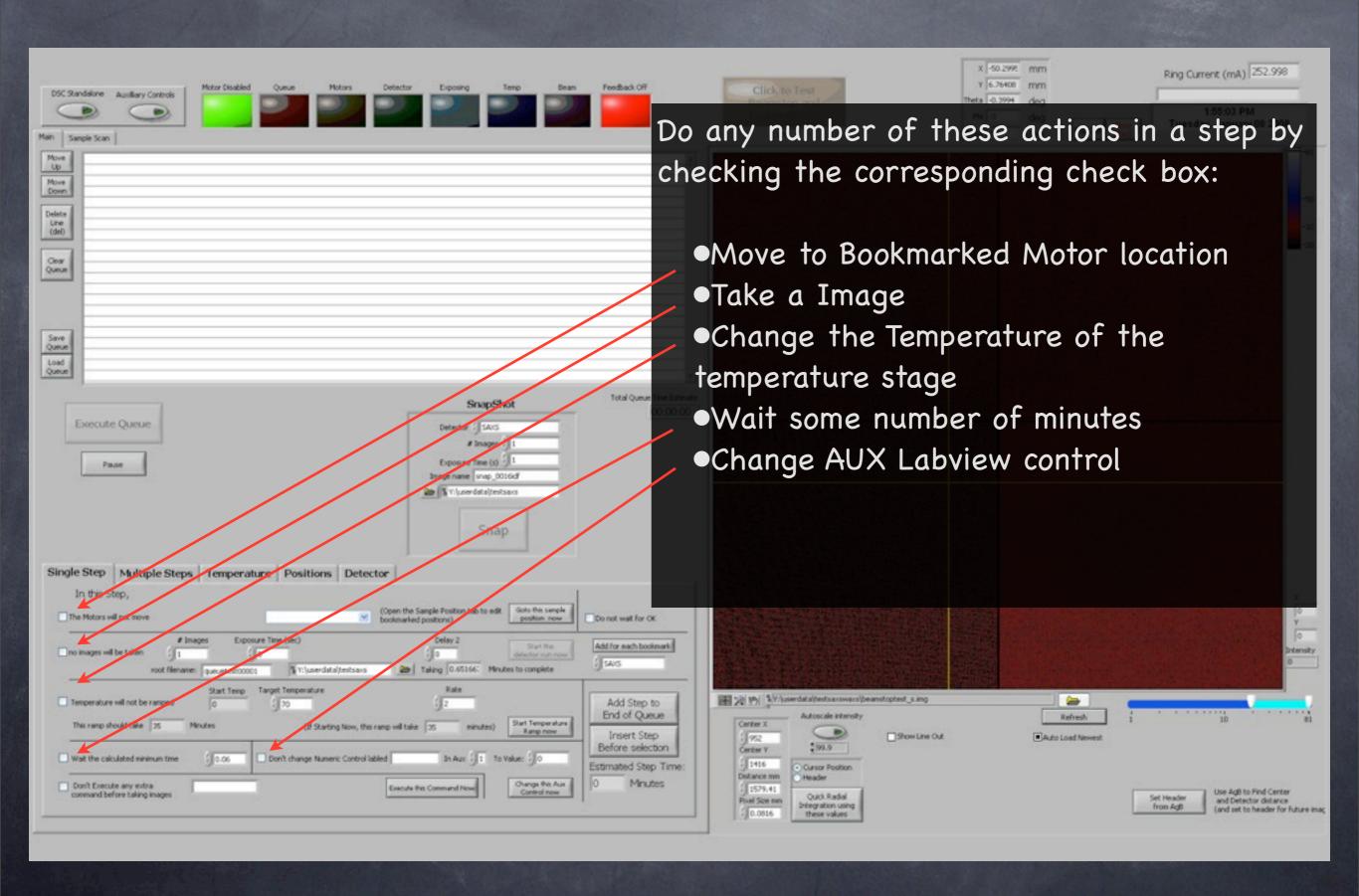


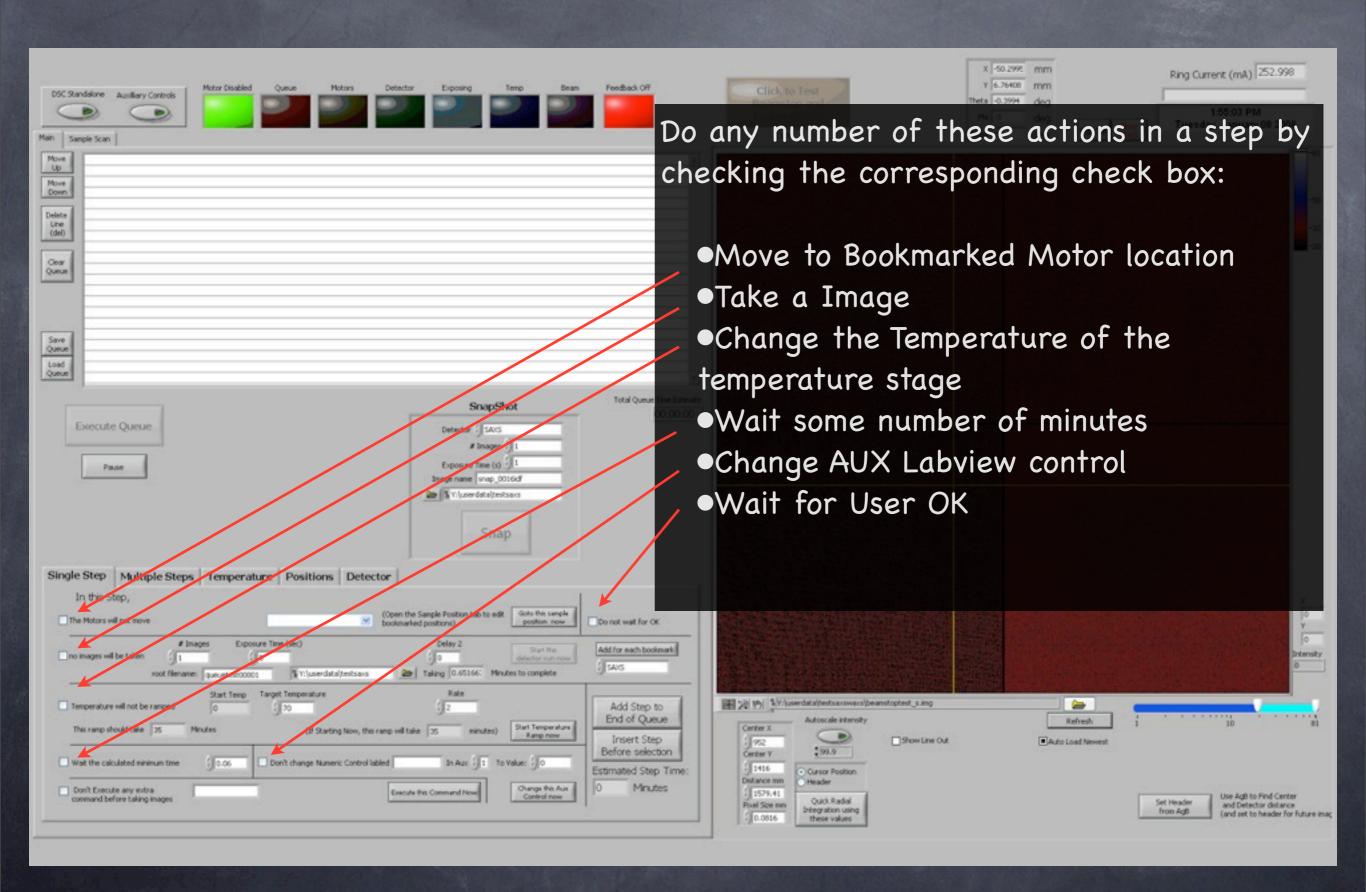


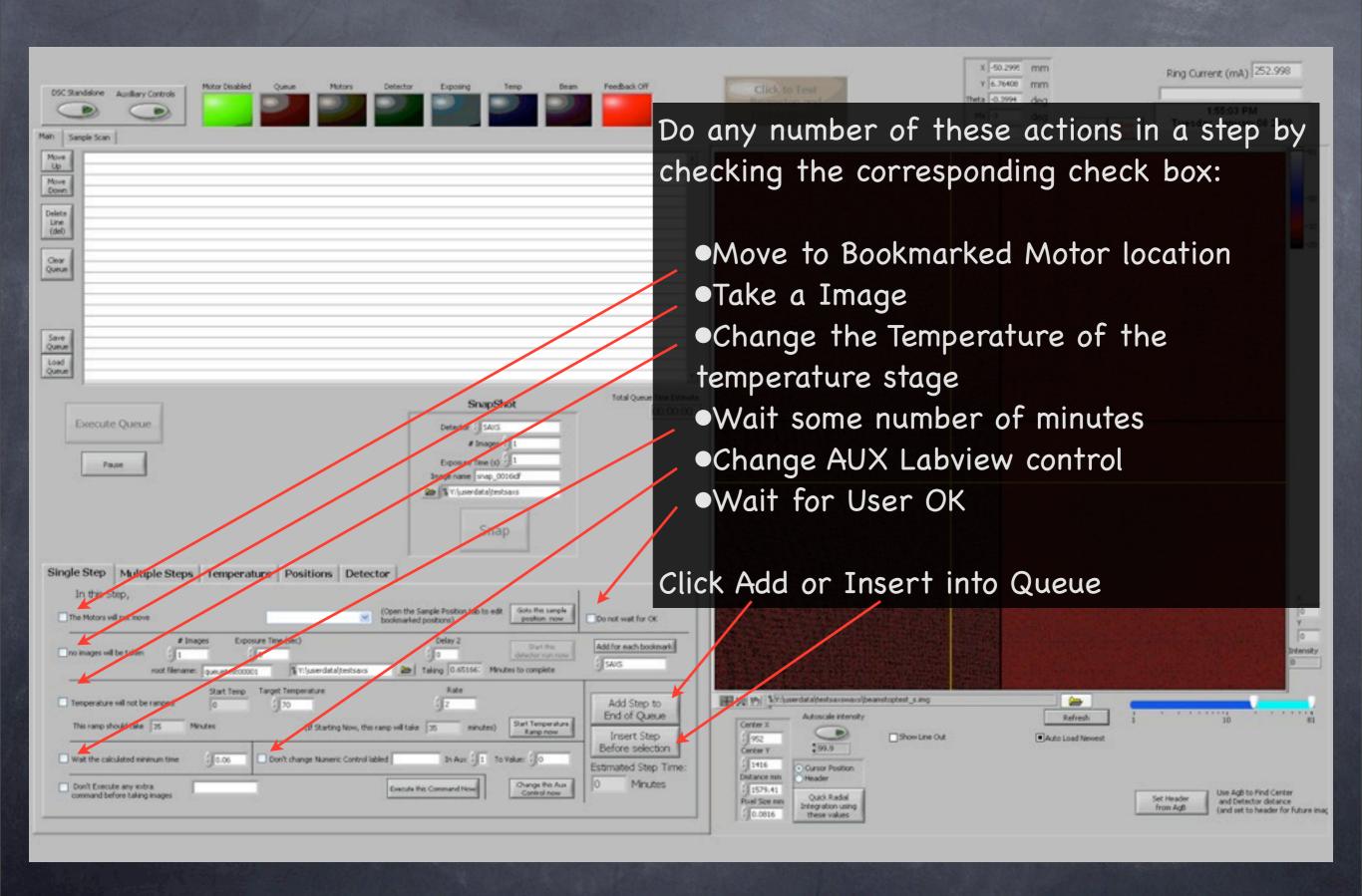


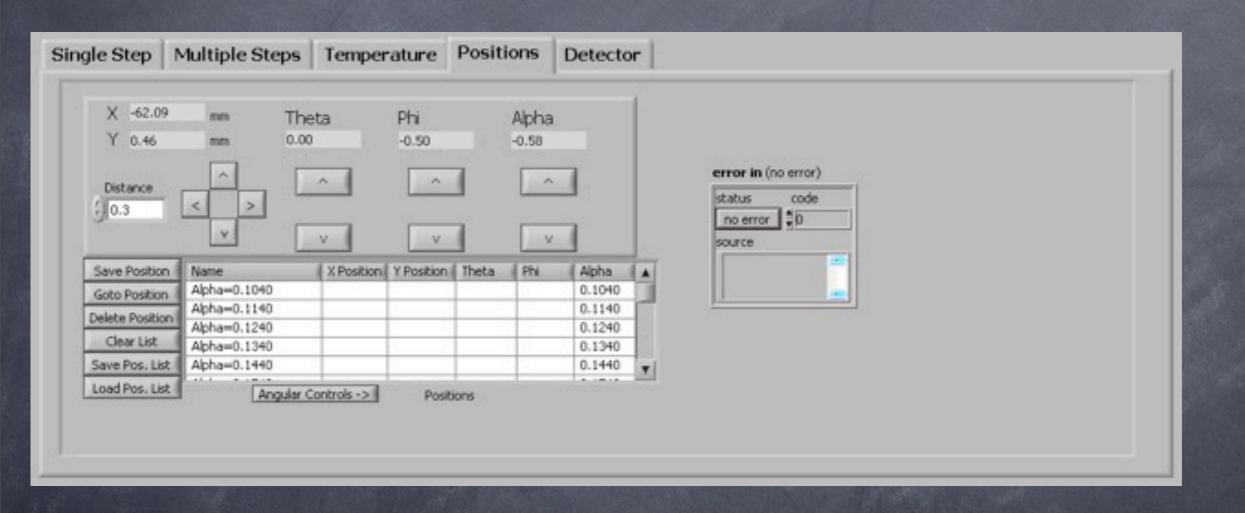










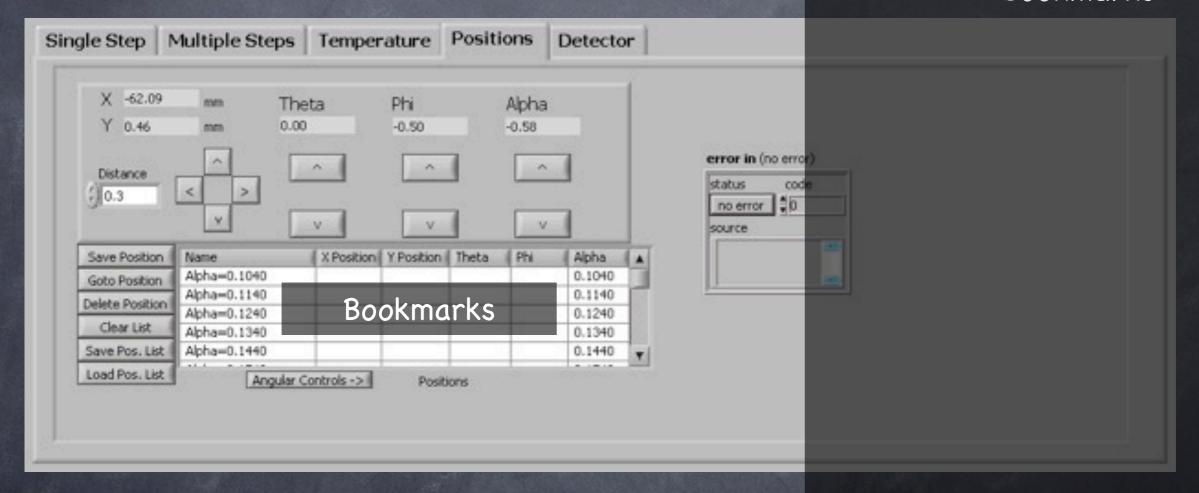


Motor control



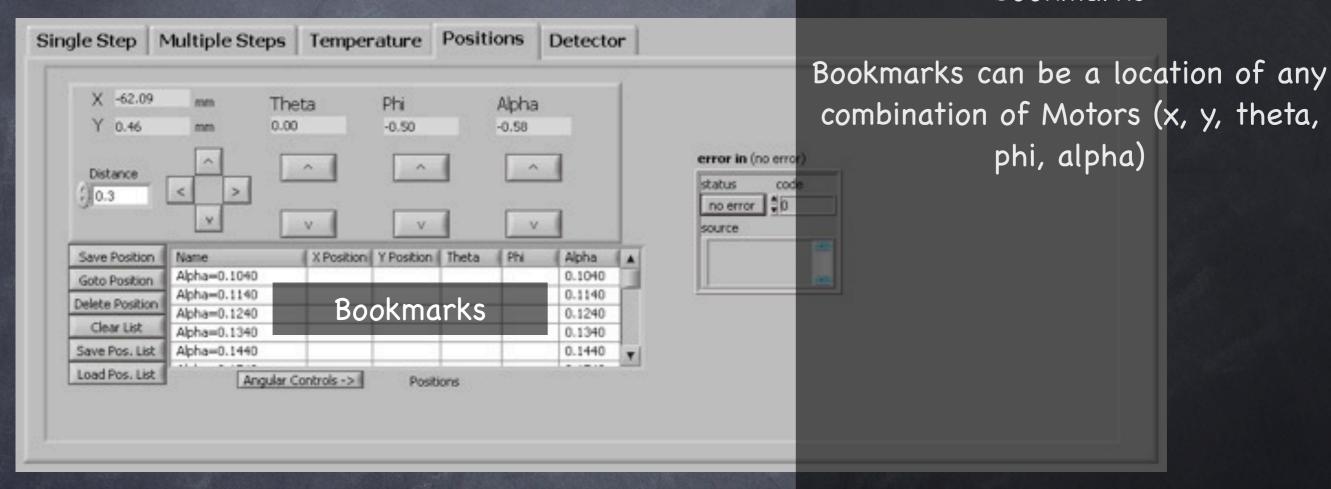
Motor control

Motor control is done through bookmarks



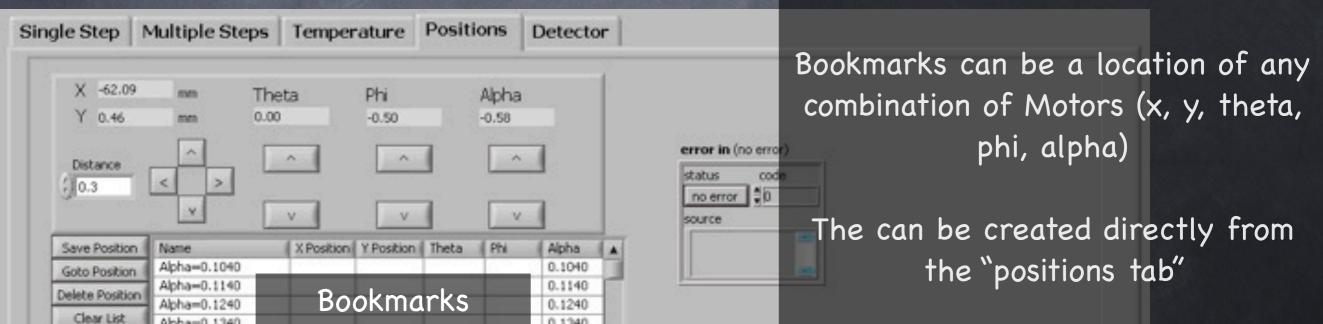
Motor control

Motor control is done through bookmarks





Motor control is done through bookmarks



0.1340

0.1440

Save Pos. List Load Pos. List Alpha=0.1340

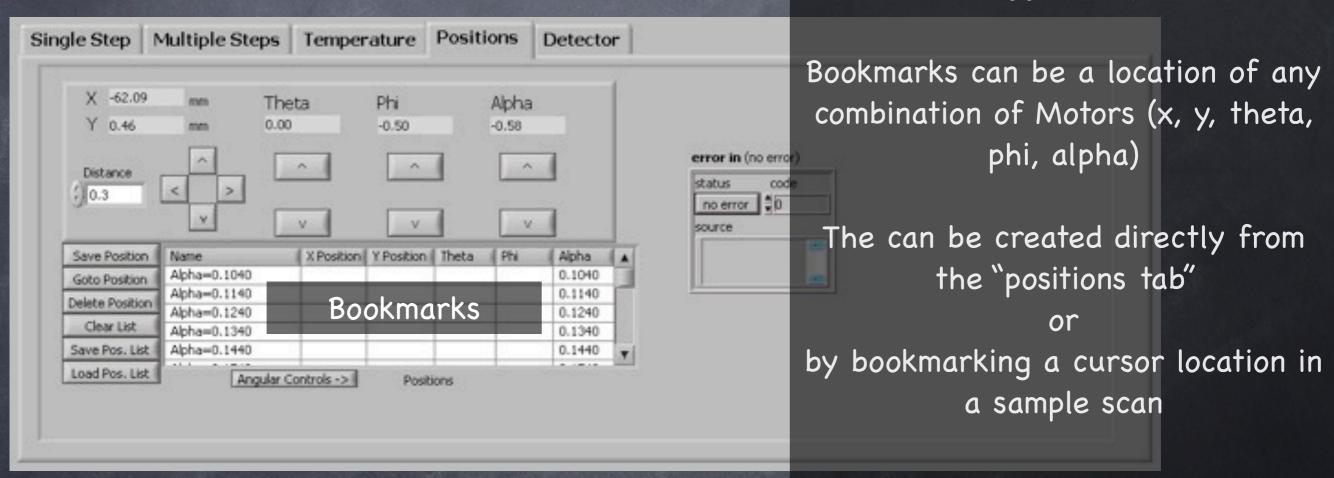
Alpha=0.1440

Angular Controls -> I

Positions

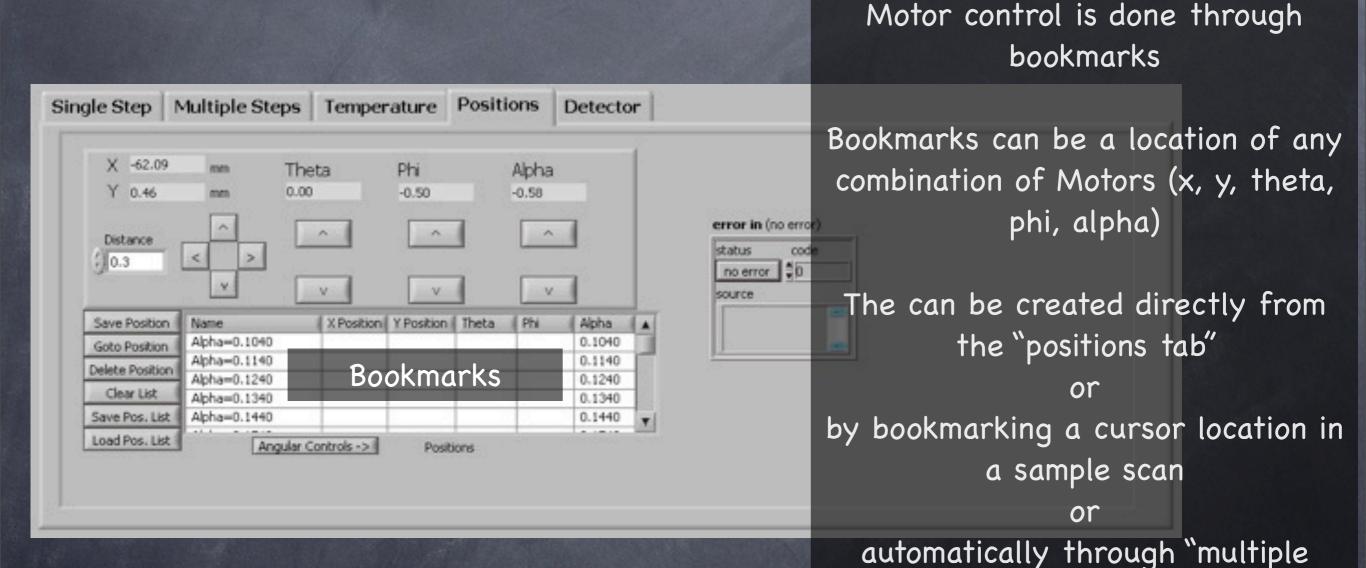
Motor control

Motor control is done through bookmarks



Motor control

steps" tab



Single Step	Multiple Steps	Temperature	Positions	Detector					
Step Settings Variable to Cl () Alpha	Start (J-0.666	Step End 10.01 1-0.53 Use List? None List will override st	13	Variable 1 Ph	Sinention to Change time after change	Start primar	Step primary 0.5 Use List? None	End primary	2 Demenitions?
Timing for each str Walt time B	rp (sec) efore Images Wait time	after images							
Image Settings a	t each step								
Exposure time Number of it	7	e Directory \$\f\frac{1}{2}\text{Vilunerde} Image Root name PS17			-	Using Detector		Build and Qu	

Multiple steps, (Queue Builder)

Single Step	Multiple Steps	Temperature	Positions	Detector				
Step Settings Variable to Ch () Alpha	hange Start	Step End () 0.01 () -0.51 Use List? () None	6 13	Prinary disertion Variable to Change J Phy Walt time after the	Start pr		End primary	2 Dimensions?
Timing for each ste Walt time B		List will override s after images	tep settings					
Image Settings a Exposure time	e (sec) (60 Imag	e Directory % Y: Juner do		-	Using Detects	w	Build and Que	

Multiple steps, (Queue Builder)

Dimensions: 1 or 2 dimensions

-		
Pick	Dim	ension





Multiple steps, (Queue Builder)

Dimensions: 1 or 2 dimensions
For each dimension, the List option determines
how the program will build the queue. The
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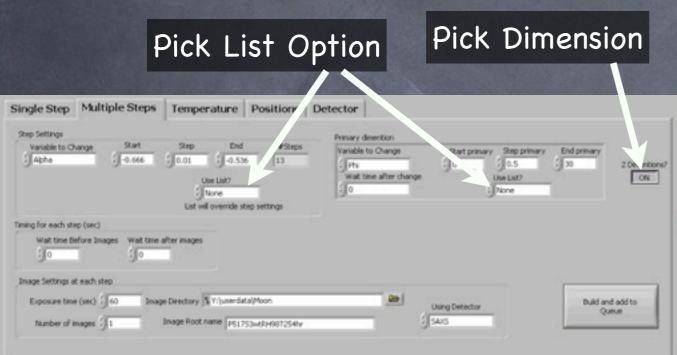
 Motor List: Use the list of Bookmarked motor Locations from positions tab



Multiple steps, (Queue Builder)

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- None: Increment whichever variable from "start" to "stop", moving by "increment" at each step

Pick List Option Single Step Multiple Steps Temperature Position Detector Step Settings Variable to Change Start 2cep End Steps II Japha 2-0.666 0.01 0.536 II Use List? Name List will override step settings Timing for each step (sec) Was time before Images was time after images Timage Settings of each step Exposure time (sec) 60 Image Directory (\$100 time after images) Image Settings of each step Image Root name Insigns II Image Ro

Click Here to Build your Queue

Multiple steps, (Queue Builder)

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The Image root name will be appended for each step



Handling Errors

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If an error occurs more than once

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If an error occurs more than once during an image

•First restart detector servers (see next slide)

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When moving a motor Make sure

Motors are enabled

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- Motors are enabled
- Motor path is not obstructed

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- •7.3.3 labview on left-most monitor doesn't have any errors

Handling Errors

Do NOT just continue through an error on a Beamstop test, without repeating the test. This can cause a false negative and allow you to hurt the detectors

In any other situation, continuing though an error is fine, although this is an indication that something may be not working correctly, and your data is suspect

If an error occurs more than once during an image

- •First restart detector servers (see next slide)
- •If that doesn't help, restart labview (save positions and queue to file first and ctrl-alt-del and end Labview process)
- •Call Eliot

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If an error occurs more than once during an image

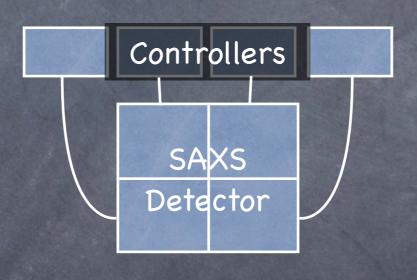
- •First restart detector servers (see next slide)
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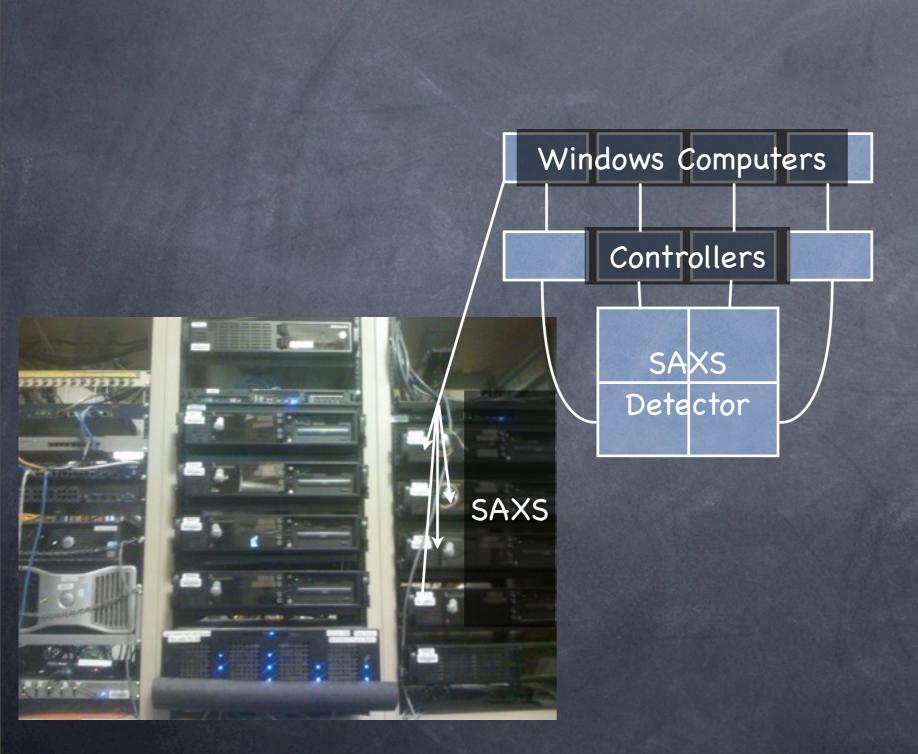
When moving a motor Make sure

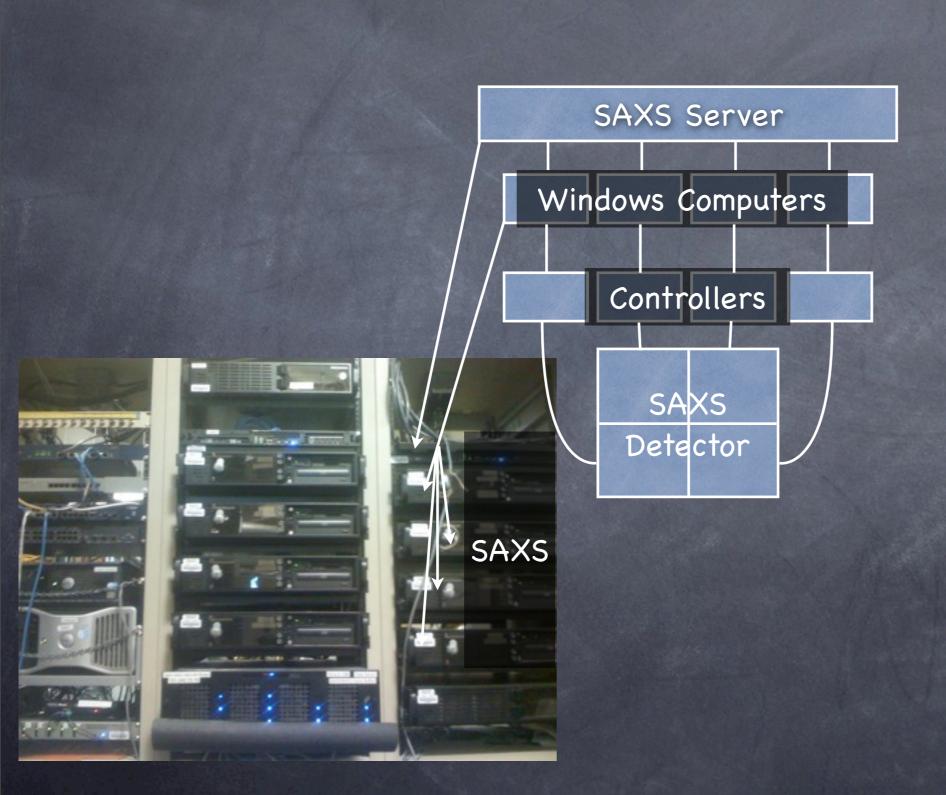
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- •Then Call Eliot

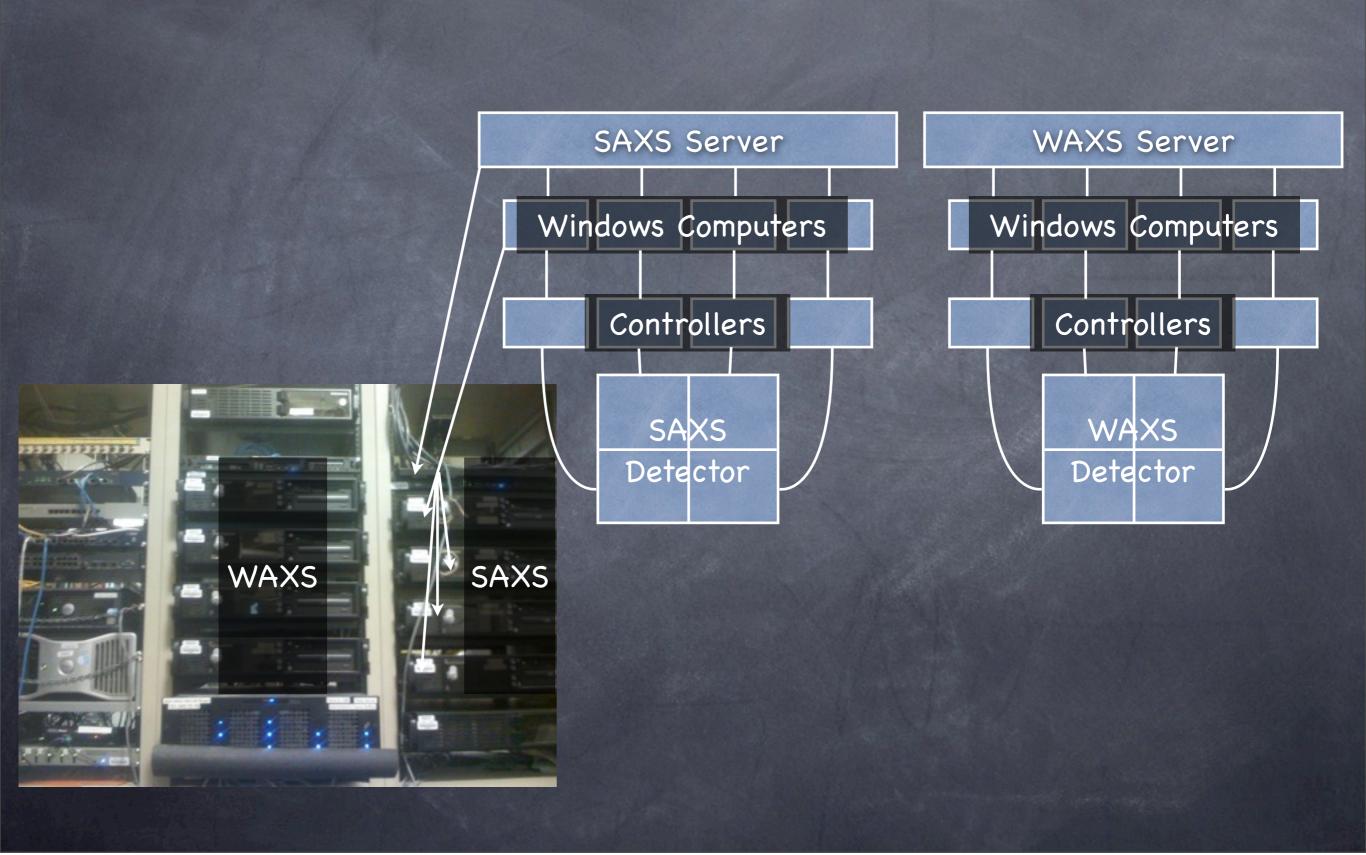
Otherwise for repeated errors, Write them down or leave them open and Call us

SAXS Detector

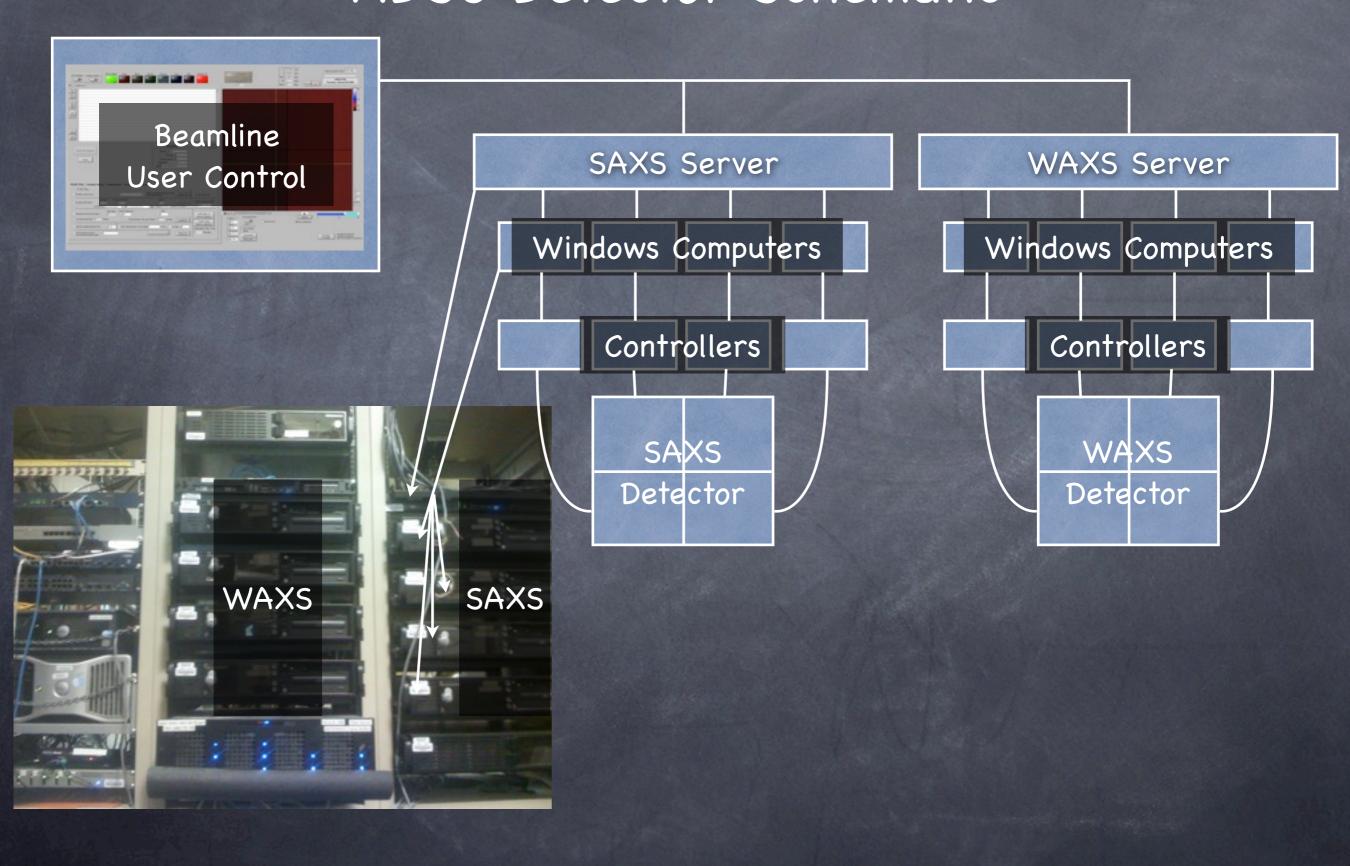




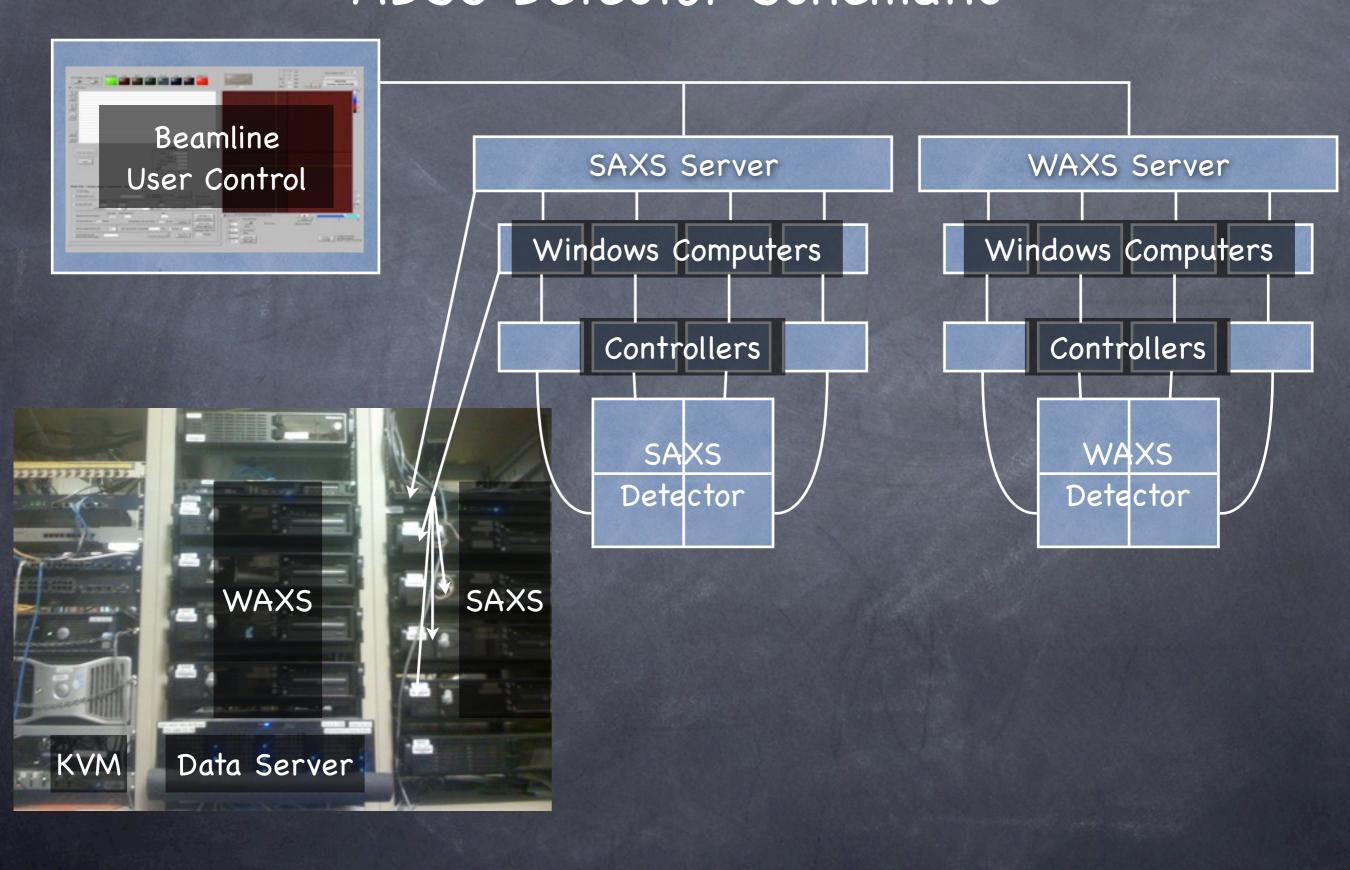




ADSC Detector Schematic



ADSC Detector Schematic







Restarting Detector Servers

When to restart the servers:

- •Error occurs when taking an image
- •Images are delayed or offset, or acting funny

How to restart the servers: Change KVM to SAXS and then WAXS

- •on each of these computers in the terminal type: "startccd"
- hit enter and wait for all four connections to be accepted





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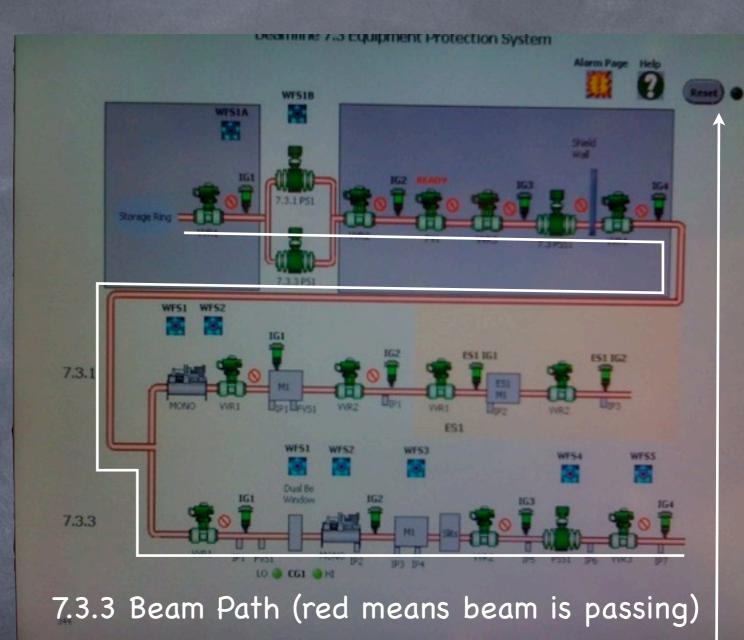
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EPS Display

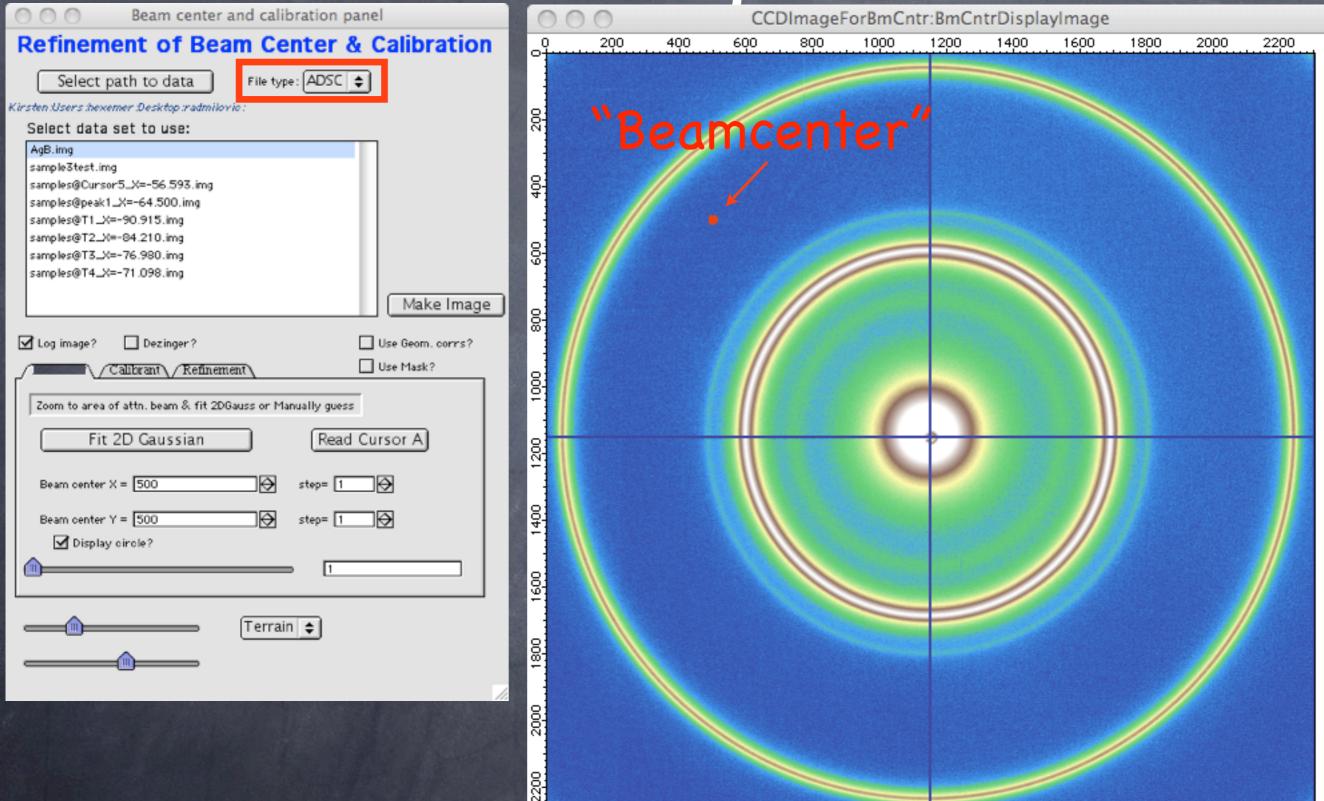
- When do we need to use it?
 - When ALS is running, PSS1 is open but no "beam pass" light is lit.
- Anything red or yellow on EPS display needs attention before beam will be available
- Report all errors
- Devices are Valves, Gauges, Pumps, and Water Flow Sensors
- Anything red is not working, ask Alex or Eliot, or beamline operators, or if none are available, the control room. they will put you in contact with the person who can fix the problem

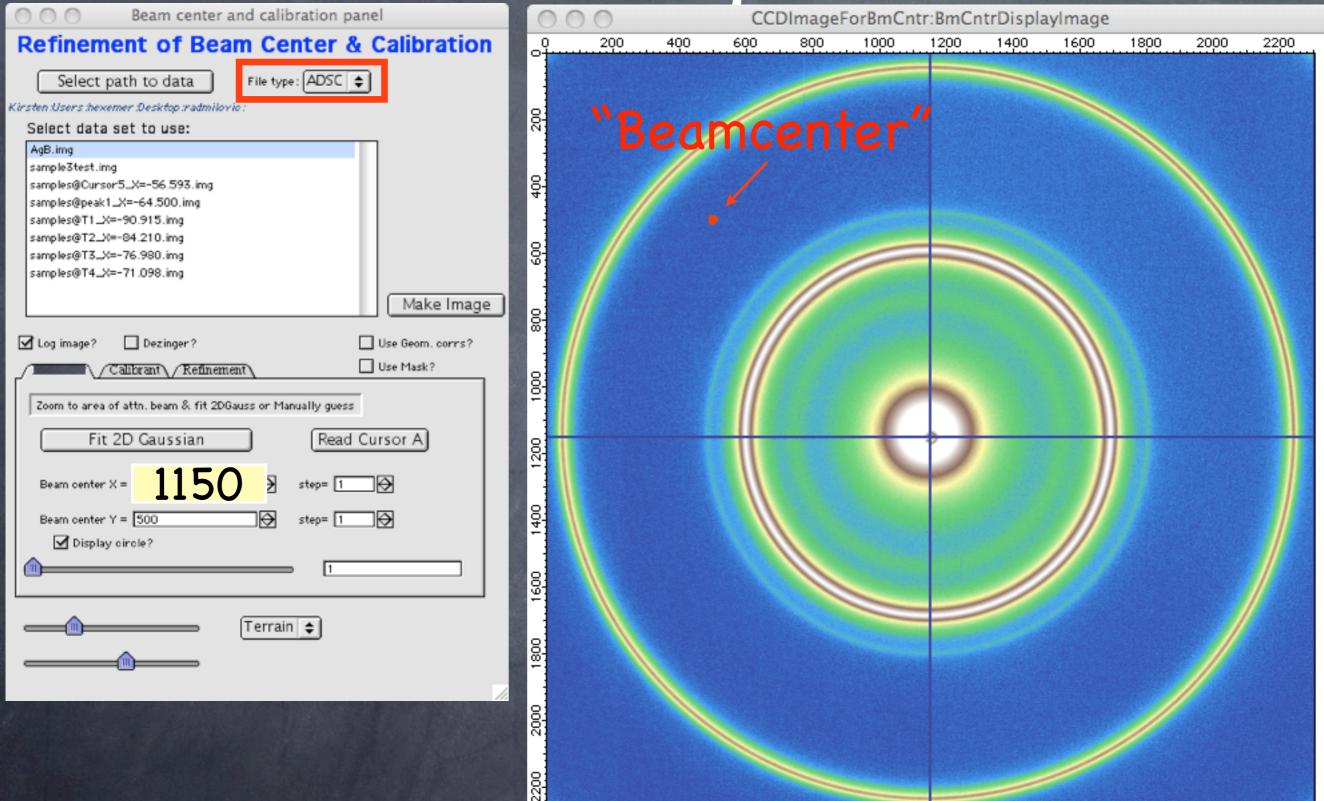


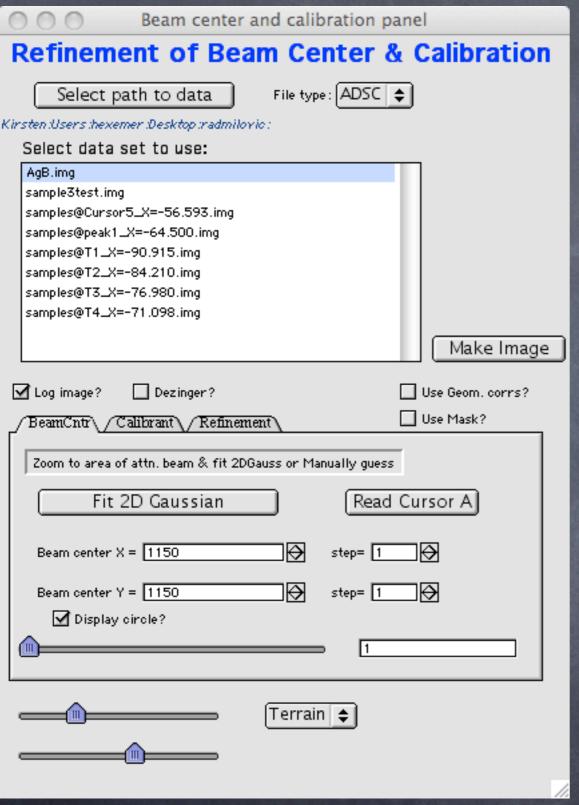
- Yellow Items can be "reset" by pressing reset
 - Still report anytime you need to reset the EPS display

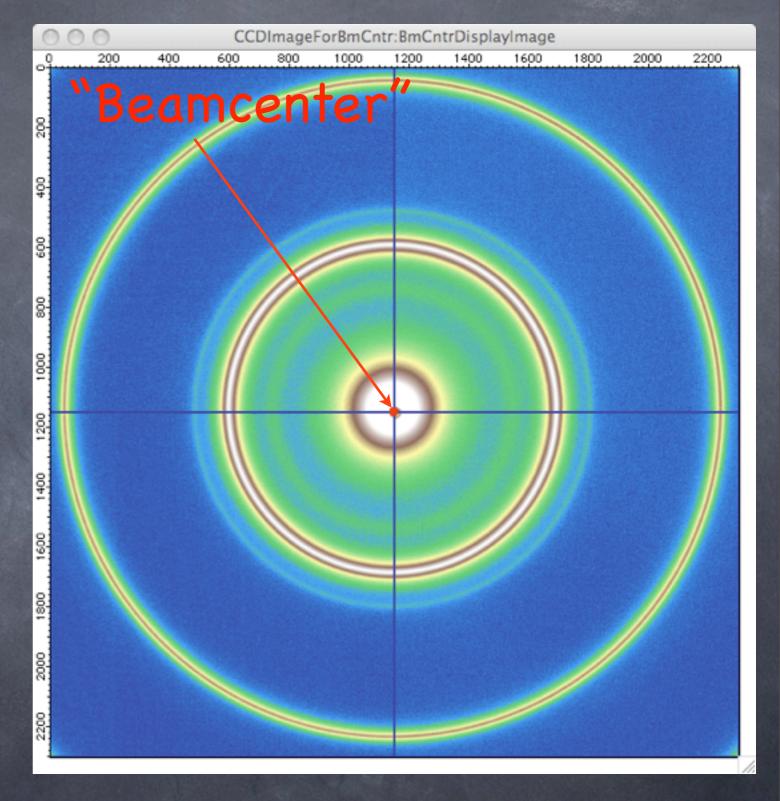
Analysis

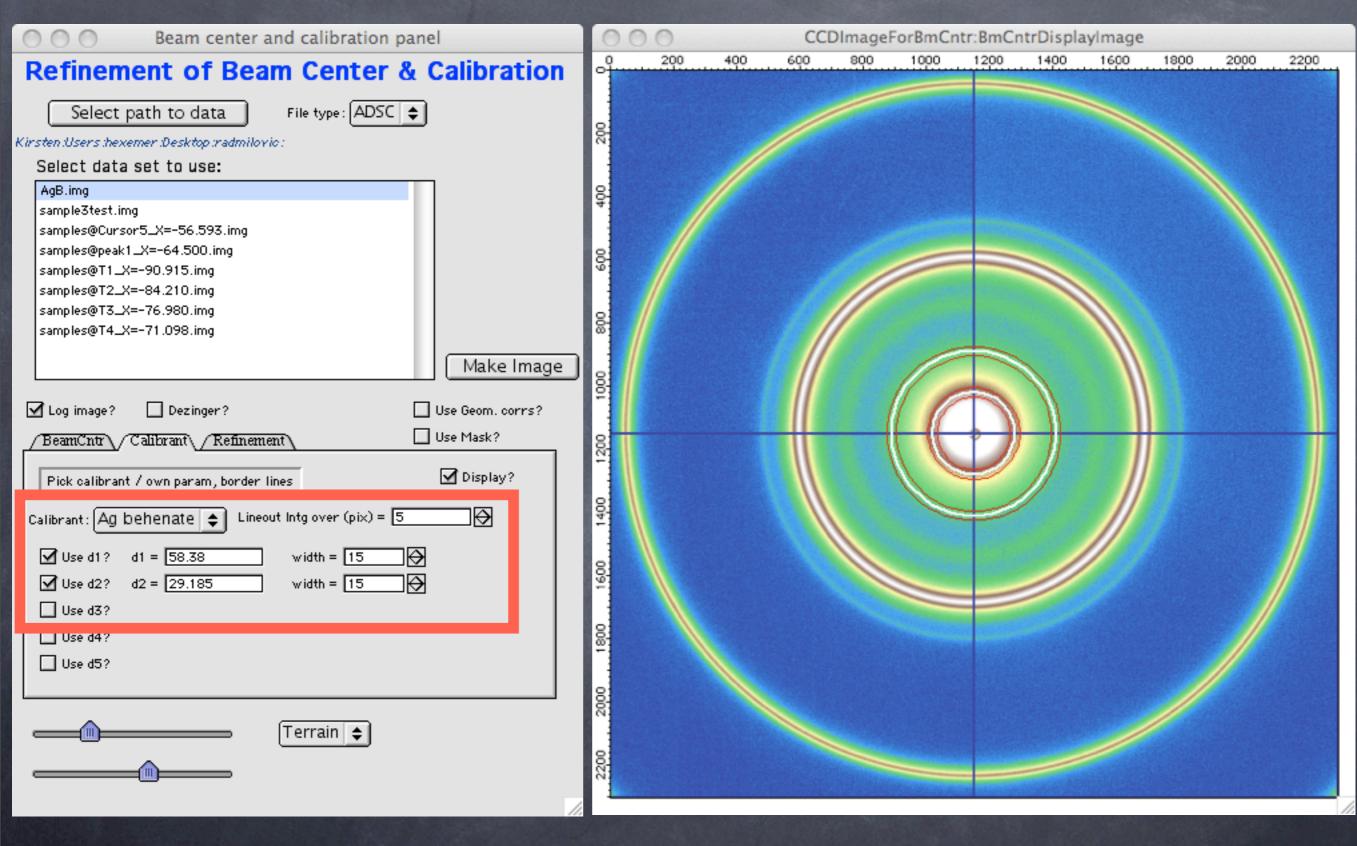
- Find direct beam position and sample detector distance
- Sector graphs
- Radial integration
- Line profile and where are they
- Getting your data in Ascii

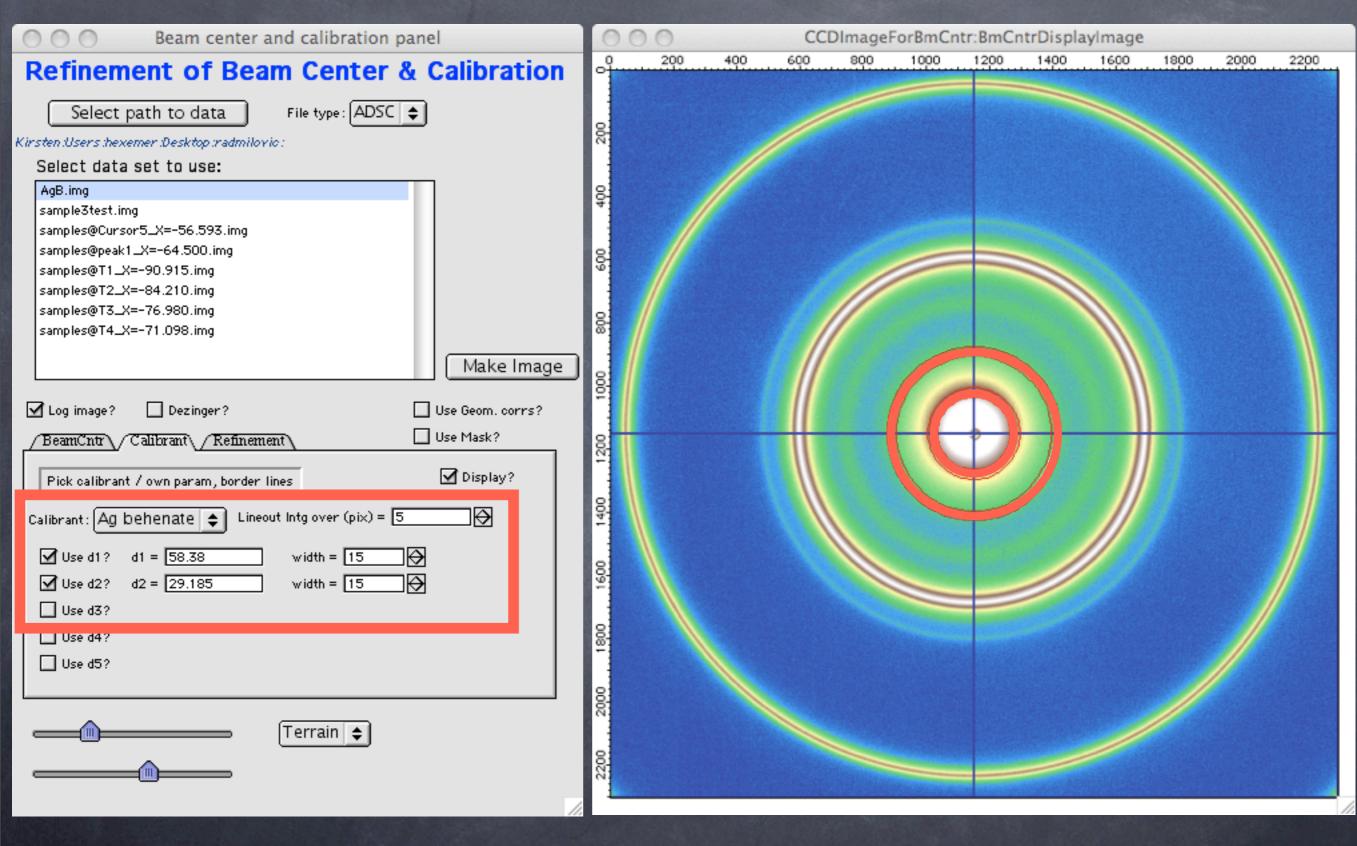


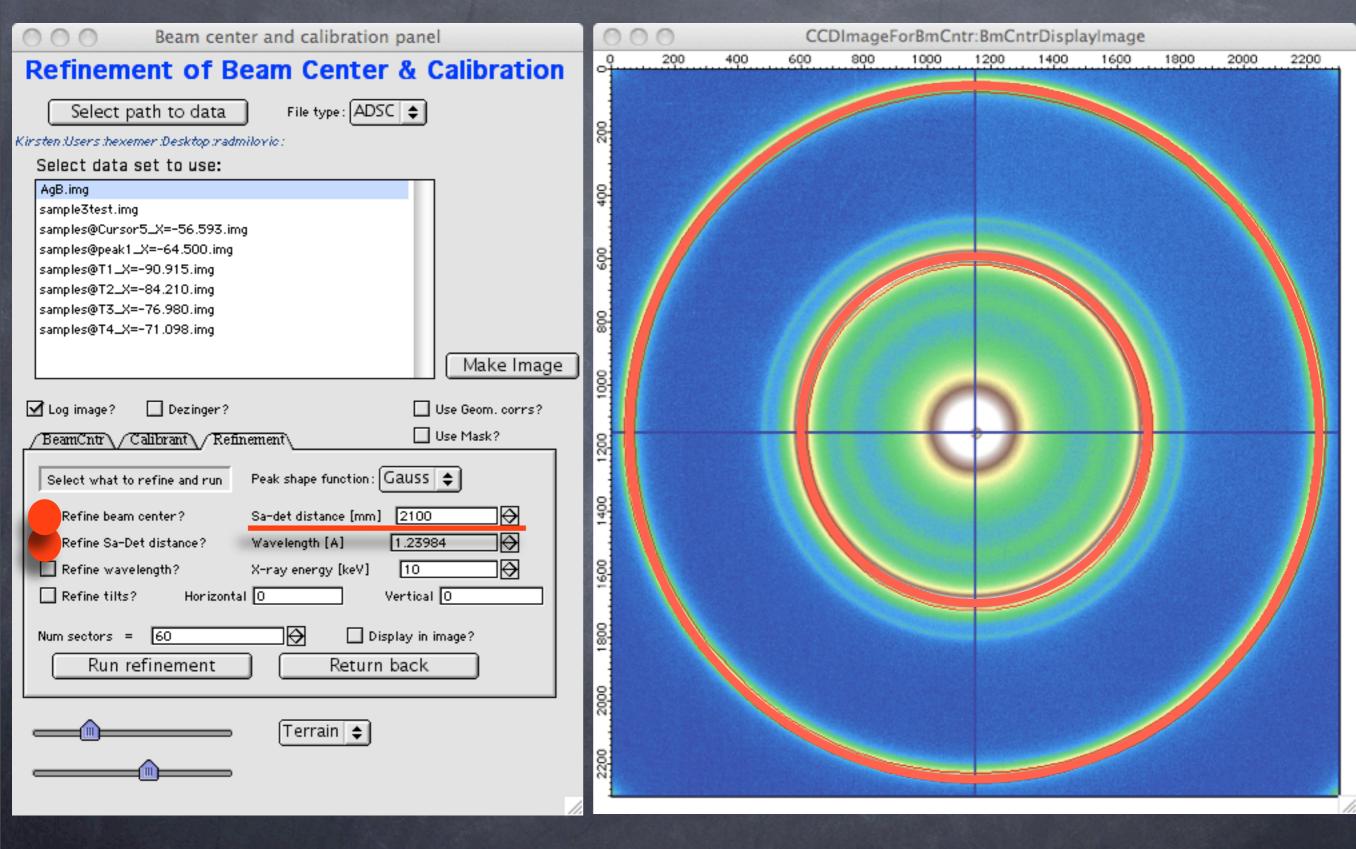


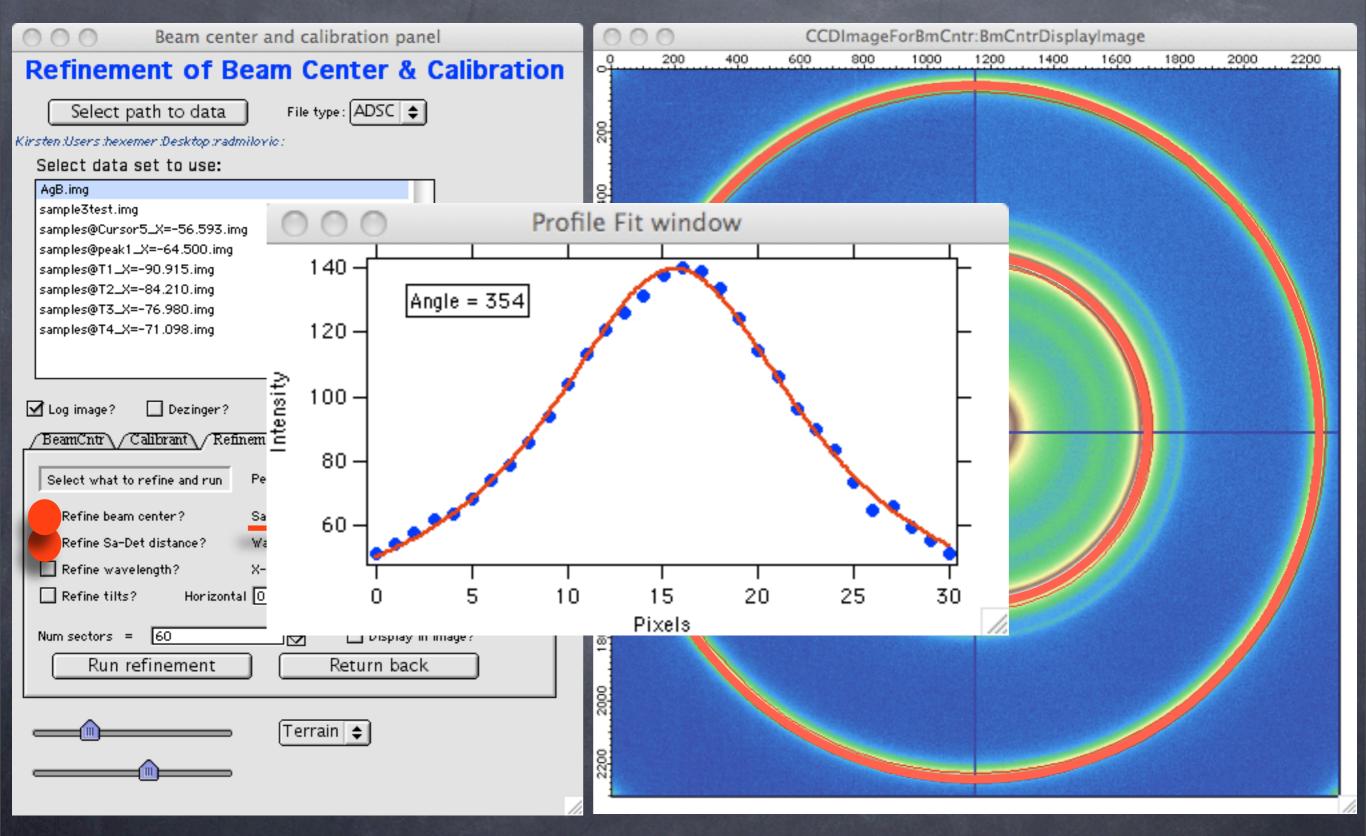


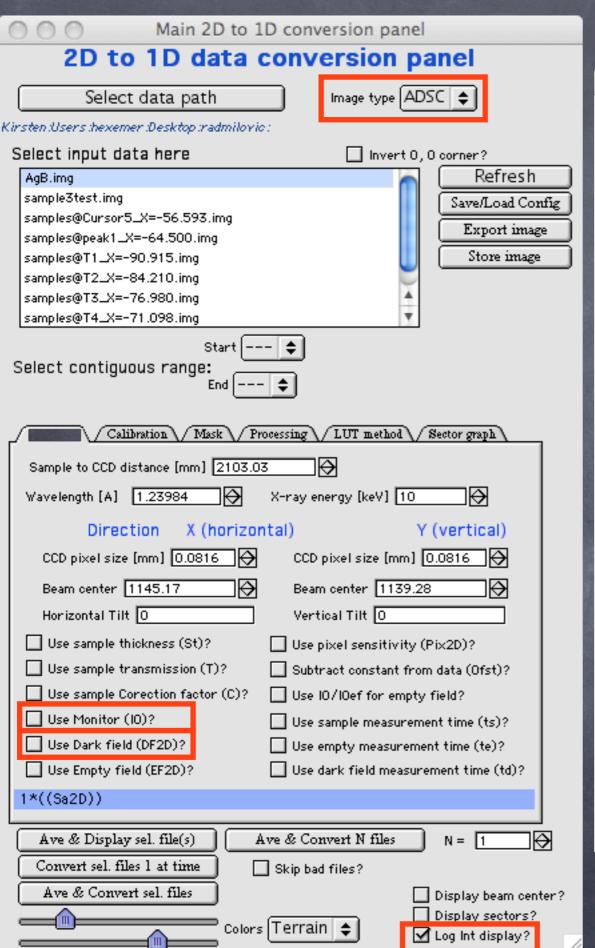


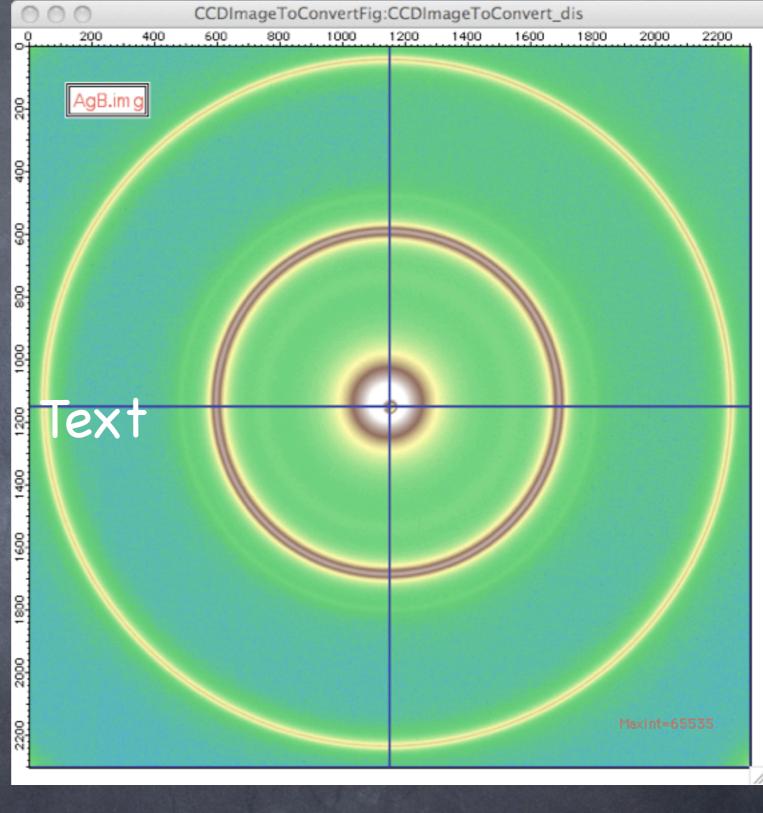


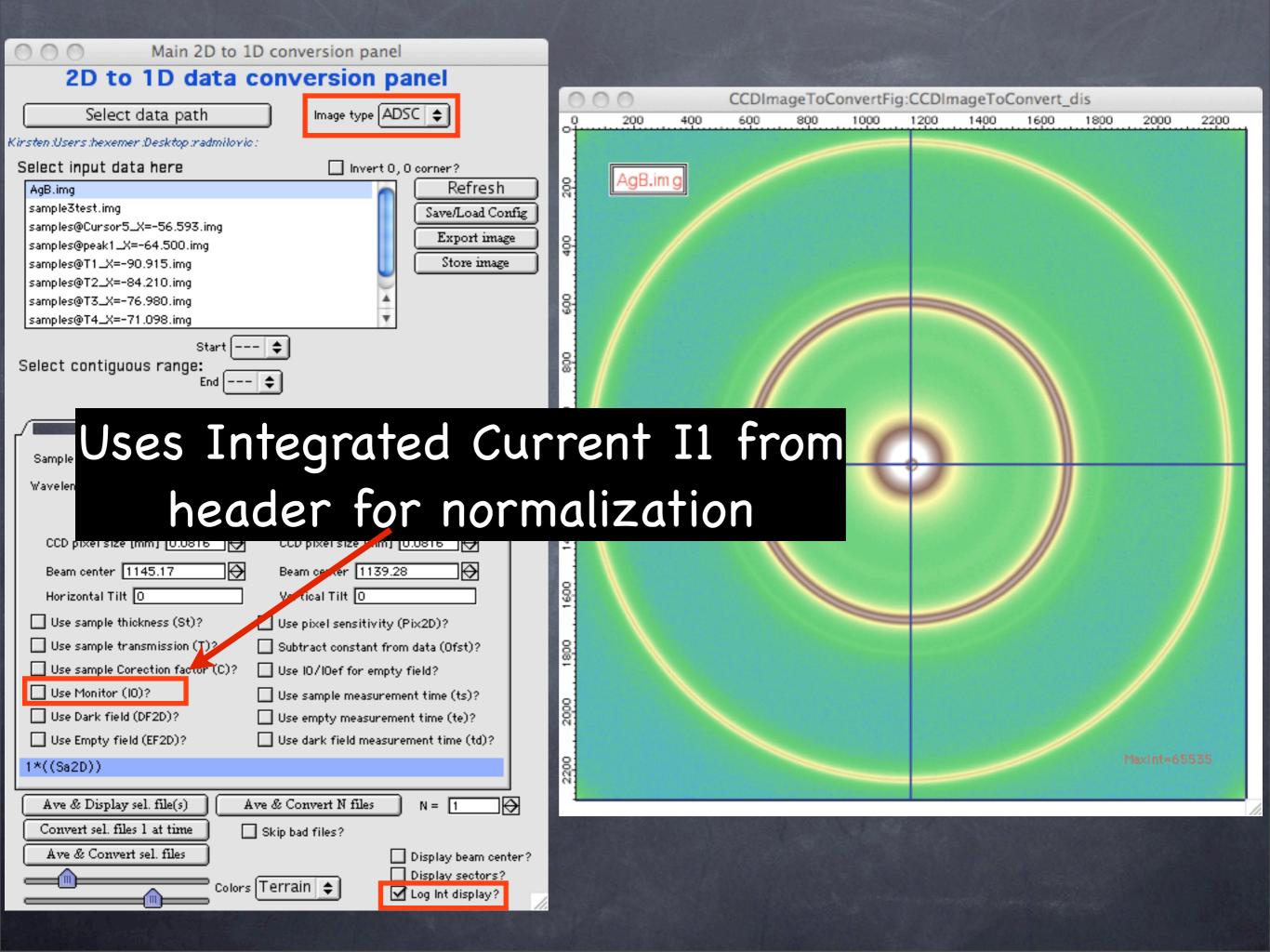


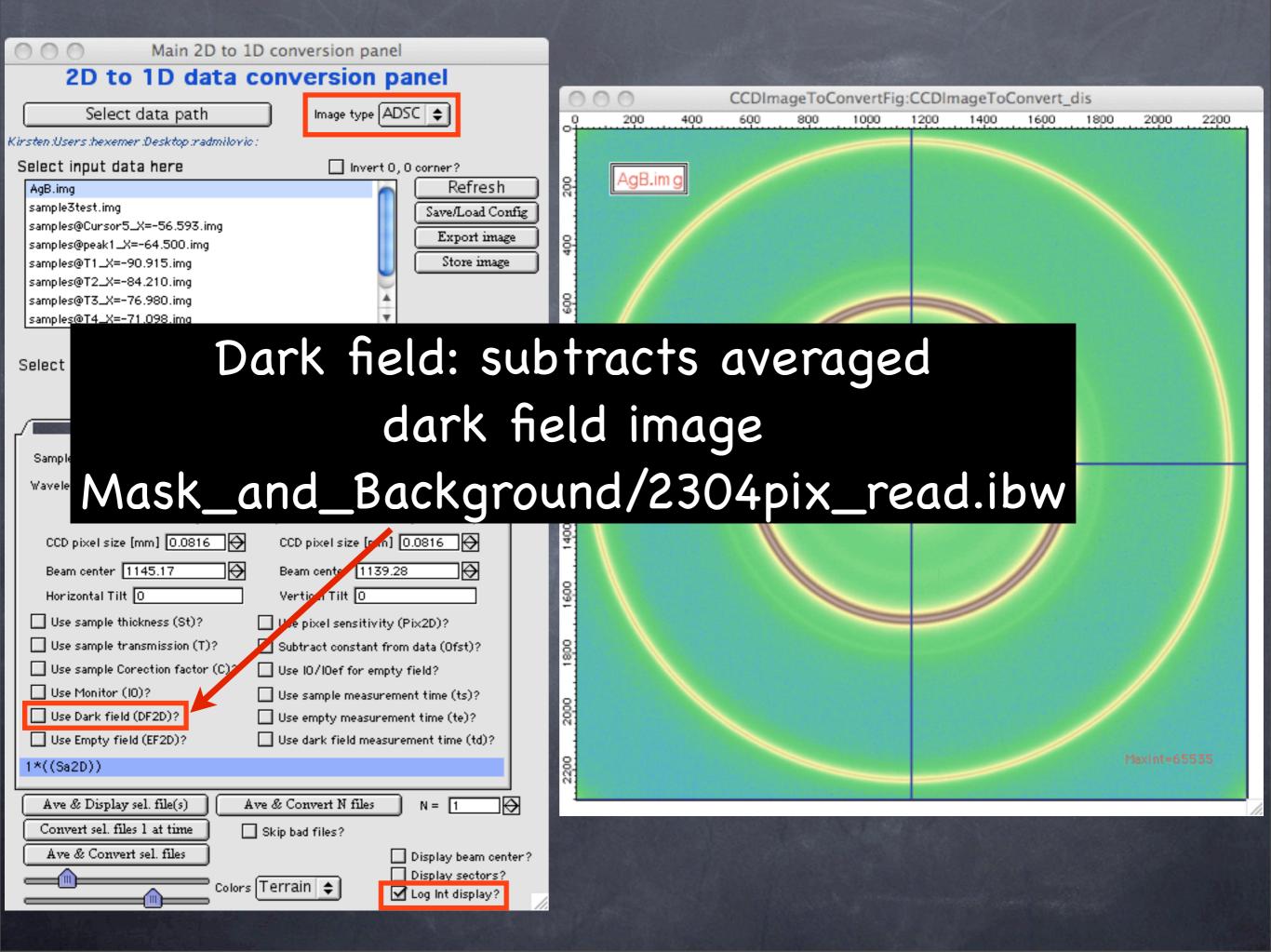


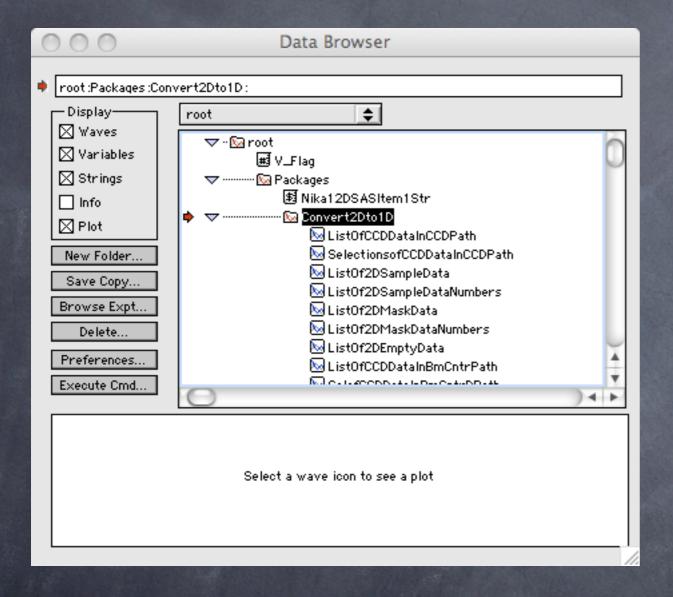


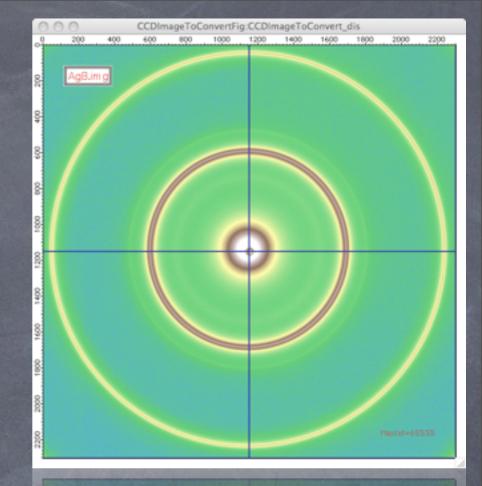


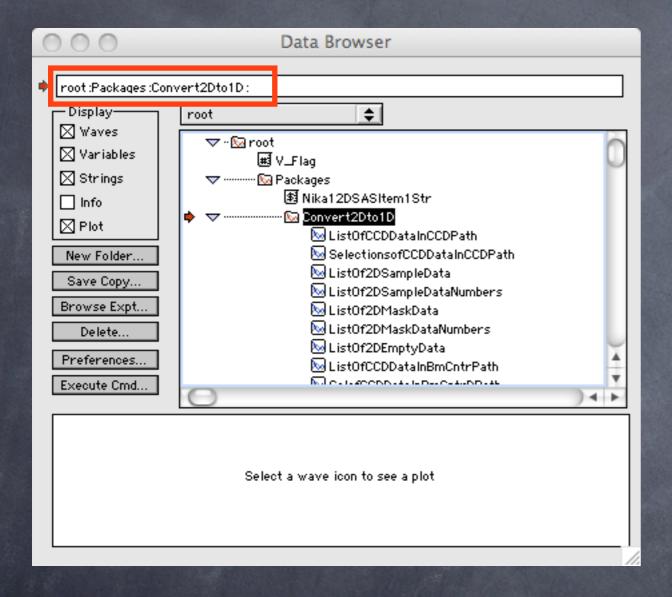


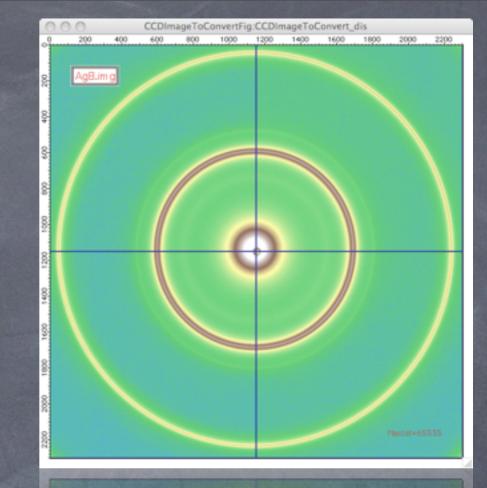


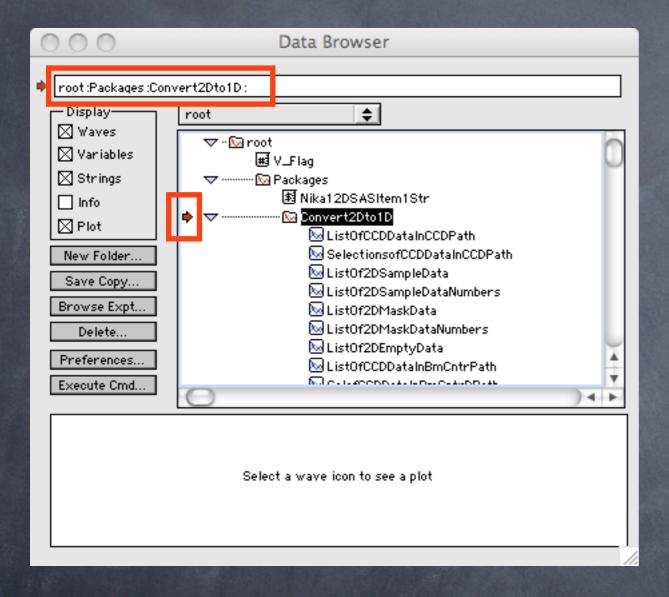


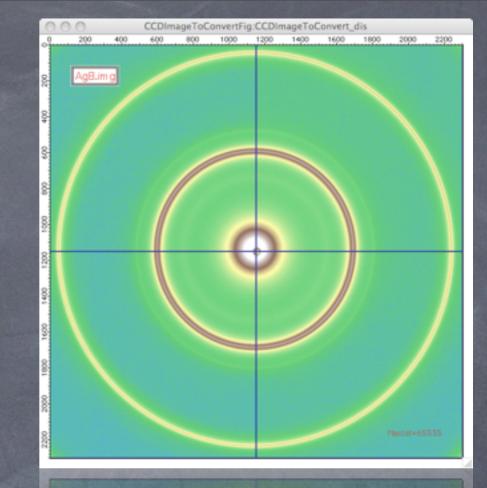


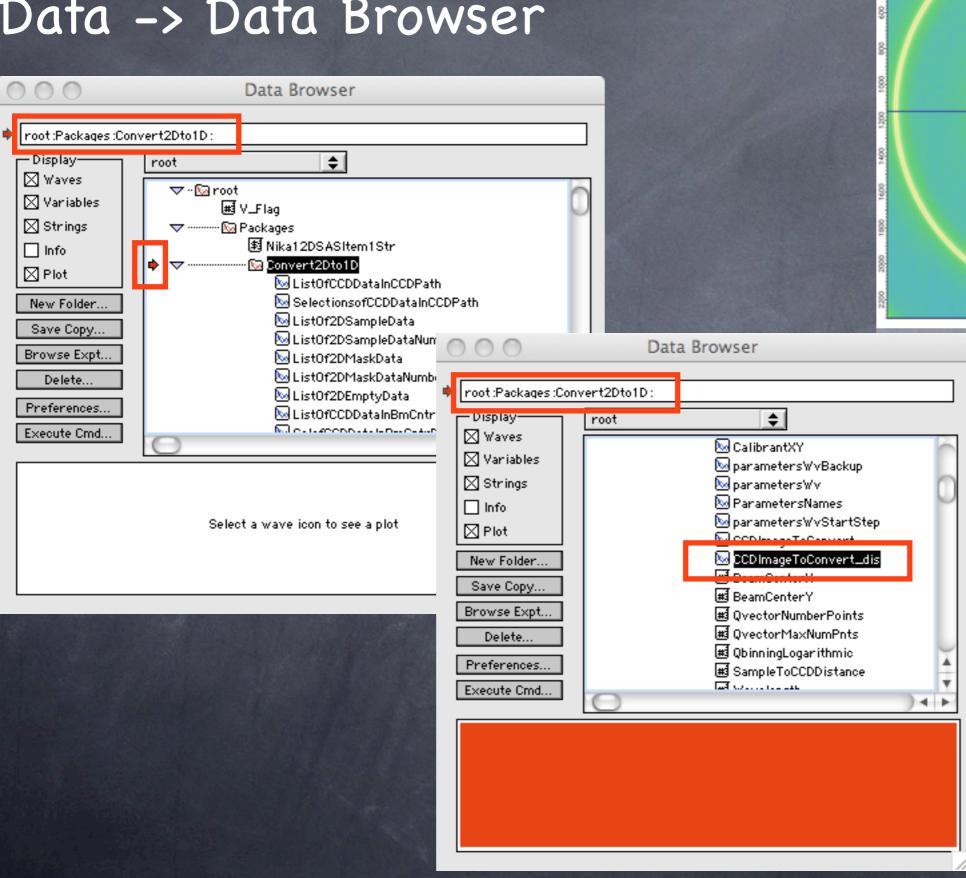


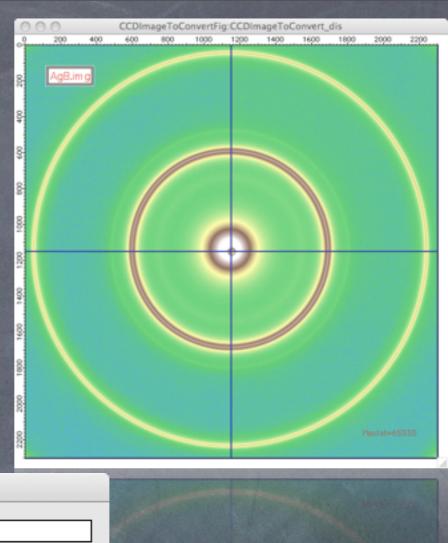


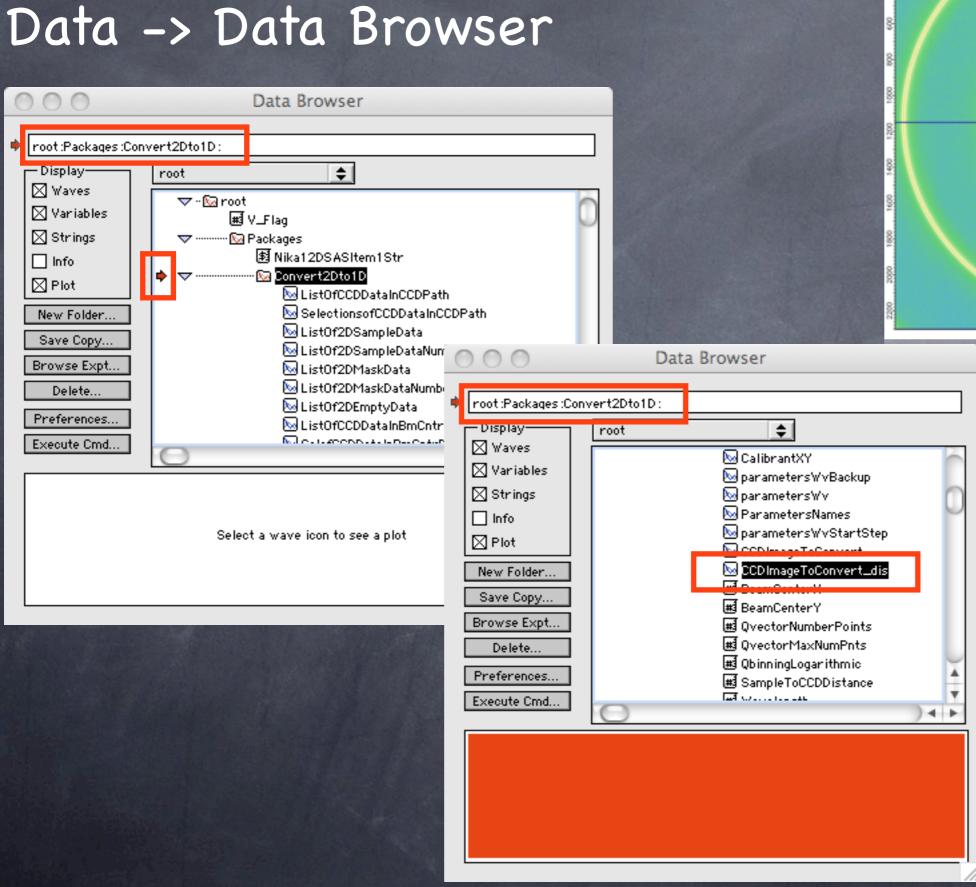


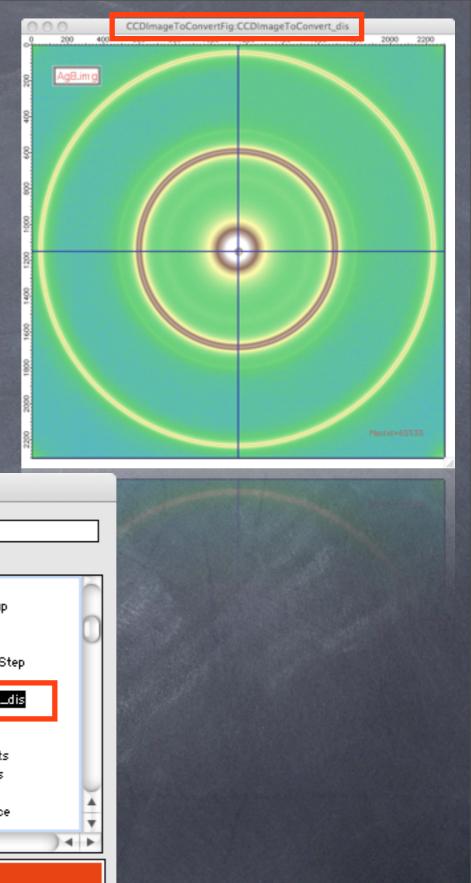


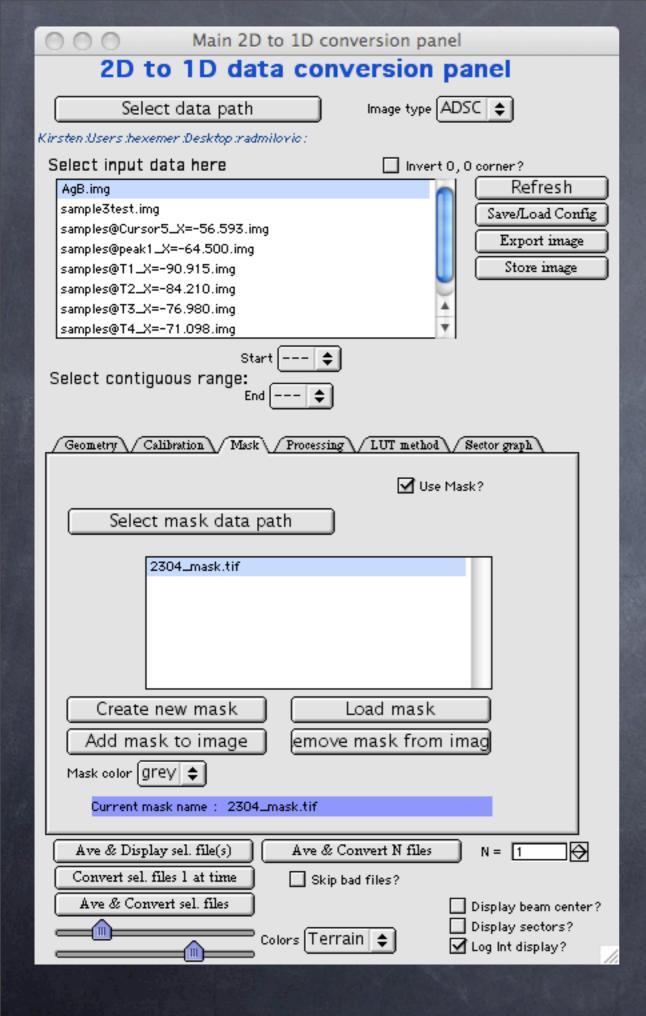






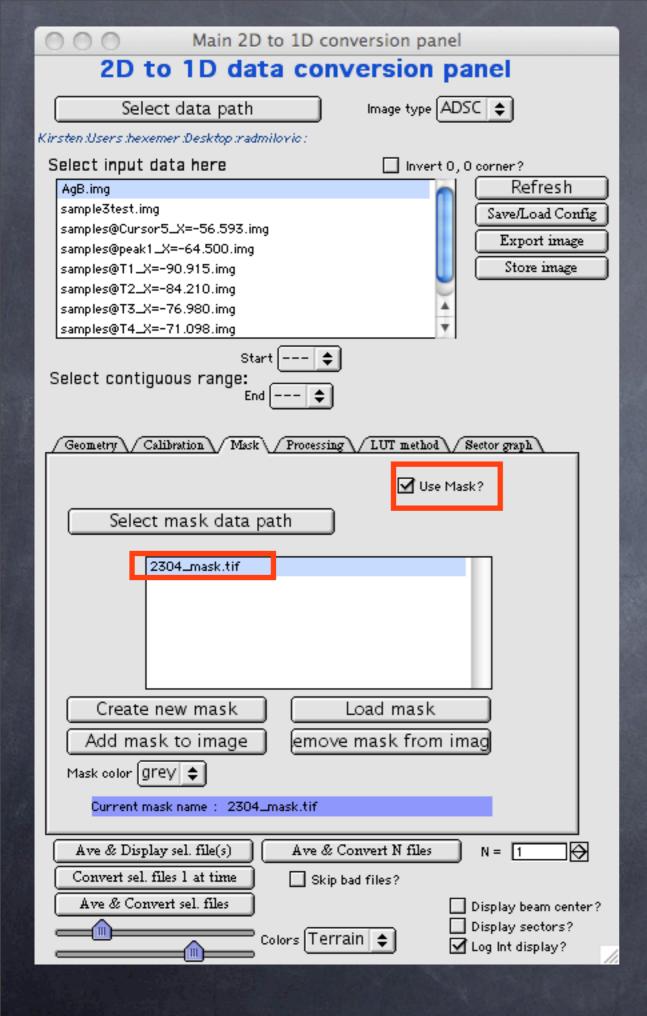






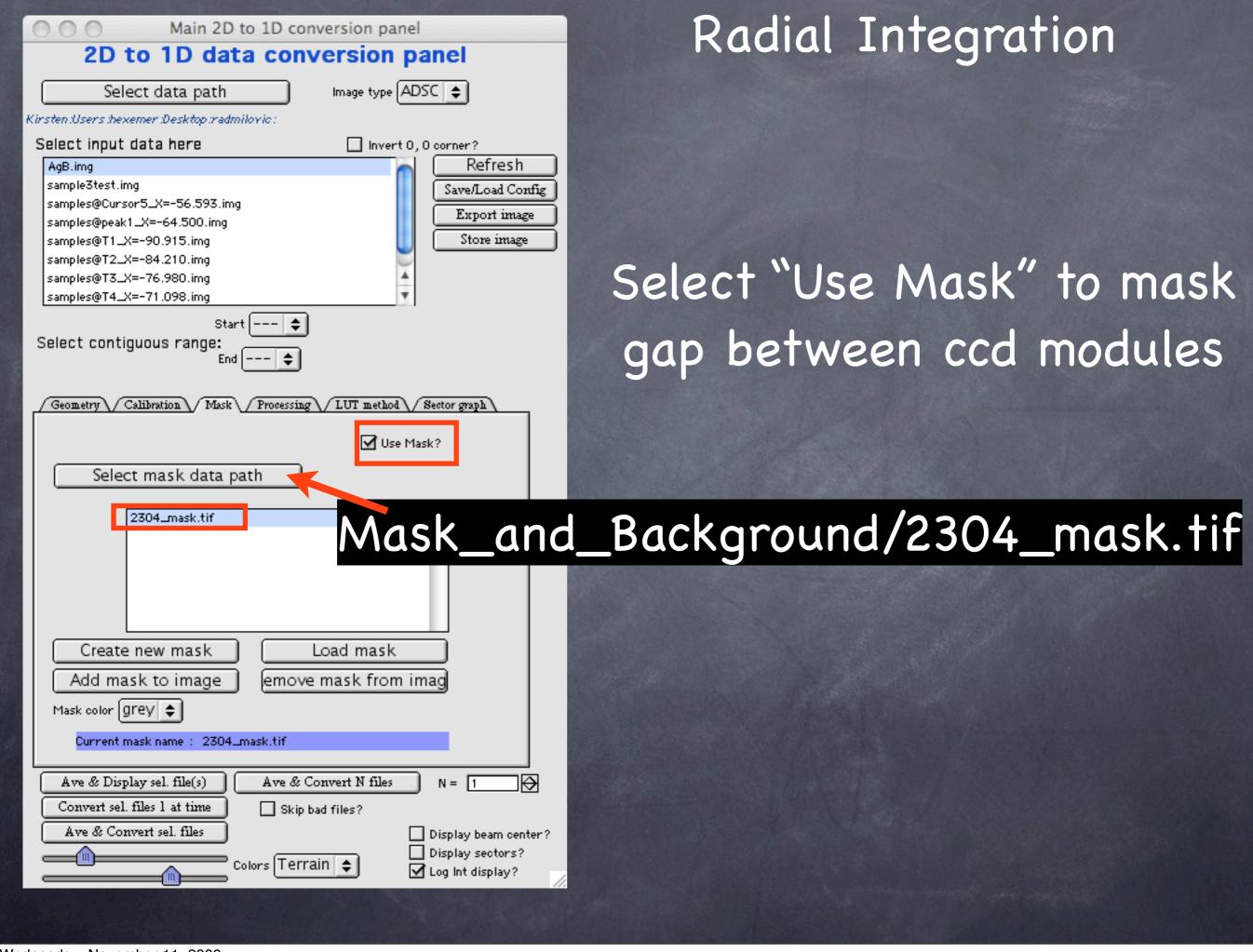
Radial Integration

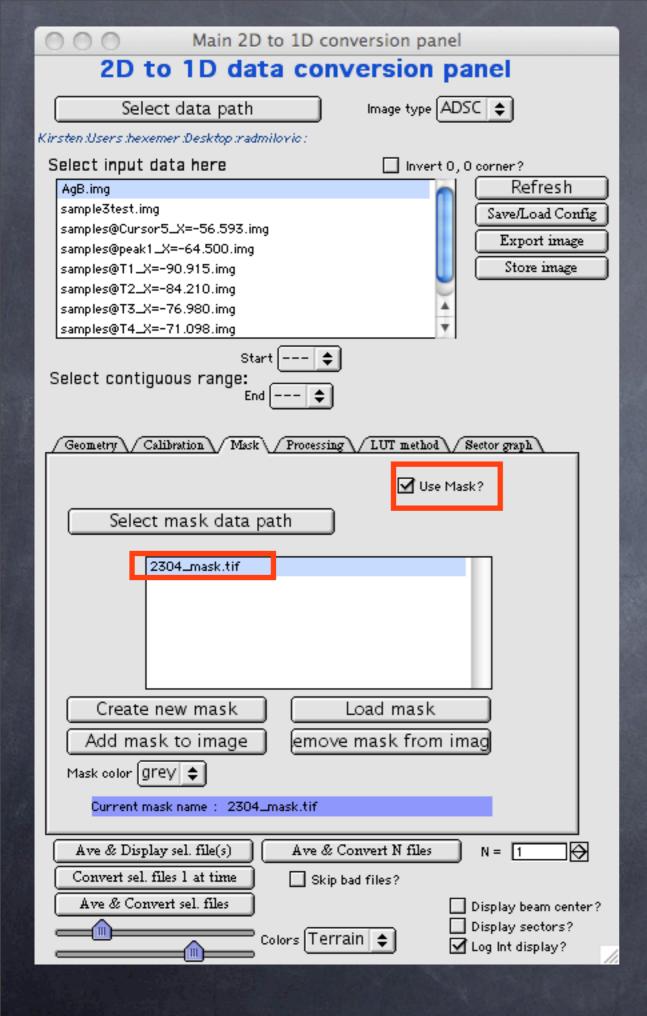
Select "Use Mask" to mask gap between ccd modules



Radial Integration

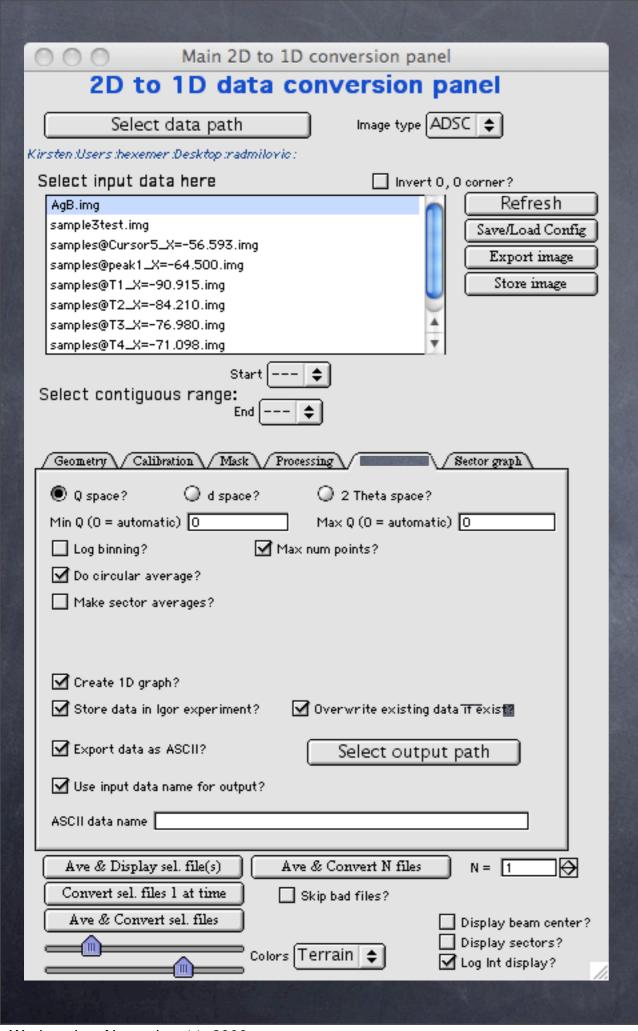
Select "Use Mask" to mask gap between ccd modules

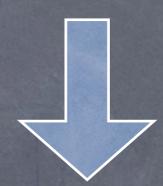


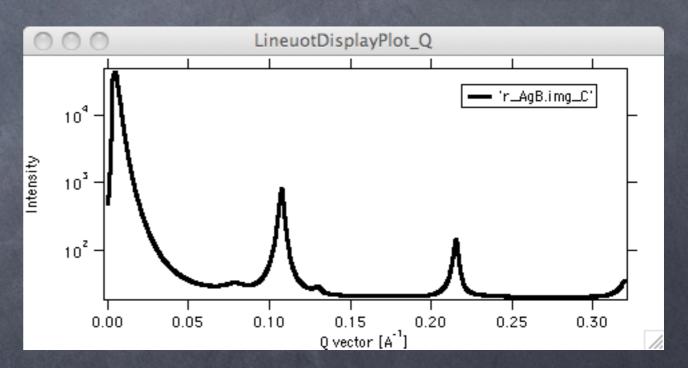


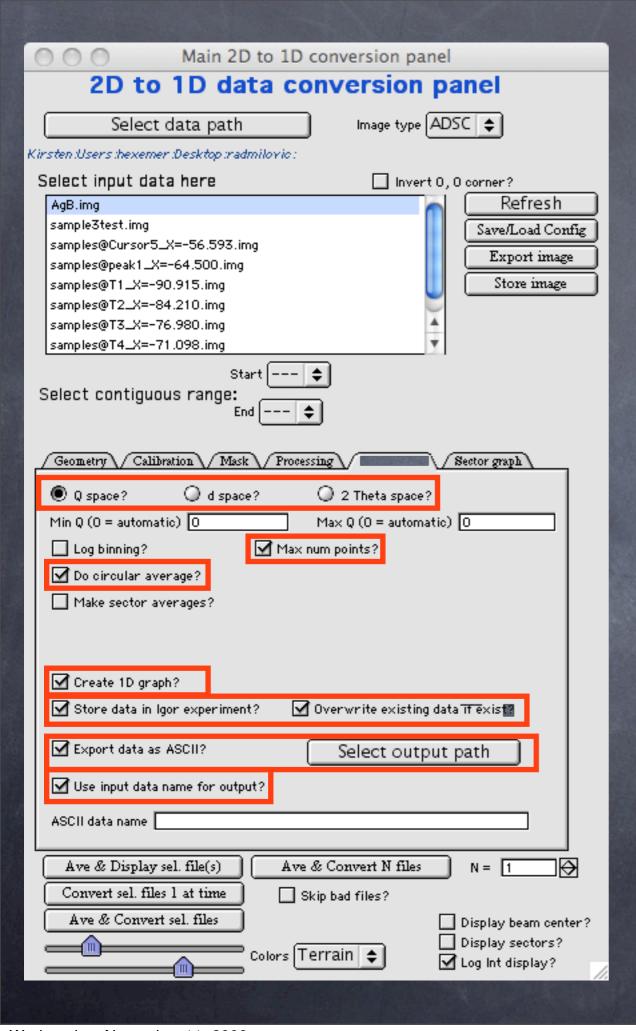
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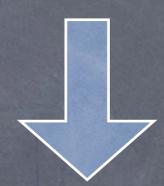
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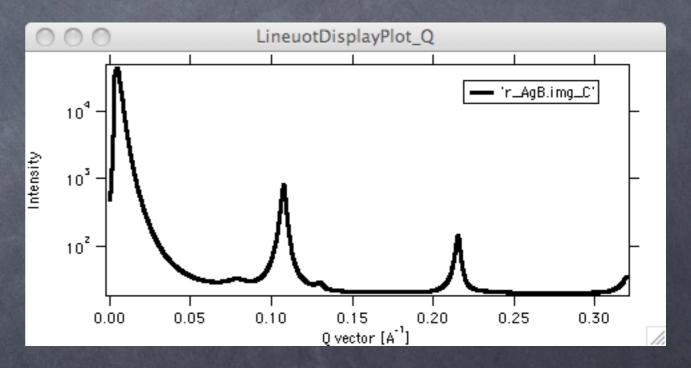


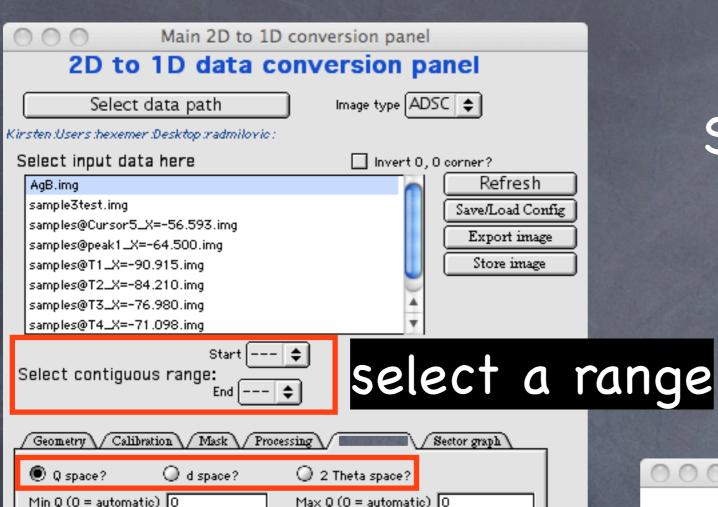


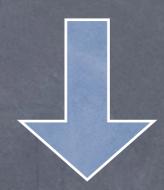


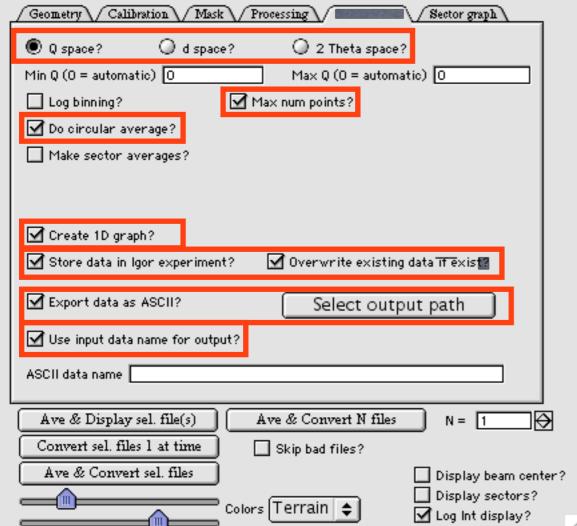


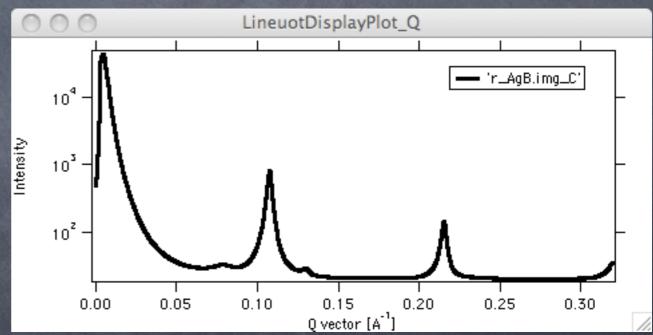


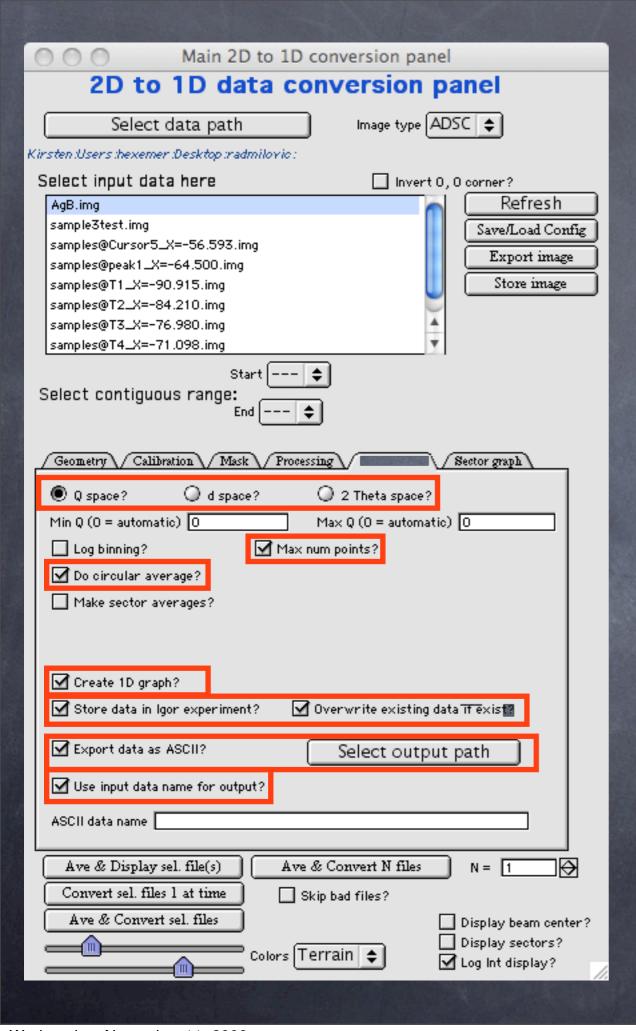


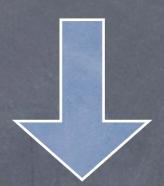


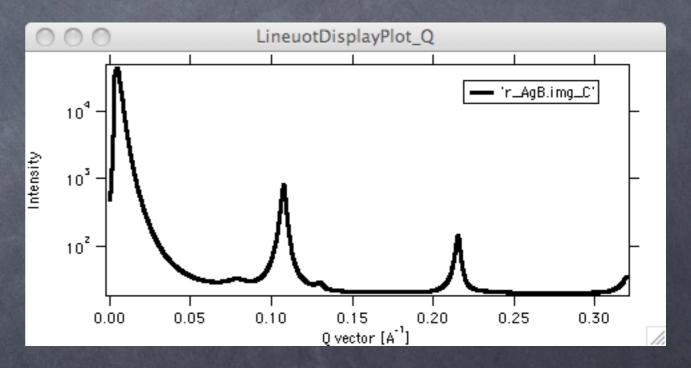




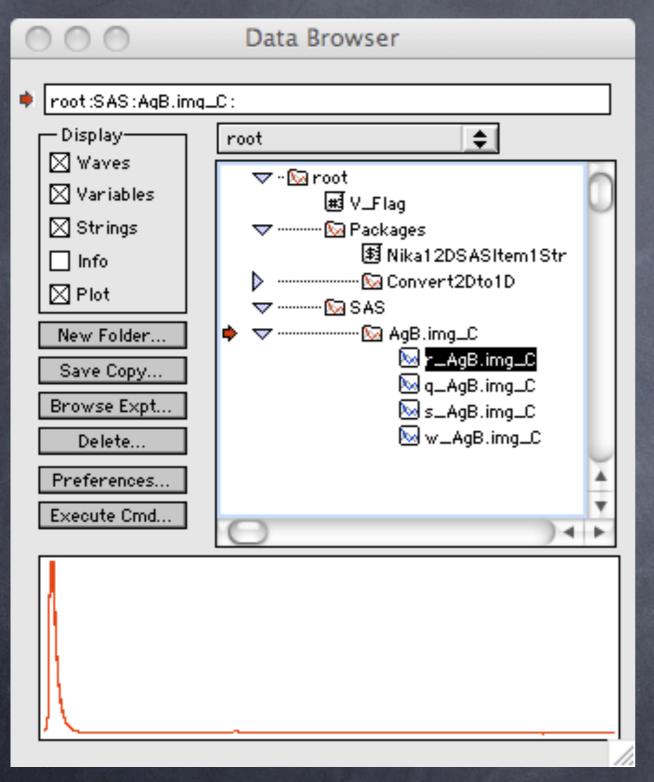


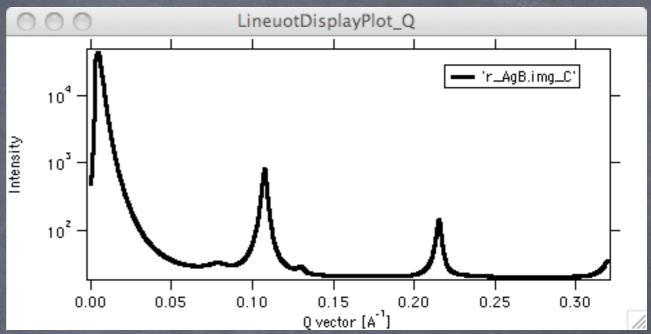






Where is the data?



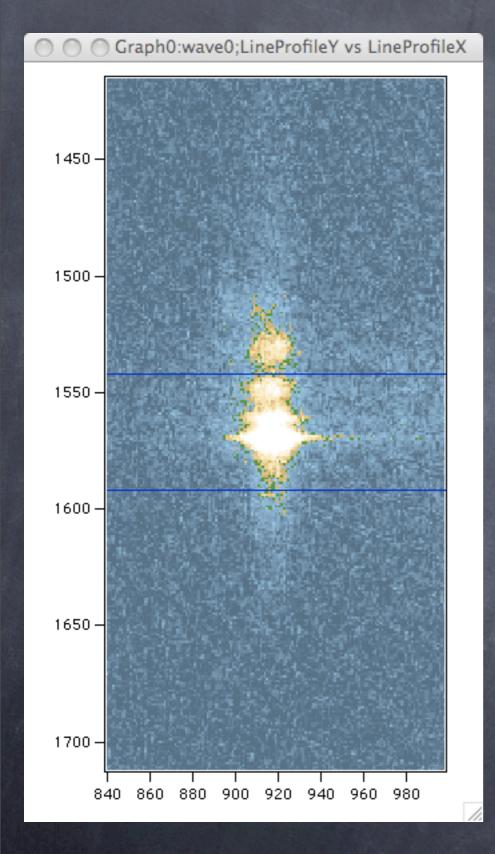


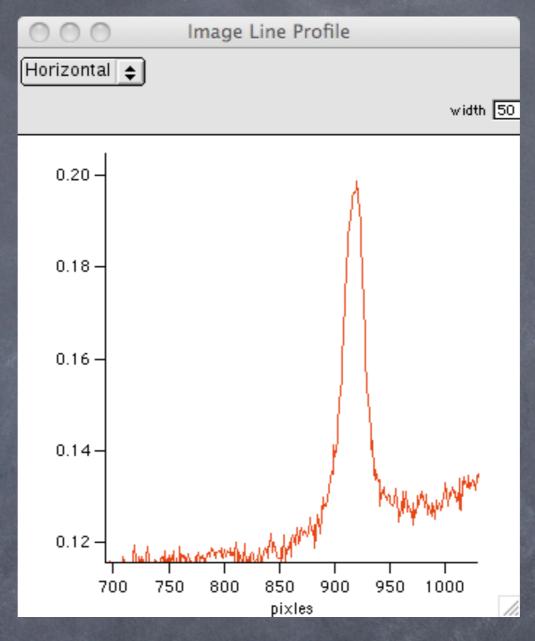
r = Radial intensity

 $q = q_value$

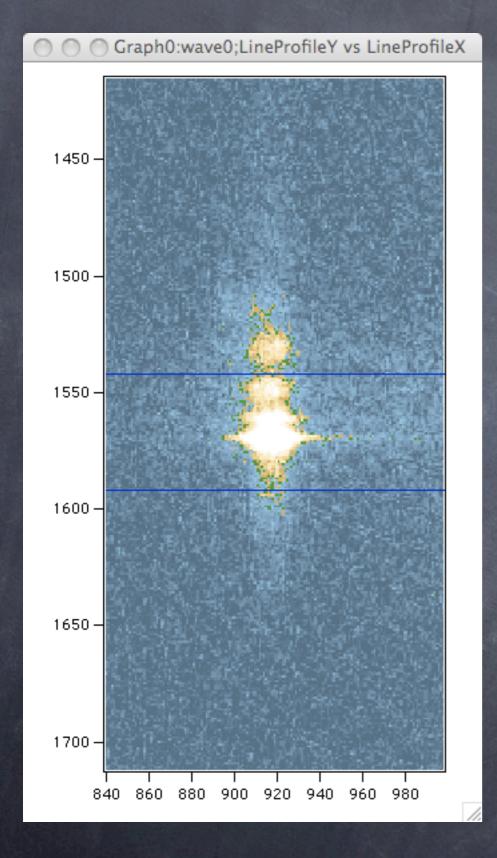
s = error (sqrt(r))

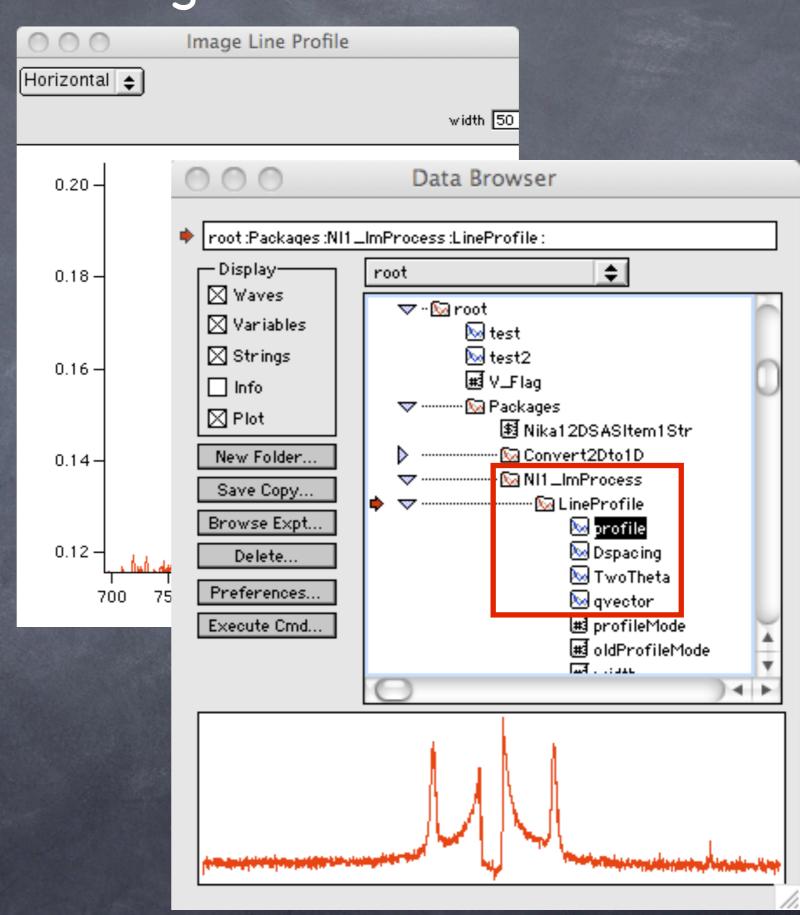
Line integration





Line integration





Before you Leave

- Clean up preparation area and remove your samples
- Close helium/other gasses
- Tell us when you are done collecting data
- Report Errors
- Suggest Improvements
- Getting your data through sftp password: