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

- Beamline Optics
- PSS
- X Ray Hutch
- Shutter & Slits
Monochromator
Monochromator

Coating:
Material pair: Mo/B₄C
d-spacing: 2.0nm +/-0.1nm
N: 250

Substrates:
Material: Silicon <100>

Target Energy: 10keV

E/ΔE = 100
Scatter Slits
Exit Slits
Predicted flux

We measured $1.8 \times 10^{12}$ ph/sec. This is 60% of predicted.

Spot size at sample position: 0.180 mm x 0.780 mm
### Detector Quantum 4

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area</td>
<td>188mm x 188mm</td>
</tr>
<tr>
<td>Pixels</td>
<td>2304 x 2304 pixels</td>
</tr>
<tr>
<td>Pixel Size</td>
<td>82um x 82um</td>
</tr>
<tr>
<td>Read Out Time</td>
<td>9 sec and 3 sec (full resolution)</td>
</tr>
<tr>
<td>Front End Gain</td>
<td>1.1 ADU/photon</td>
</tr>
<tr>
<td>Bits</td>
<td>16</td>
</tr>
</tbody>
</table>
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

Feedback will stir M1 to move beam back between slits

Screen blinking RED means feedback is not working

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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)
Alignment

Beamstop

Direct Beam
Alignment

Hold F and F11 and Click here

Beamstop

Direct Beam
Alignment

Flight Tube Movement Vector

Hold F and F11 and Click here

Beamstop

Direct Beam
Alignment

Flight Tube Movement Vector

Hold F and F11 and Click here

Beamstop Direct Beam
Alignment

Beamstop

Hold d and f and f11 and Click here

Direct Beam
Alignment

Flight tube and detector Movement Vector

Beamstop

Direct Beam

Hold d and f and f11 and Click here
Alignment

Hold d and f11 and Click here

Beamstop

Direct Beam
Alignment

Detector Movement Vector

Hold d and f11 and Click here

Beamstop
Direct Beam

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Alignment

Detector
Movement
Vector

Beamstop

Direct Beam

Hold d and f11
and Click here
Alignment
Alignment

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

x-axis
y-axis

1 -> 0.3 mm
2 -> 1 mm
3 -> 3 mm
4 -> 10 mm

DSC stage
alignment
spot (black)

x-rays

step size

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DSC stage alignment

alignment
spot (black)

step size
1 -> 0.3 mm
2 -> 1 mm
3 -> 3 mm
4 -> 10 mm

DSC stage

x-rays

x-axis

y-axis
DSC stage alignment

alignment spot (black)

step size
1 -> 0.3 mm
2 -> 1 mm
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DSC stage

x-rays

x-axis

y-axis
DSC stage alignment

alignment spot (black)

step size
1 -> 0.3 mm
2 -> 1 mm
3 -> 3 mm
4 -> 10 mm

DSC stage

x-rays

then move 17.22 mm to the right

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GISAXS scan y: set to 1/2 intensity
Adjust x to center sample in beam

GISAXS
GISAXS

adjust x to center sample in beam

scan y: set to 1/2 intensity
GISAXS

adjust x to center sample in beam

scan y: set to 1/2 intensity

Go to Cursor
GISAXS

adjust x to center sample in beam

scan y: set to 1/2 intensity

scan alpha angle: set to max point

Go to Cursor

Go to Cursor
GISAXS

critical angle polymer

critical angle substrate

area to take images

alpha scan
GISAXS

critical angle polymer

critical angle substrate

double click to create cursor

area to take images

alpha scan
GISAXS

critical angle polymer

critical angle substrate

area to take images

alpha scan
GISAXS

critical angle polymer

critical angle substrate

create more cursors

area to take images

alpha scan

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GISAXS

- Critical angle polymer
- Critical angle substrate
- Create more cursors
- Area to take images

Alpha scan

Bookmark Cursors

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GISAXS

move to mark angle

create

alpha scan

Bookmark cursors

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

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GISAXS

move to mark angle

create

alpha scan

Bookmark cursors

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

alpha scan

Bookmark cursors

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

Control Lights

(what's the program doing?)
Motor Disabled
Hutch is not made up
All motors cannot be enabled while Hutch is open
Hutch is made up but motors are disabled
click Enable Motors
Queue is running (scanning and taking images is disabled)
Beamline Software

Motors

Motors are moving, do not scan or move motors while this light is lit.

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Beamline Software

Detector

Detectors are working, do not start another detector run now
Beamline Software

Exposing

X-rays are actually in the hutch

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Temp
Temperature controller is actively cooling or heating
Beam is ready to go in hutch
Beamline Software

If Hutch is closed, and beam is on, feedback should be on if the feedback stays off, even after enabling it. Let us know, image taking is possible, but the beam center may wander.
What is a Queue
Do any number of these actions in a step by checking the corresponding check box:
Do any number of these actions in a step by checking the corresponding check box:

- Move to Bookmarked Motor location
Do any number of these actions in a step by checking the corresponding check box:

- Move to Bookmarked Motor location
- Take a Image
Do any number of these actions in a step by checking the corresponding check box:

- Move to Bookmarked Motor location
- Take a Image
- Change the Temperature of the temperature stage
Do any number of these actions in a step by checking the corresponding check box:

- Move to Bookmarked Motor location
- Take a Image
- Change the Temperature of the temperature stage
- Wait some number of minutes
Do any number of these actions in a step by checking the corresponding check box:

- Move to Bookmarked Motor location
- Take a Image
- Change the Temperature of the temperature stage
- Wait some number of minutes
- Change AUX Labview control
Do any number of these actions in a step by checking the corresponding check box:

- Move to Bookmarked Motor location
- Take a Image
- Change the Temperature of the temperature stage
- Wait some number of minutes
- Change AUX Labview control
- Wait for User OK
Do any number of these actions in a step by checking the corresponding check box:

- Move to Bookmarked Motor location
- Take a Image
- Change the Temperature of the temperature stage
- Wait some number of minutes
- Change AUX Labview control
- Wait for User OK

Click Add or Insert into Queue
Beamline Software
Beamline Software

Motor control

![Motor control interface](image-url)
Motor control
Motor control is done through bookmarks
Motor control
Motor control is done through bookmarks

Bookmarks can be a location of any combination of Motors (x, y, theta, phi, alpha)
Motor control
Motor control is done through bookmarks

Bookmarks can be a location of any combination of Motors (x, y, theta, phi, alpha)
The can be created directly from the “positions tab”
Motor control

Motor control is done through bookmarks

Bookmarks can be a location of any combination of Motors (x, y, theta, phi, alpha)

The can be created directly from the “positions tab” or by bookmarking a cursor location in a sample scan
Motor control
Motor control is done through bookmarks

Bookmarks can be a location of any combination of Motors (x, y, theta, phi, alpha)

The can be created directly from the “positions tab”
or
by bookmarking a cursor location in a sample scan
or
automatically through “multiple steps” tab
Beamline Software

Multiple steps, (Queue Builder)
Beamline Software

Multiple steps, (Queue Builder)

Dimensions : 1 or 2 dimensions

Pick Dimension
Beamline Software

Multiple steps, (Queue Builder)

Dimensions: 1 or 2 dimensions
For each dimension, the List option determines how the program will build the queue. The Options are:

**Pick Dimension**

**Pick List Option**
Beamline Software

Multiple steps, (Queue Builder)

Dimensions: 1 or 2 dimensions
For each dimension, the List option determines how the program will build the queue. The Options are:

- Motor List: Use the list of Bookmarked motor Locations from positions tab
Beamline Software

Multiple steps, (Queue Builder)

Dimensions: 1 or 2 dimensions
For each dimension, the List option determines how the program will build the queue. The Options are:

- **Motor List**: Use the list of Bookmarked motor Locations from positions tab
- **Temperature List**: Prompts for a file which contains columns of Temperature, rate to ramp, and wait time at that temperature, all separated by tabs
Multiple steps, (Queue Builder)

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- **None**: Increment whichever variable from “start” to “stop”, moving by “increment” at each step
Beamline Software

Multiple steps, (Queue Builder)

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The Image root name will be appended for each step.
Handling 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.
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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.
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If an error occurs more than once
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  • First restart detector servers (see next slide)
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When moving a motor Make sure

- Motors are enabled
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When moving a motor, make sure:
- Motors are enabled
- Motor path is not obstructed
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- Motors are enabled
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- Motors are not at end of travel
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- 7.3.3 labview on left-most monitor doesn’t have any errors
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  • Motors are enabled
  • Motor path is not obstructed
  • Motors are not at end of travel
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When moving a motor Make sure:
  • Motors are enabled
  • Motor path is not obstructed
  • Motors are not at end of travel
  • 7.3.3 labview on left-most monitor doesn’t have any errors
  • Then Call Eliot

Otherwise for repeated errors, Write them down or leave them open and Call us.
Beamline Software
ADSC Detector Schematic
Beamline Software
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

If this doesn’t work let us know
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- hit enter and wait for all four connections to be accepted

If this doesn’t work let us know

Wednesday, November 11, 2009
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 inAscii
Beam alignment

Refinement of Beam Center & Calibration

Select data set to use:
- AgB.png
- sample3test.png
- samples@Cursor5_Xc=56.593.png
- samples@peak1_Xc=64.500.png
- samples@T1_Yc=90.915.png
- samples@T2_Yc=64.210.png
- samples@T3_Yc=76.980.png
- samples@T4_Yc=71.098.png

Make Image

Log image?  
Check for Geom. corrs?
Use Mask?

Zoom to area of attention, beam & fit 2D Gaussian or Manually guess

Fit 2D Gaussian

Beam center X = 500  
Beam center Y = 500

Read Cursor A

Display circle?  
Terrain

"Beamcenter"

Wednesday, November 11, 2009
Beam alignment

Select path to data

File type: ADSC

Select data set to use:

Ag8_img
sample1test_img
samples@Cursor5_X=56.593_img
samples@peak1_X=64.500_img
samples@T1_X=90.915_img
samples@T2_X=84.210_img
samples@T3_X=76.980_img
samples@T4_X=71.098_img

Make Image

Log image?  Dezinger?  Use Geom. corrs?  Use Mask?

Fit 2D Gaussian  Read Cursor A

Beam center X = 1150
Beam center Y = 500

Display circle?

"Beamcenter"
Beam alignment

Refinement of Beam Center & Calibration

Select data set to use:

- AgB.img
- sample3test.img
- samples@Cursor5_x=-56.593.img
- samples@peak1_y=-64.500.img
- samples@T1_x=-90.915.img
- samples@T2_x=-84.210.img
- samples@T3_y=-76.980.img
- samples@T4_x=-71.098.img

Make Image

- Log image?
- Dezinger?
- Use Geom. corr?
- Use Mask?

 BeamCntr Calibrant Refinement

Zoom to area of attn. beam & fit 2D Gauss or Manually guess

Fit 2D Gaussian Read Cursor A

Beam center X = 1150  step = 1
Beam center Y = 1150  step = 1

Display circle? 1

Terrain

"Beamcenter"

Wednesday, November 11, 2009
Beam alignment

Refinement of Beam Center & Calibration

Select path to data: ADSC

Select data set to use:
- AgB.img
- sample3test.img
- samples@Cursor5_X=-56.593.img
- samples@peak1_X=-64.500.img
- samples@T1_X=-90.915.img
- samples@T2_X=-84.210.img
- samples@T3_X=-76.980.img
- samples@T4_X=-71.998.img

- Log image?
- No Decinger?
- Use Geom. corrs?
- Use Mask?

- BeamCntr
- Calibrant
- Refinement

Pick calibrant / own param, border lines
- Display?

Calibrant: Ag behenate
- Lineout Intg over (pix) = 5

- Use d1? d1 = 58.38
- Use d2? d2 = 29.185
- Use d3?
- Use d4?
- Use d5?

Terrain

Wednesday, November 11, 2009
Beam alignment

Refinement of Beam Center & Calibration

Select path to data: ADSC

Select data set to use:
- AgB.jpg
- sample3test.jpg
- samples@Cursor5_X=-56.593.jpg
- samples@peak1_X=-64.500.jpg
- samples@T1_X=-90.915.jpg
- samples@T2_X=-84.210.jpg
- samples@T3_X=-76.980.jpg
- samples@T4_X=-71.096.jpg

Log image?  Dezinger?  Use Geom. corrs?  Use Mask?

Calibrant: Ag behenate

Lineout Intg over (pix) = 5

Use d1?  d1 = 58.38  width = 15
Use d2?  d2 = 29.185  width = 15
Use d3?
Use d4?
Use d5?

Terrain
Beam alignment

Refinement of Beam Center & Calibration

Select path to data

File type: ADSC

Select data set to use:
- AgB.img
- sample3test.img
- samples@Cursor5_X=56.593.img
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- samples@T1_X=90.915.img
- samples@T2_X=84.210.img
- samples@T3_X=76.980.img
- samples@T4_X=71.998.img

Make Image

[Checkboxes for Log image, Dezinger, Use Geom. corrs, Use Mask]

[Radio buttons for BeamCntr, Calibr, Refinement]

Select what to refine and run
- Refine beam center?
- Refine Sa-Det distance [mm]
- Refine Sa-Det distance [Wavelength [Å]]
- Refine wavelength [Å]
- Refine tilts
  - Horizontal: 0
  - Vertical: 0

Num sectors = 60

[Checkboxes for Display in image]

Run refinement

Return back

Terrain
Uses Integrated Current I1 from header for normalization
Dark field: subtracts averaged dark field image

Mask_and_Background/2304pix_read.ibw
Where is the data?

Data -> Data Browser
Where is the data?

Data -> Data Browser
Where is the data?

Data -> Data Browser
Where is the data?

Data -> Data Browser
Where is the data?

Data -> Data Browser
Select “Use Mask” to mask gap between ccd modules
Radial Integration

Select “Use Mask” to mask gap between ccd modules
Radial Integration

Select "Use Mask" to mask gap between ccd modules

Mask_and_Background/2304_mask.tif
Radial Integration

Select “Use Mask” to mask gap between ccd modules.
Radial Integration

Select “LUT method”

why does it take a long time for the first image?
Radial Integration
Select “LUT method”

why does it take a long time for the first image?
Radial Integration
Select "LUT method"

Select contiguous range: select a range

why does it take a long time for the first image?

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Radial Integration
Select “LUT method”

why does it take a long time for the first image?
Where is the data?

\[ r = \text{Radial intensity} \]
\[ q = q\_value \]
\[ s = \text{error (sqrt}(r)) \]
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: