



3. SAXS Data Acquisition and Reduction

– a “how to” guide

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Data Acquisition and Reduction

- **Objective**

Obtain 1D scattering curves from 2D patterns of best quality possible, ready to analyze. These curves may have :

- Good sample signal
- Low background noise
- Appropriate q-range and enough angular span

- **Good alignment of holder and selection of geometry**
 - If incorrect, both can create extra background to data
- **Selecting camera configuration to have best instrumental function**
 - Appropriate Distance
 - Adequate slits configuration
 - Correct sample thickness
- **Properly do the calibrations and subtractions**
 - Exposure time and transmission
 - Sample-to-detector distance
 - Absolute Intensity
- **Perform careful SAXS experiment and correct data treatment**

Experiment Overview

Preparing the Experiment

- Select the instrument and choose energy (wavelength) and tube power
- Choose detector(s)
- Select and set sample-to-detector distance (SD)
- Choose sample holder and sample environment
- Select the slit configuration (mirror, HiRes, HiFlux,...)
- Do a sample holder alignment

Data acquisition

- Measure Calibrants:
 - Direct Beam
 - Silver Behenate
 - Glassy Carbon
 - Empty
- **Measure sample(s)**
- Extra Measurements :
 - Windows or empty containers (empty cuvette)
 - Buffer or solution

Data reduction

- Make angular averages of data
- Calculate sample-to-detector distance and center of the beam
- Subtract the background of camera or sample buffer or empty cuvette
- Normalization to: Exposure time, Transmission and Absolute Intensity (if necessary)

Data analysis

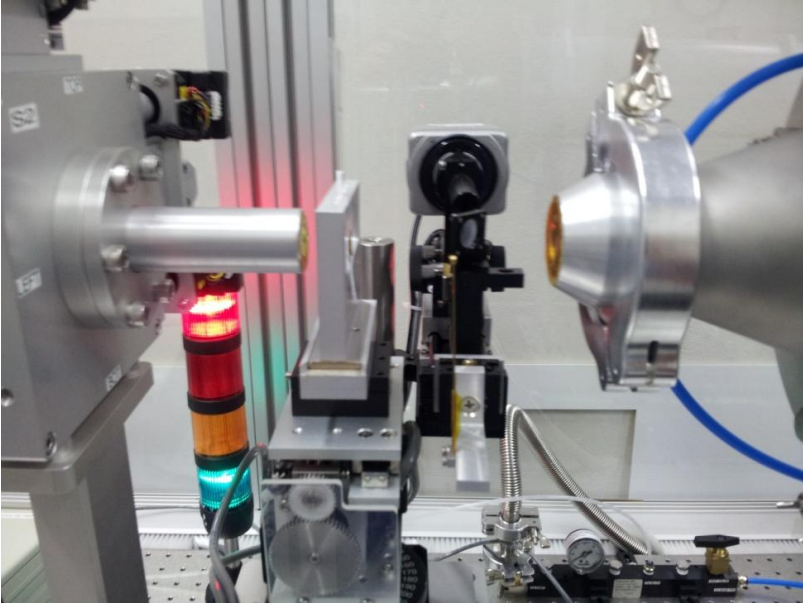
- Modelling and fit of 1-dimensional data (curves)

Data acquisition and treatment

- 1) Measure calibrants and direct beam to do **Angular Integrations** (transforms 2D into 1D)
 - Need the distance SD
 - Need center of beam
 - Need to calculate q-vector
- 2) Measure sample and empties
- 3) Measure transmissions of sample and empties
- 4) Normalize to exposure time and transmission
- 5) Subtract the empties
- 6) Save the data for analyze

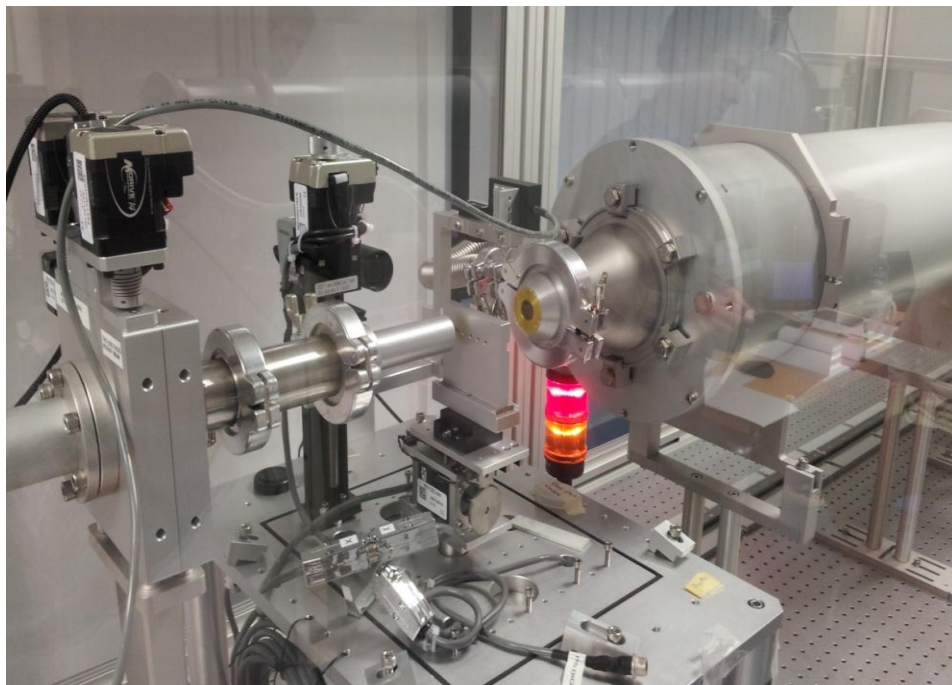
Preparing and Making a Measurement

Prior to measurement

- Fixed Instrument (Xeuss)
 - Multilayer, collimating mirrors
 - Energy and tube power
 - Select Detector type
 - SAXS, WAXS or both
 - Selection of sample holder
 - Large choice for Xeuss
 - Selection of distance sample-to-detector.
 - Modular camera length
- 
- Selection of slit collimation and flux.
 - Double set of slits with motorized independent blades
 - Select Exposure time
 - Noise and statistics

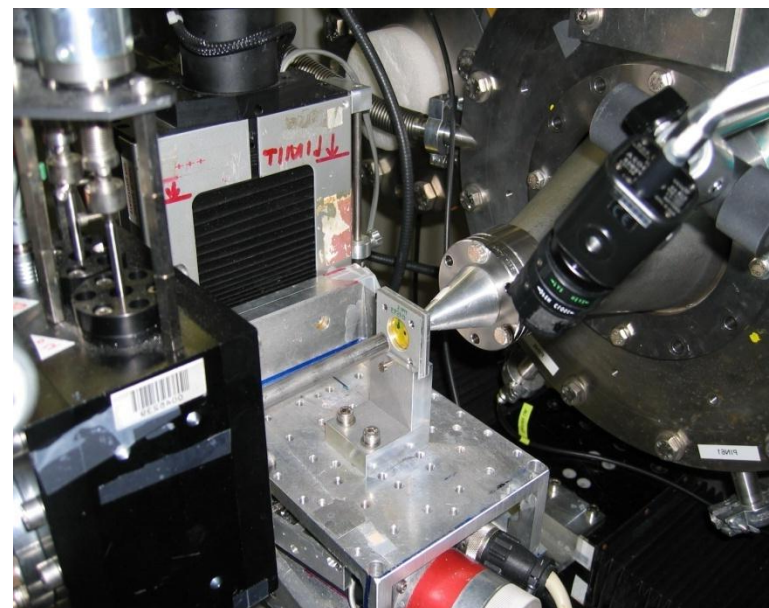
Experiment set-up

Xeuss



- Cu micro source 8 keV.
- Pencil beam 800 x 800 μm or 500 x 500 μm
- Two-dimensional Hybrid pixel detector situated at 0.1 to 6 m from the sample.
- Flux ~ 40 Mph/s
- Scan steps in Z and X and rotation

ID02 ESRF



- Undulator synchrotron source at approximately 50m from the sample. Beam monochromatized to a wavelength of 0.1 nm –or an energy of 12 keV.
- Pencil beam 200 x 200 μm .
- Two-dimensional CCD detector situated at 3-10 m from the sample.
- Flux very high
- Scan steps in Z and Y 1-0.25 mm.

Selecting the sample-to-detector Distance

Q_{\min} , is defined by the beamstop and the divergency

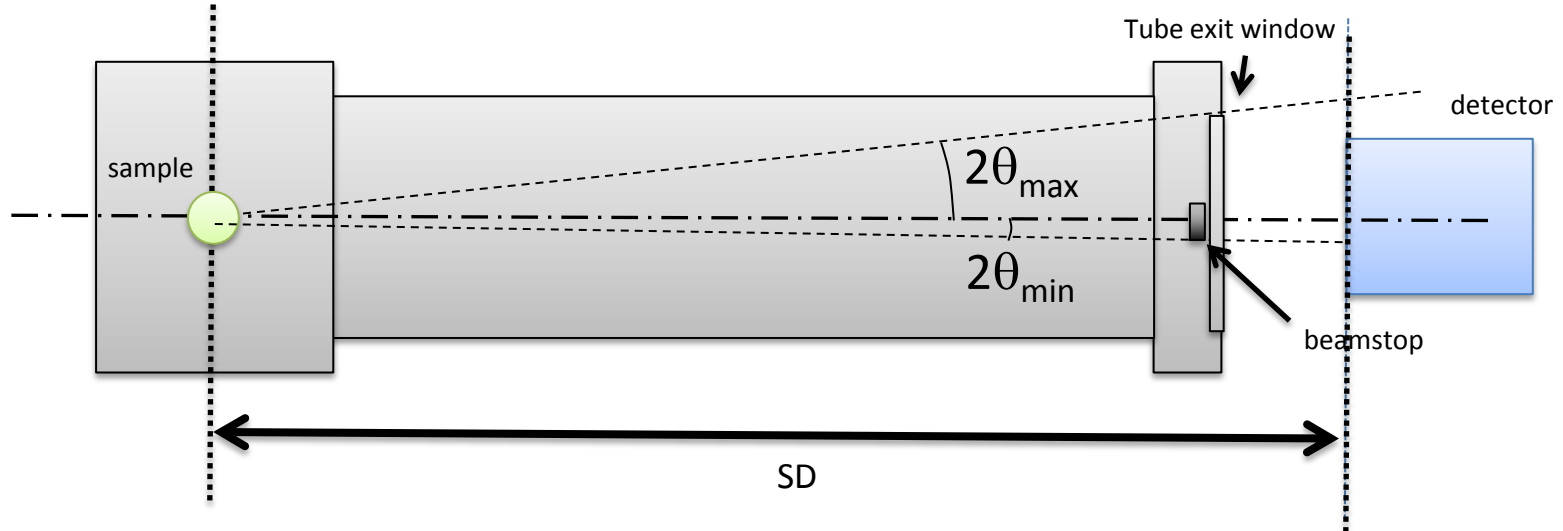
$$Q_{\min} = \frac{4\pi \sin \theta_{\min}}{\lambda}$$

$$d_{\min} = 2\pi / Q_{\max}$$

Q_{\max} absolute, is defined by the exit window. Q_{\max} defined by the farrest edge of the detector.




$$Q_{\max} = \frac{4\pi \sin \theta_{\max}}{\lambda}$$

$$d_{\max} = 2\pi / Q_{\min}$$

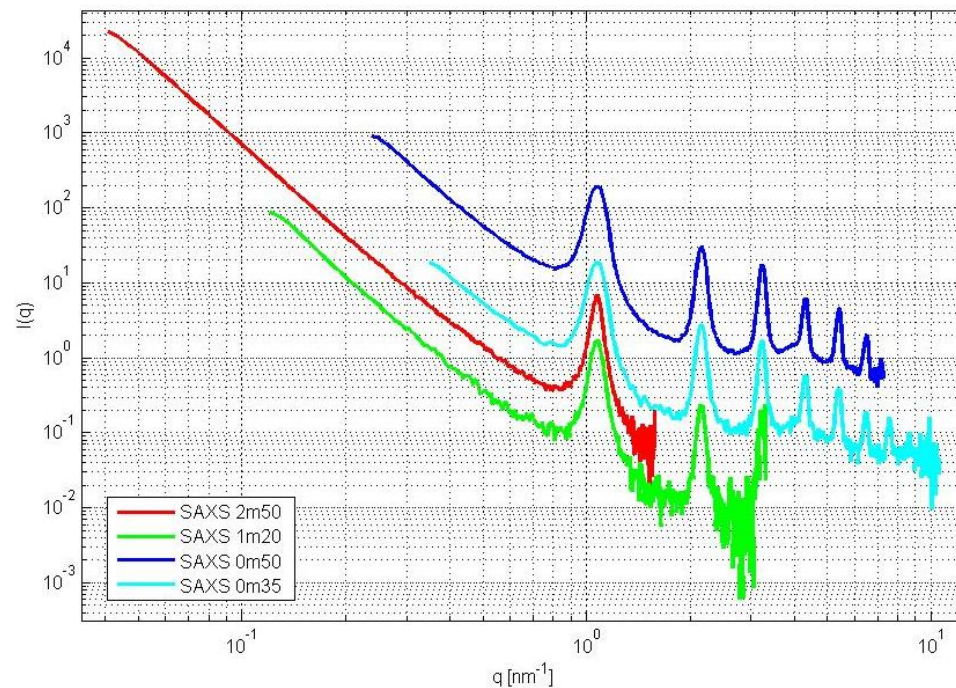


Q-range (aprox.) as function of SD distance

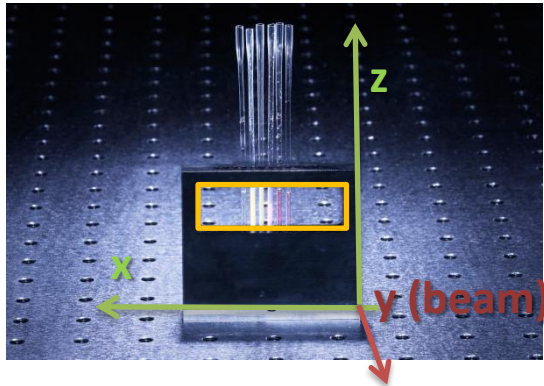
Beam on geometrical center of detector and beamstop

SD [mm]	Pipe Sections	q_{\min} [nm^{-1}]	q^*_{\max} [nm^{-1}]	Characteristic Dimension [nm]
2485		0.042	2.21	from 2.8 to 150
1190		0.085	4.58	from 1.4 to 73
538		0.18	9.8	from 0.64 to 34
360		0.27	14.2	from 0.44 to 23

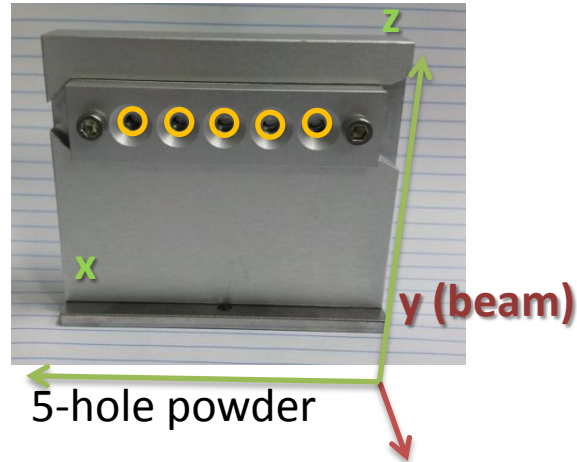
Cu radiation



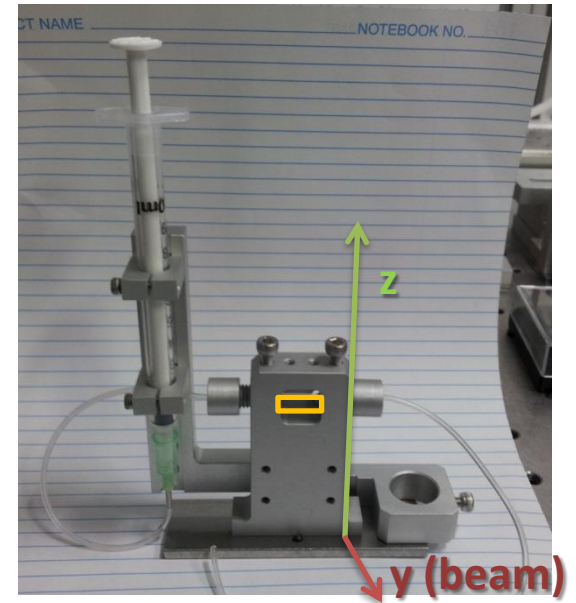
Select Appropriate Sample holder



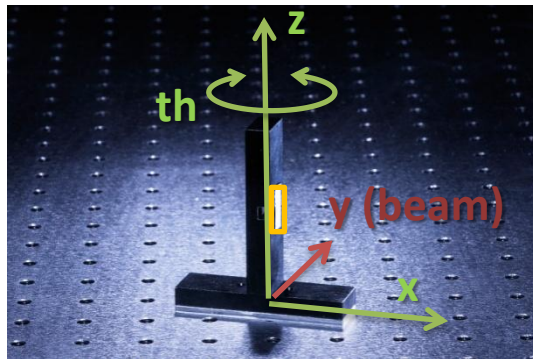
Multicapillary



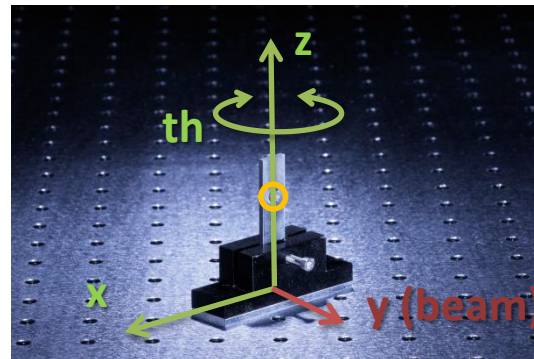
5-hole powder



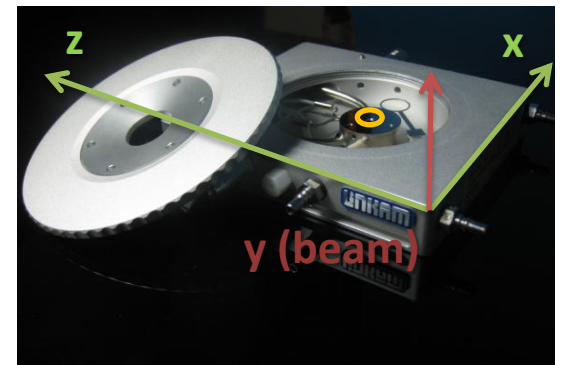
Flow-through



GiSAXS



Alignment tool



Linkam temperature

X-ray energy and sample thickness

- Optimal thickness

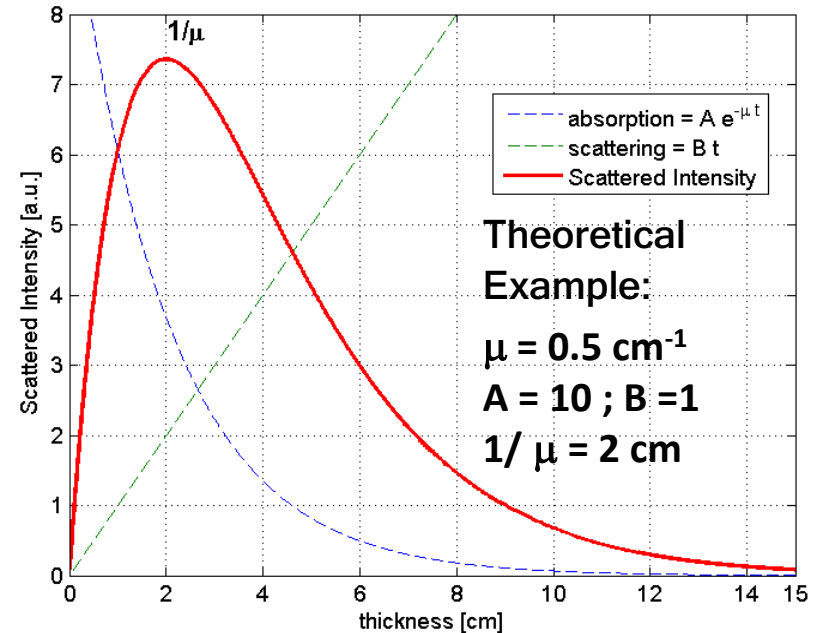
Are we going through? Is the energy enough?

Absorption $\exp(-\mu t)$

How much the radiation is absorbed? Scattering is linear with thickness

- Fluorescence. Is Cu radiation ok if sample contains iron?

Fluorescence (resonant) gives high background level and spoils scattering signal (non-resonant).

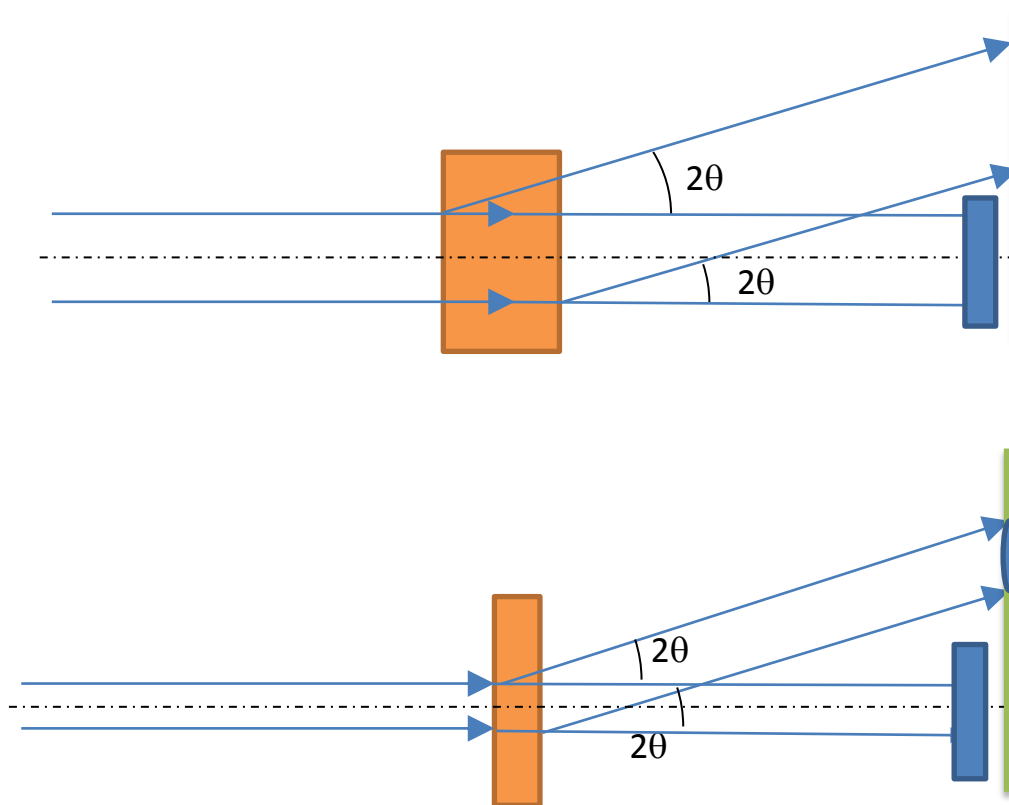


Optimal thickness : $1/\mu$

m : function of radiation energy and atomic number

Cu Radiation and Carbon: $1/\mu = 1 \text{ mm}$

Choice of Slits – Instrumental Function



Slits and Flux

Two set of slits S1 and S2. Every slit set consists of 4 independent blades. Apertures are usually squared, but asymmetrical geometries are also possible.

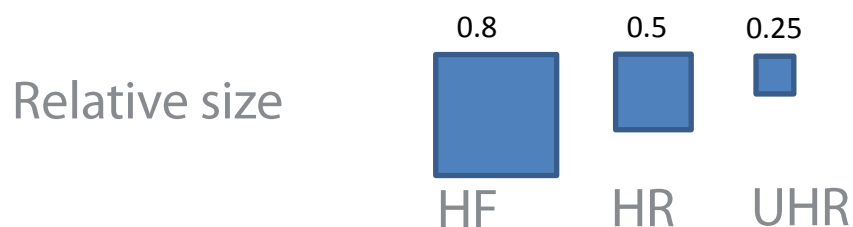
- Close the slits for **High Resolution (HR)** and better resolution. Flux drops. Instrumental function thinner.
- Open the slits for **High Flux (HF)**. But divergency increases. Instrumental function widens.

Compromise between flux and resolution!

Slits and Flux

Name		S1 (mm)	S2 (mm)	Relative Flux ¹
UHR	Ultra-high Resolution	0.3 x 0.3	0.25 x 0.25	0.097
HR	High Resolution	0.6 x 0.6	0.5 x 0.5	0.39
HF	High Flux	1.2 x 1.2	0.8 x 0.8	1.00
VHF	Very high Flux	1.5x1.5	1.0x1.0	--
FO	Full Open	8.0 x 8.0	8.0 x 8.0	No collimation

¹Relative to High Flux



Preparing a Measurement

Calibration

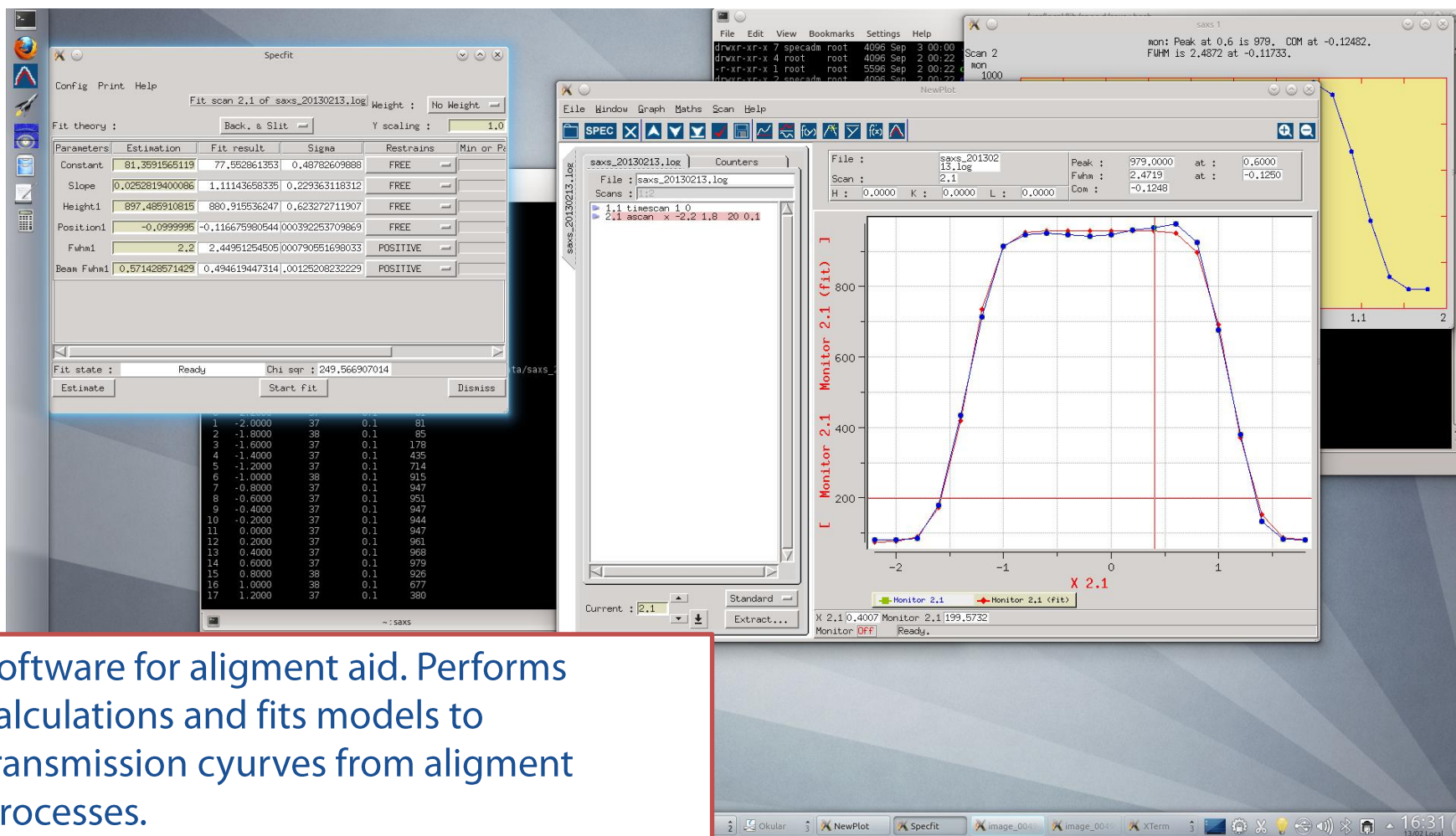
- Empty camera
- Ag Behenate
 - For distance calibration and q-vector construction
- Glassy carbon
 - For absolute intensity normalization and creation of mask (shadows)
- Direct Beam
 - For center determination of the center of the beam





Data Acquisition software

Newplot



Software for alignment aid. Performs calculations and fits models to transmission curves from alignment processes.

SPEC front-end graphical interface / SPECfe

Both command-line and graphical interface control.

The screenshot displays the SPECfe graphical user interface (GUI) and a terminal window. The GUI window, titled 'specfe', features a menu bar (File, Edit, Graph, Scans, Commands, Help) and a toolbar with buttons for Abort, Print, Prev Scan, Next Scan, Legend, Autoscale, Save Reference, Clear Reference, Open Shutter, and Close Shutter. The status bar indicates 'Status: Scan 31' and 'Xenocs Interface to Spec // specfe 1.5.32'. The main window contains a graph titled 'Graph 1' with a y-axis from 0 to 1 and an x-axis labeled 'Time' from 0 to 1. A legend on the right shows two 'Monitor' entries. Below the graph is a table for 'Current Scan' and 'Reference' with columns for Column, X, Y1, Y2, and Colour. The terminal window, titled '~soft_for_saxs/specfe/specfe-xenocs_dist : wish', shows the following output:

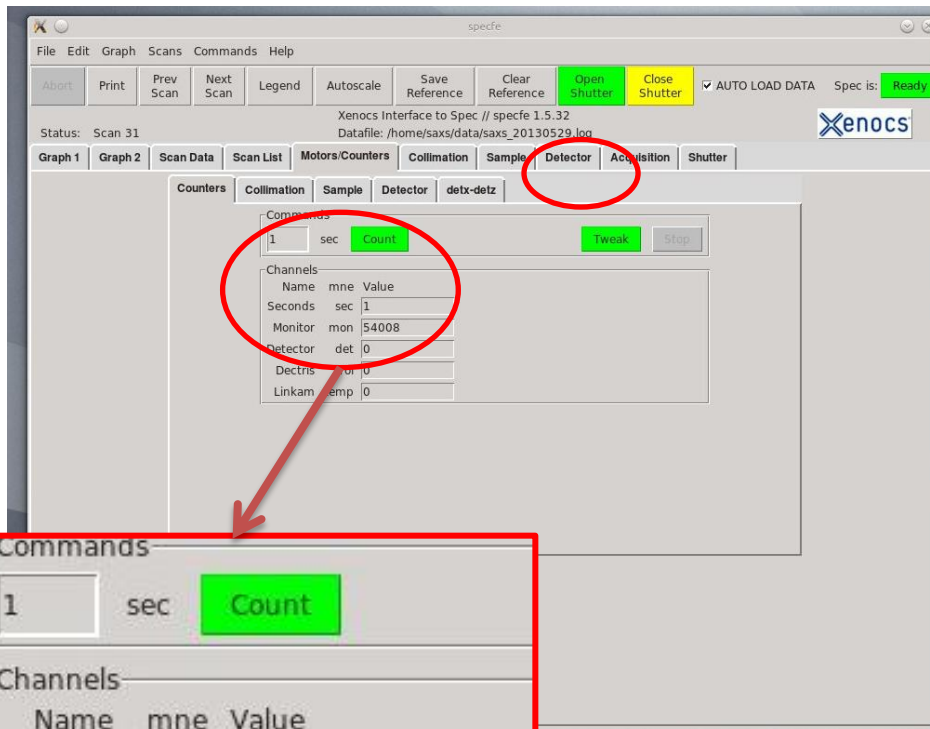
```

Found MDrive motor "Q" (bs2) with firmware 3.006.
Found MDrive motor "T" (th) with firmware 3.013.
Found MDrive motor "U" (detc) with firmware 3.013.
Found MDrive motor "V" (detc) with firmware 3.013.
Found MDrive motor "X" (s) with firmware 3.010.
Found MDrive motor "Z" (z) with firmware 3.013.
Not compiled for the "EPICS_PV" controller needed for scaler "roi".
=
spec Hot Line: (617) 576-1610.
Type h changes for info on latest changes.
Browse to http://www.certif.com for complete documentation.
=
Reading file "/home/saxs/soft_for_saxs/specfe/specfe-xenocs_dist/spec.mac".
detc
detc
1641_SAXS> so
X-RAY SHUTTER OPEN
1642_SAXS> sc
X-RAY SHUTTER CLOSED
1643_SAXS> so
X-RAY SHUTTER OPEN
1644_SAXS> sc
X-RAY SHUTTER CLOSED
1645_SAXS> -|
  
```

The taskbar at the bottom shows several open applications, including 'specfe', 'NewPlot', 'Specfit', and 'temp.tif'. The system tray on the right shows the time as 09:21 and the date as 31/03/2011.

SPECfe

Counting



```
1647.SAXS> ct 1  
  
Fri May 31 09:22:52 2013  
  
Seconds = 1  
Monitor = 54008 (54008/s)  
Detector = 0 (0/s)  
Dectris = 0 (0/s)  
Linkam = 0 (0/s)  
  
1648.SAXS> |
```

```
~/soft_for_saxs/specfe/specfe-xenocs_dist: wish  
File Edit View Bookmarks Settings Help  
1643.SAXS> so  
X-RAY SHUTTER OPEN  
1644.SAXS> sc  
X-RAY SHUTTER CLOSED  
1645.SAXS> ct 1  
Fri May 31 09:22:46 2013  
  
Seconds = 1  
Monitor = 813 (813/s)  
Detector = 0 (0/s)  
Dectris = 0 (0/s)  
Linkam = 0 (0/s)  
1646.SAXS> so  
X-RAY SHUTTER OPEN  
1647.SAXS> ct  
Fri May 31 09:22:52 2013  
  
Seconds = 1  
Monitor = 54008 (54008/s)  
Detector = 0 (0/s)  
Dectris = 0 (0/s)  
Linkam = 0 (0/s)  
1648.SAXS> |
```

Commands

1 sec **Count**

Channels

Name	mne	Value
Seconds	sec	1
Monitor	mon	54008
Detector	det	0

SPECfe

Slit collimation and Flux

- High flux (HF). Nominal aperture.
S1 1.2 mm / S2 0.8 mm
 - High resolution (HR).
S1 0.6 mm / S2 0.5 mm
- Slits are smaller and but the flux is much reduced.

File Edit Graph Scans Commands Help

Abort Print Prev Scan Next Scan Legend Autoscale Save Reference Clear Reference Open Shutter Close Shutter AUTO LOAD DATA Spec is: Ready

Xenocs Interface to Spec // specfe 1.5.32
Status: Scan 33 Datafile: /home/saxs/data/saxs_20130529.log

Graph 1 Graph 2 Scan Data Scan List Motors/Counters Collimation Sample Detector Acquisition Shutter

Center Beam
 S1 S2 Final Gap : 2 Center

Collimation Mode
High Resol. High Flux Full Open

Slits	Vert. Gap	Hor. Gap	Vert. Off	Hor. Off	
S1	1.20	x 1.20	-0.00	0.00	Move
S2	0.80	x 0.80	-0.00	-0.00	Move

Reset

Predefined apertures

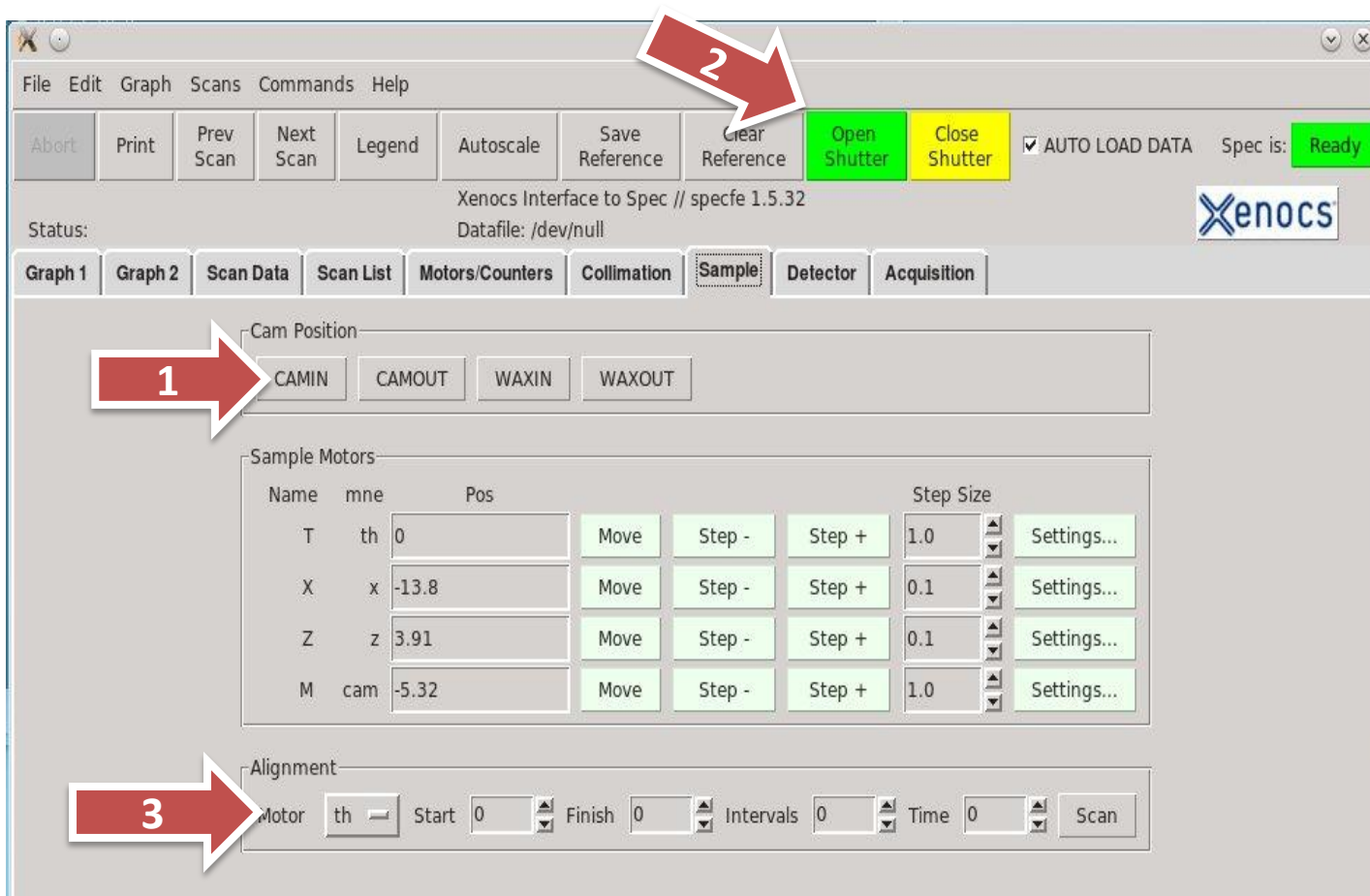
-Any aperture - Asymmetry

Auto alignment.

SPECfe

Sample holder alignment

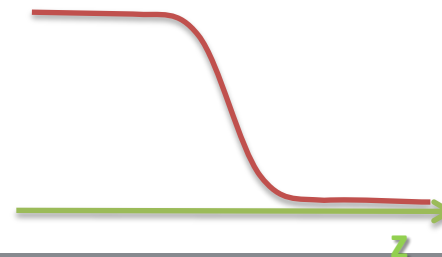
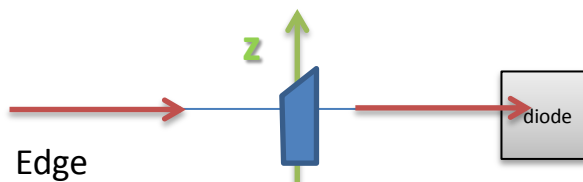
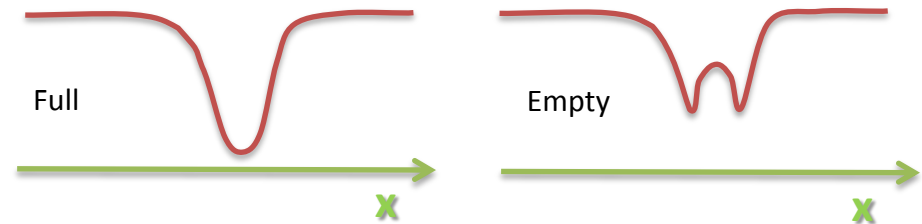
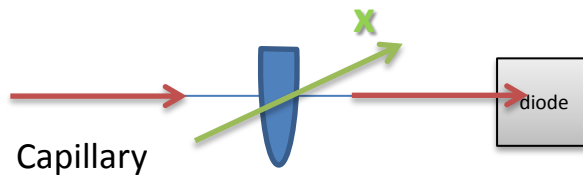
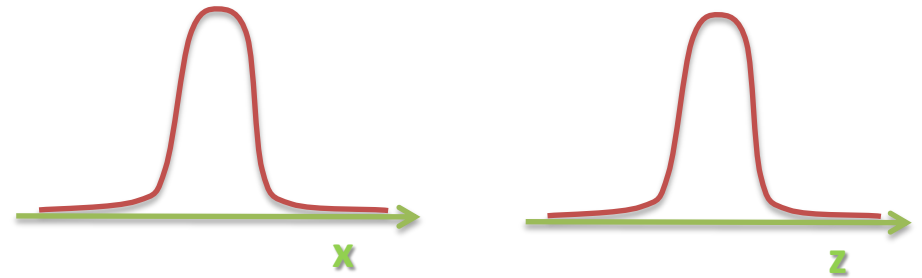
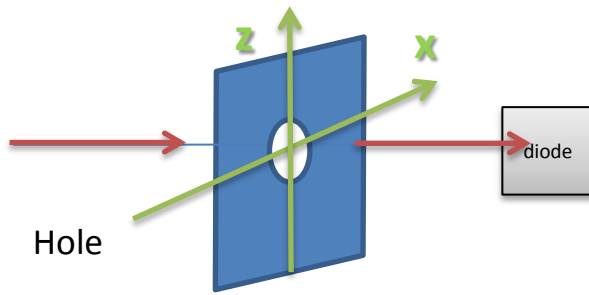
- 1) Insert the PINdiode (if needed)
- 2) Open the shutter
- 3) Scan the appropriate motor



Sample holder alignment

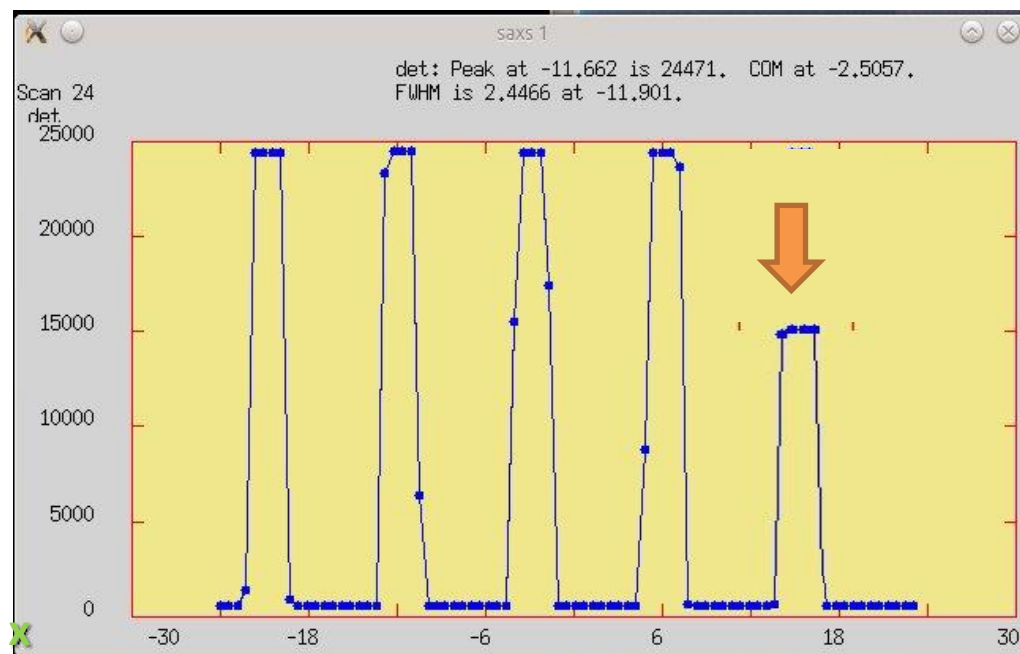
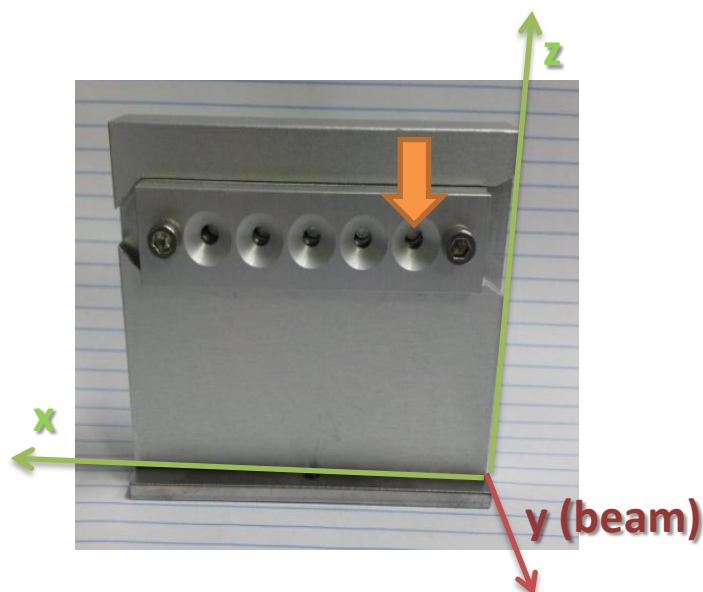
Motor Scan

Transmission curves of sample holder across the beam.
Intensity of beam measured with PIN diode, as function of motor position.



Sample holder alignment

5-hole holder

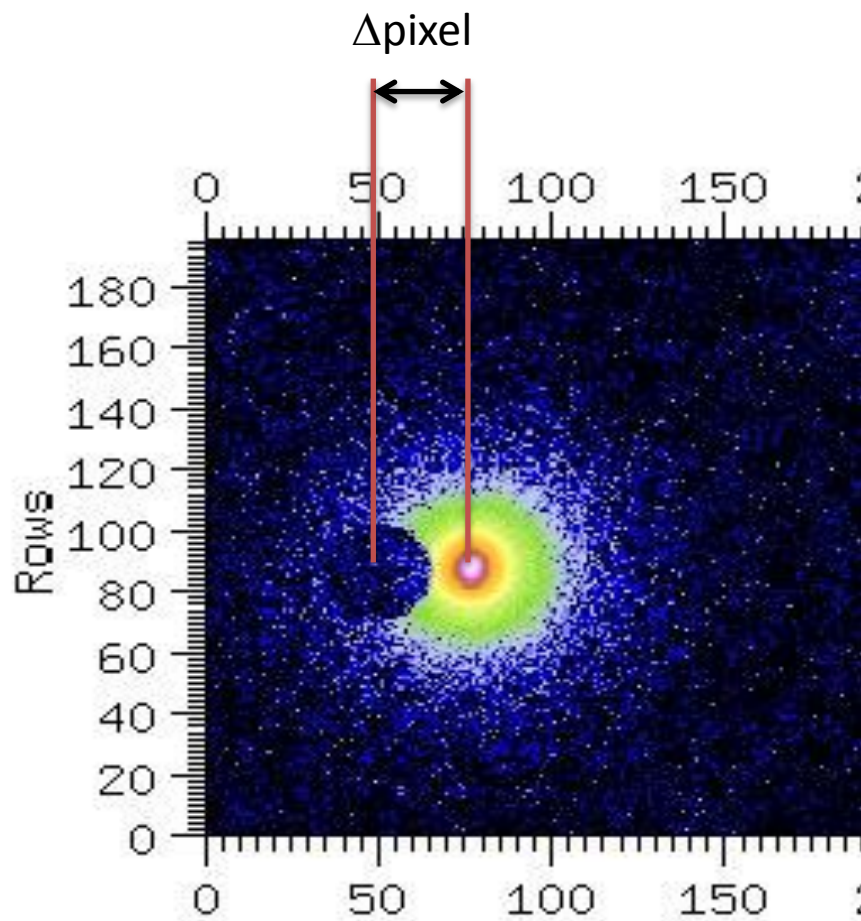


- Image of the 5-hole powder sample holder.
- Arrow marks position of a sample in the holder.

- Transmission plot of the intensity of the beam across the 5-hole powder sample holder. Five peaks represent the five holes.
- At the arrow position, the hole with sample appears to absorb more x-ray than empty holder, as expected.

SAXS beamstop alignment

SAXS



Pixel size: $172\ \mu\text{m}$

Motor shift = $\Delta\text{pixel} \times 0.172\ \text{mm}$

Motors:

bsx (beamstop horizontal)

bsz (beamstop vertical)

SPECfe

Data files Binary file : *.edf (ESRF Data Files) ASCII header in the file between curly brackets {}

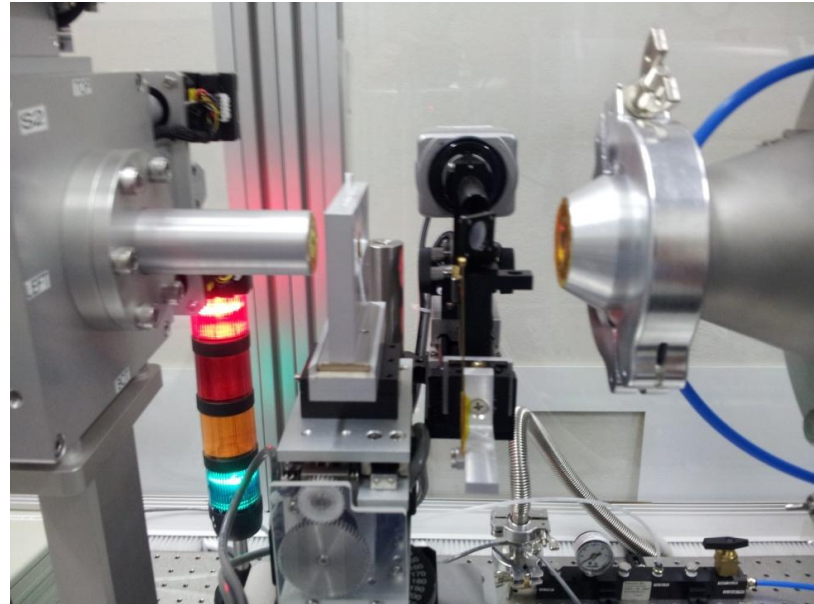
```
{
EDF_DataBlockID = 1.Image.Psd ;
EDF_BinarySize = 1206272 ;
EDF_HeaderSize = 8192 ;
ByteOrder      = LowByteFirst ;
DataType       = SignedInteger ;
Dim_1         = 487 ;
Dim_2         = 619 ;
title         = virtual_detector_001_0_00009.edf ;
Intensity1 = 1 ;
ExposureTime = 60 ;
Dummy = -10 ;
DDummy = 0.1 ;
Offset_1 = 0 ;
Offset_2 = 0 ;
Center_1 = 380.56 ;
Center_2 = 183.98 ;
PSize_1 = 0.000172 ;
PSize_2 = 0.000172 ;
SampleDistance = 1.04322 ;
Wavelength = 1.5411e-10 ;
RasterOrientation = 1 ;
Detector = 1656726 ;
History-1 = saxs_mac -p +pass -omod n -ildis 1.04322 -ilwvl
1.5411e-10 -ilcen 244.65 513.13 -type SignedInteger
/data0/images/external/virtual_detector_001/virtual_detector_001
_0_00009.edf
/data0/images/external/virtual_detector_001/virtual_detector_001
_0_00009.edftmp ;
HeaderID      = EH:000001:000000:000000 ;

bsx = 2.59301 ;
bsz = 0.97 ;
cam = 100.975 ;
Compression = None ;
count_time = 60.000000000 ;
Date = Wed Feb 5 18:16:26
2014 ;
detx = -25 ;
detz = -25 ;
Image = 1 ;
run = 0 ;
s1bot = 0.6 ;
s1hl = 0.6 ;
s1hr = 0.6 ;
s1top = 0.600003 ;
s2bot = 0.4 ;
s2hl = 0.4 ;
s2hr = 0.4 ;
s2top = 0.4 ;
SaxsDataVersion = 2.40 ;
Size = 1206272 ;
th = 0 ;
VersionNumber = 0.10 ;
x = -19.2 ;
z = -1.4 ;
Temperature = 20.3
}
```


Making the actual measurement

Sample(s)

- Measurement of :
 - Empty cuvette (buffer or solution)
 - Sample in its cuvette or in its buffer or solution
 - Transmissions





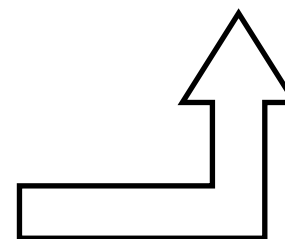
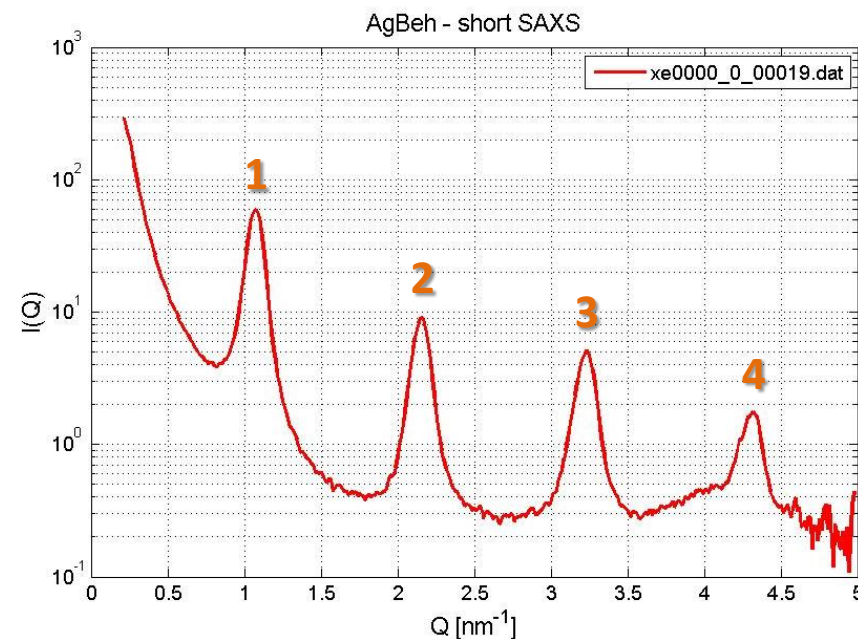
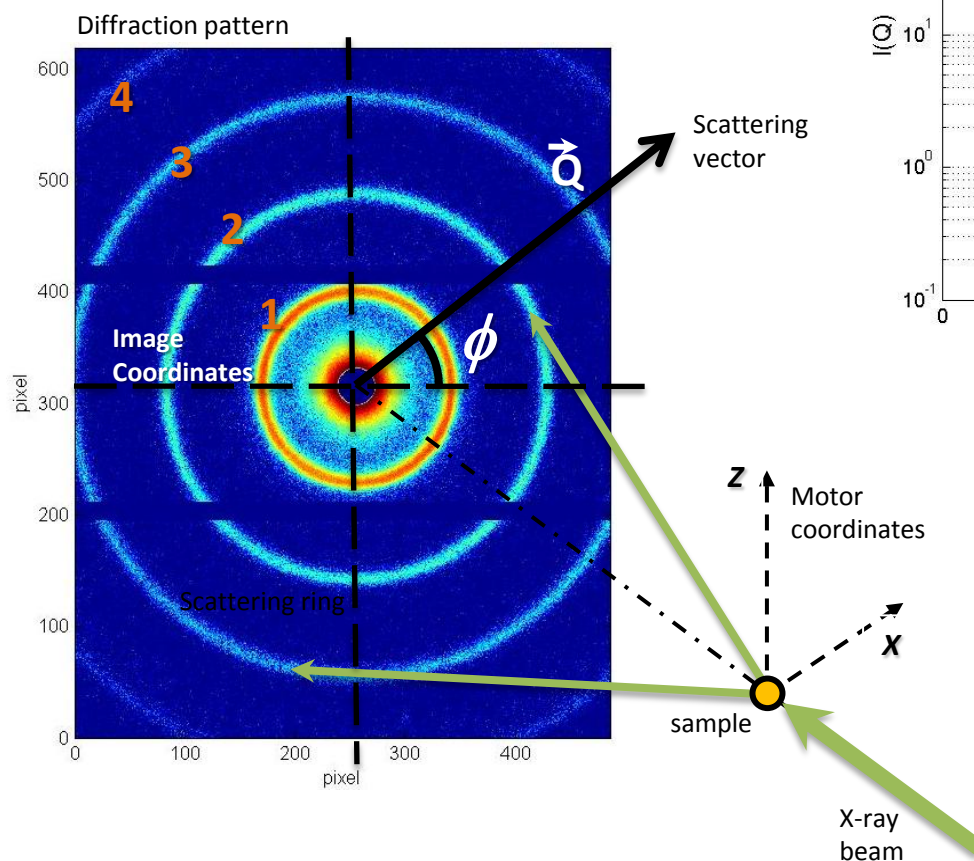
Data Reduction

Data Reduction

- 1) Measure calibrants and direct beam to do **Angular Integrations** (transforms 2D into 1D)
 - Need the distance SD
 - Need center of beam
 - Need to calculate q-vector
- 2) Measure sample and empties
- 3) Measure transmissions of sample and empties
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- 6) Save the data for analyze

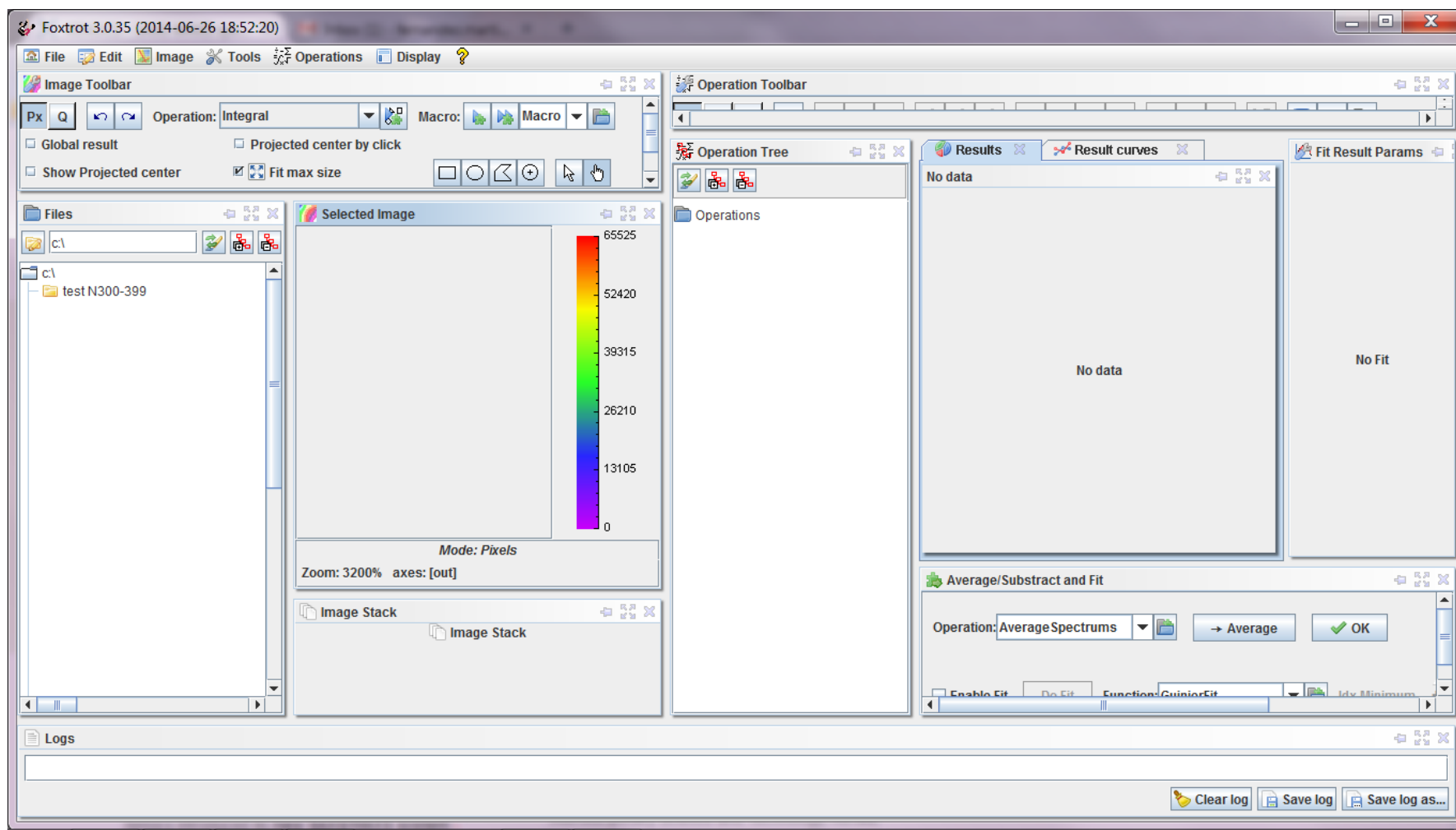
Angular Integration

2D images into 1D curves



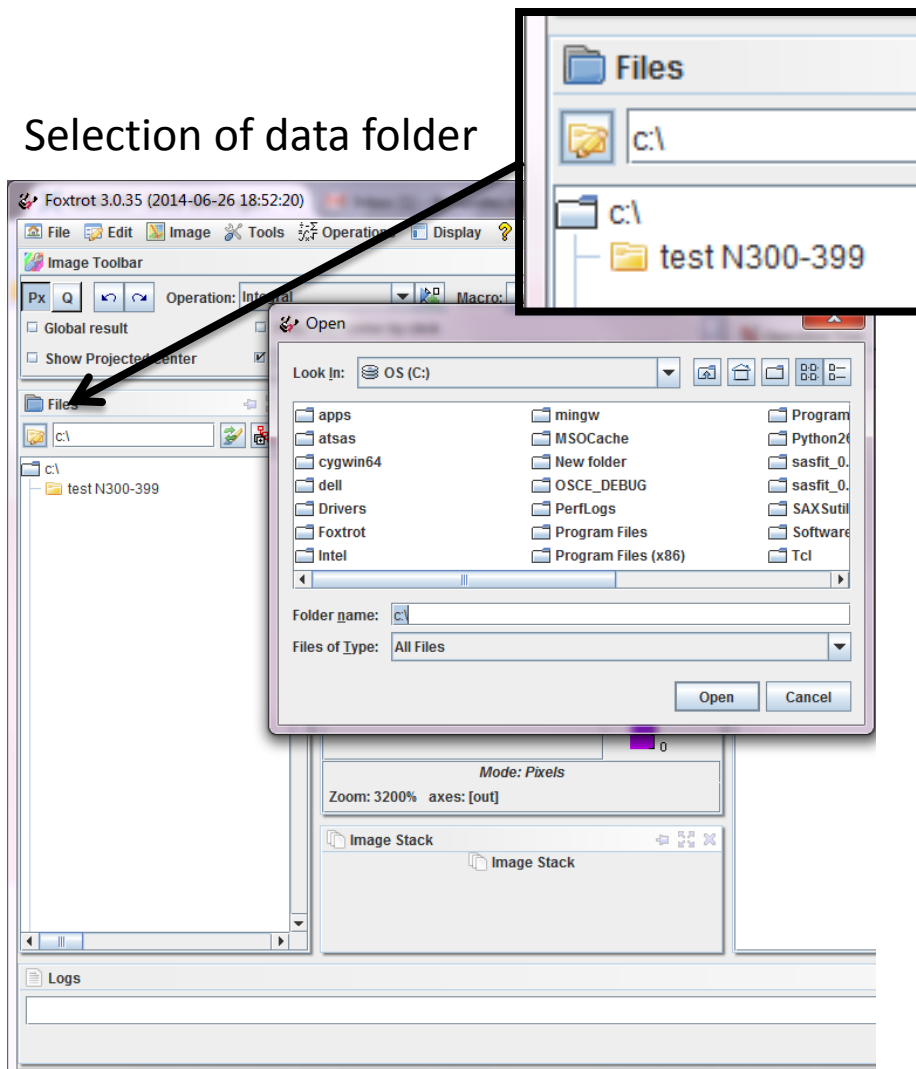
Quick guide to FOXTROT Data Reduction

Foxtrot environment

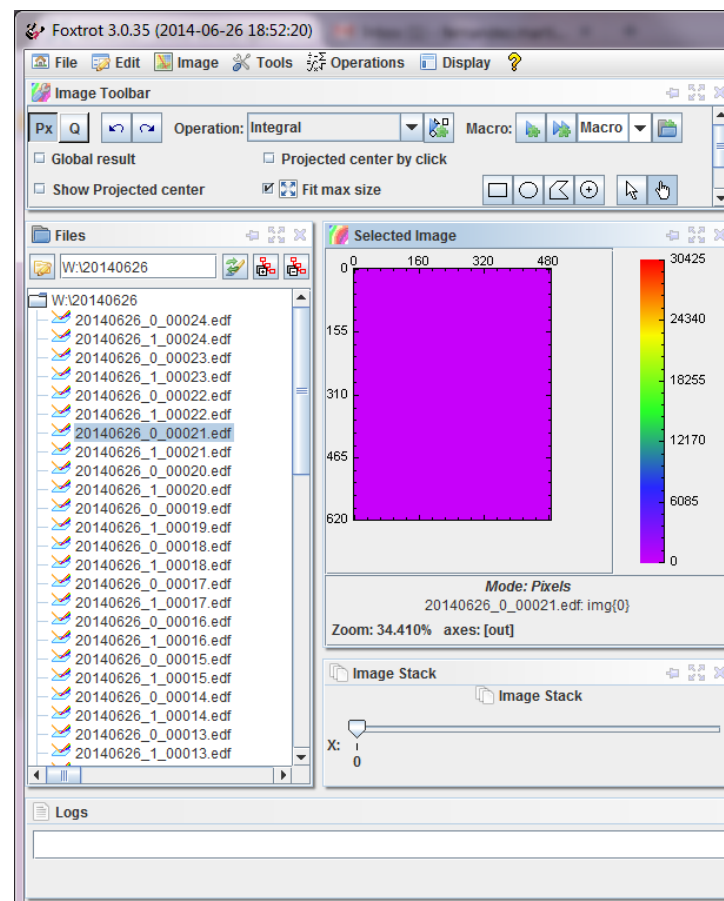


Quick guide to FOXTROT Data Reduction

Selection of data folder

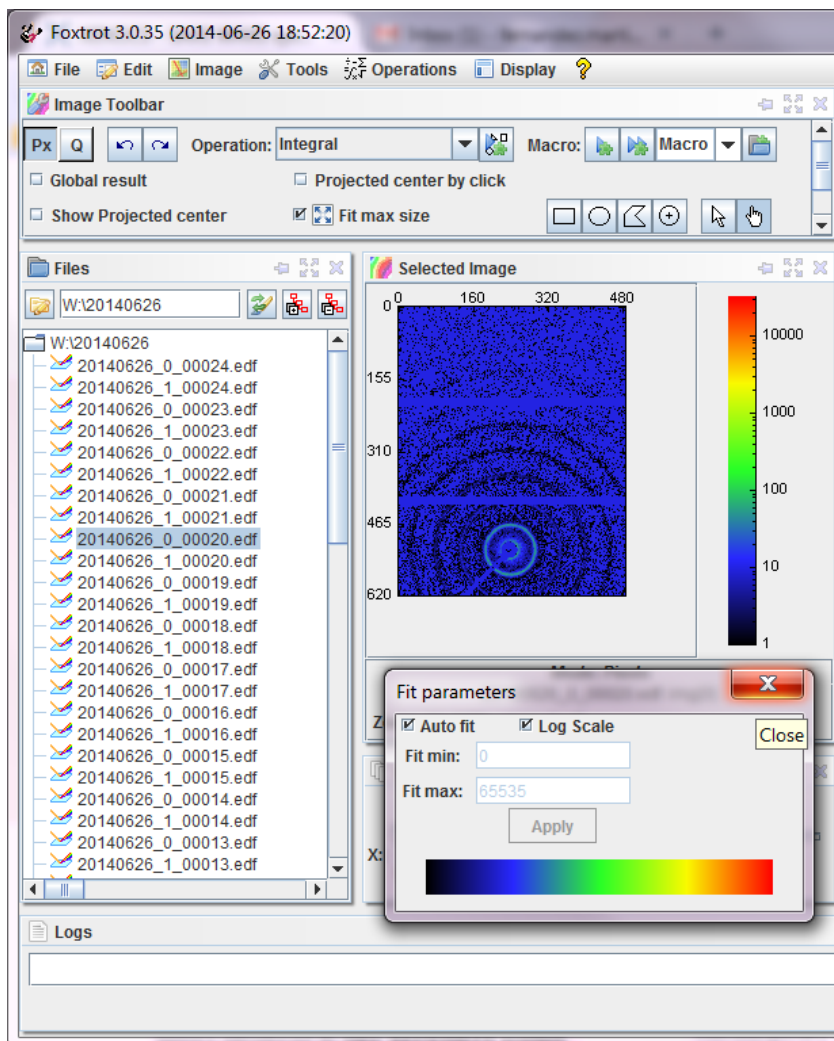


Selection of file / two clicks

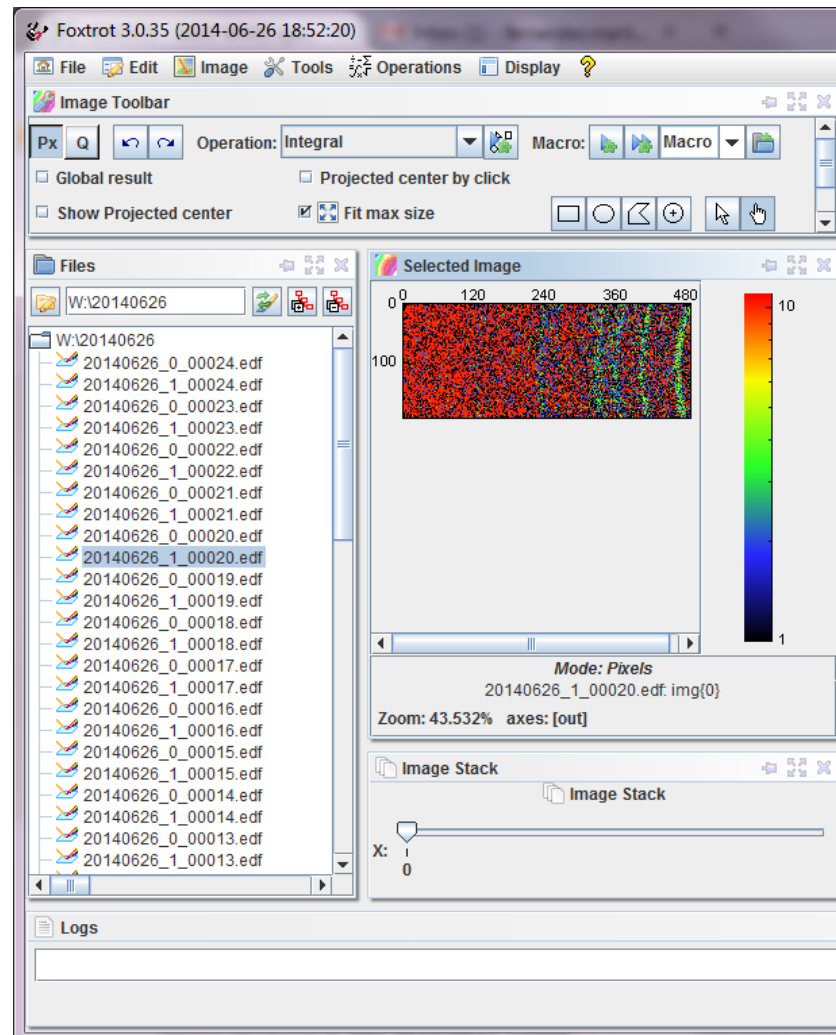


Quick guide to FOXTROT Data Reduction

Changing scale of image

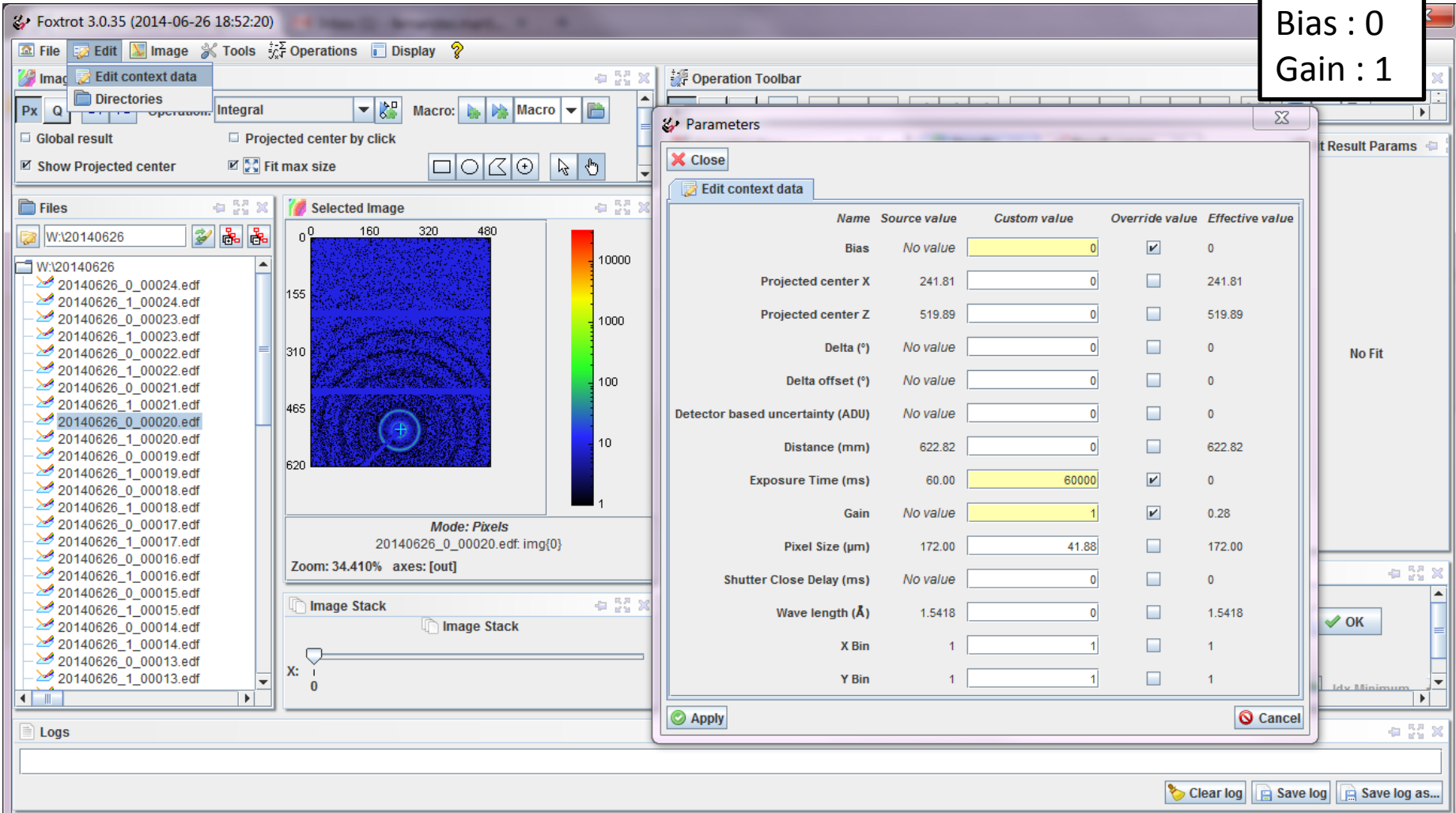


Opening other file (different size)



Quick guide to FOXTROT Data Reduction

Edit context data (partially read from header) used for Angular Integration



Parameters

Close

Edit context data

Name	Source value	Custom value	Override value	Effective value
Bias	No value	0	<input checked="" type="checkbox"/>	0
Projected center X	241.81	0	<input type="checkbox"/>	241.81
Projected center Z	519.89	0	<input type="checkbox"/>	519.89
Delta (°)	No value	0	<input type="checkbox"/>	0
Delta offset (°)	No value	0	<input type="checkbox"/>	0
Detector based uncertainty (ADU)	No value	0	<input type="checkbox"/>	0
Distance (mm)	622.82	0	<input type="checkbox"/>	622.82
Exposure Time (ms)	60.00	60000	<input checked="" type="checkbox"/>	0
Gain	No value	1	<input checked="" type="checkbox"/>	0.28
Pixel Size (µm)	172.00	41.88	<input type="checkbox"/>	172.00
Shutter Close Delay (ms)	No value	0	<input type="checkbox"/>	0
Wave length (Å)	1.5418	0	<input type="checkbox"/>	1.5418
X Bin	1	1	<input type="checkbox"/>	1
Y Bin	1	1	<input type="checkbox"/>	1

Apply Cancel

Bias : 0
Gain : 1

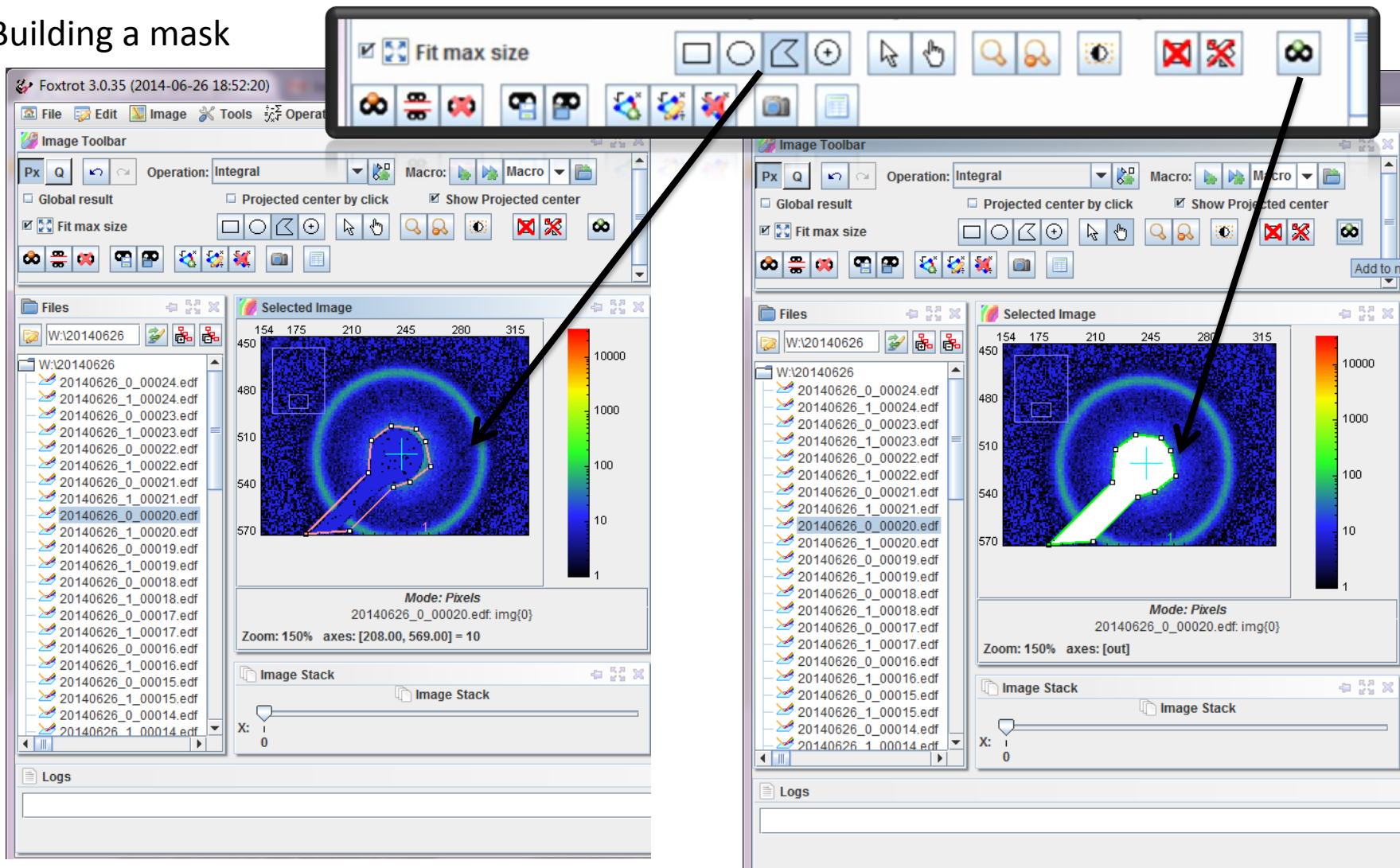
No Fit

OK

Clear log Save log Save log as...

Quick guide to FOXTROT Data Reduction

Building a mask



Quick guide to FOXTROT Data Reduction

Building a mask / Threshold mask

Mask all pixels with values:

- smaller than 0 (blind stripes)
- Bigger than 1000 (cosmics)

Min: 0.0 Max: 1000.0

Apply Cancel

Quick guide to FOXTROT Data Reduction

Angular Integration/ Circle Gathering

The screenshot displays the Foxtrot 3.0.35 software interface. The main window shows a list of files on the left, a central image stack with a color scale from 1 to 10000, and a bottom status bar. The 'Operations' menu is open, highlighting 'Circle Gathering'. A 'Parameters' dialog box is overlaid on the right, showing settings for 'Circle Gathering'.

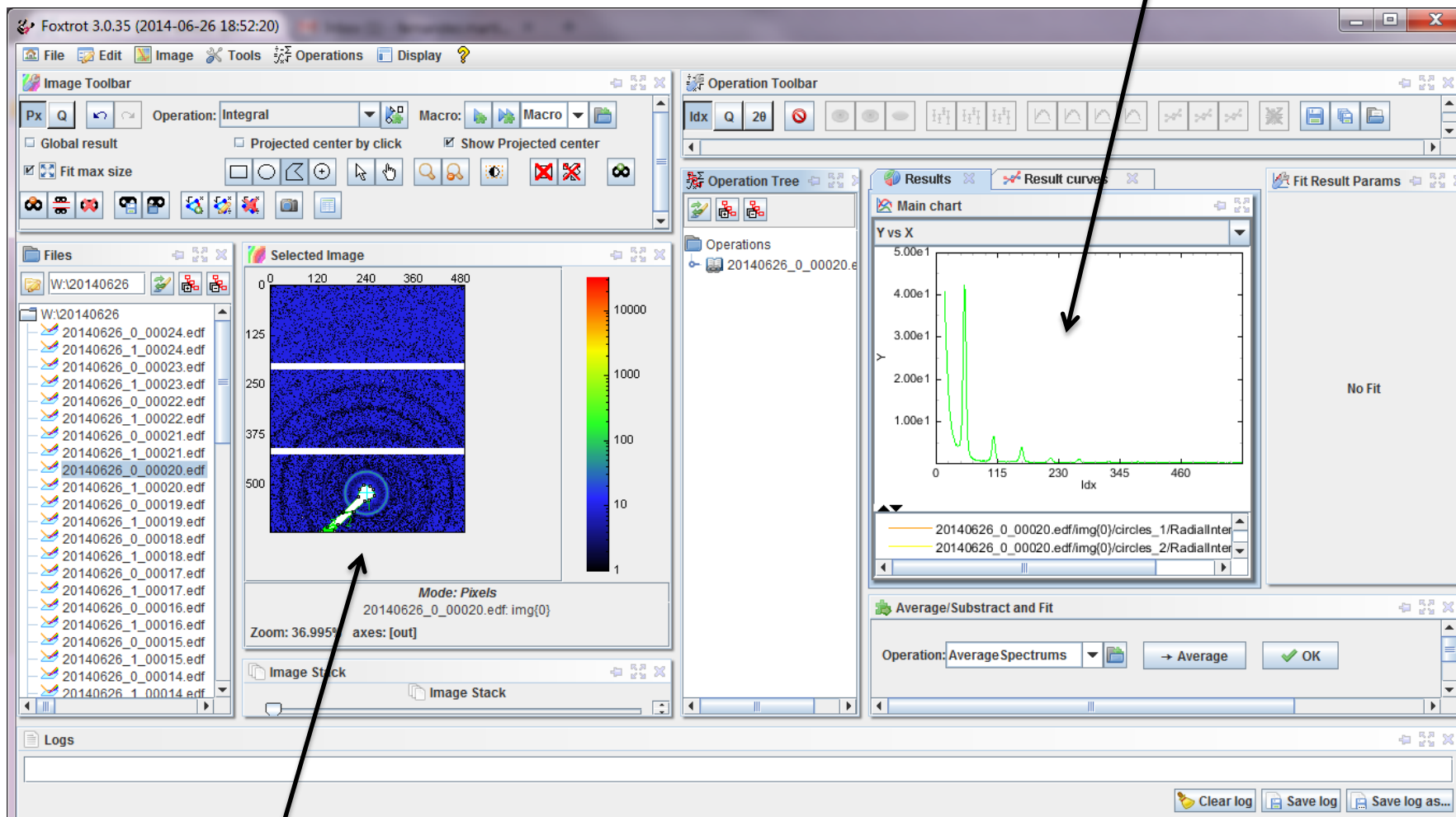
Parameters Dialog Box:

- Close** button
- Tabs: **Circle Gathering** (selected), Threshold Mask
- Projected center coordinates (pixels):**
 - X: 241.81000
 - Z: 519.88900
 - Use context coordinates
- Use pixel splitting
- Wave length (Å): 1.54180
- Bias: 0
- Distance (mm): 622.82200
- Pixel Size (µm): 172.00000 × 172.00000
- Normalization** section:
 - Apply Normalization
 - Exposure time (ms): 60000.00
 - Shutter Close Delay (ms): 0.00
 - Number of MI: 1
 - Intensity monitor: [dropdown]
 - Factor: 1.0
- Apply** and **Cancel** buttons

Quick guide to FOXTROT Data Reduction

Angular Integration/ Circle Gathering

1-dimensional result of angular integration

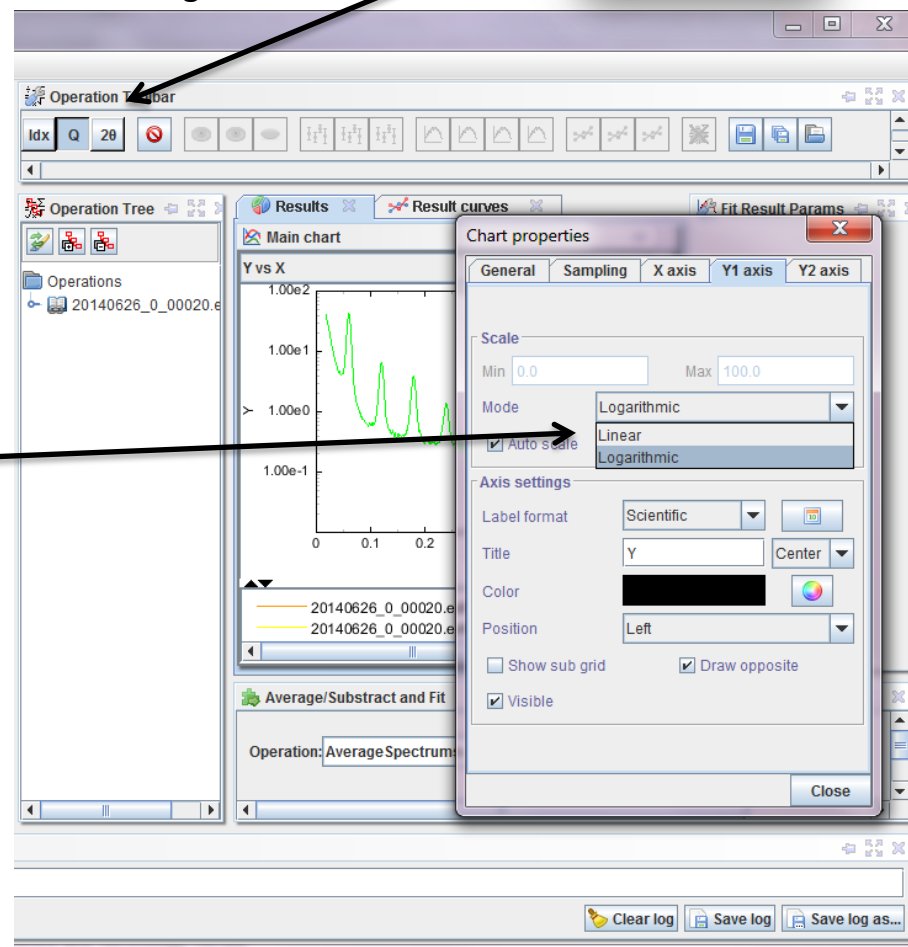
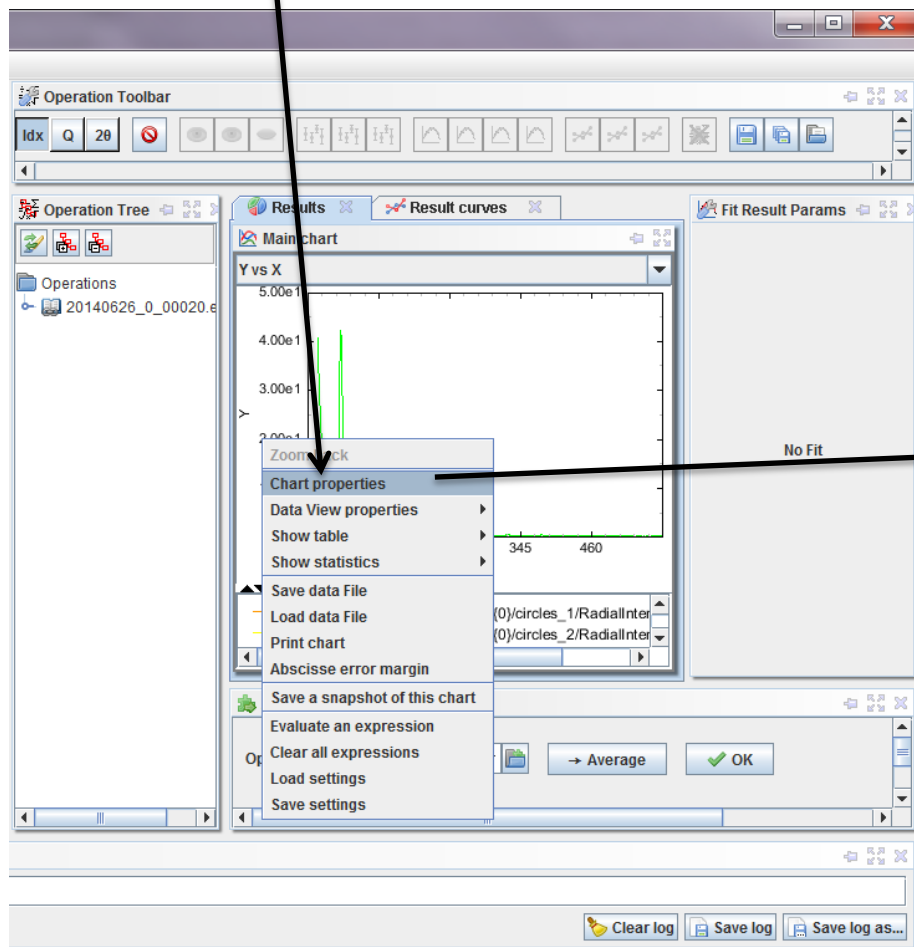
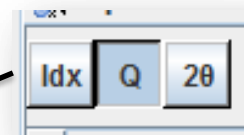


2-dimensional image + mask

Quick guide to FOXTROT Data Reduction

Chart Properties / Click on axis

- Changing scale of axis
- Selecting Q-vector



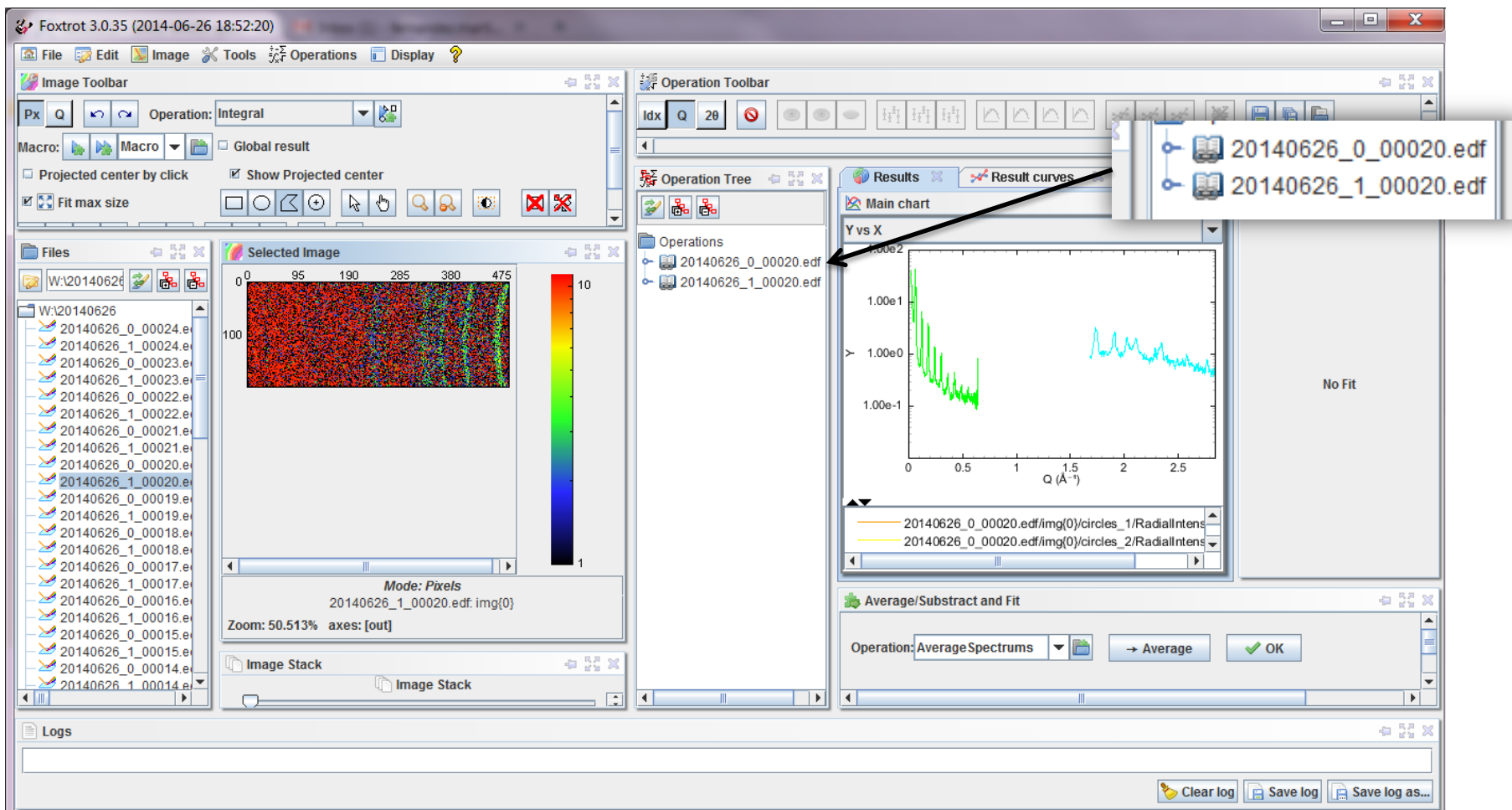
Quick guide to FOXTROT Data Reduction

Saving Data / Nexus file (ASCII)

The screenshot displays the FoxTrot 3.0.35 software interface. A 'Save' dialog box is open, showing the file system structure with 'Files of Type' set to 'Nexus files'. The 'Results' window shows a plot of 'Y vs X' with a logarithmic y-axis ranging from 1.00e-1 to 1.00e2 and an x-axis labeled 'Q (Å⁻¹)' ranging from 0 to 0.6. The plot shows a series of peaks. Below the plot, there are two entries in the legend: '20140626_0_00020.edf/img(0)/circles_1/RadialInter' and '20140626_0_00020.edf/img(0)/circles_2/RadialInter'. The 'Fit Result Params' window shows 'No Fit'. The 'Average/Subtract and Fit' window shows 'Operation: Average Spectrums' and 'Average' and 'OK' buttons. The 'Image Stack' window shows 'Mode: Pixels' and 'Zoom: 36.995% axes: [out]'. The 'Logs' window is empty.

Quick guide to FOXTROT Data Reduction

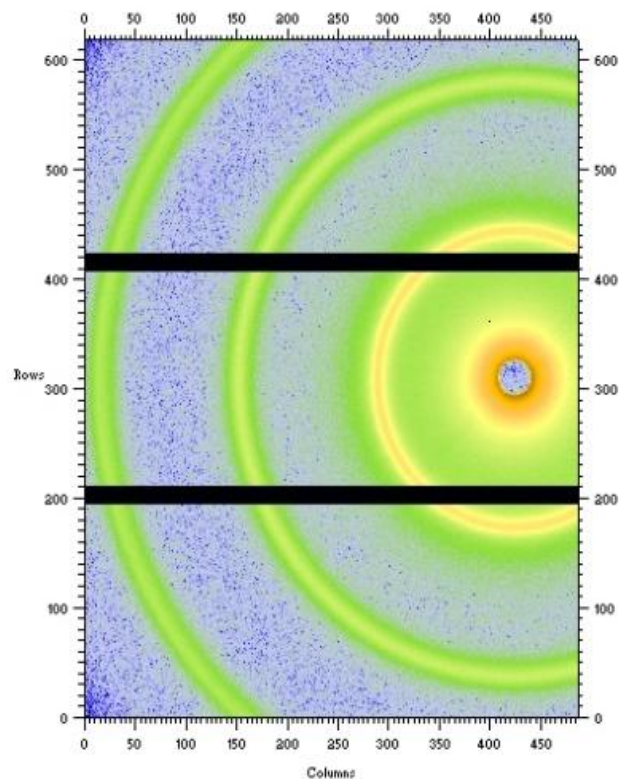
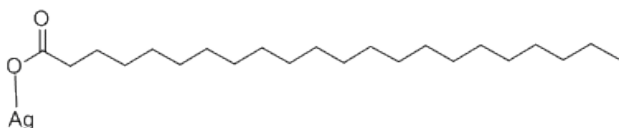
Doing a similar process for other file (WAXS), the two curves can be plotted in the same graph. Files with name '0' are SAXS, files with '1' are WAXS.



The screenshot displays the Foxtrot 3.0.35 software interface. The 'Files' panel on the left shows a list of data files for the date 20140626, including SAXS files (ending in '_0_') and WAXS files (ending in '_1_'). The 'Selected Image' panel shows a 2D diffraction pattern. The 'Results' panel shows a 'Main chart' of Y vs X (Intensity vs Q) with two curves: a green curve for the SAXS file (20140626_0_00020.edf) and a cyan curve for the WAXS file (20140626_1_00020.edf). A callout box highlights these two files. The 'Average/Subtract and Fit' panel shows the 'AverageSpectrums' operation.

Calibration of Q-vector and distance calculation

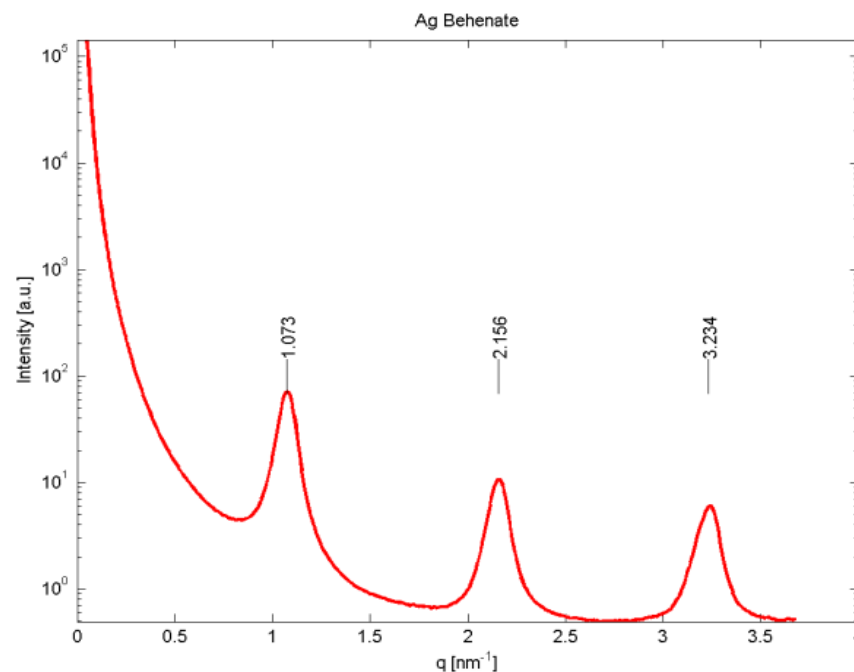
Silver Behenate (AgBeh)



Lamellar structure with :

$$d_{001} = 58.7 \text{ \AA}$$

$$Q_{001} = 1.07 \text{ nm}^{-1}$$



Distance Calculation

SPEC function (by Xenocs):

SAXS>**dist_calc** d_ q_ q0

d = real distance

d_ = distance used to first integration

q_ = q-value measured in the first integration from the calibration peak

q₀ = q-value tabulated for the calibration peak
 (AgBeh : q₀ = 1.070 nm⁻¹)

$$d = \frac{d_{\text{tan}} \left[2 \sin^{-1} \left(\frac{\lambda q_{-}}{4\pi} \right) \right]}{\tan \left[2 \sin^{-1} \left(\frac{\lambda q_0}{4\pi} \right) \right]}$$

Experiment Info into the image header

Info for Header

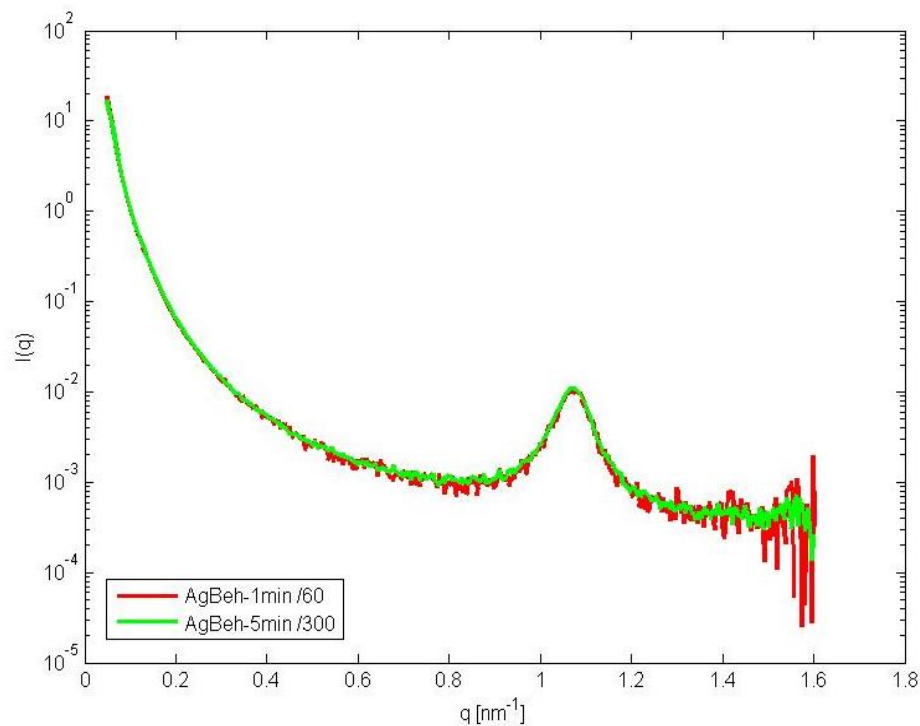
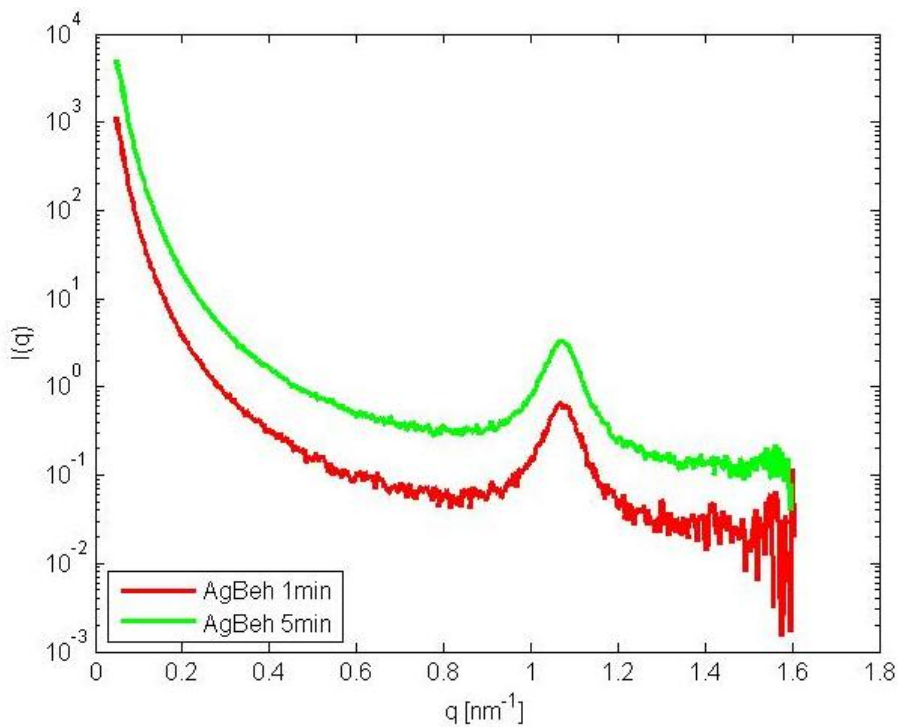
The screenshot shows the Xenocs software interface with the following details:

- Menu Bar:** File, Edit, Graph, Scans, Commands, Help
- Toolbar:** Abort, Print, Prev Scan, Next Scan, Legend, Autoscale, Save Reference, Clear Reference, Open Shutter, Close Shutter, AUTO LOAD DATA, Spec is: Ready
- Status Bar:** Xenocs Interface to Spec // specfe-xenocs 1.0, Status: Scan 128 loaded, Datafile: /home/saxs/data/saxs_20140325.log
- Navigation Tabs:** Graph 1, Graph 2, Scan Data, Scan List, Motors/Counters, Collimation, Sample, Detector, Acquisition
- Saving options:** External, Inhouse (selected), Exper. name: test, First Number: 1201
- Acq. Mode:** Saxs (selected), Waxs, Both
- SPEC/Detector:** Use in ct/scan, ROIs
- Virtual Detector (SAXS):** Use virtual det. mode, Geometry: g_vert2, Change
- Settings:**
 - Wavelength: 1.5411
 - Beam Center X: 69.86234 (SAXS), 19.87554 (WAXS)
 - Beam Center Z: 536.2469 (SAXS), 15.65453 (WAXS)
 - Pixel Size: 172 (SAXS), 172 (WAXS)
 - Sample-Det Distance: 2609.15 (SAXS), 137.072 (WAXS)
- Standard / Mapping:** Exposure Time: 1, Number of Shots: 1, Cycle Time: 2, Acquire



Data Normalization

Normalization to Exposure Time



Normalization to transmission

Use the values from a sample scan to get the transmission T ,
 ans then divide the scattering curves by the measured T

$$T = \frac{I - I_b}{I_0 - I_b}$$

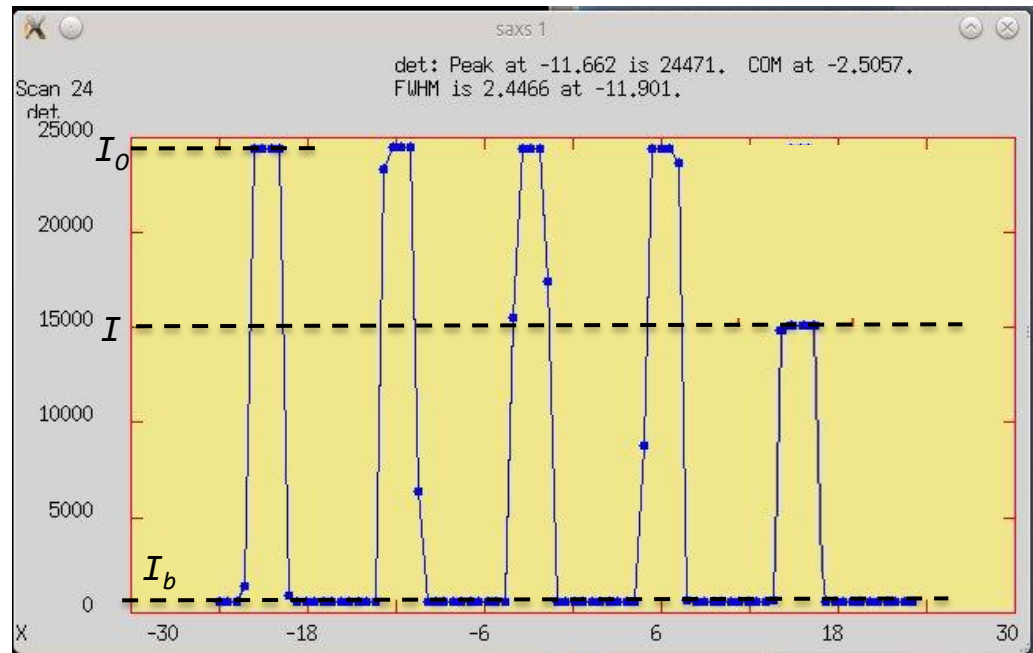
$$I_0 = 24900$$

$$I = 15000$$

$$I_b = 400$$

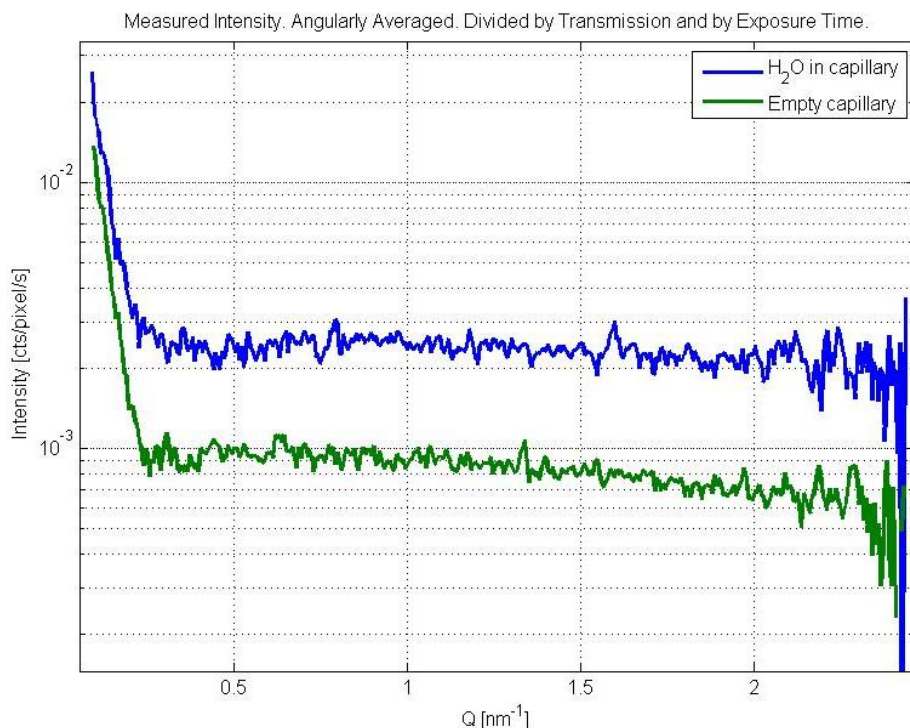
$$T = (15000 - 400) / (24900 - 400)$$

$$T = 0.595918 \text{ (~60\%)}$$



Absolute Intensity Calibration (with H₂O)

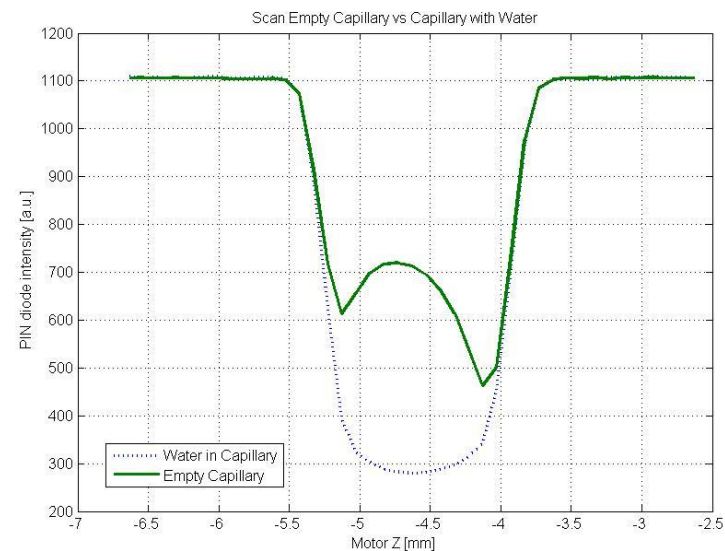
Measure scattering from empty capillary and capillary full of water



Data

Measure transmission (I/I_0) of empty capillary and capillary full of water

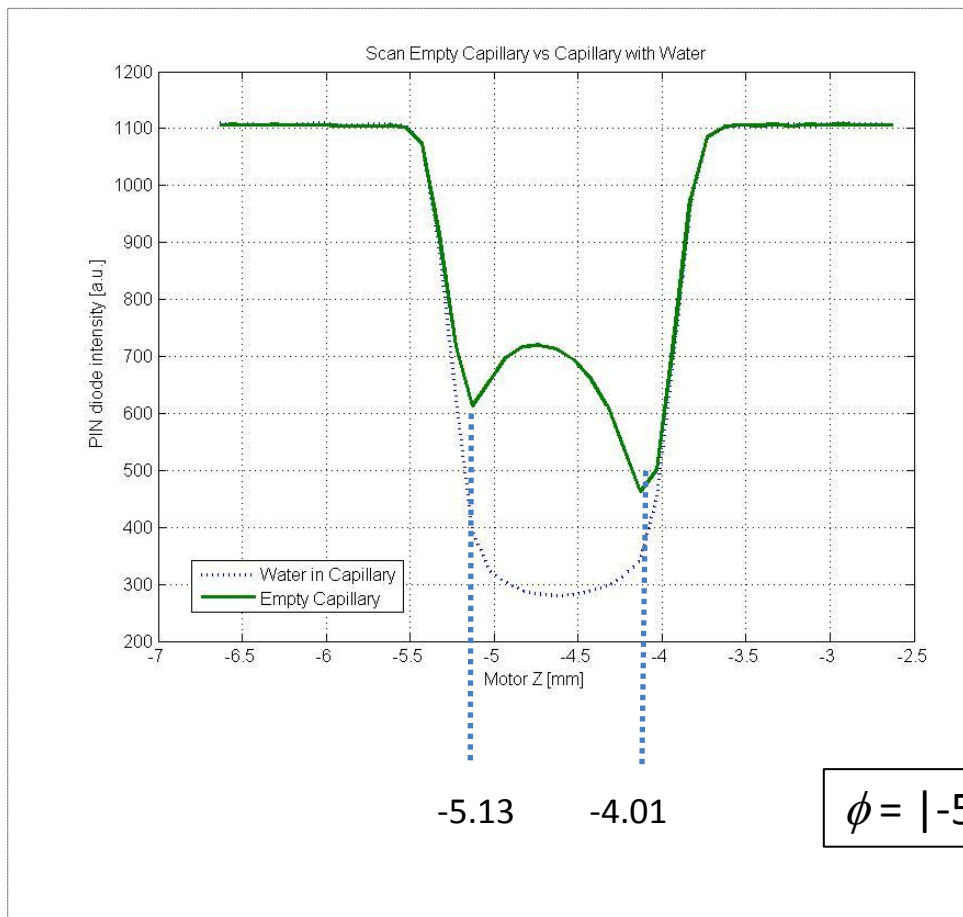
```
SAXS>dscan z -1.5 1.5 60 0.1
```



Capillary : $T_c = 710/1110 = 0.6396$
 Water : $T_{w+c} = 290/1110 = 0.2613$

Absolute Intensity Calibration (with H₂O)

Measure of the diameter f of the capillary



Absolute Intensity Calibration (with H₂O)

Subtract the scattering curve of empty capillary from that of the water in capillary, to obtain the contribution of water alone

Tabulated value for water intensity

$$I_w(0) = 1.66 \cdot 10^{-3} \text{ mm}^{-1}$$

Linear fit (or extrapolation) to $Q = 0 \text{ nm}^{-1}$ and measure $I_w(0)$

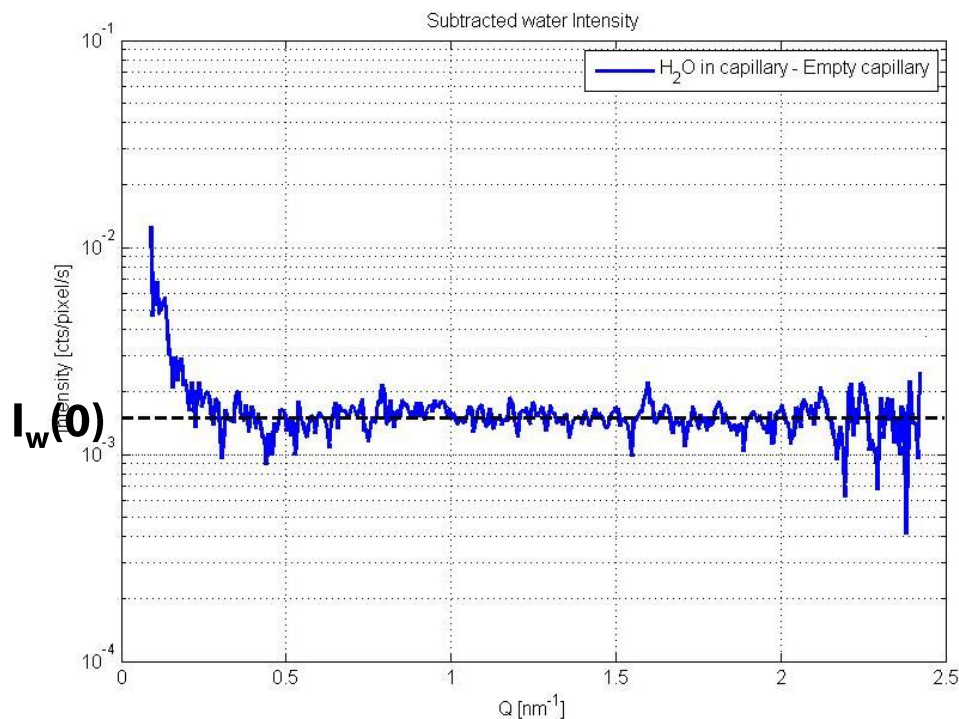
$$I_w(0) = 1.00 \cdot 10^{-3} \text{ a.u.} / 1.12 \text{ mm} = 1.79 \cdot 10^{-3} \text{ a.u./mm}$$

Correction factor

$$\kappa = 1.66/1.79$$

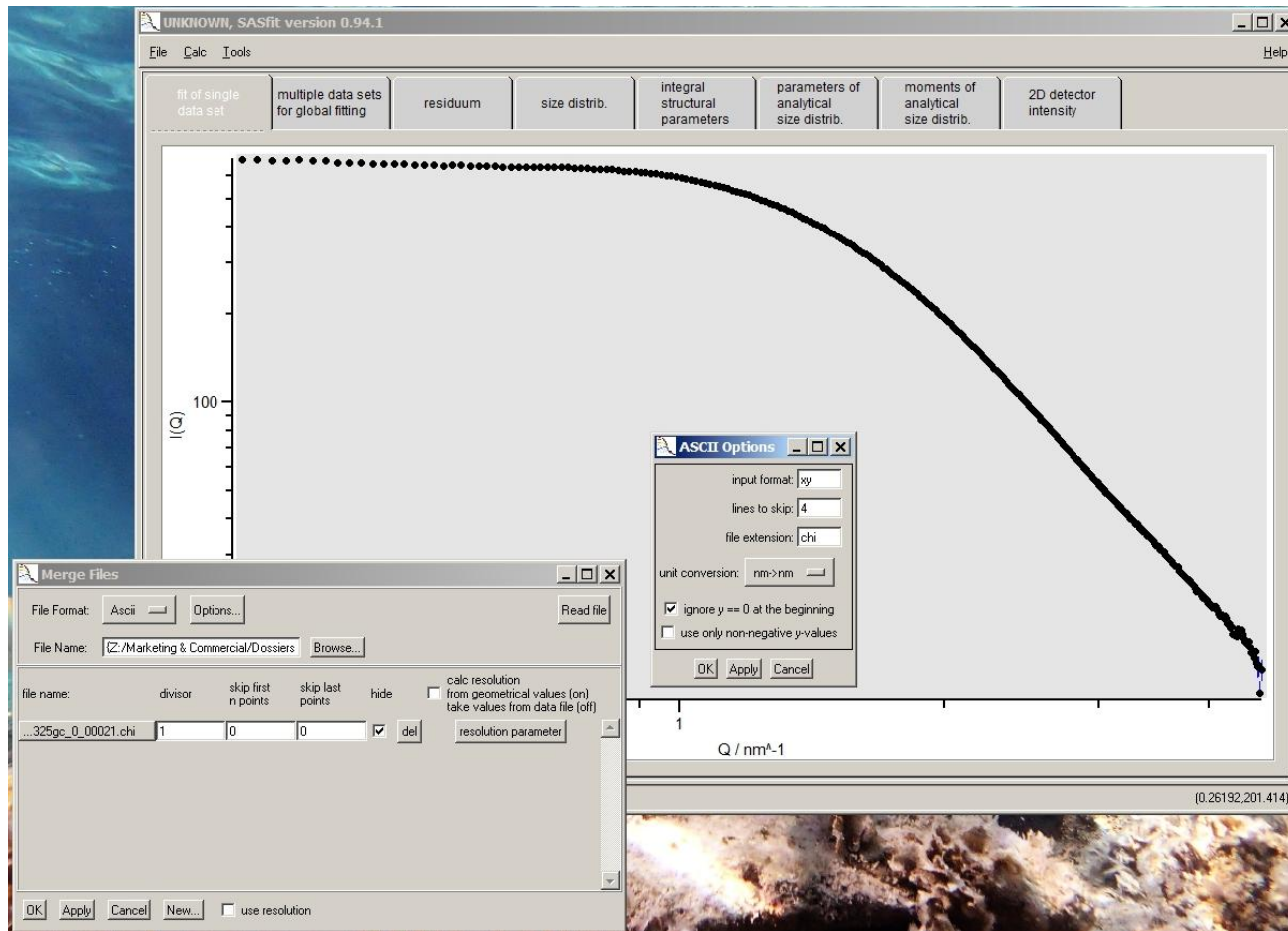
$$\kappa = 0.927$$

Finally, the intensity from the a given sample has to be divided by its thickness. Units will be in **mm⁻¹**



Absolute Intensity Calibration (with Glassy Carbon)

Load into SASfit (or other) the Glassy Carbon data from the calibration sample provided.

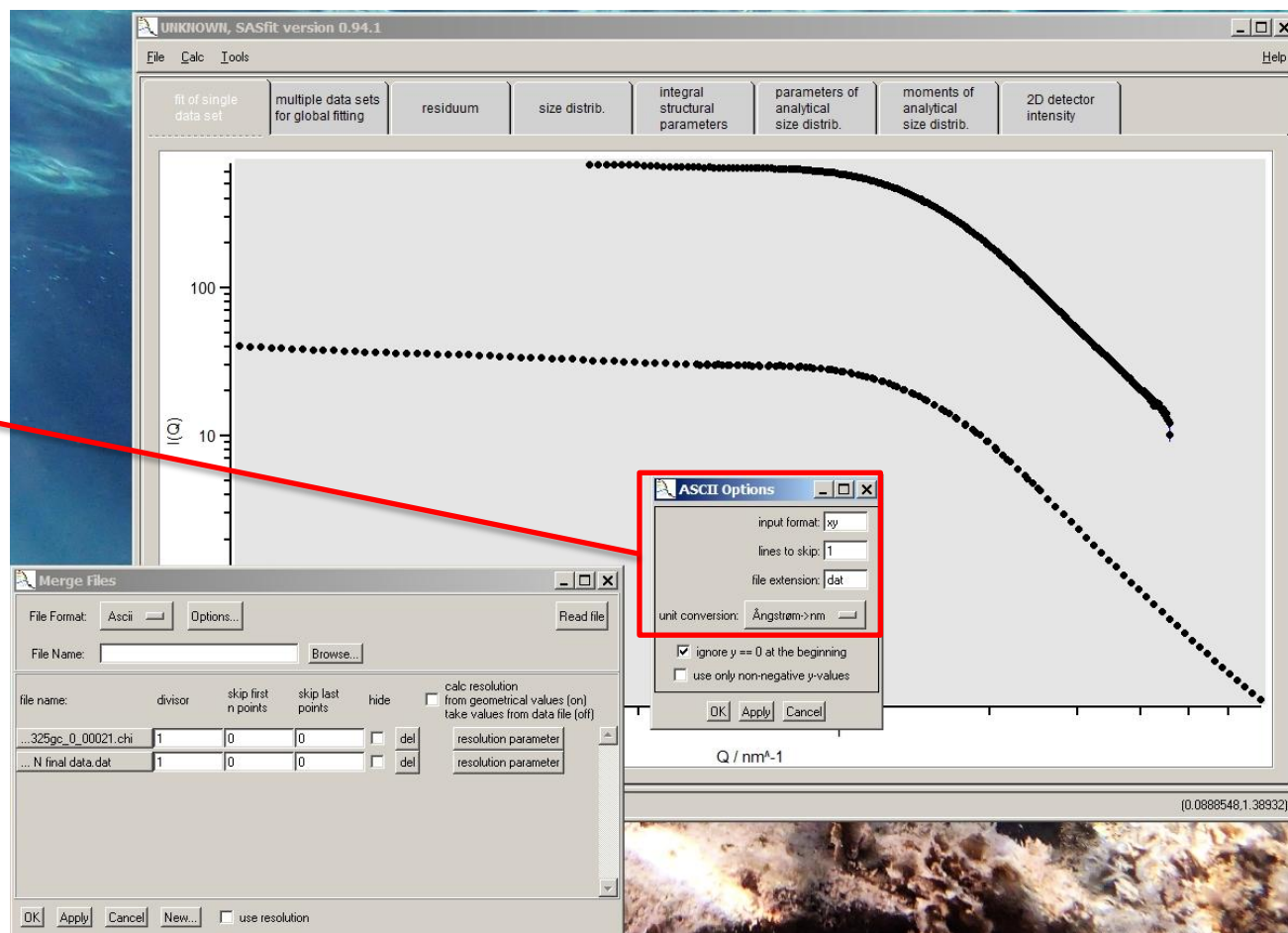


Absolute Intensity Calibration (with Glassy Carbon)

Load the calibration data of the Glassy Carbon data (APS): **Glassy Carbon N final data.dat**

Note!

- Unit conversion : Å -> nm
- lines to skip : 1
- file extension : dat



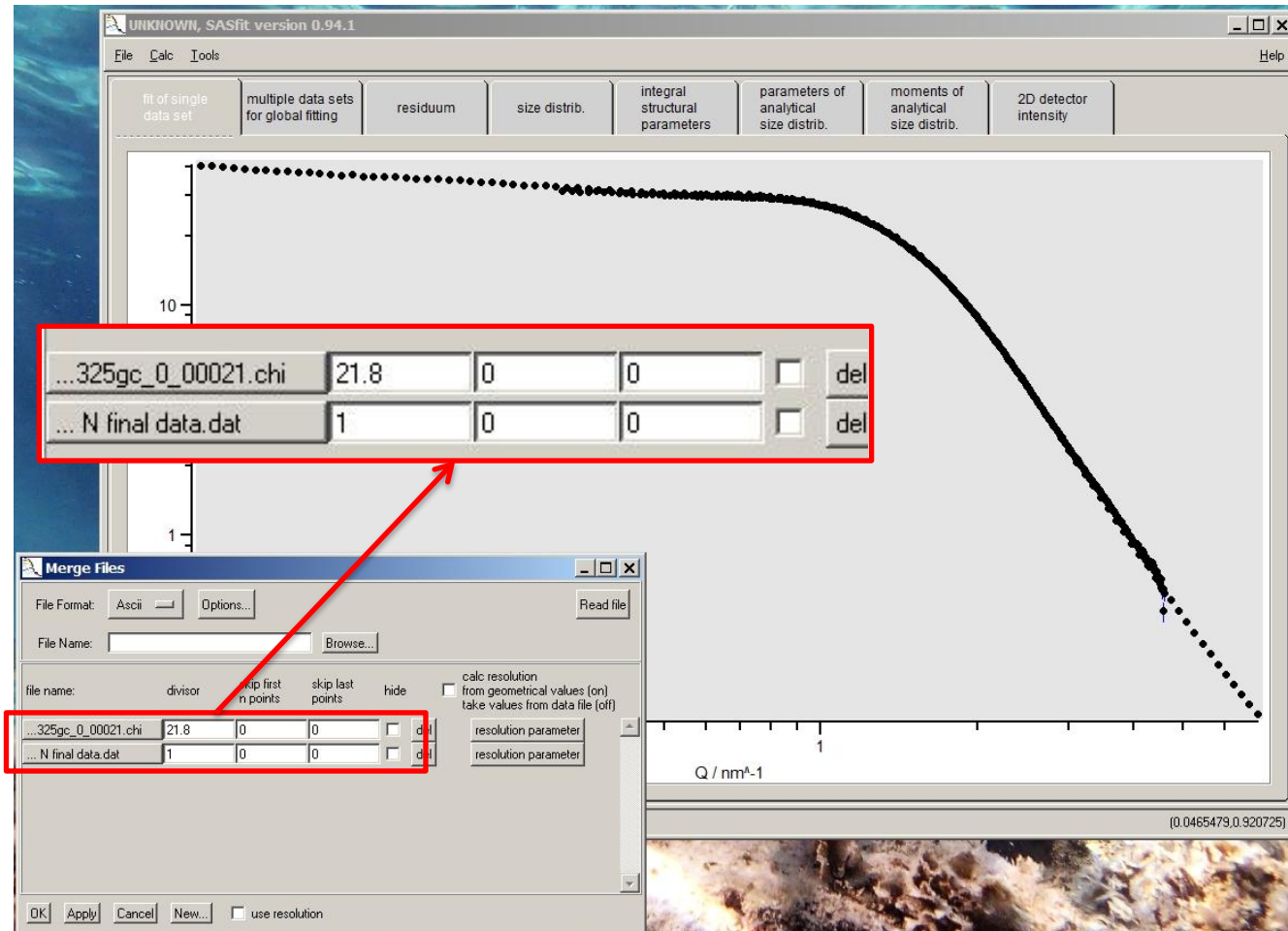
Absolute Intensity Calibration (with Glassy Carbon)

Calculate the factor to be applied to your data in order to overlap with the calibration curve. In this case : 21.8. Calibration sample thickness is 1 mm. No need to normalize to this thickness.

Measured Glassy Carbon data had 600 s exposure time.

Data to be calibrated has to be normalized to 600 s exposure time, and the intensity divided by the sample thickness.

Units will be in mm^{-1}





Empty cuvette and buffer subtraction

Subtraction

Scattering is additive

All objects in the beam contribute to the total scattering : camera (windows, environment, collimation), buffer, sample container, etc... The contribution of these elements is additive.

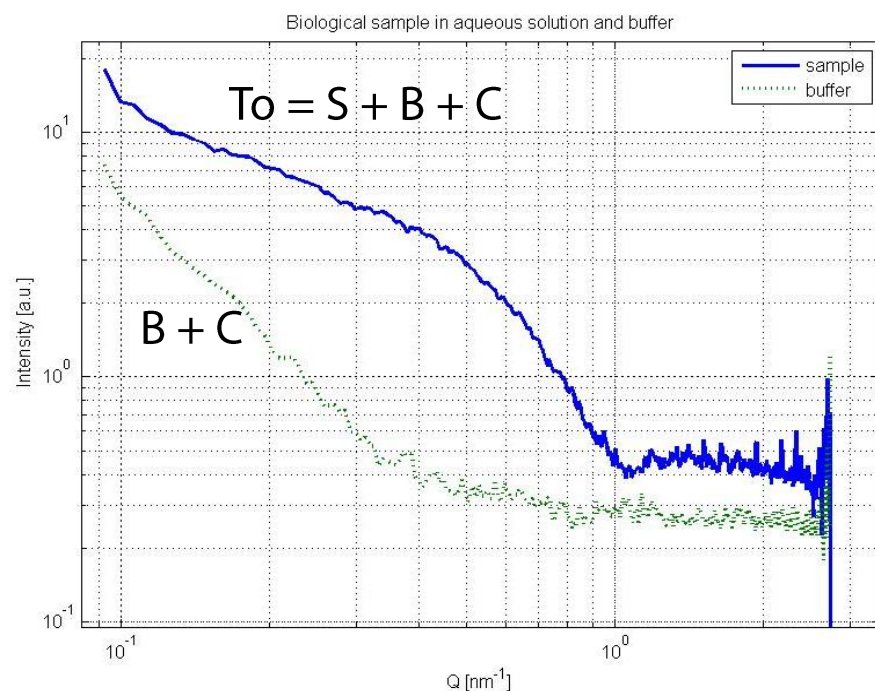
Unknown (and wanted) contribution to the total scattering coming from **the sample** may be extracted from the total scattering data by simple subtraction of the known contributions : camera and/or buffers or containers.

Subtraction can be done directly from the 2D images
or from the 1D curves

Buffer subtraction : aqueous solutions

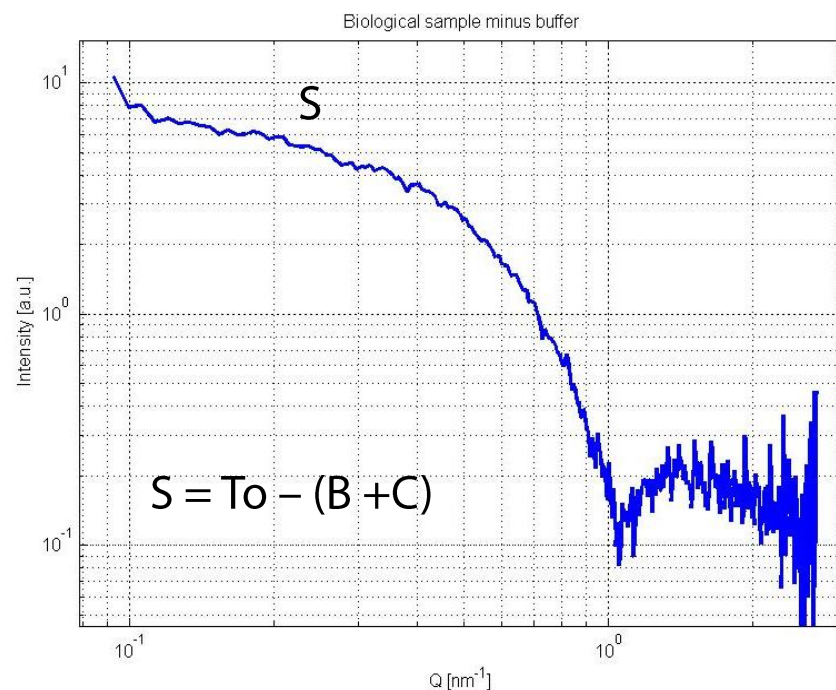
The total scattered intensity from a liquid solutions (i.e. proteins) is a contribution of the camera, the cell or capillary, the protein signal and the solvent (buffer) signal.

In order to obtain the scattering arising from the protein alone a subtraction has to be performed : data with sample minus data of buffer alone.



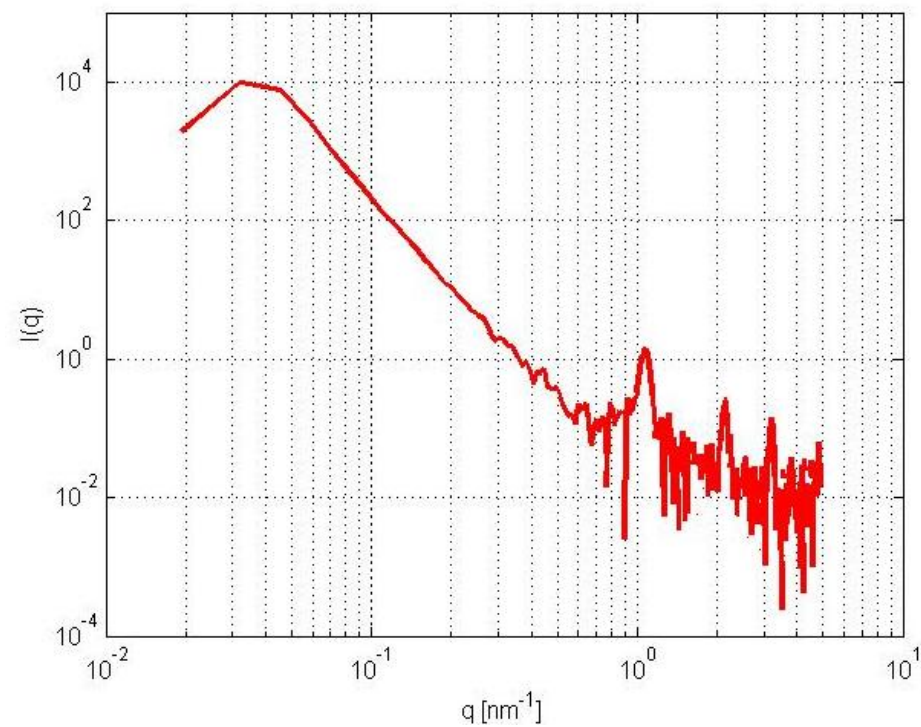
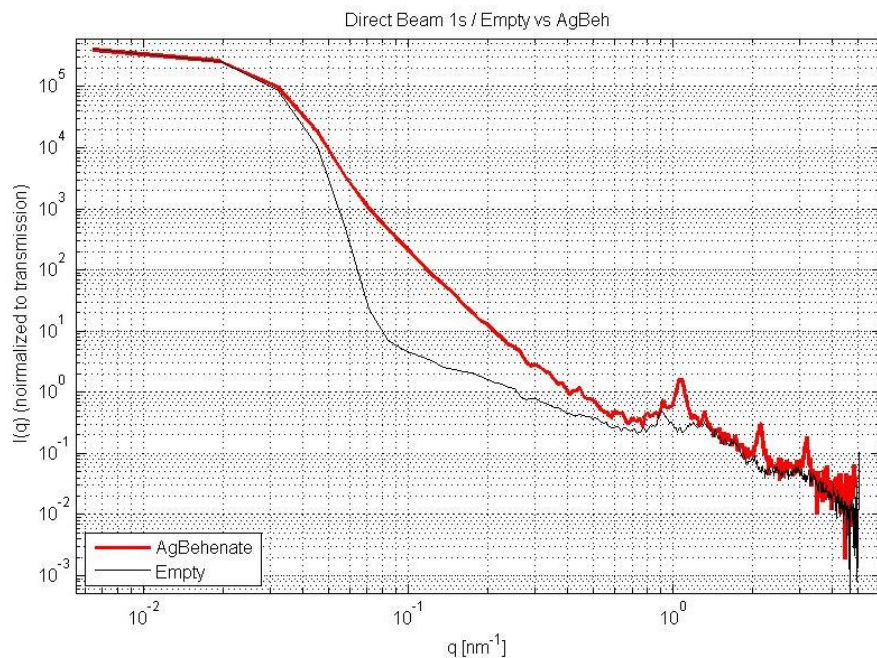
- Protein in its buffer (S+B)
- Buffer alone (B)

Both patterns contain the SAME contribution from the camera (C).



Camera and cell contribution are included in sample and buffer signal, and subtracted at the same time.

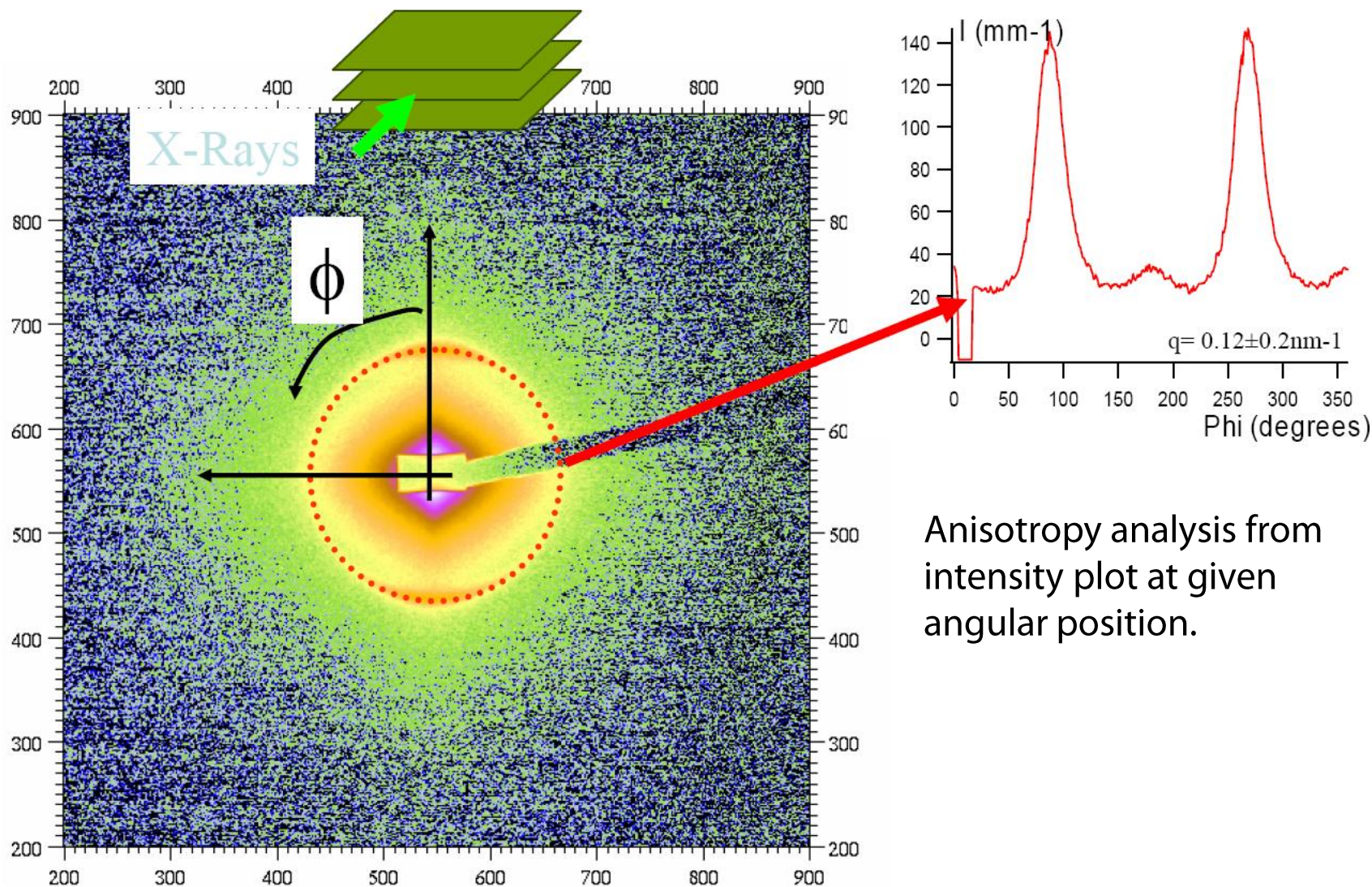
Empty cuvette subtraction





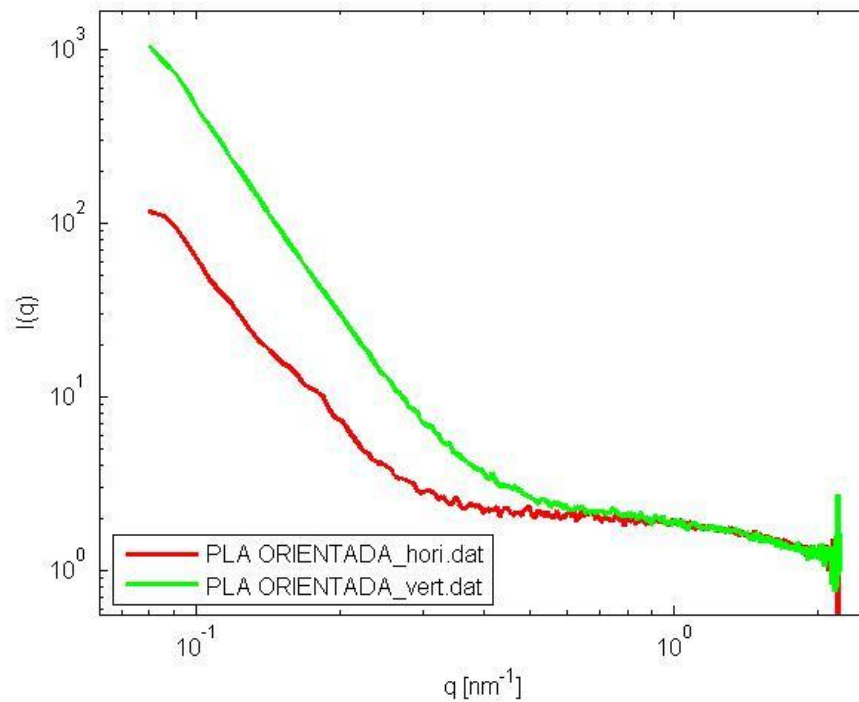
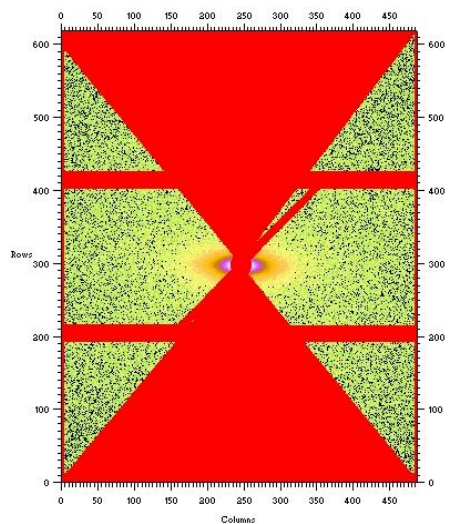
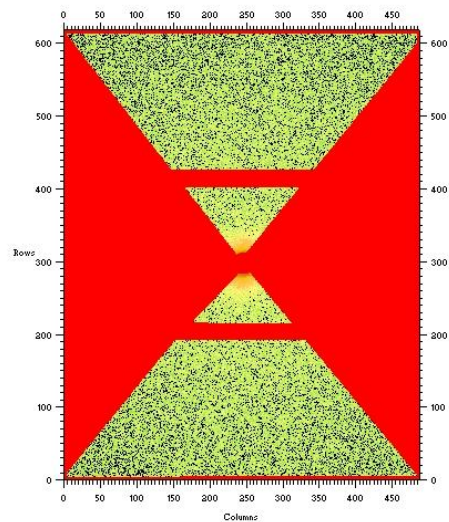
Especial Cases / Alignment

Anisotropy analysis

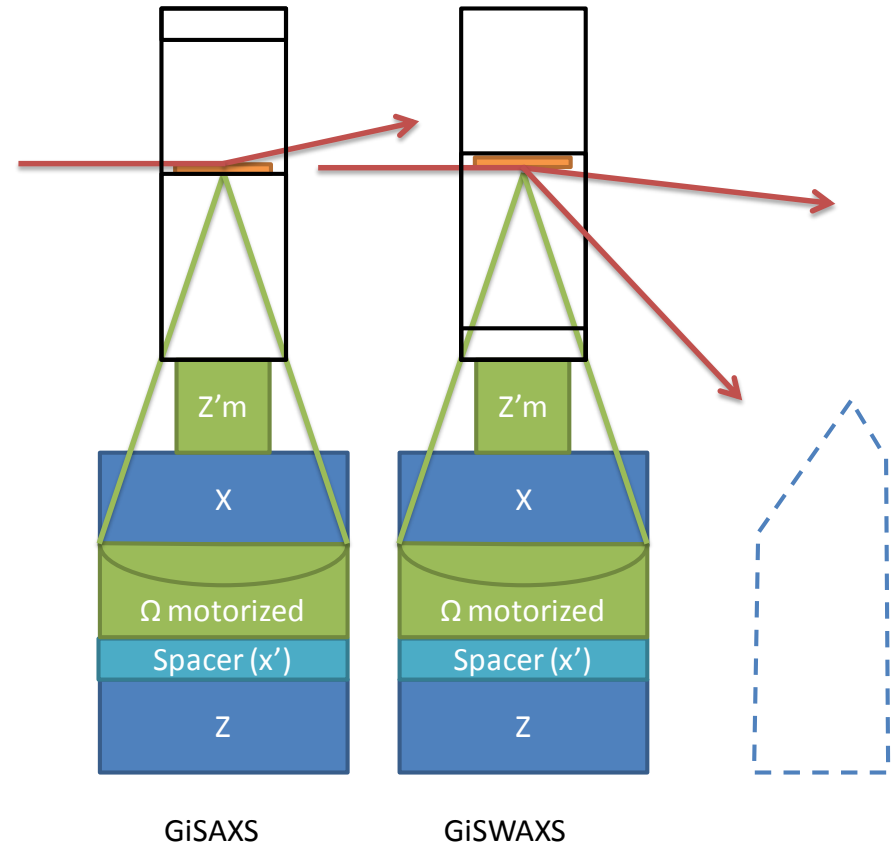
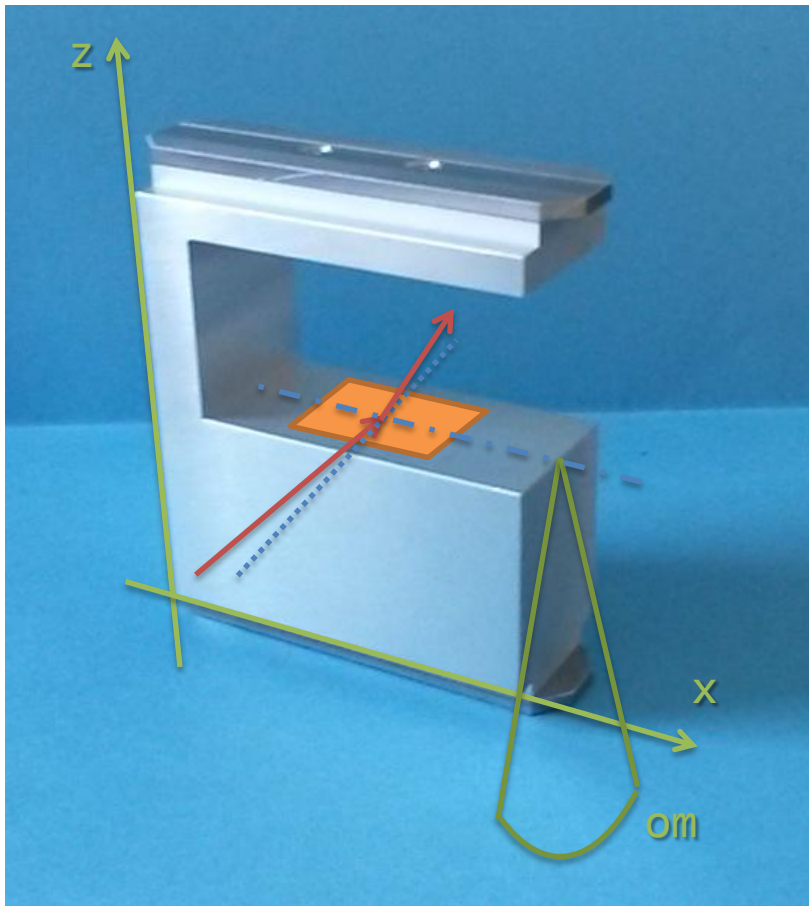


Anisotropy analysis from intensity plot at given angular position.

Anisotropy analysis

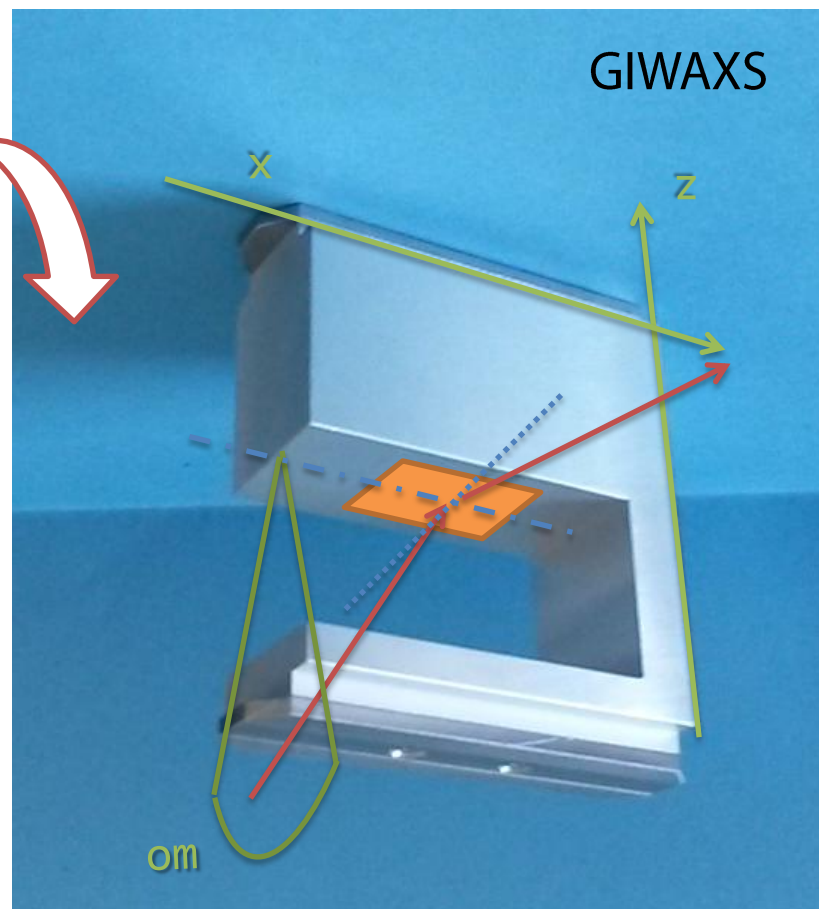
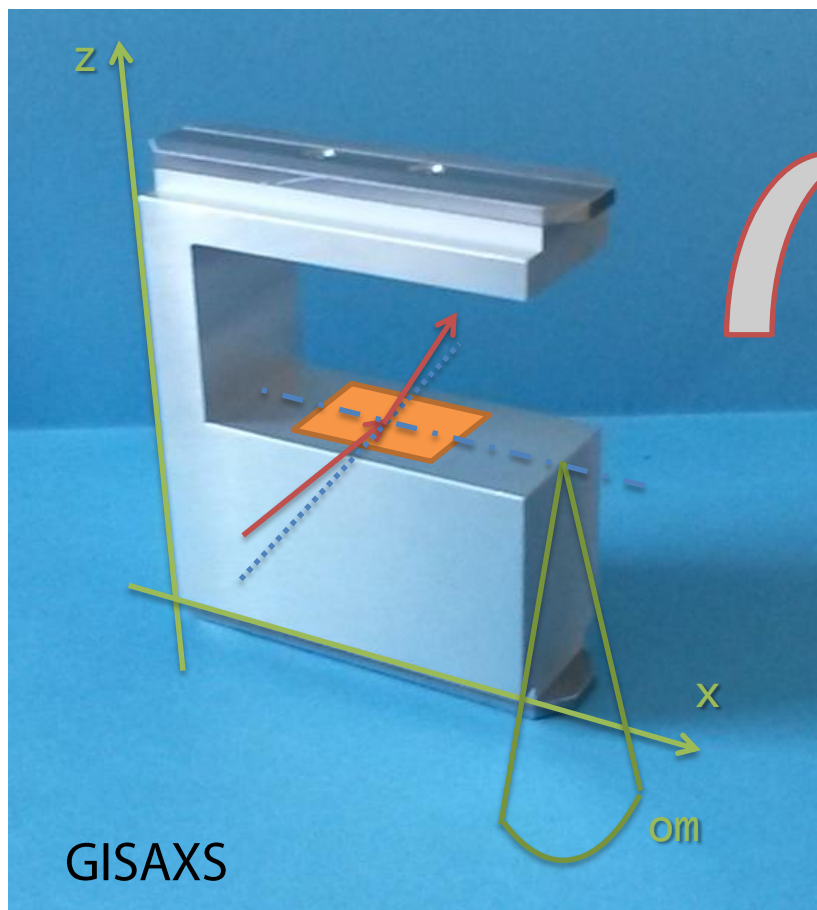


Simple GISAXS stage



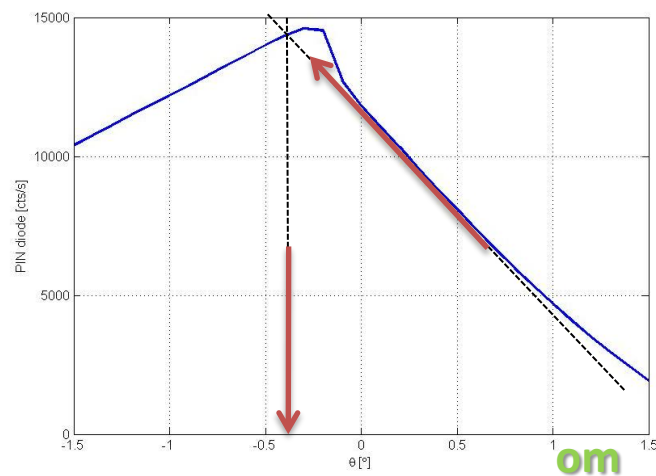
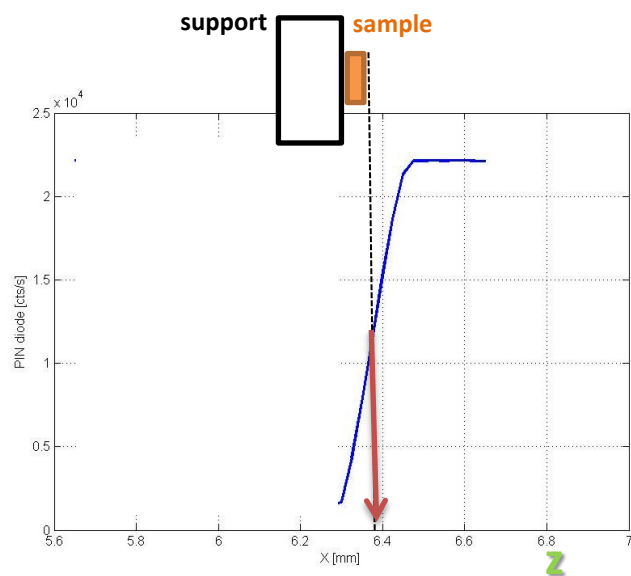
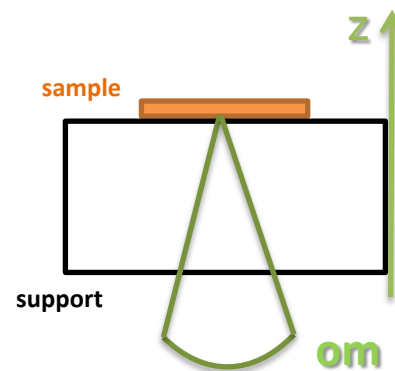
GISAXS / GIWAXS

Flip-flop the stage, not removing the sample



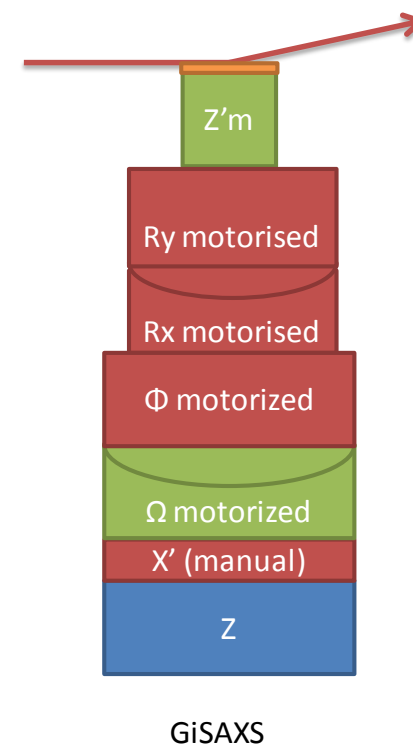
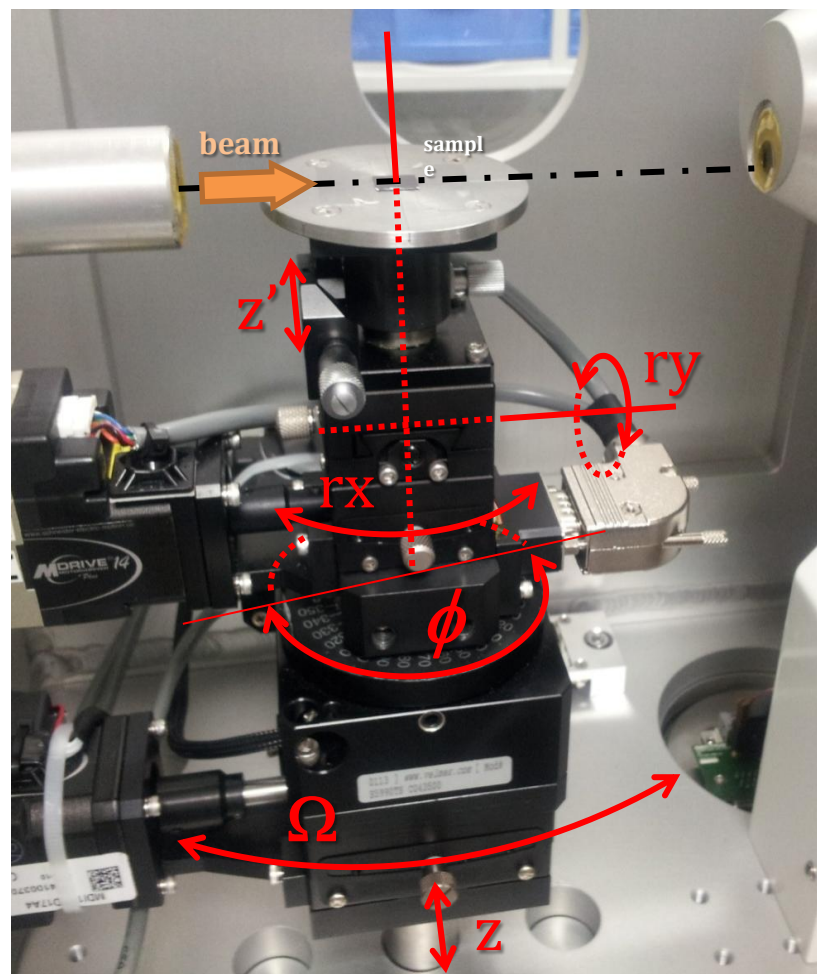
GISAXS alignment

Simple Stage



Advanced GISAXS stage

z : vertical translation
 ω : omega, reflection tilt
 ϕ : vertical rotation
 rx : fine tilt
 ry : fine reflection tilt
 z' : manual vertical translation

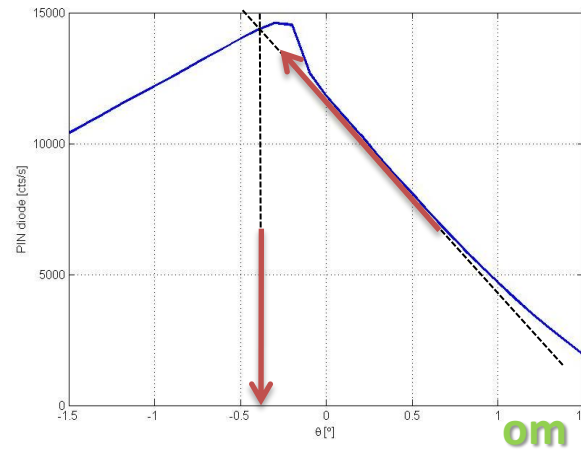
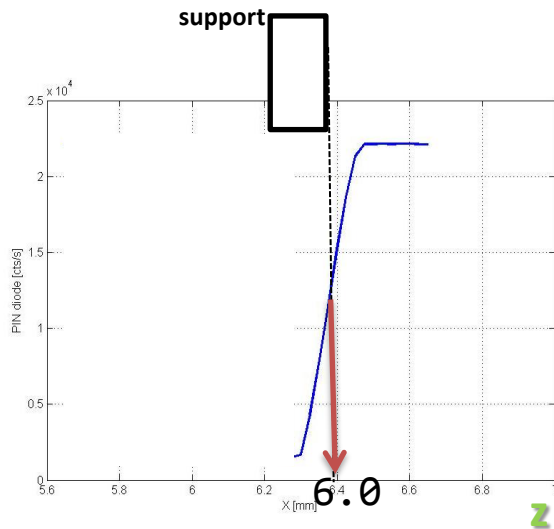


GISAXS alignment

Advanced Stage

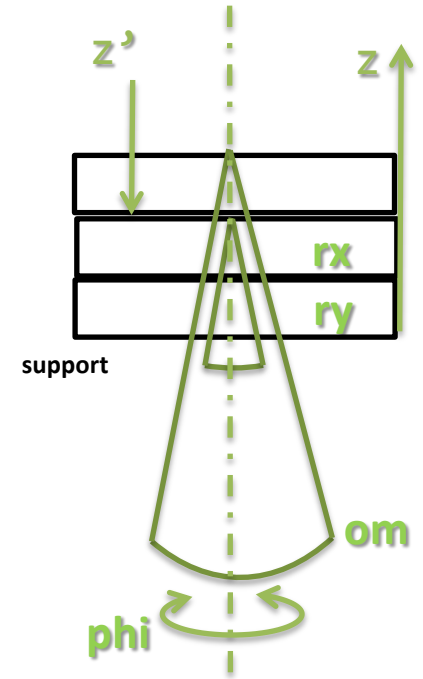
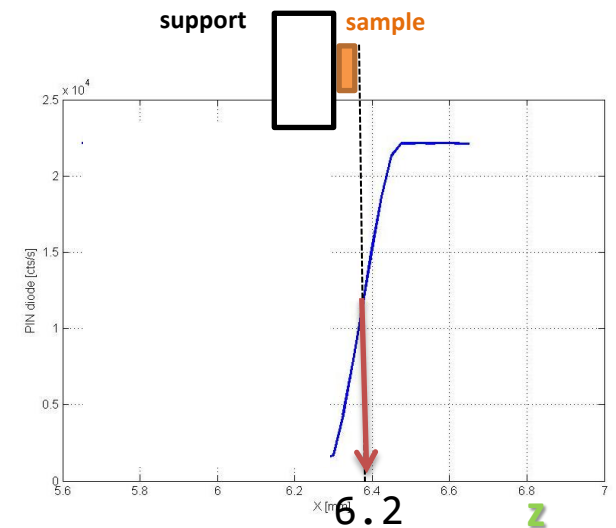
Align first the sample support into the beam.

- 1) check for edge
- 2) check for horizontality



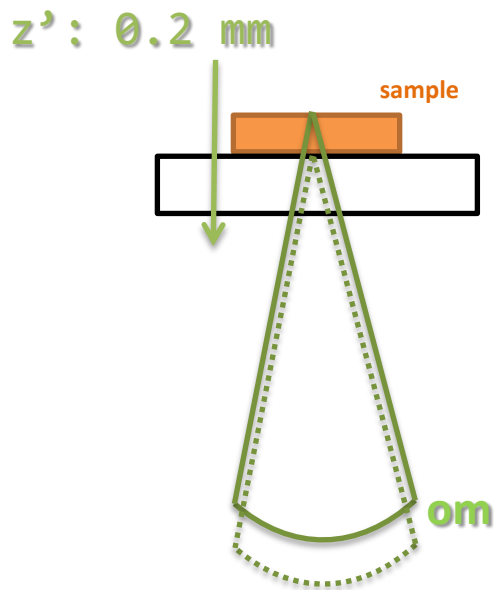
Install the sample and repeat the edge scan. This will give the sample thickness.

Sample thickness :
 $6.2 - 6.0 = 0.2$ mm

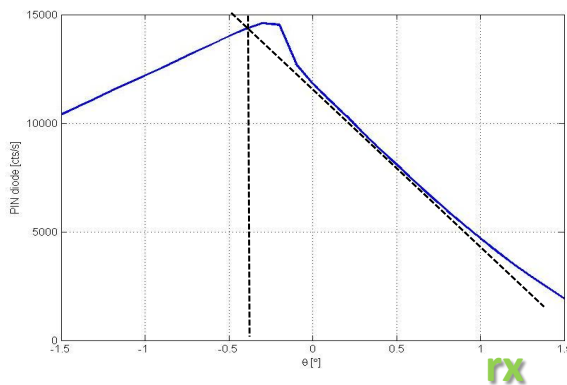


GISAXS alignment

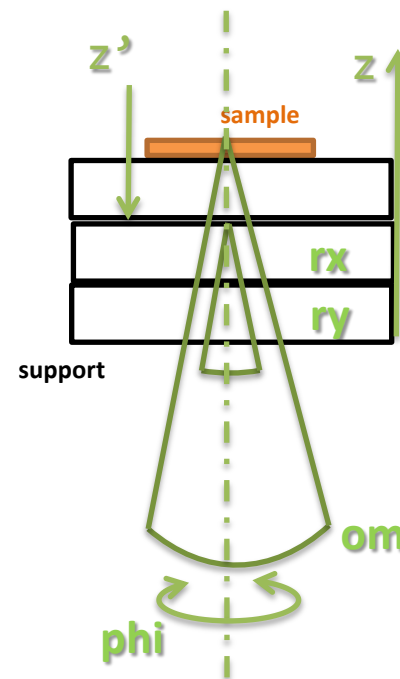
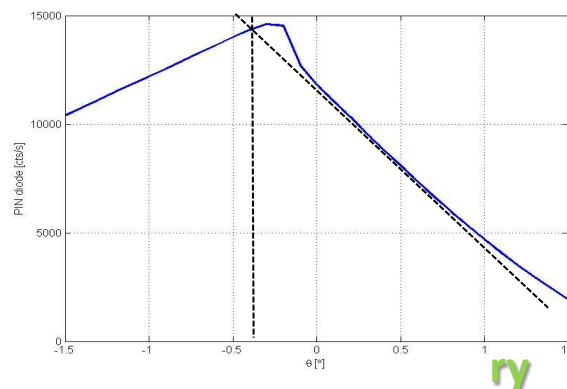
Advanced Stage



Correction of z' to shift rotation point from the support surface to the sample surface.



$\Delta\text{phi} = 180^\circ$



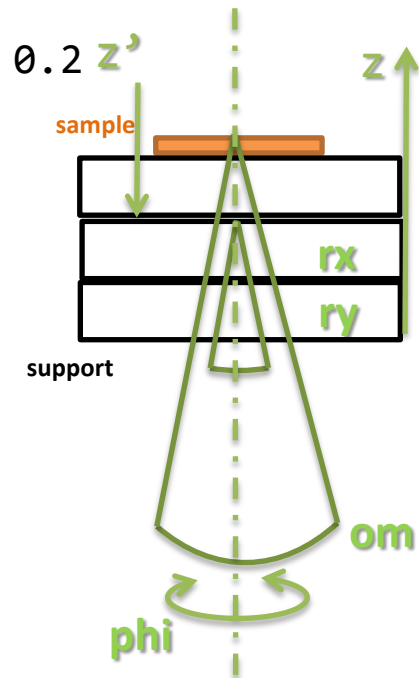
Now align the sample for horizontality in both directions

- 1) Direction 'rx'
- 2) Turn 'phi' motor 180°
- 3) Direction 'ry'

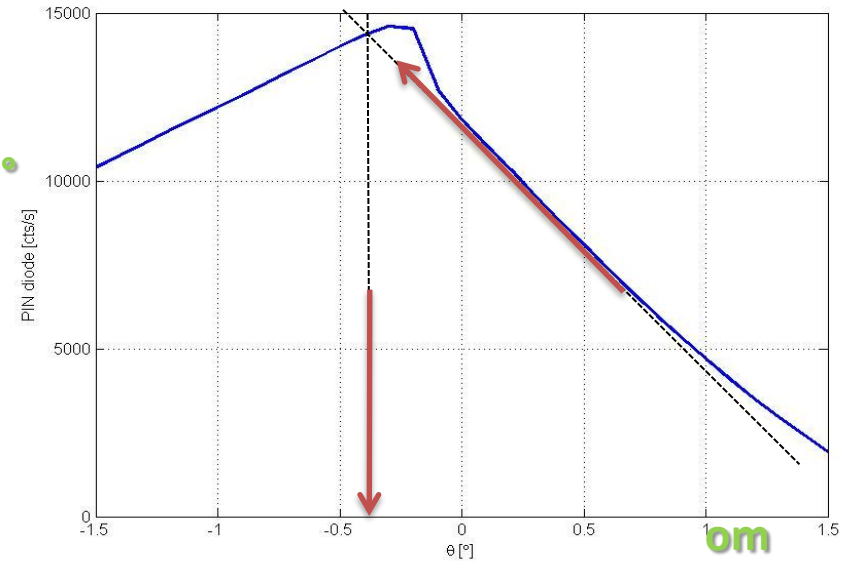
GISAXS alignment

Advanced stage

$$6.2 - 6.0 = 0.2$$



$$\Delta\phi = -180^\circ$$



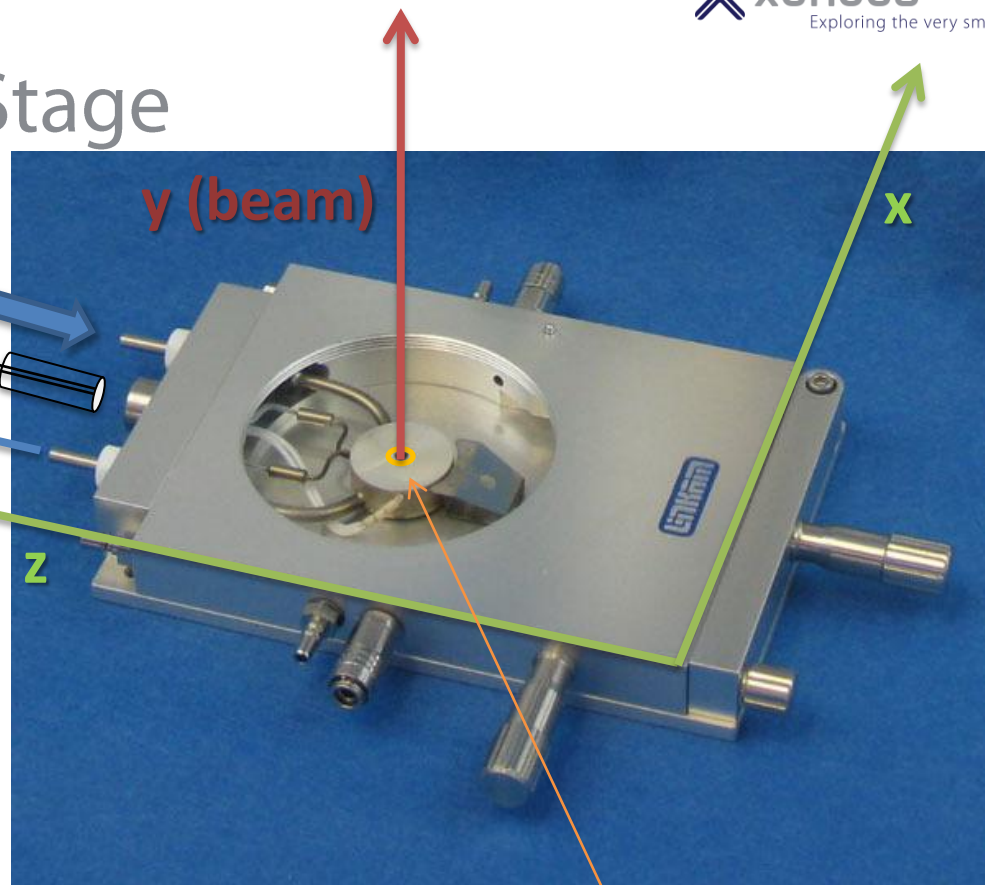
Linkam Temperature Stage

Temperature controller

LN₂ in

LN₂ out

Temperature range :
-196 °C to 350 °C

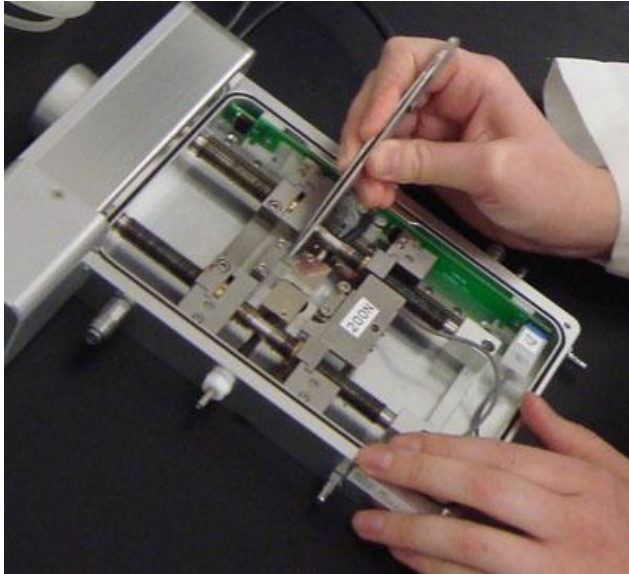


LN₂ reservoir

sample

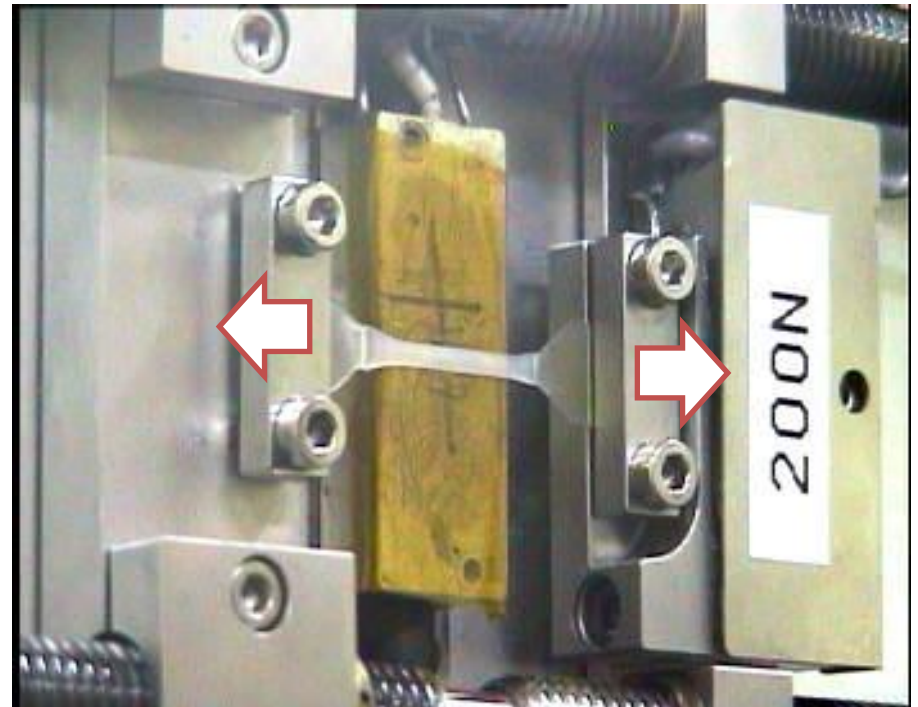
Single sample : self-standing or capillary

Linkam Tensile Stage



Temperature range :
-196 °C to 350 °C

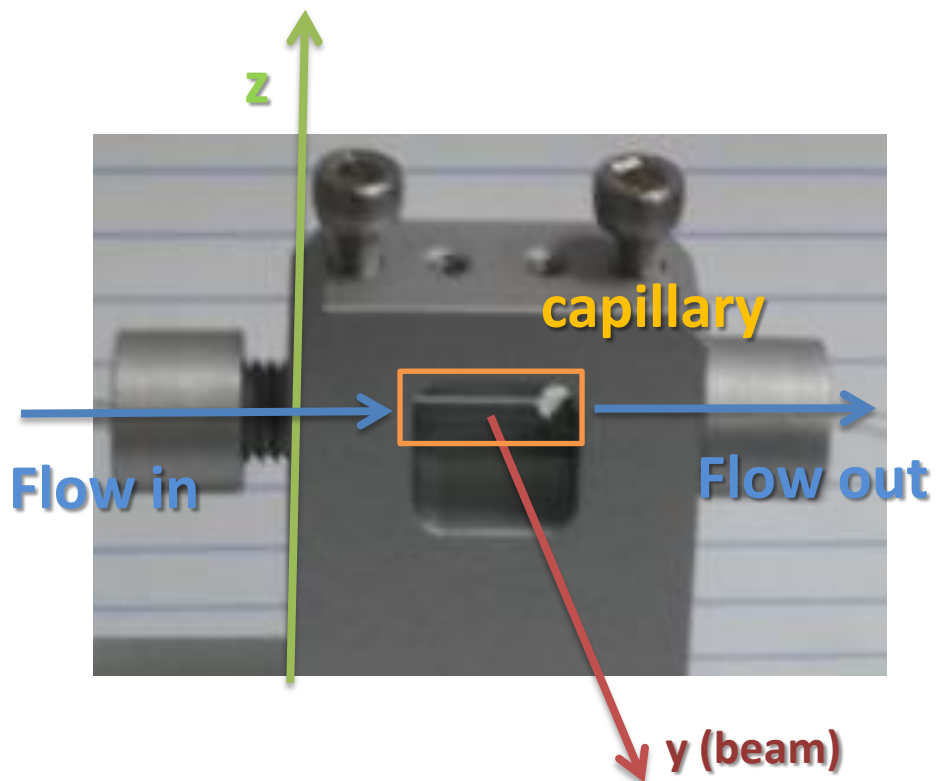
Single sample : self-standing



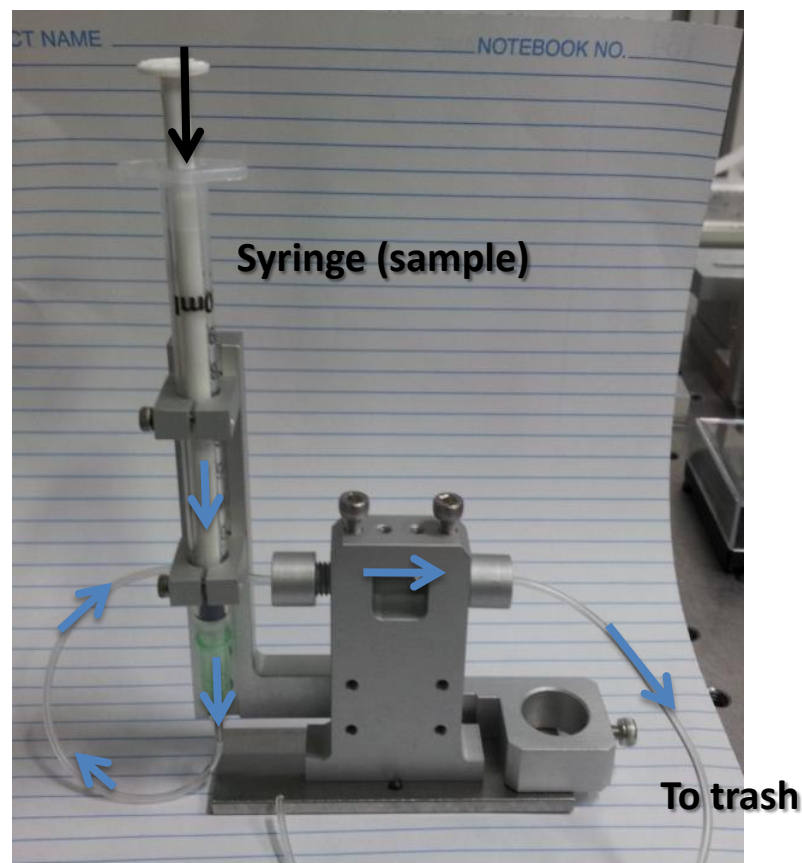
Flow-through

No temperature controlled.

For ideal buffer subtraction, measure of sample and buffer made at exactly the same position of capillary.



Push piston for flow in



Virtual Detector

Acquisition

specfe

File Edit Graph Scans Commands Help

Abort Print Prev Scan Next Scan Legend Autoscale Save Reference Clear Reference Open Shutter Close Shutter AUTO LOAD DATA Spec is: Ready

Xenocs Interface to Spec // specfe-xenocs 1.0
Datafile: /home/saxs/data/saxs_20140325.log

Status: Scan 128 loaded

Graph 1 Graph 2 Scan Data Scan List Motors/Counters Collimation Sample Detector Acquisition

Saving options
 External Inhouse
Exper. name: test
First Number: 1201

Acq. Mode
 Saxs Waxs Both

SPEC/Detector
 Use in ct/scan ROIs

Virtual Detector (SAXS)
 Use virtual det. mode
Geometry: g_vert2 Change

Settings

	SAXS	WAXS
Wavelength:	1.5411	
Beam Center X:	69.86234	19.87554
Beam Center Z:	536.2469	15.65453
Pixel Size:	172	172
Sample Det Distance:	2609.15	137.072

Standard Mapping
Exposure Time: 1
Number of Shots: 1
Cycle Time: 2
Acquire

Virtual detector / Geometry select

Square Modes
 g4x4 g_diag1 g_diag2

Horizontal Modes
 g_hor1 g_hor2 g_hor3

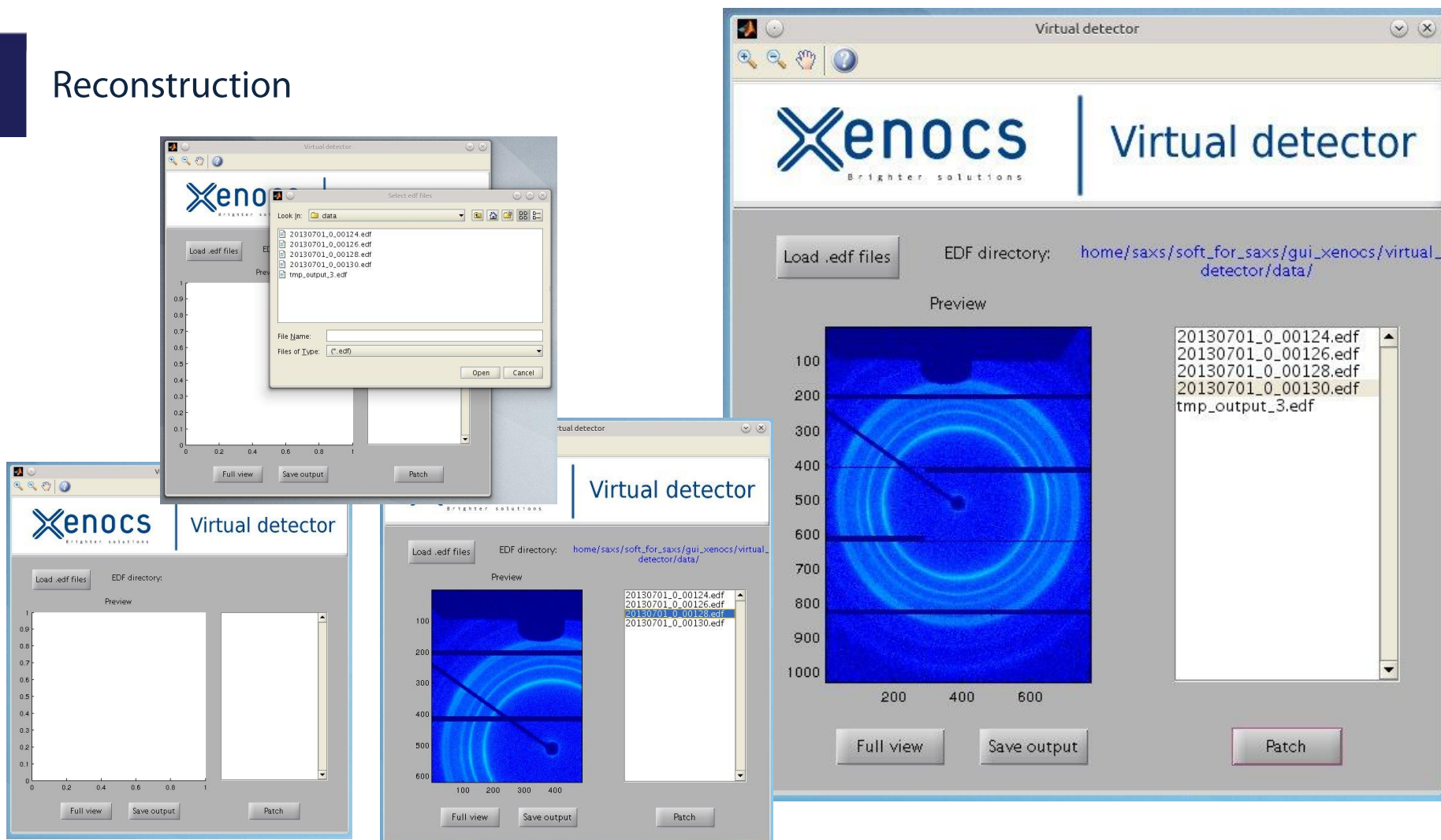
Vertical Modes
 g_vert1 g_vert2 g_vert3

OK Cancel

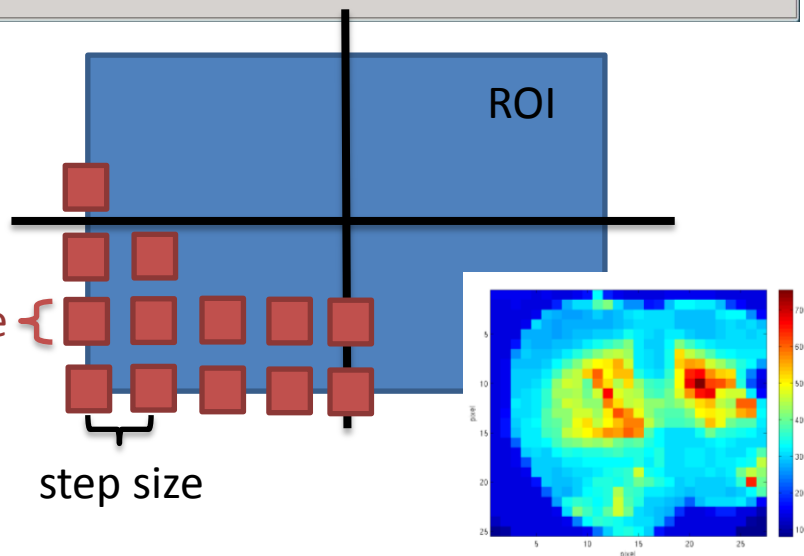
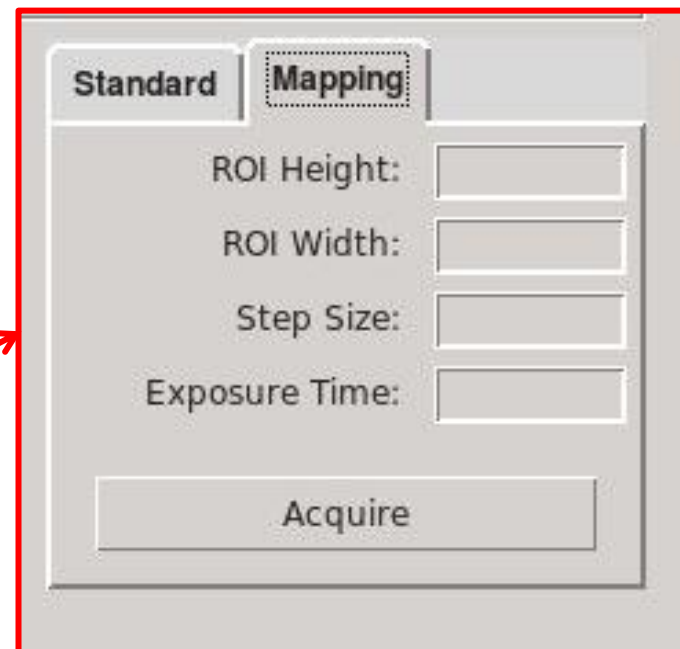
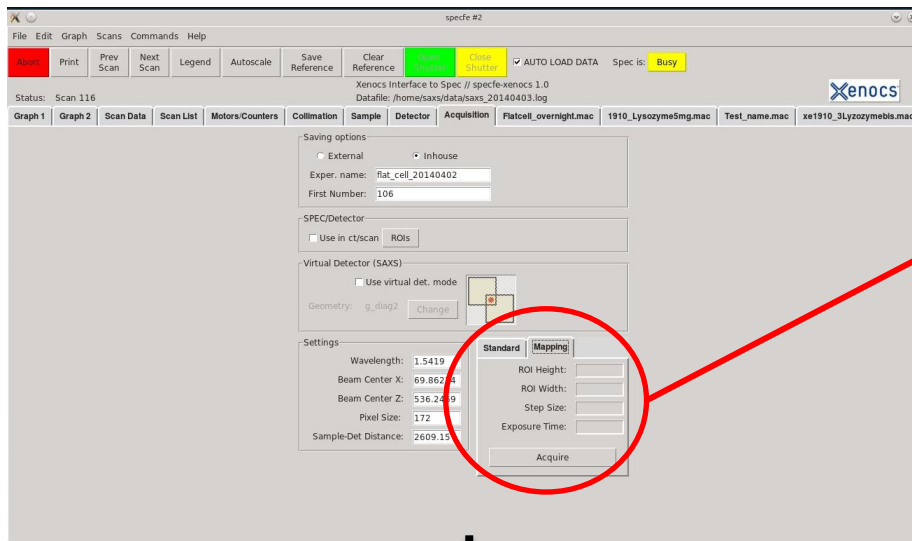
Info for Header

Virtual Detector

Reconstruction



Mapping Acquisition



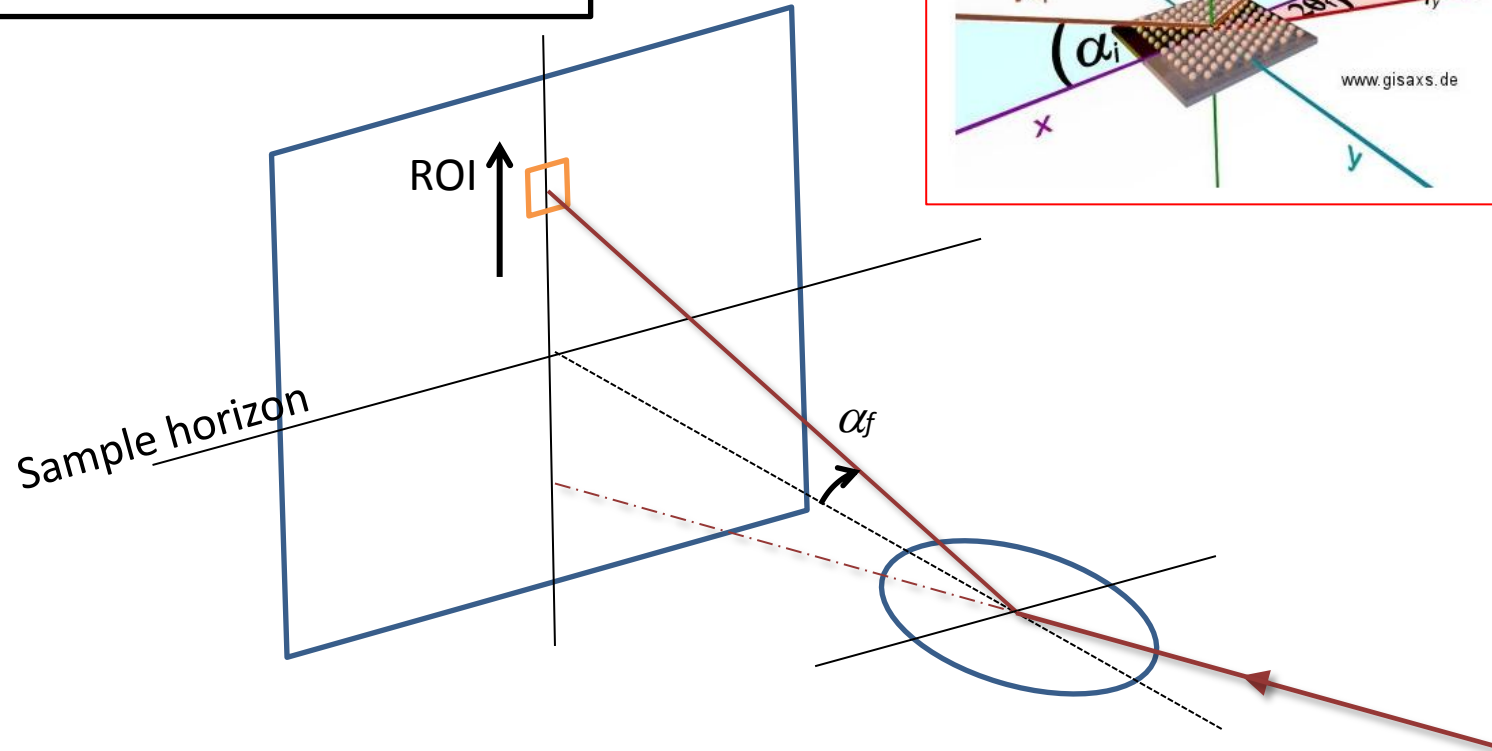
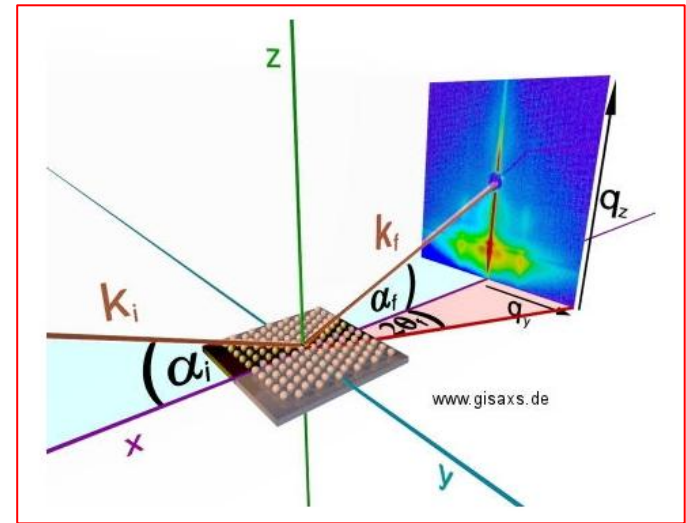
RASTER SCAN:

- The acquisition program calculates the number of measured points as function of the size of the ROI and the size of the beam, and calculates the motor positions for the sample stage to move.
- Takes one SAXS/WAXS exposure for every position (moves automatically) of the scan starting from the actual motor position at center of ROI.
- If $\text{step-size} = \text{beam-size}$: total coverage of area

Reflectometry

Follow-up of the intensity of the specular reflection while scanning the incident angle.

The software creates a drifting ROI, and measures the intensity, frame-by-frame. A reflectivity curve (Intensity versus angle) can therefore be reconstructed.



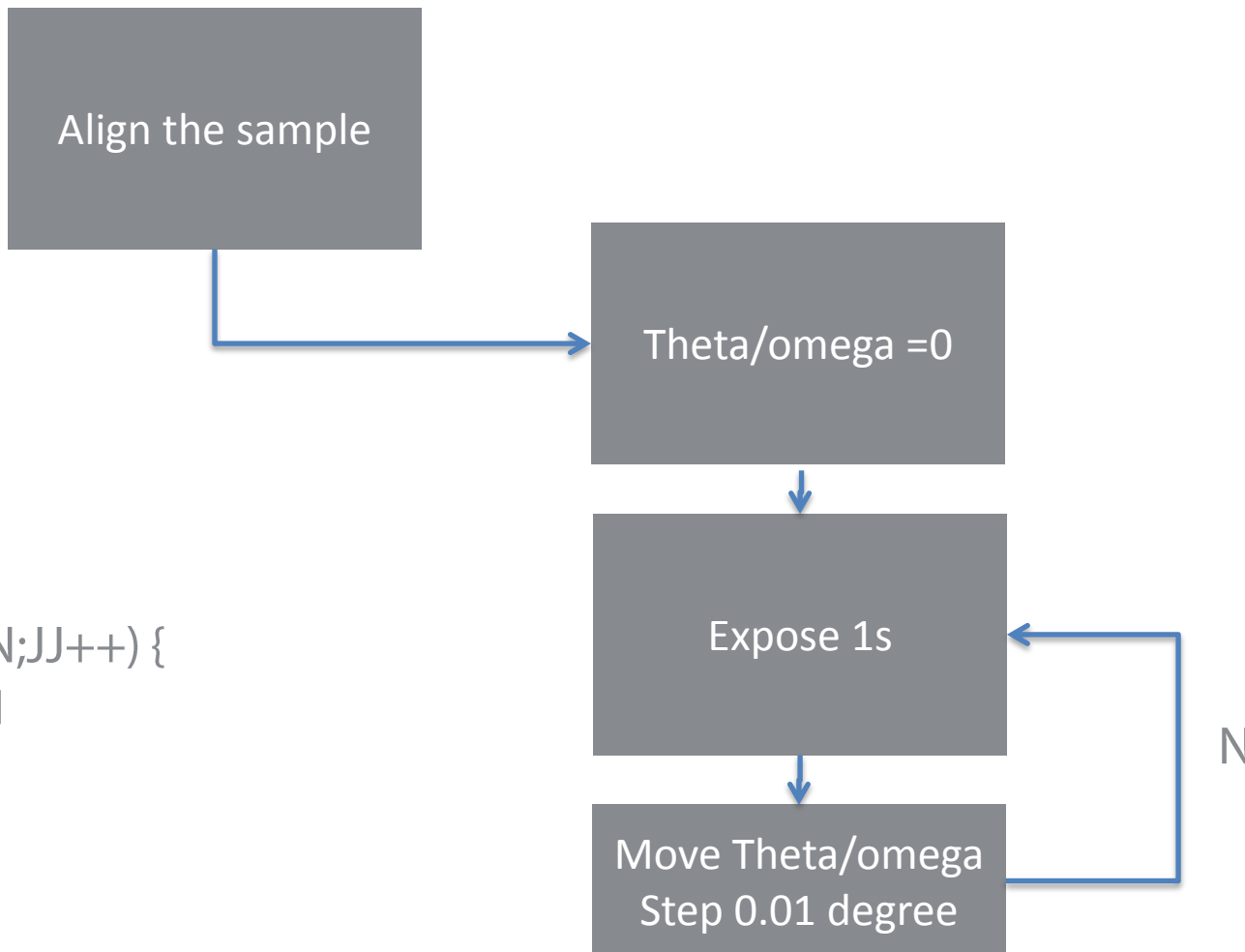
Reflectometry

Acquisition

- SPEC macro

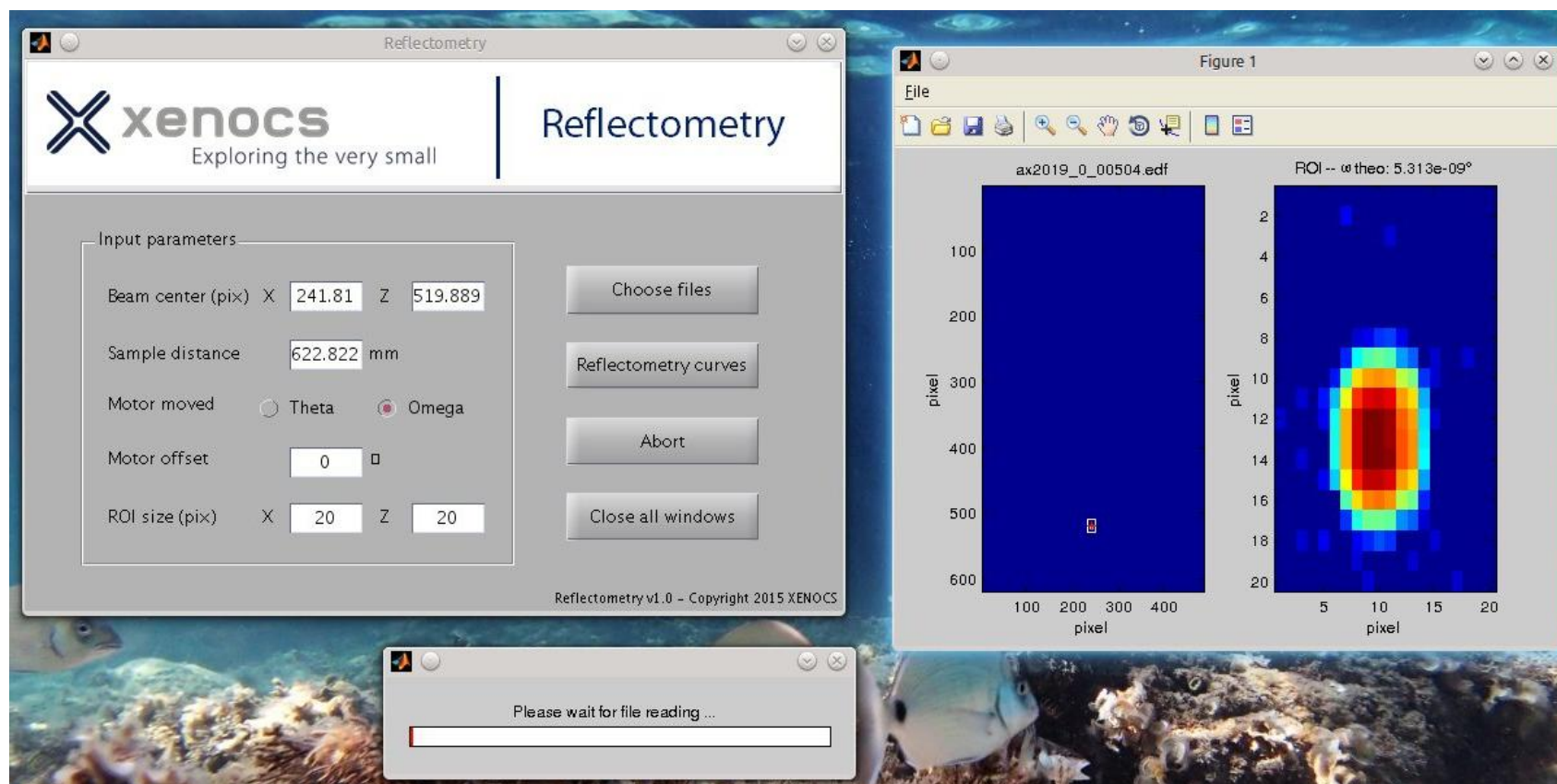
```
umv th 0
```

```
for (jj=0;jj<N;JJ++) {
multiexp 1 1
umv th 0.01
}
```

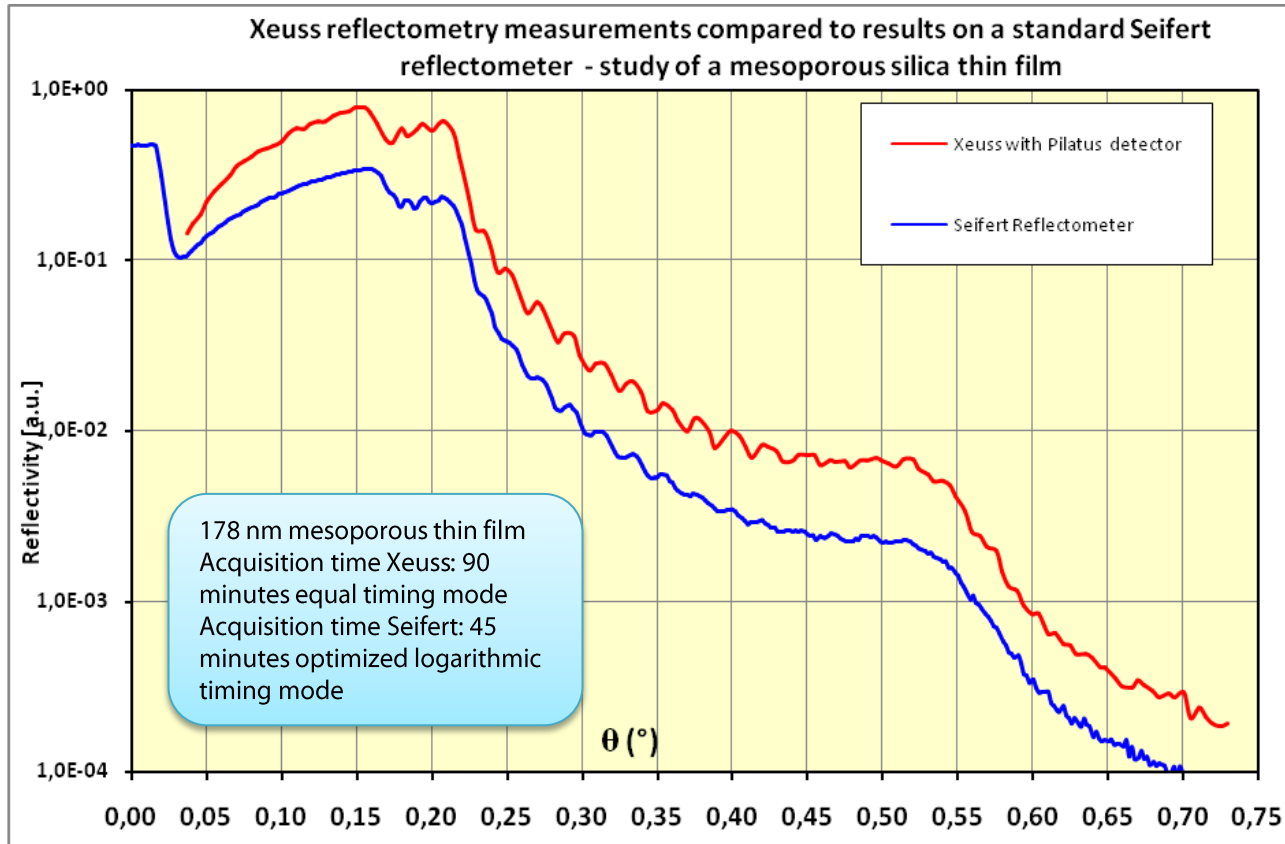


Reflectometry

Reconstruction



Reflectometry



Comparison of a Xeuss reflectometry measurement (sample to detector distance of 2.4 meters, θ step of 0.005°) with an acquisition on a standard reflectometer (seifert reflectometer). Measurement on the mesoporous thin film.

Thank you
for your attention!