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. Theses 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 transmisison
  - 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 emtpy containers (empty cuvette)
  - Buffer or solution

Data reduction

- Make angular averages of data
- Calculate sample-to-detetctor 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

- Modellisation 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

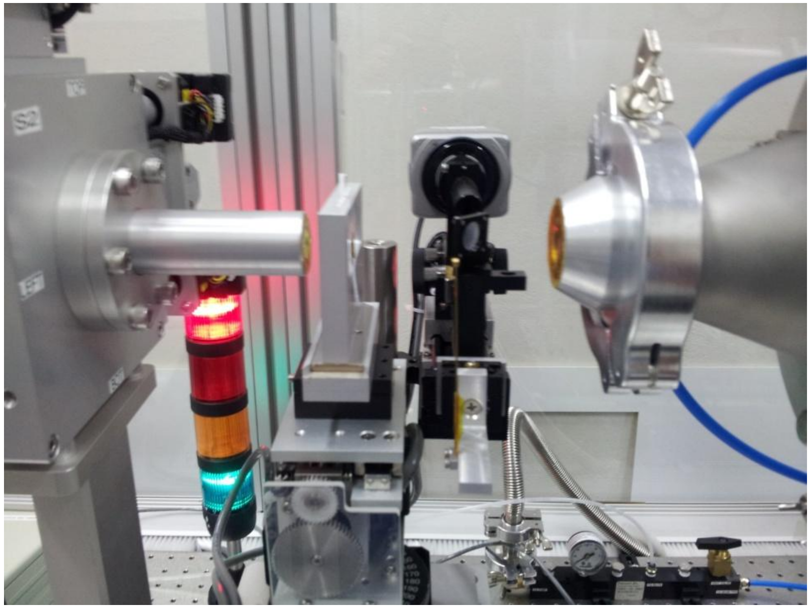
5) Subtract the empties

6) Save the data for analyze

Preparing and Making a Measurement

Prior to measurement

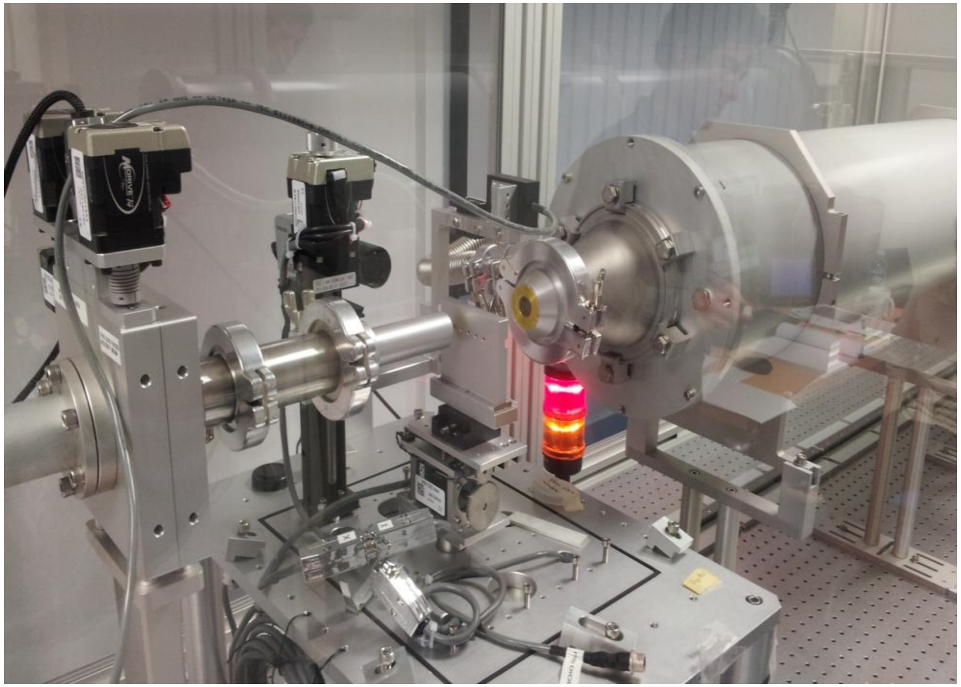
- Fixed Instrument (Xeuss)
  - Multilayer, collimating mirrors
  - Energy and tube power
- Select Detector type
  - SAXS, WAXS or both
- Selection of sample holder
  - Large choice for Xeuss
- Selection of distance sample-to-detector.
  - Modular camera length



- Selection of slit collimation and flux.
  - Double set of slits with motorized independent blades
- Select Exposure time
  - Noise and statistics

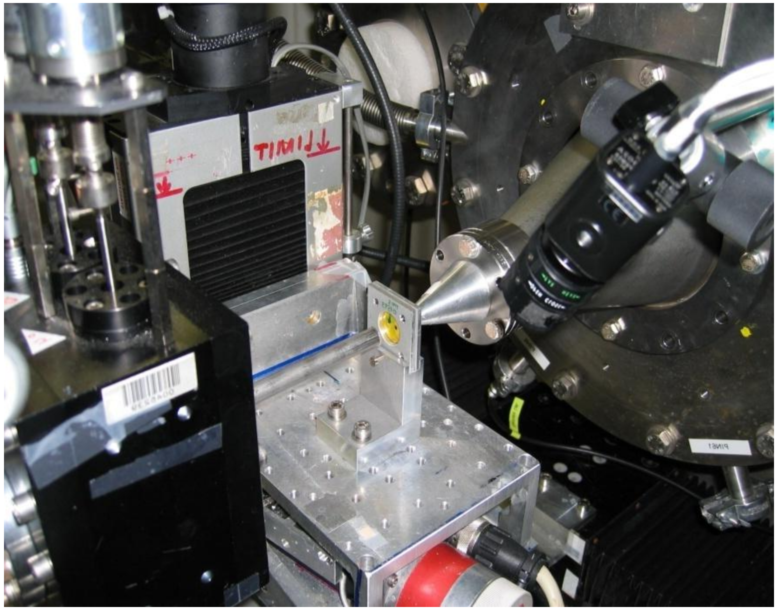
Experiment set-up

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

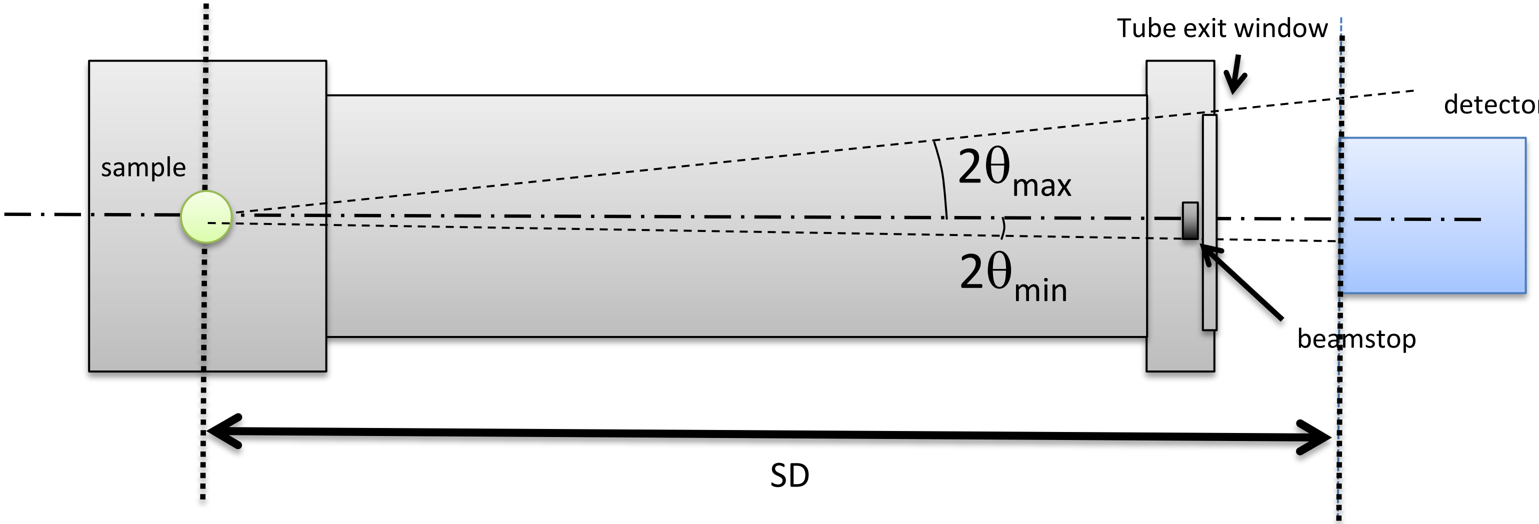
$$Q_{min} = \frac{4\pi \sin\theta_{min}}{\lambda}$$

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

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

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

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

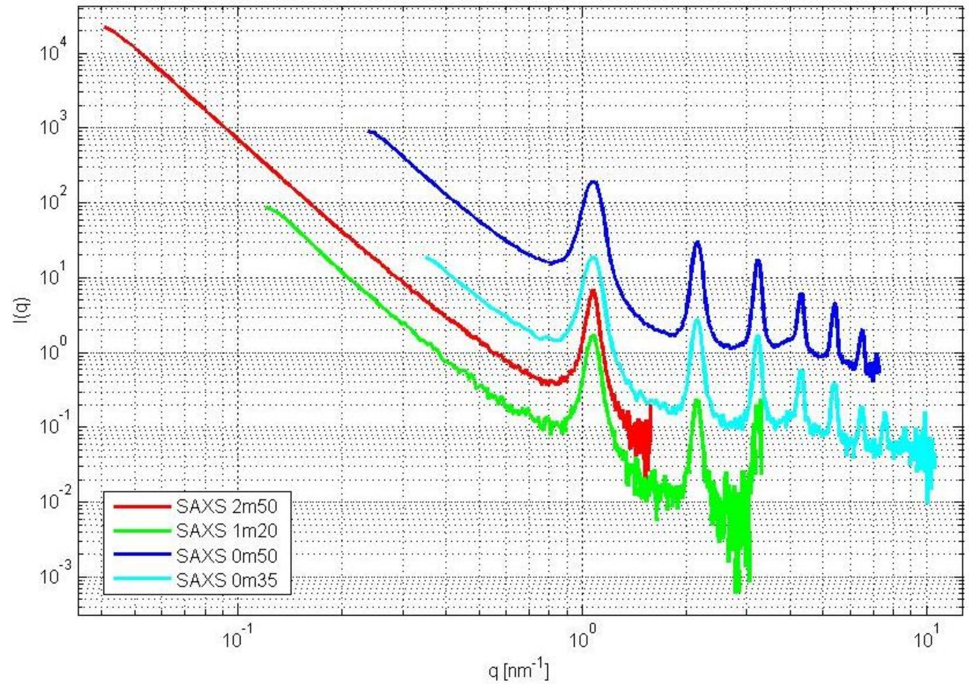


Q-range (aprox.) as function of SD distance

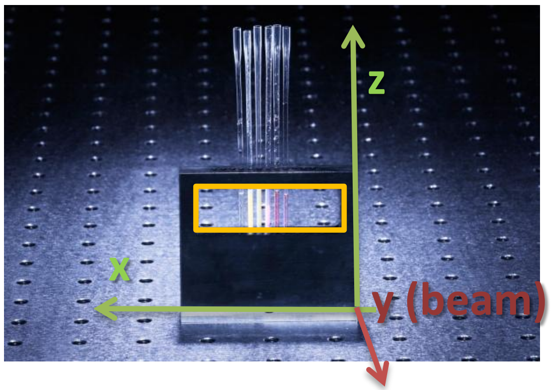
Beam on geometrical center of detector and beamstop

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

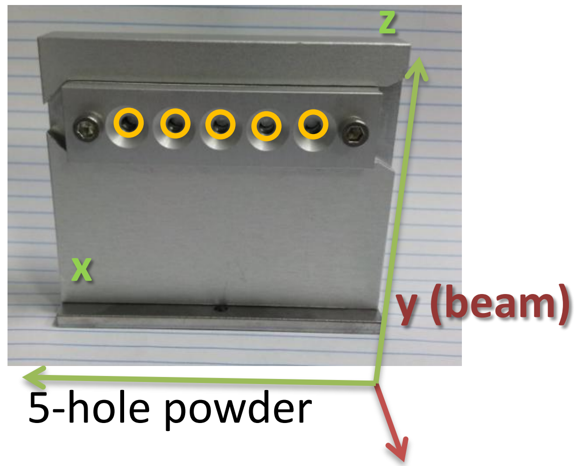
Cu radiation



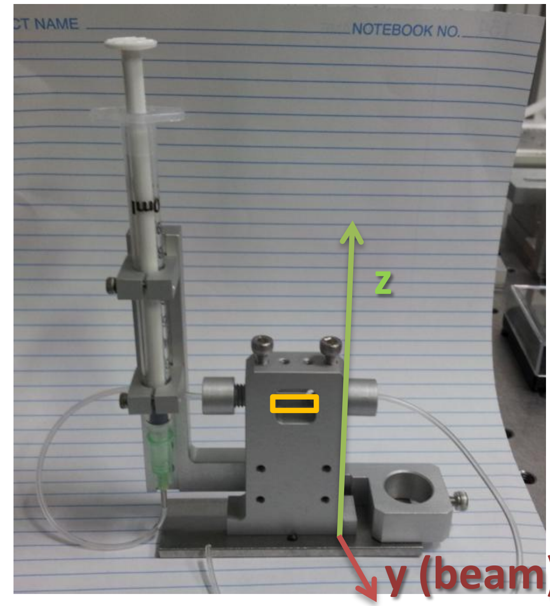
Select Appropriate Sample holder



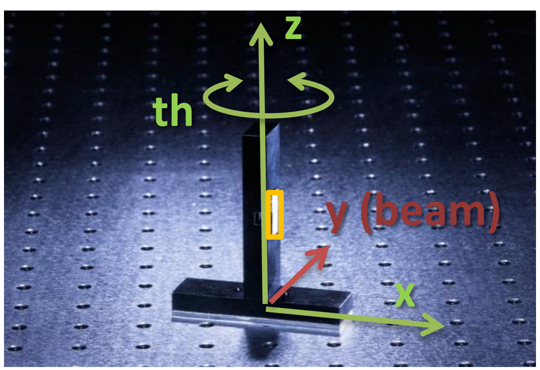
Multicapillary



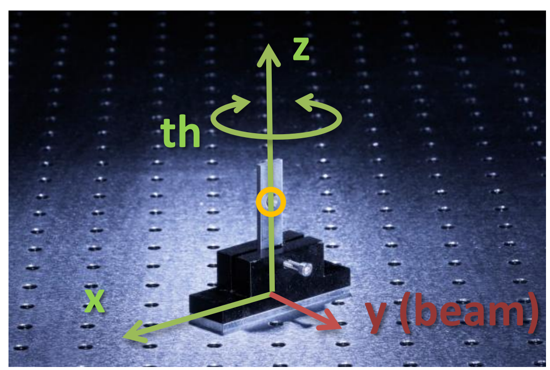
5-hole powder



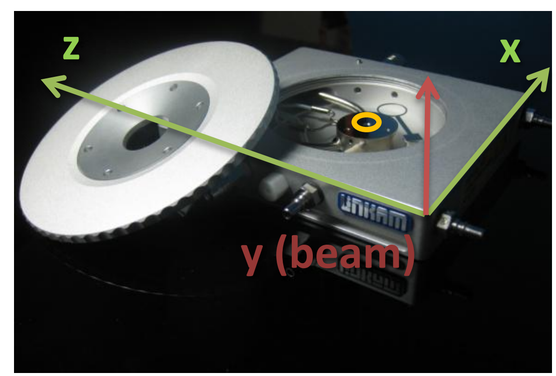
Flow-through



GiSAXS



Alignment tool



Linkam temperature

X-ray energy and sample thickness

- Optimal thickness

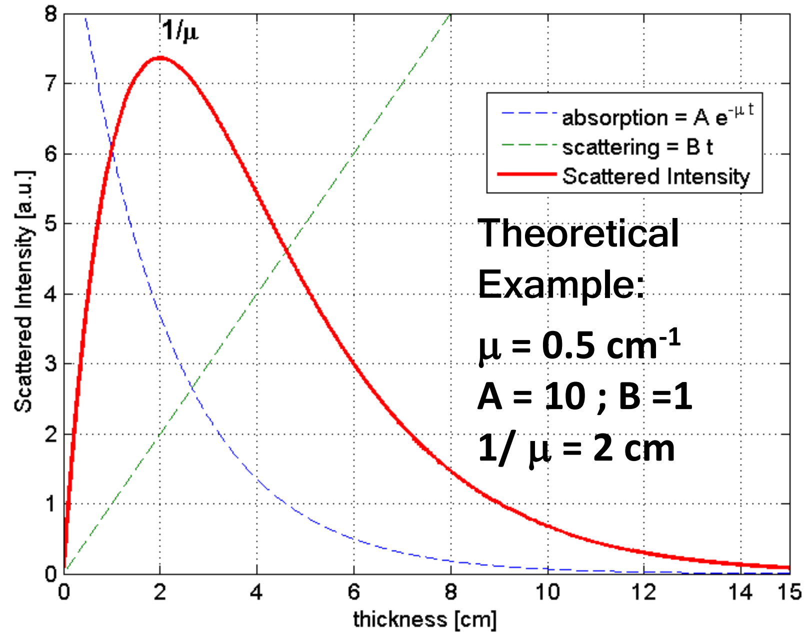
  Are we going through? Is the energy enough?

  Absorption exp(-μt)

  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 andspoils scattering signal (non-resonant).



Theoretical Example:

μ = 0.5 cm^{-1}

A = 10 ; B =1

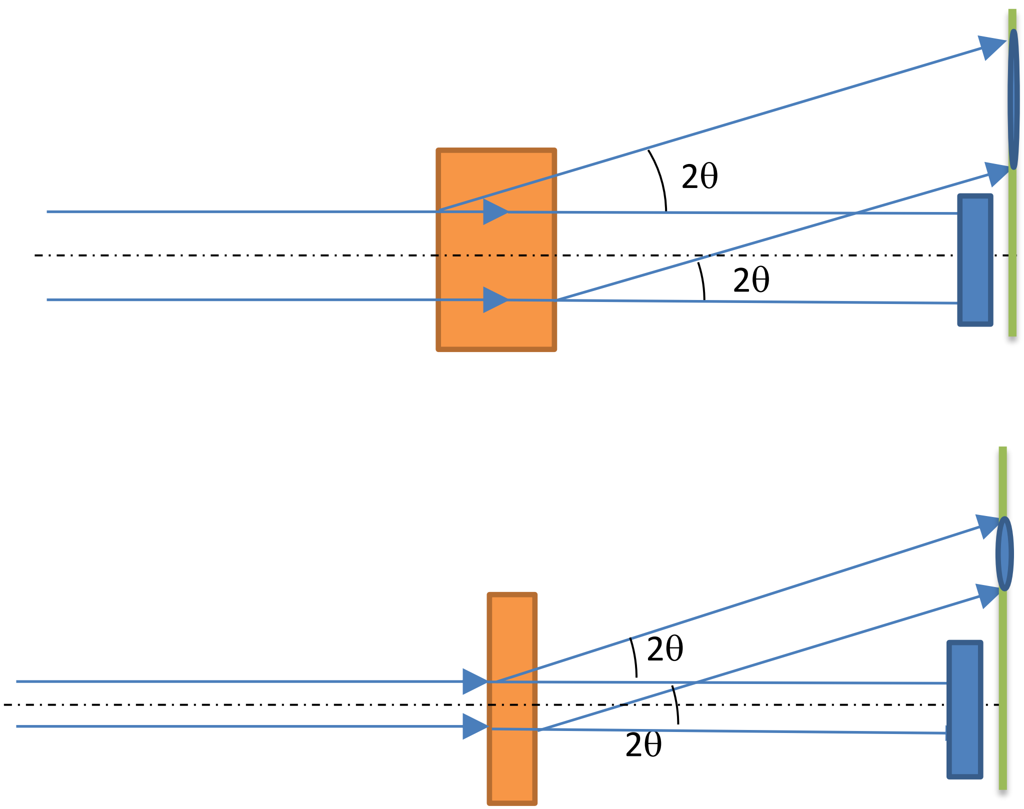
1/ μ = 2 cm

Optimal thickness : 1/μ

m : function of radiation energy and atomic number

Cu Radiation and Carbon: **1/μ = 1 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.

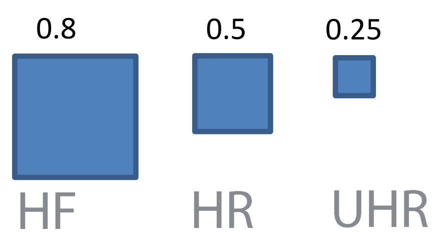
Compromise between flux and resolution!

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

[1] Relative to High Flux

Relative size



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

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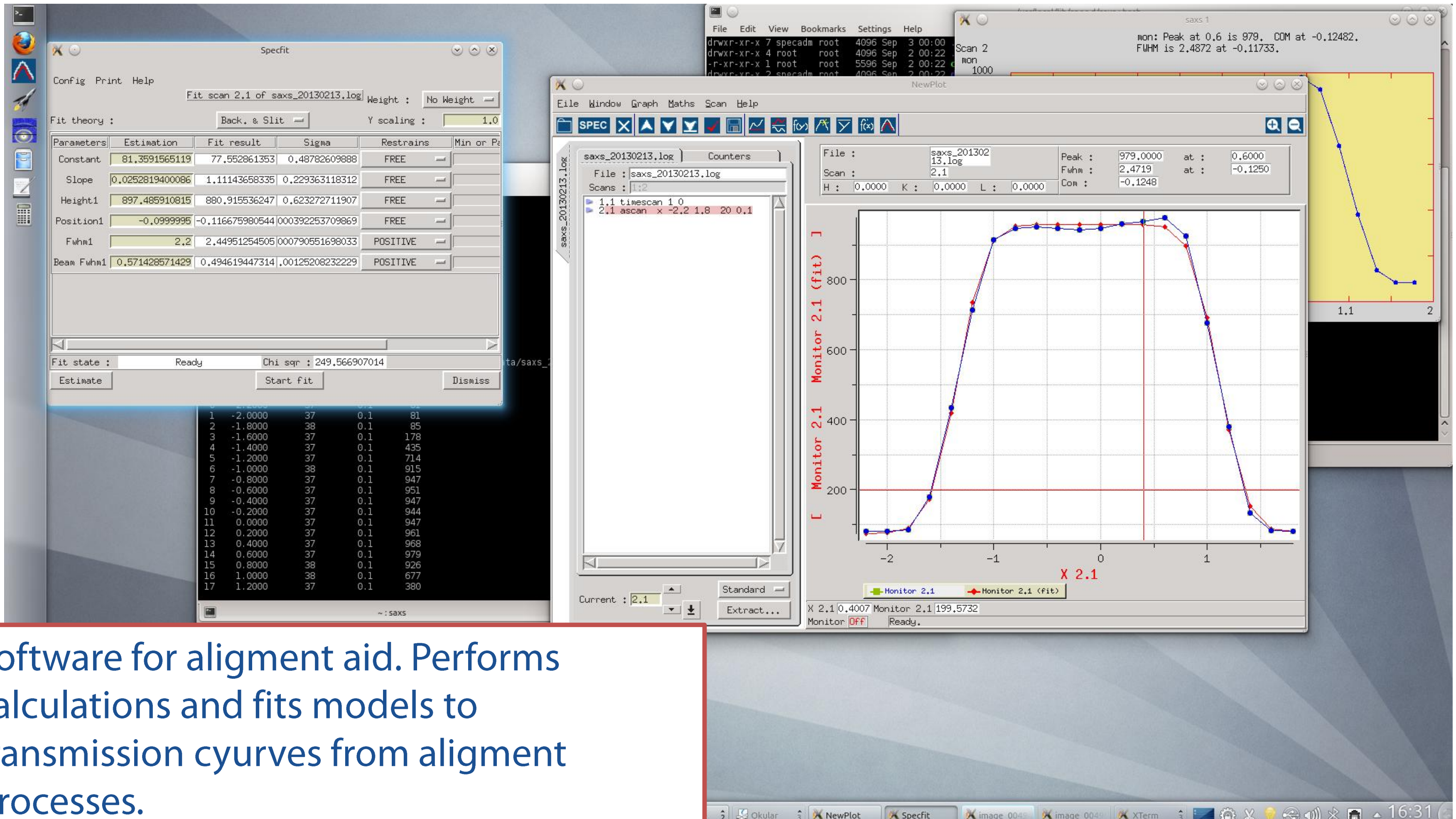
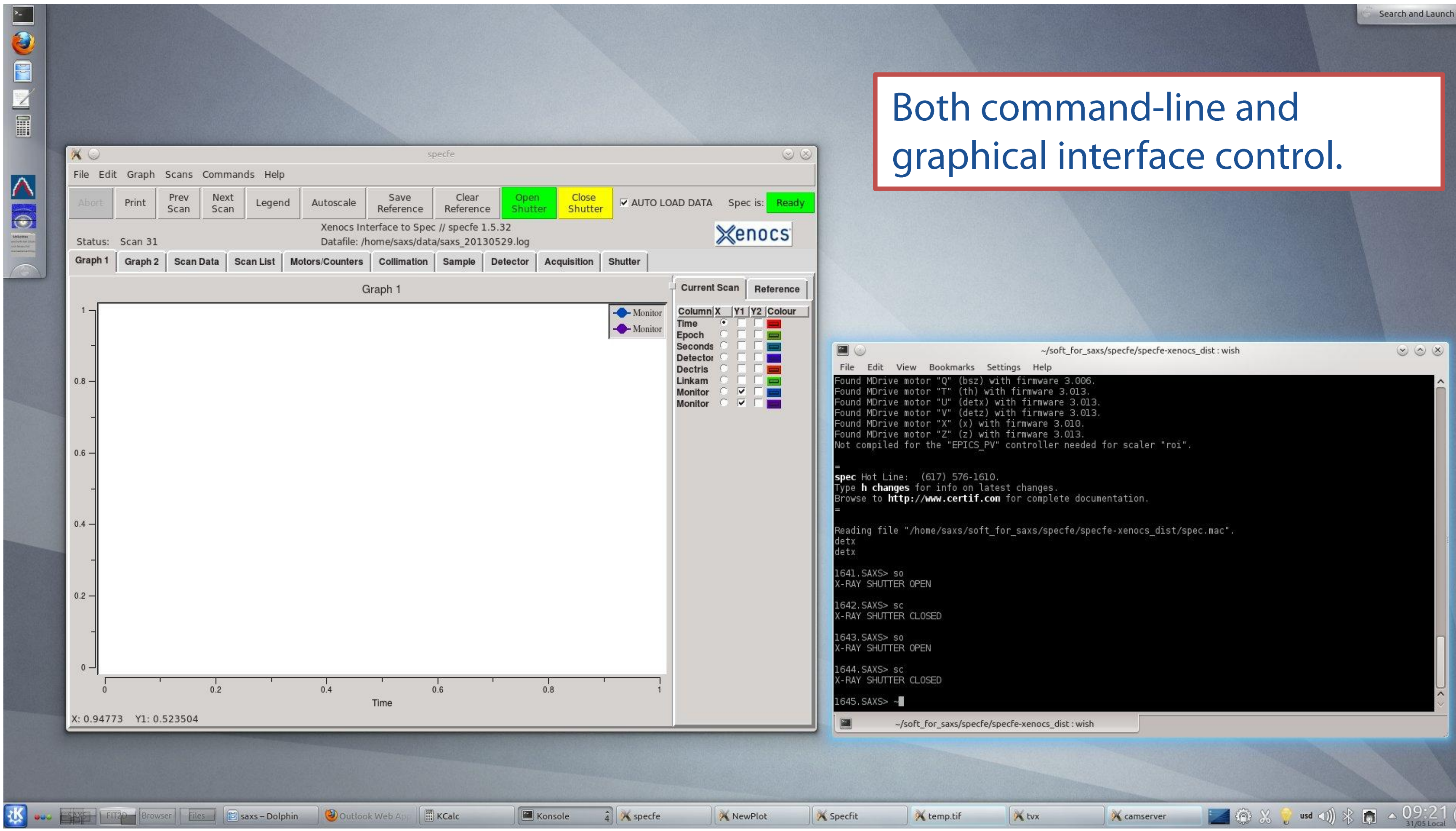
Data Acquisition software

Newplot



Software for aligment aid. Performs calculations and fits models to transmission cyurves from aligment processes.

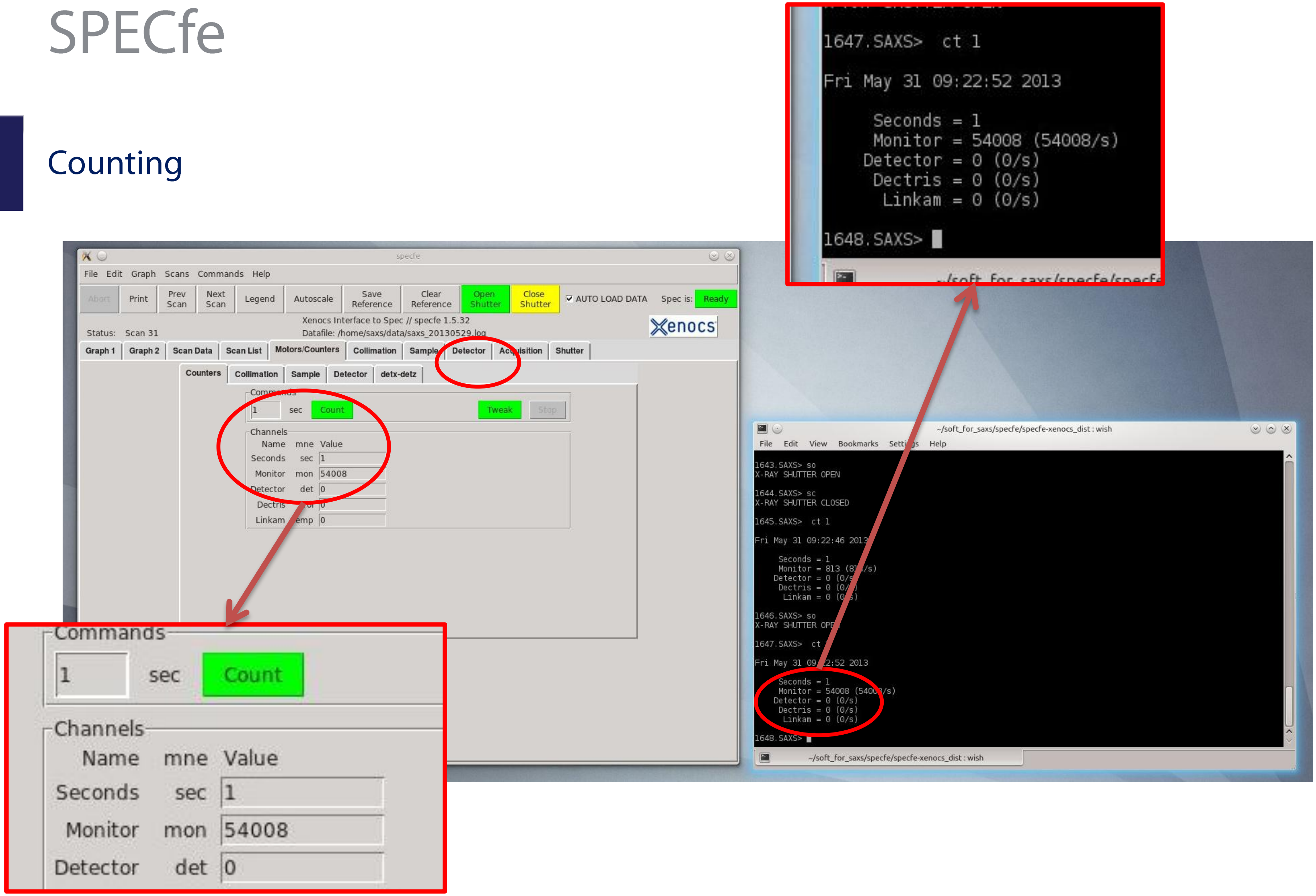
SPEC front-end graphical interface / SPECfe



Both command-line and graphical interface control.

SPECfe

Counting



SPECfe

Slit collimation and Flux

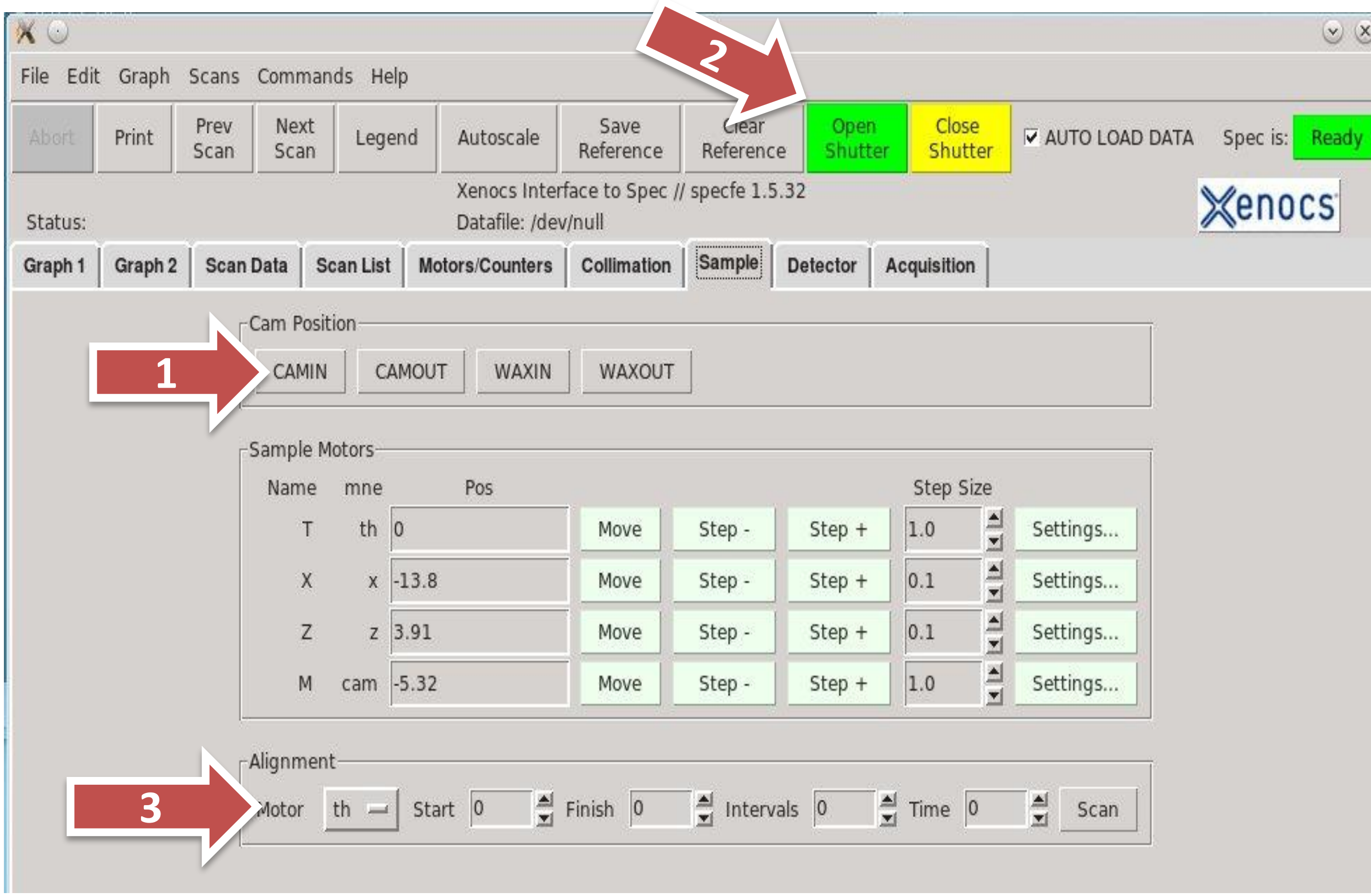
- High flux (HF). Nominal aperture.
  S1 1.2 mm / S2 0.8 mm
- High resolution (HR).
  S1 0.6 mm / S2 0.5 mm

Slits are smaller and but the flux is much reduced.

SPECfe

Sample holder alignment

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



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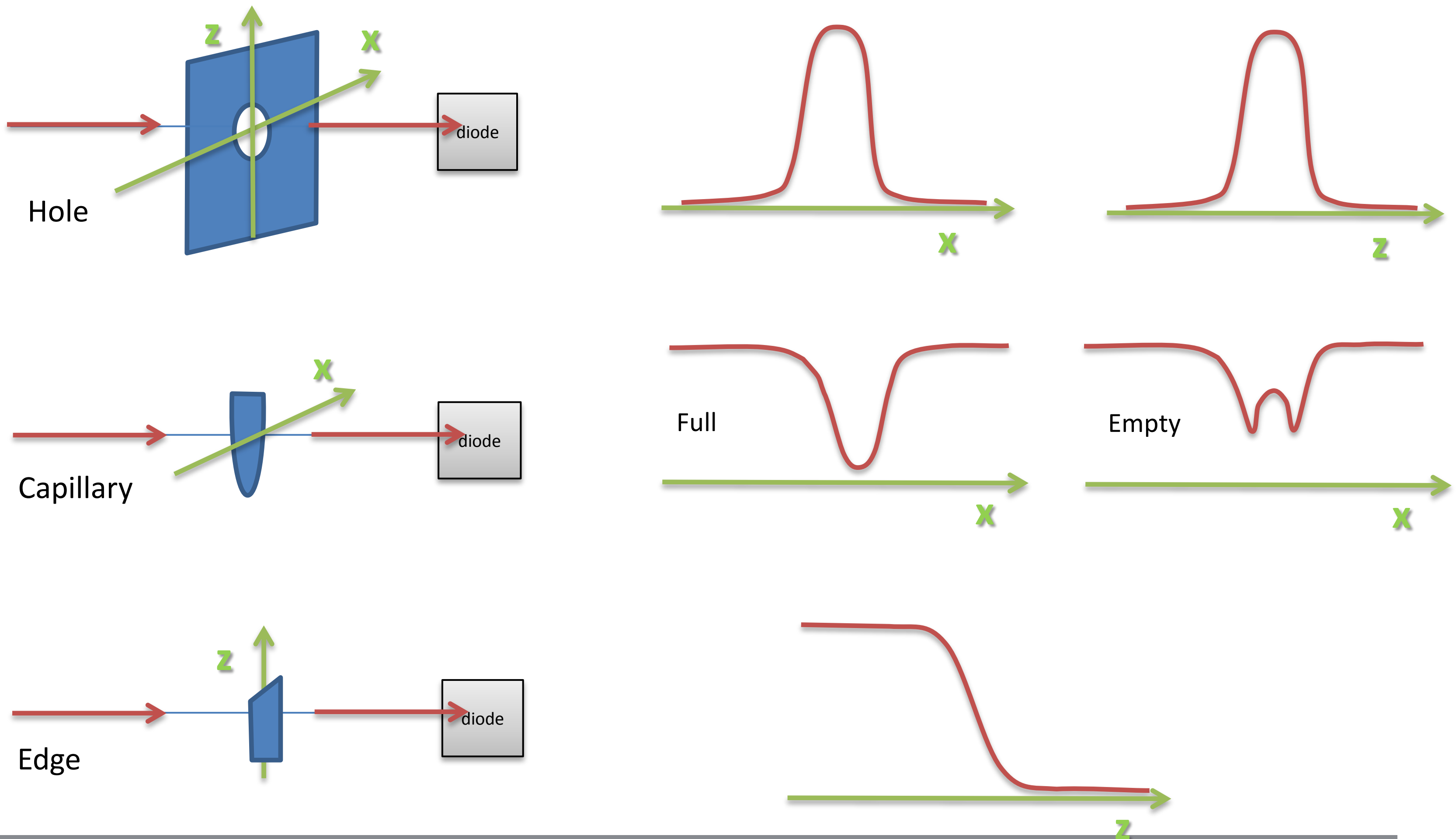
Sample holder alignment

Motor Scan

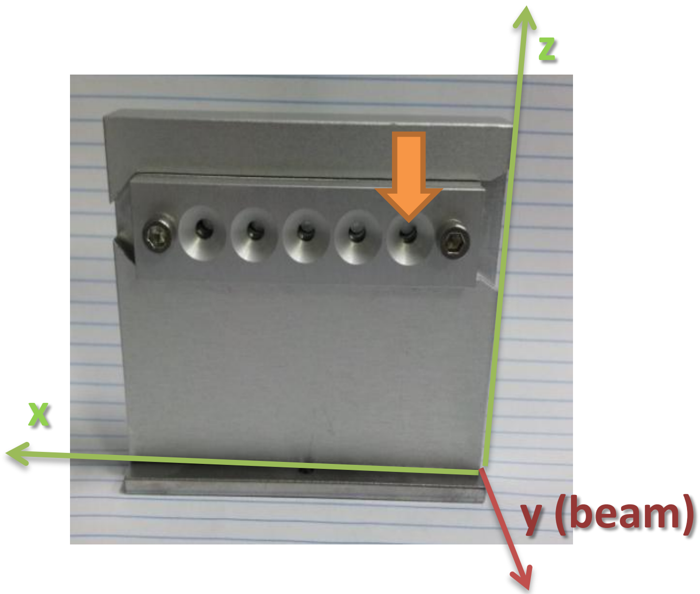
Transmission curves of sample holder across the beam.

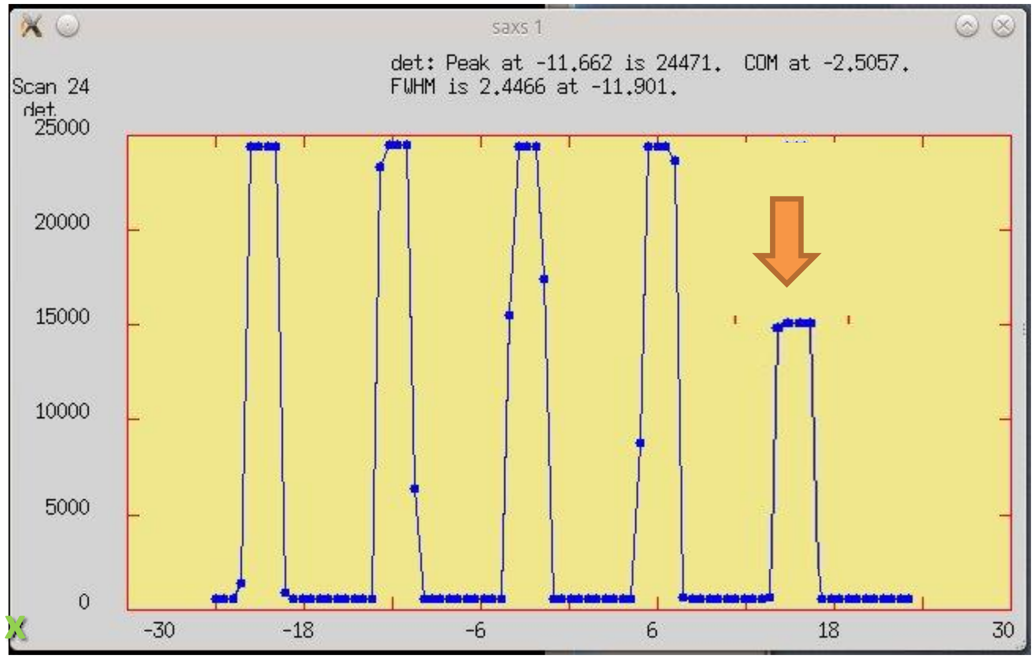
Intensity of beam measured with PIN diode, as function of motor position.



Sample holder alignment

5-hole holder



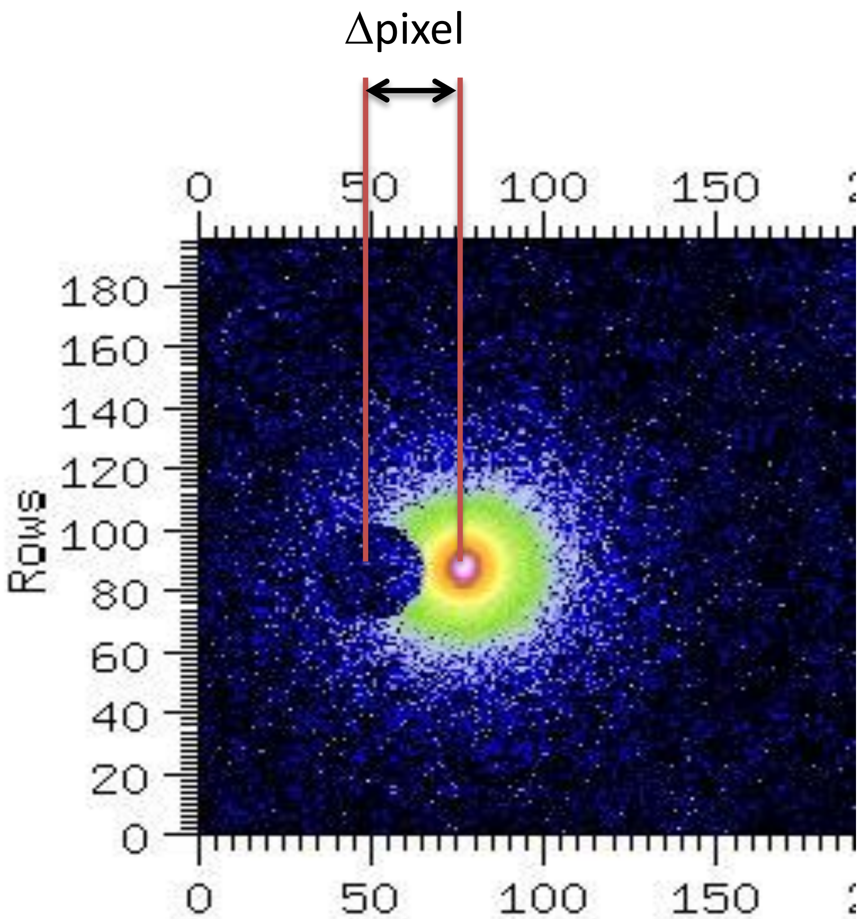


- Image of the 5-hole powder sample holder.
- Arrow marks position of a sample in the holder.

- 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 that empty holder, as expected.

SAXS beamstop alignment

SAXS



Pixel size: 172 μm

Motor shift = Δpixel x 0.172 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 {}

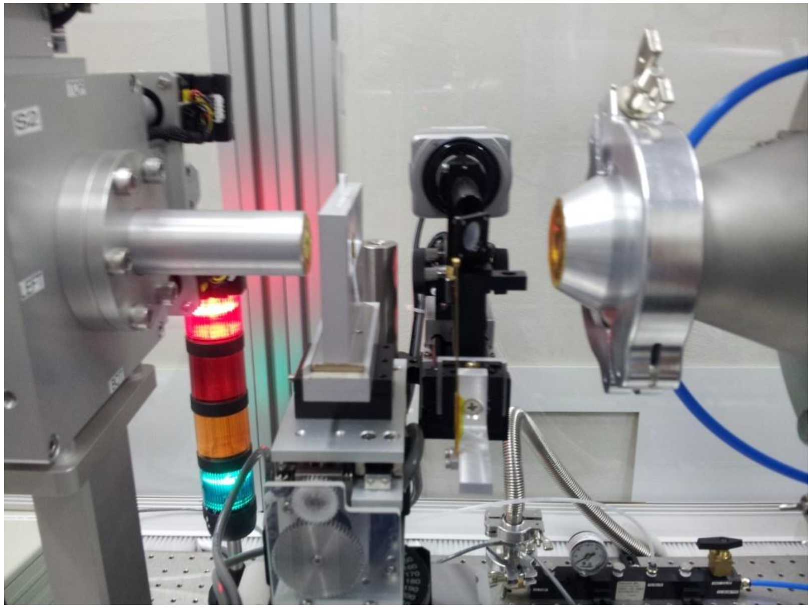
```
{
EDF_DataBlockID = 1.Image.Psd ;
EDF_BinarySize = 1206272 ;
EDF_HeaderSize = 8192 ;
ByteOrder      = LowByteFirst ;
DataType       = SignedInteger ;
Dim_1          = 487 ;
Dim_2          = 619 ;
title          = virtual_detector_001_0_00009.edf ;
Intensity1 = 1 ;
ExposureTime = 60 ;
Dummy = -10 ;
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 -i1dis 1.04322 -i1wvl
1.5411e-10 -i1cen 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



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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

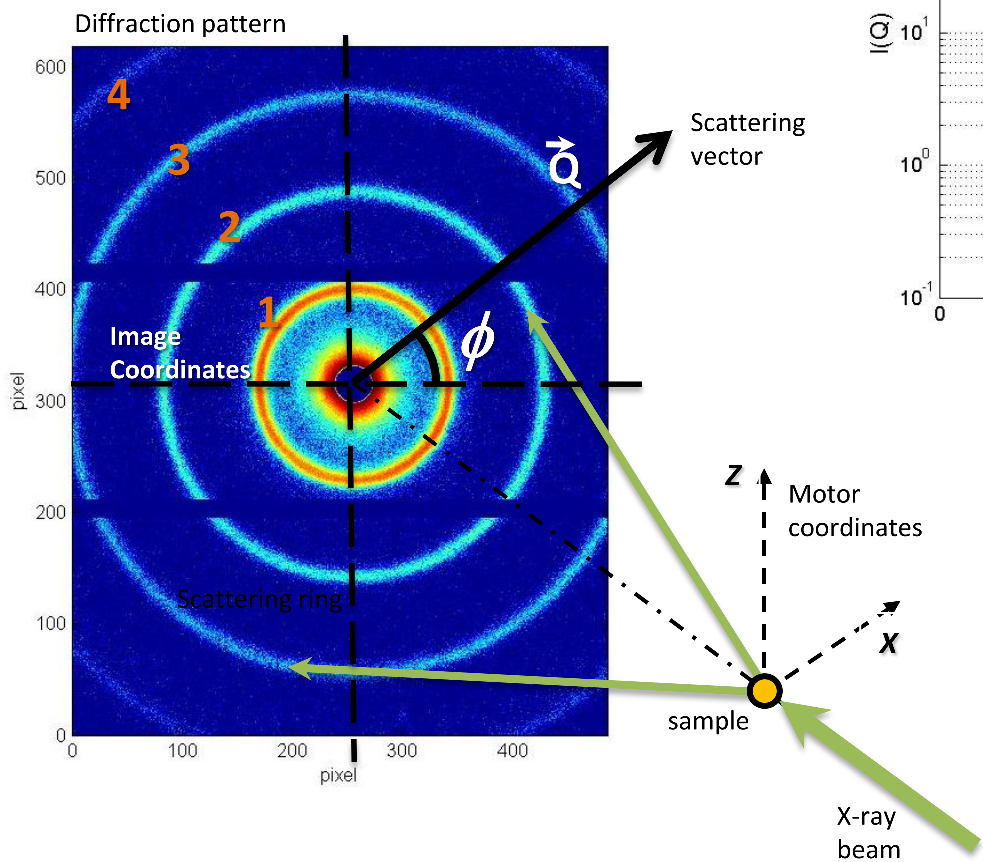
4) Normalize to exposure time and transmission

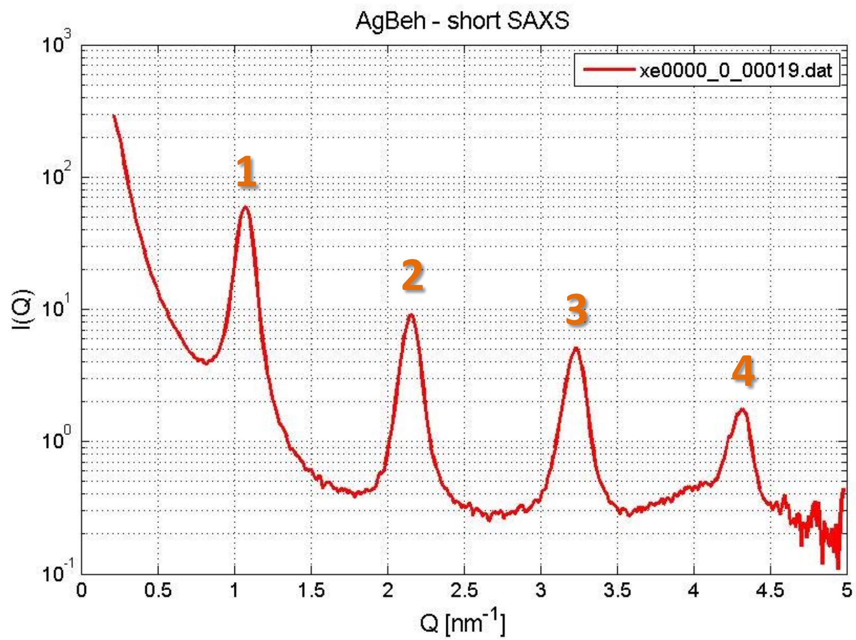
5) Subtract the empties

6) Save the data for analyze

Angular Integration

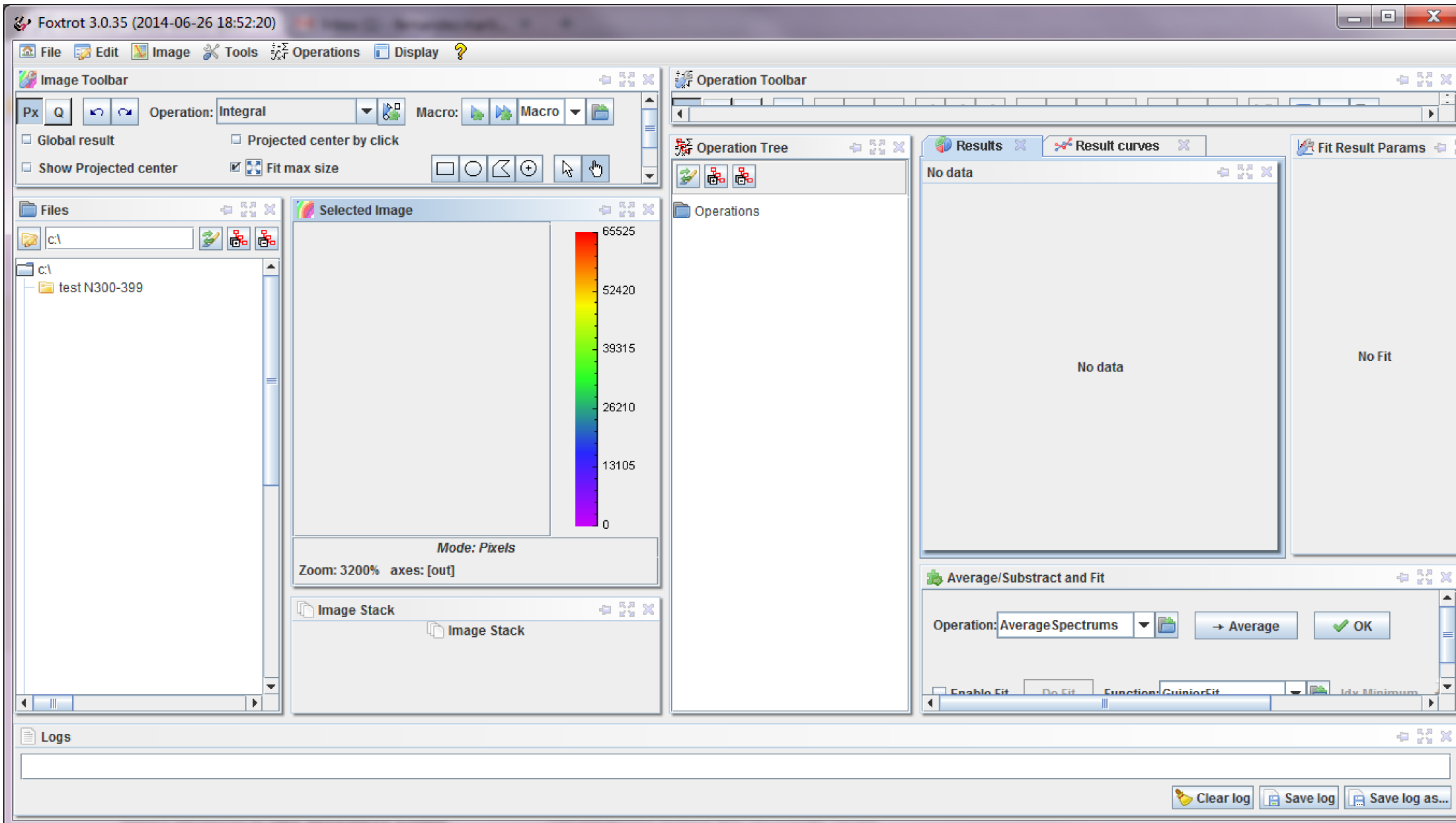
2D images into 1D curves





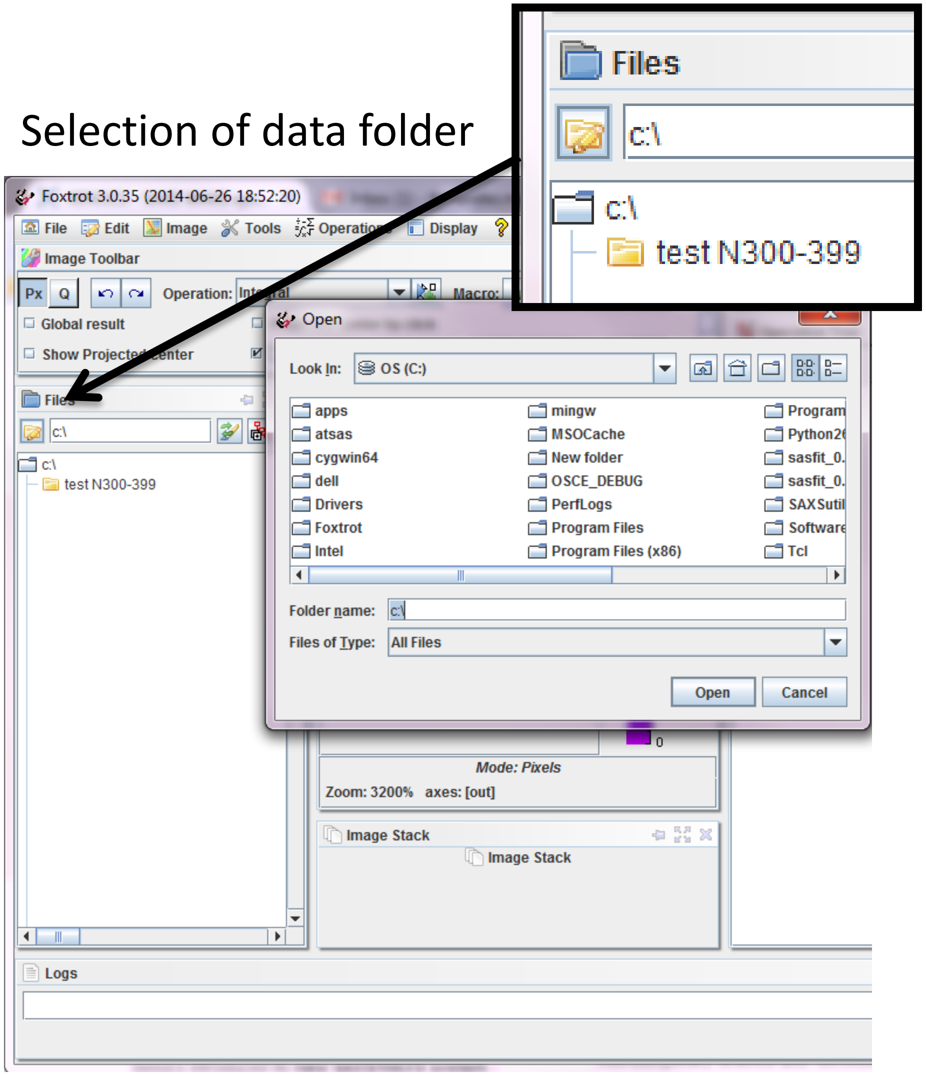
Quick guide to FOXTROT Data Reduction

Foxtrot environment

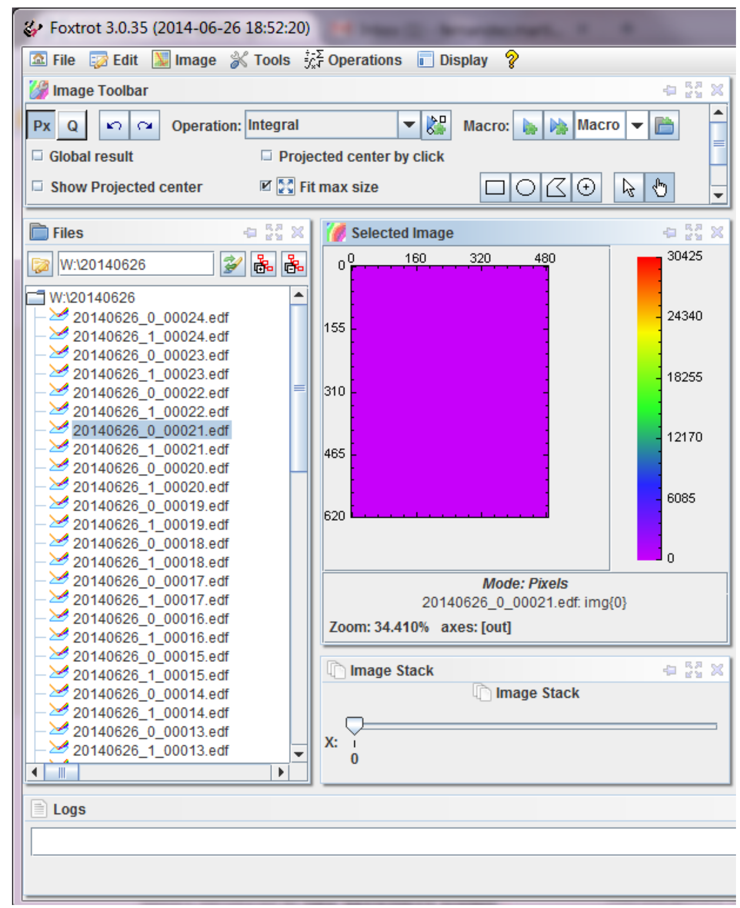


Quick guide to FOXTROT Data Reduction

Selection of data folder

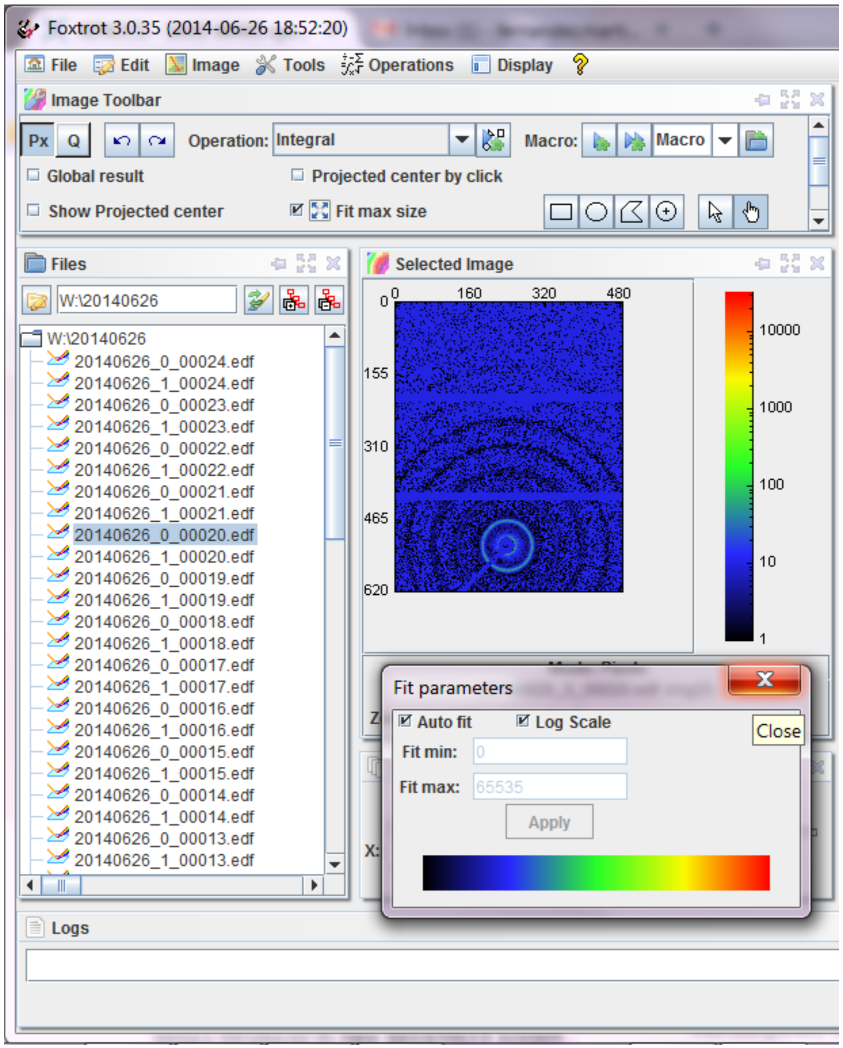


Selection of file / two clicks

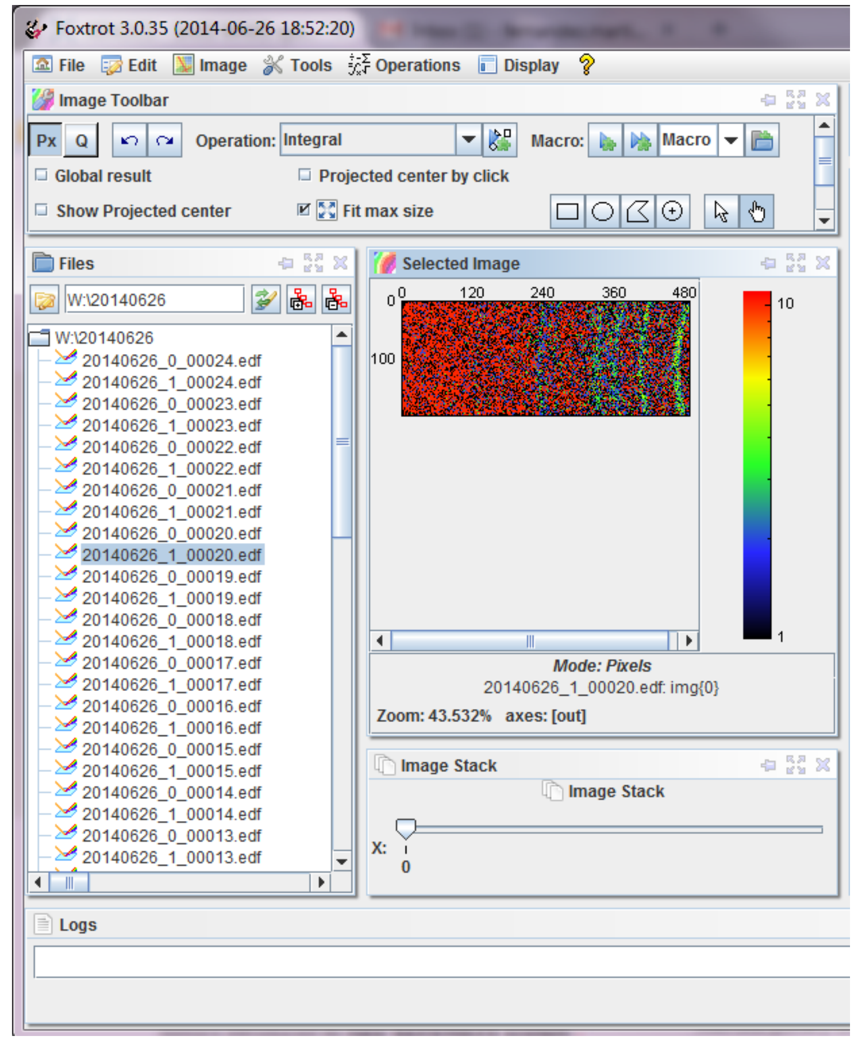


Quick guide to FOXTROT Data Reduction

Changing scale of image



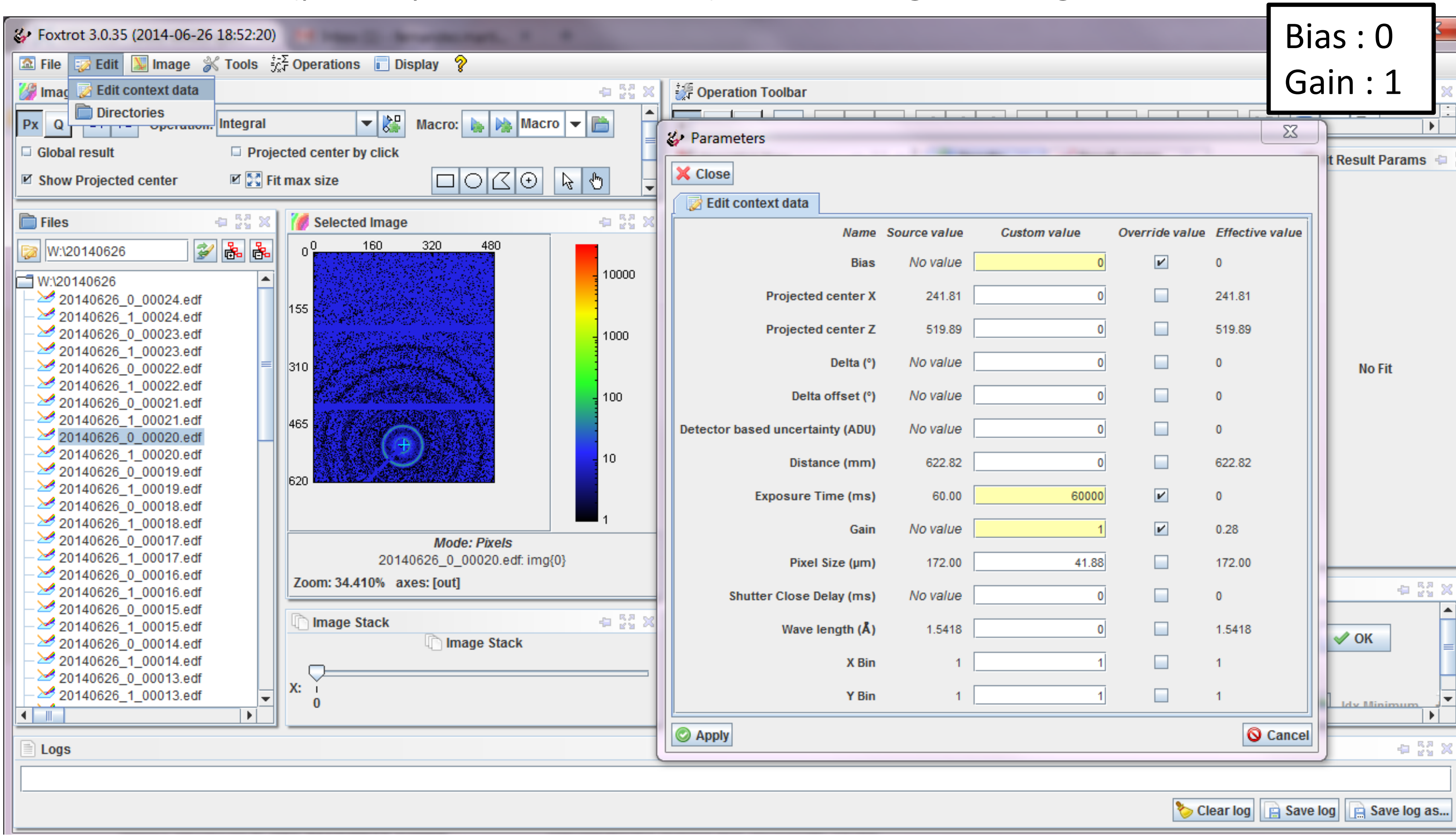
Opening other file (different size)



Quick guide to FOXTROT Data Reduction

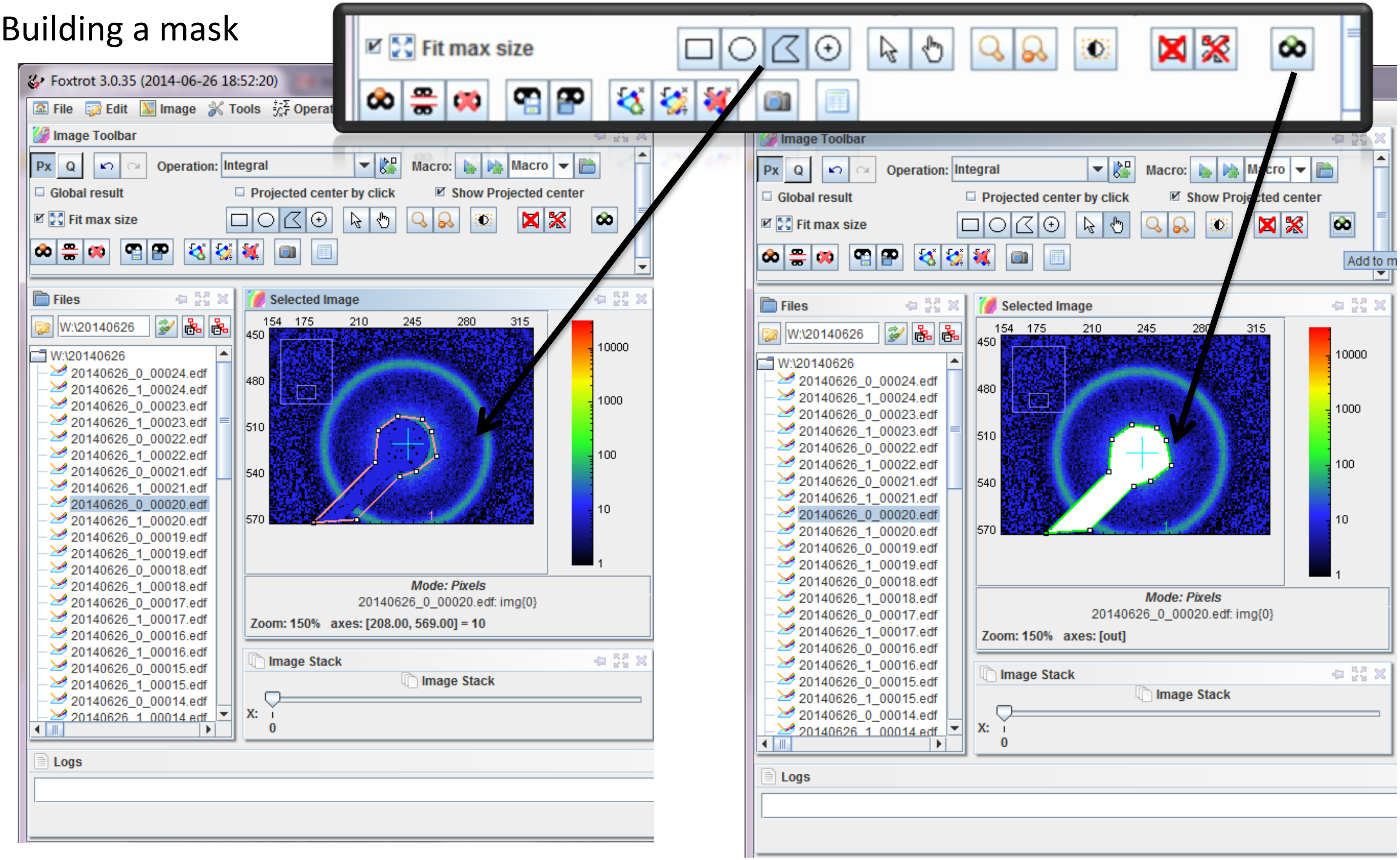
Edit context data (partially read from header) used for Angular Integration

Bias : 0
Gain : 1



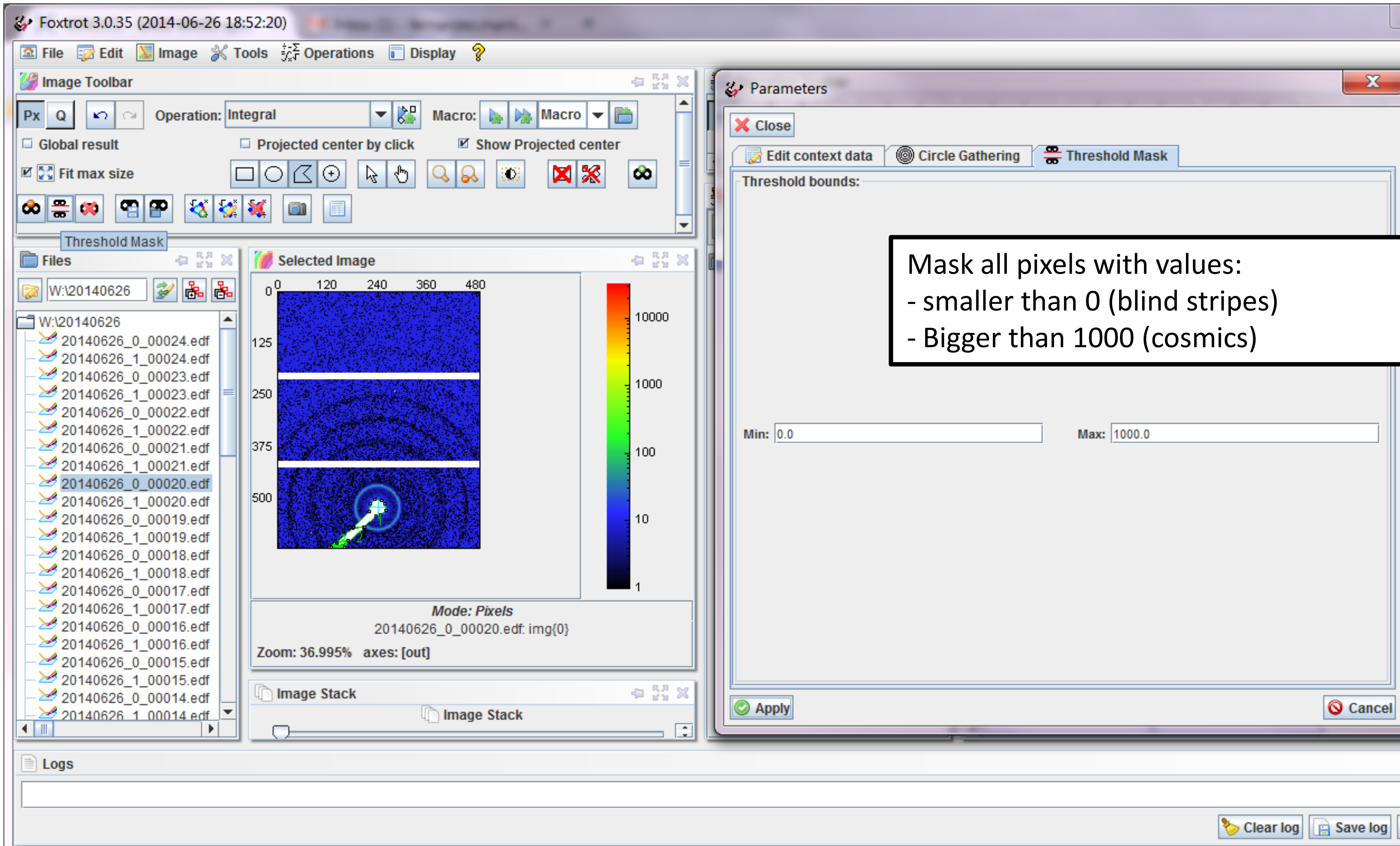
Quick guide to FOXTROT Data Reduction

Building a mask



Quick guide to FOXTROT Data Reduction

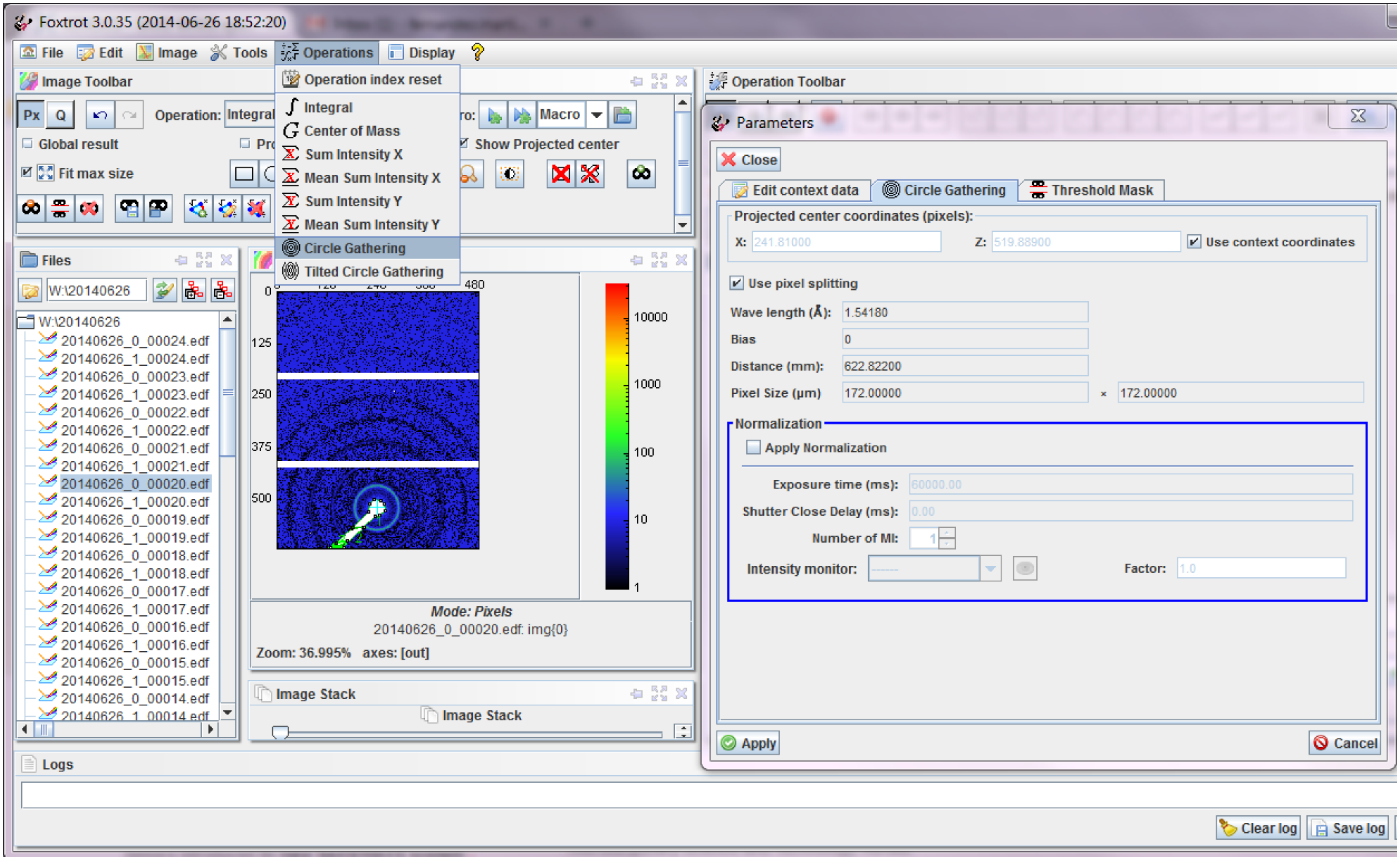
Building a mask / Threshold mask



Mask all pixels with values:
- smaller than 0 (blind stripes)
- Bigger than 1000 (cosmics)

Quick guide to FOXTROT Data Reduction

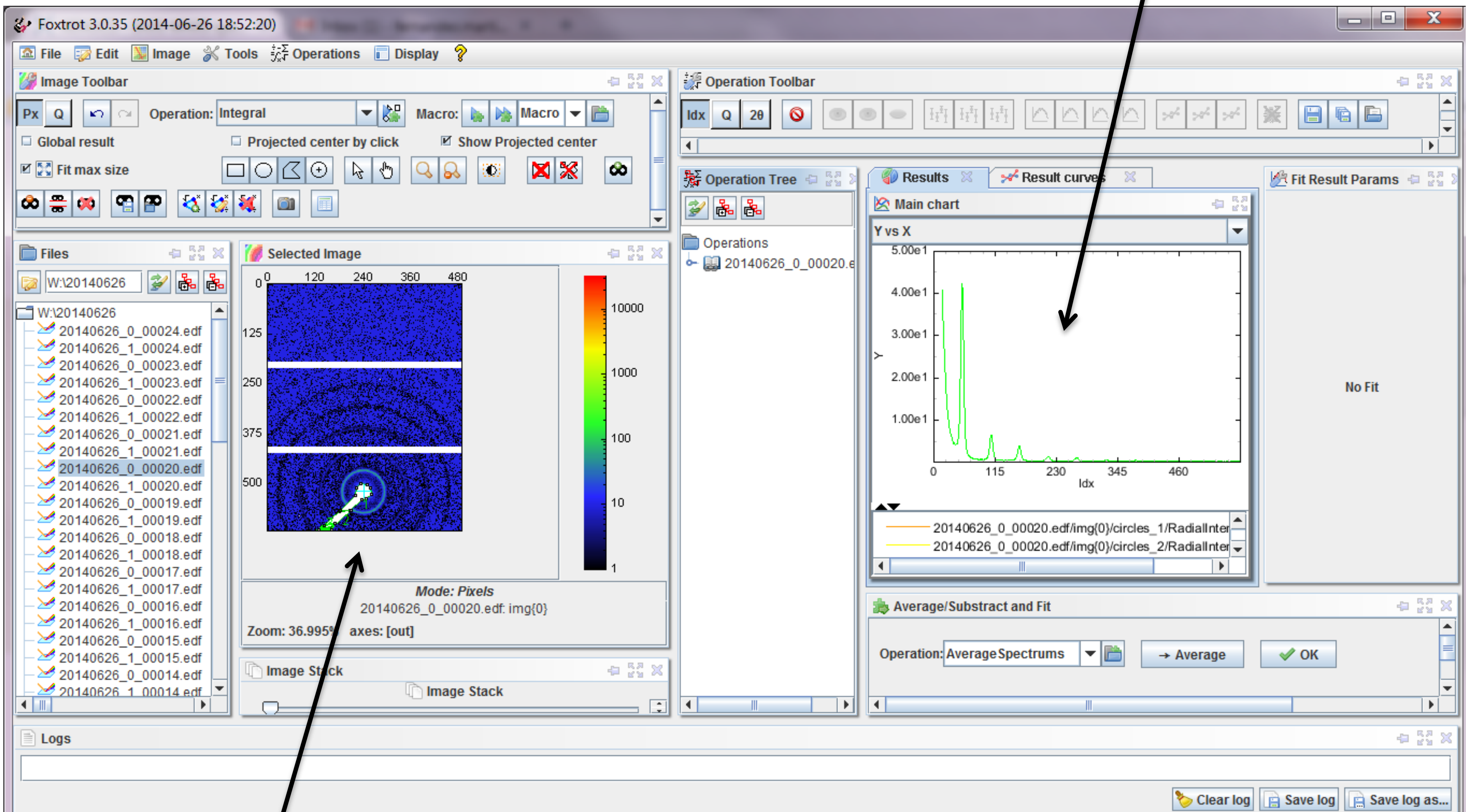
Angular Integration/ Circle Gathering



Quick guide to FOXTROT Data Reduction

Angular Integration/ Circle Gathering

1-dimensional result of angular integration

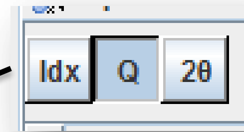


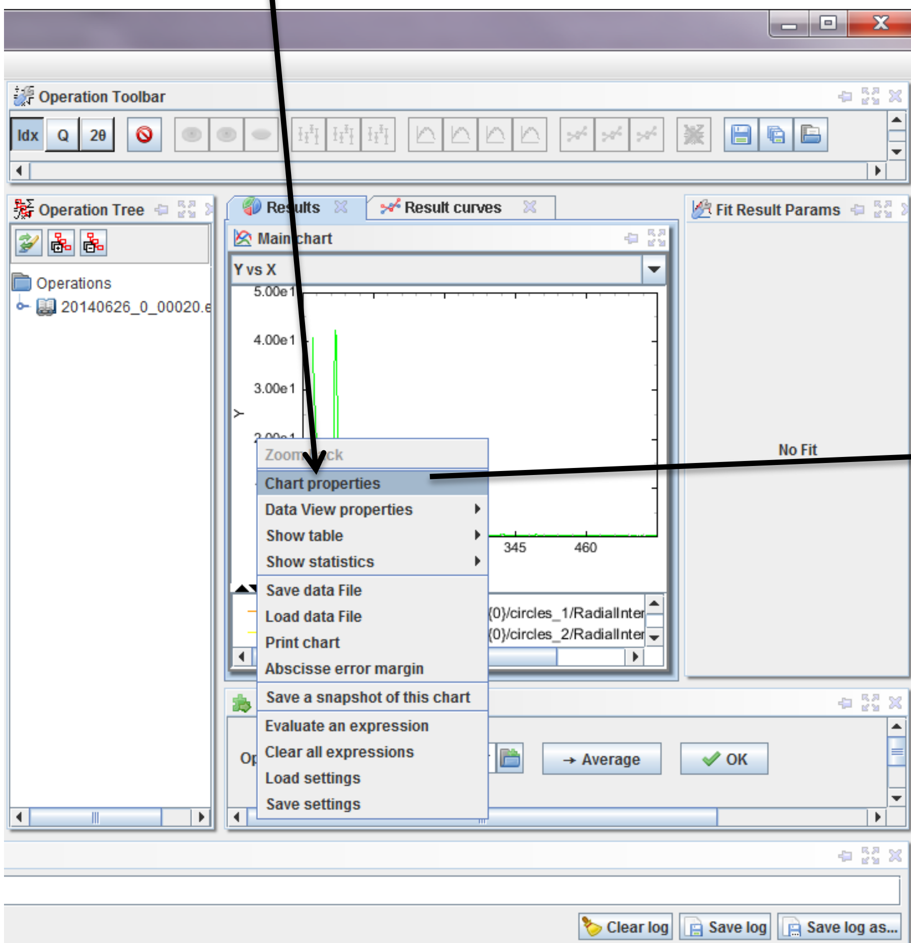
2-dimensional image + mask

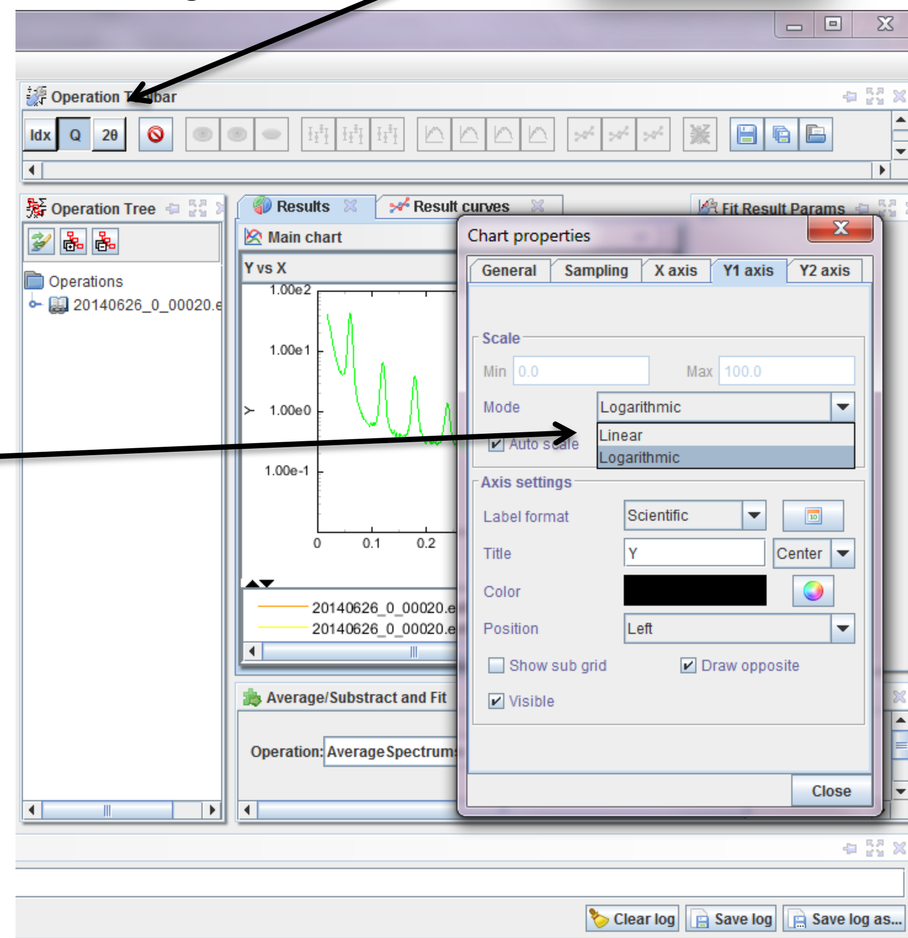
Quick guide to FOXTROT Data Reduction

Chart Properties / Click on axis

- Changing scale of axis
- Selecting Q-vector

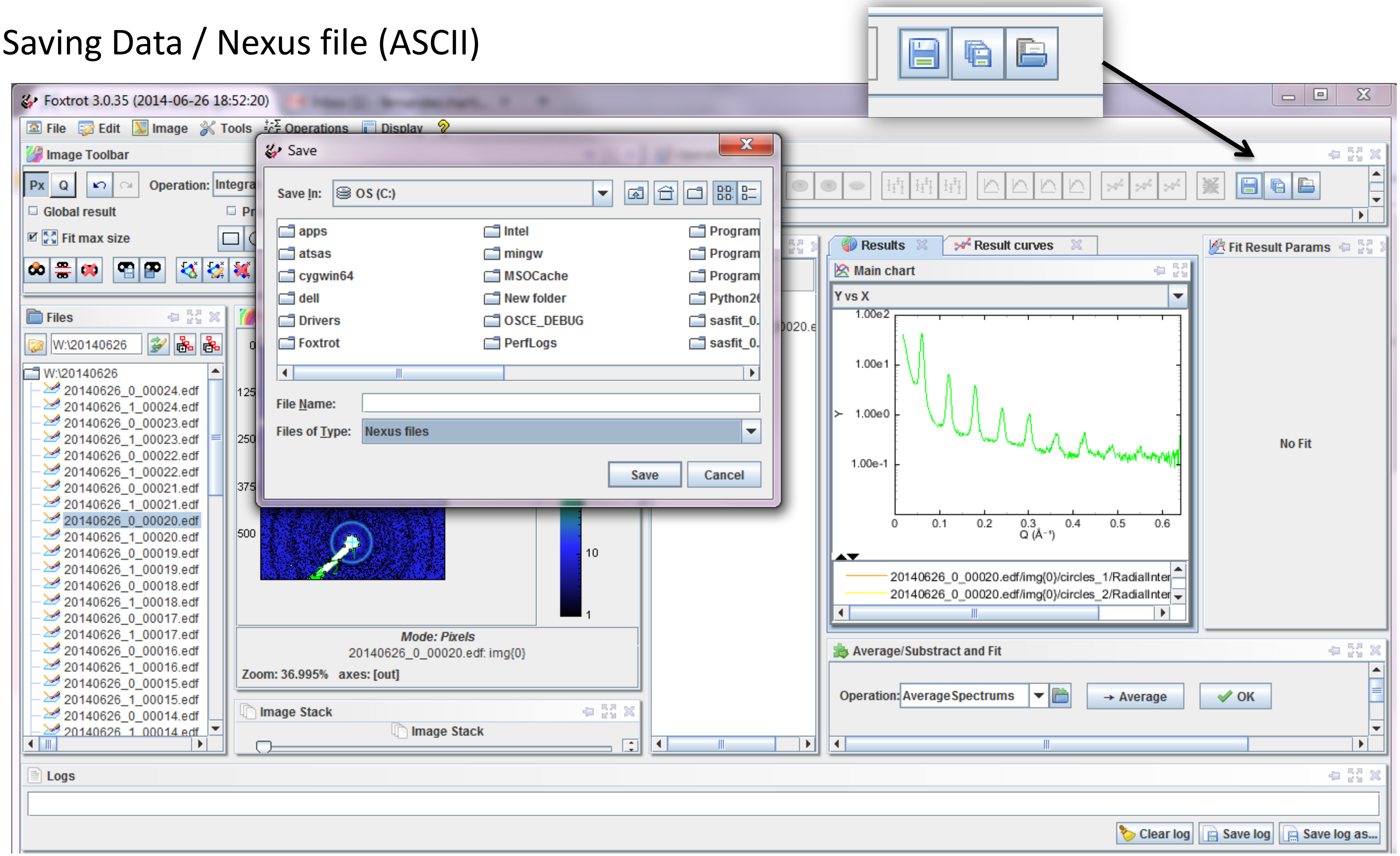






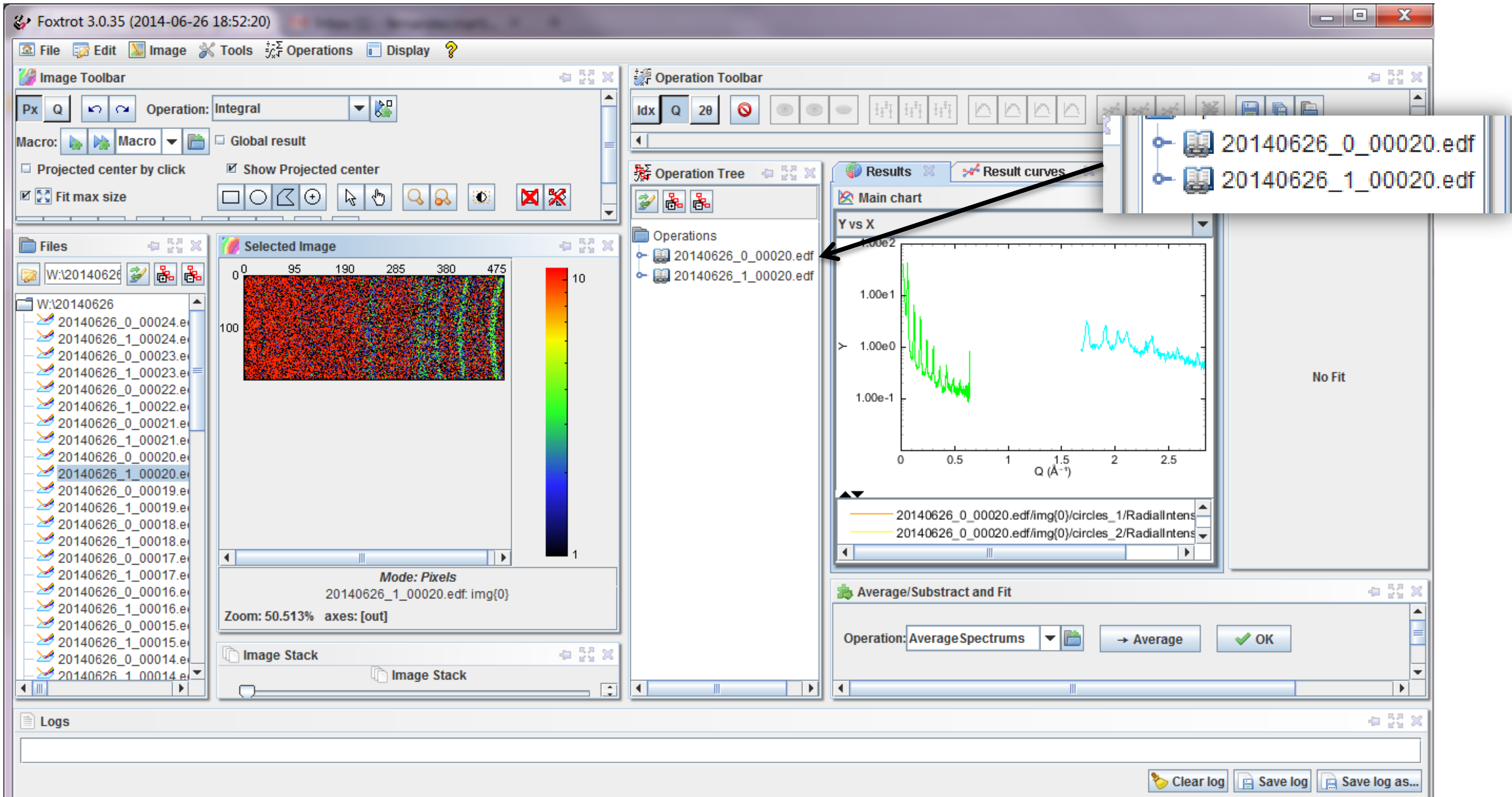
Quick guide to FOXTROT Data Reduction

Saving Data / Nexus file (ASCII)



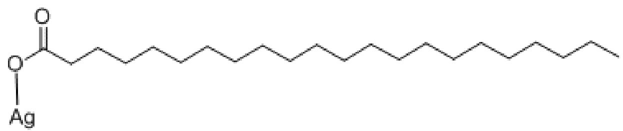
Quick guide to FOXTROT Data Reduction

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.

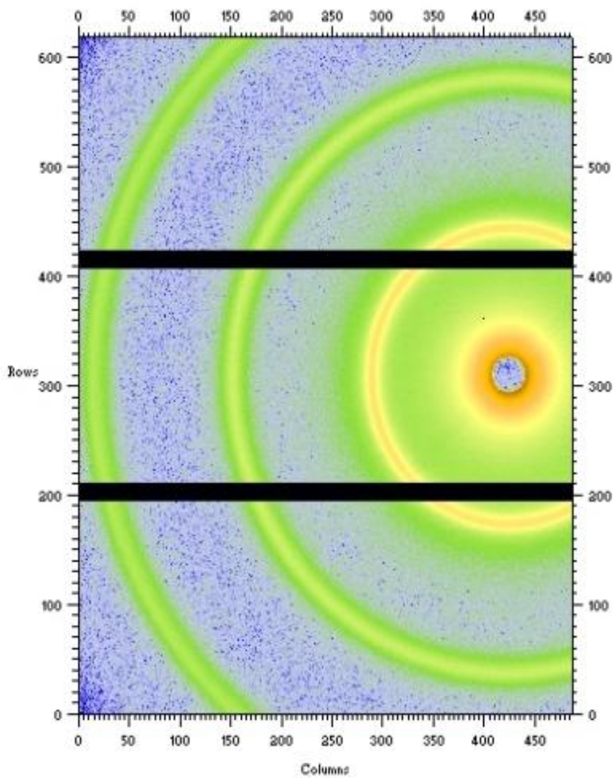


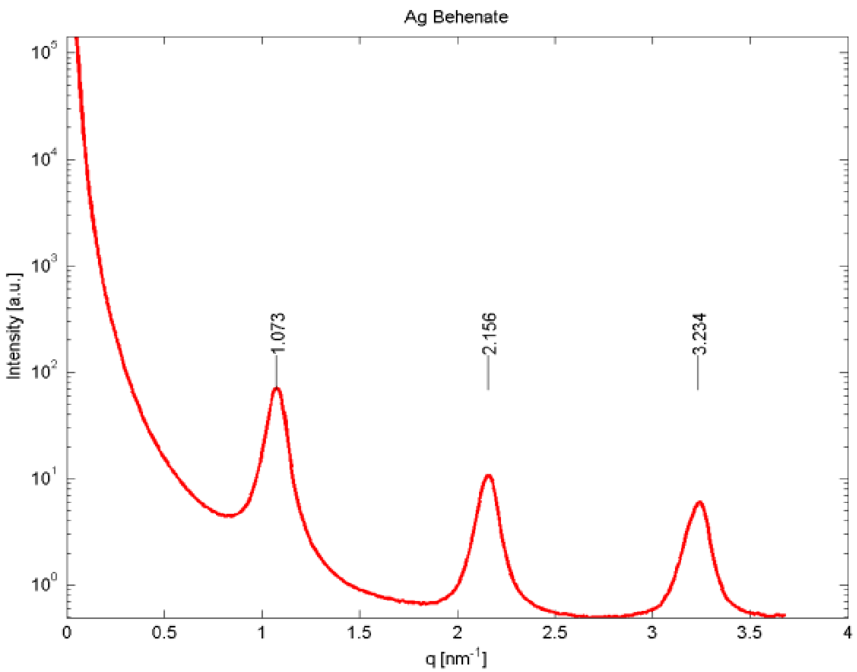
Calibration of Q-vector and distance calculation

Silver Behenate (AgBeh)



Lamellar structure with :
d_{001} = 58.7 Å
Q_{001} = 1.07 nm^{-1}





Distance Calculation

SPEC function (by Xenocs):

SAXS>**dist_calc** d_ q_ qo

$$d = \frac{d\_ \tan\left[2\sin^{-1}\left(\lambda q\_ / 4\pi\right)\right]}{\tan\left[2\sin^{-1}\left(\lambda q_0 / 4\pi\right)\right]}$$

d = real distance

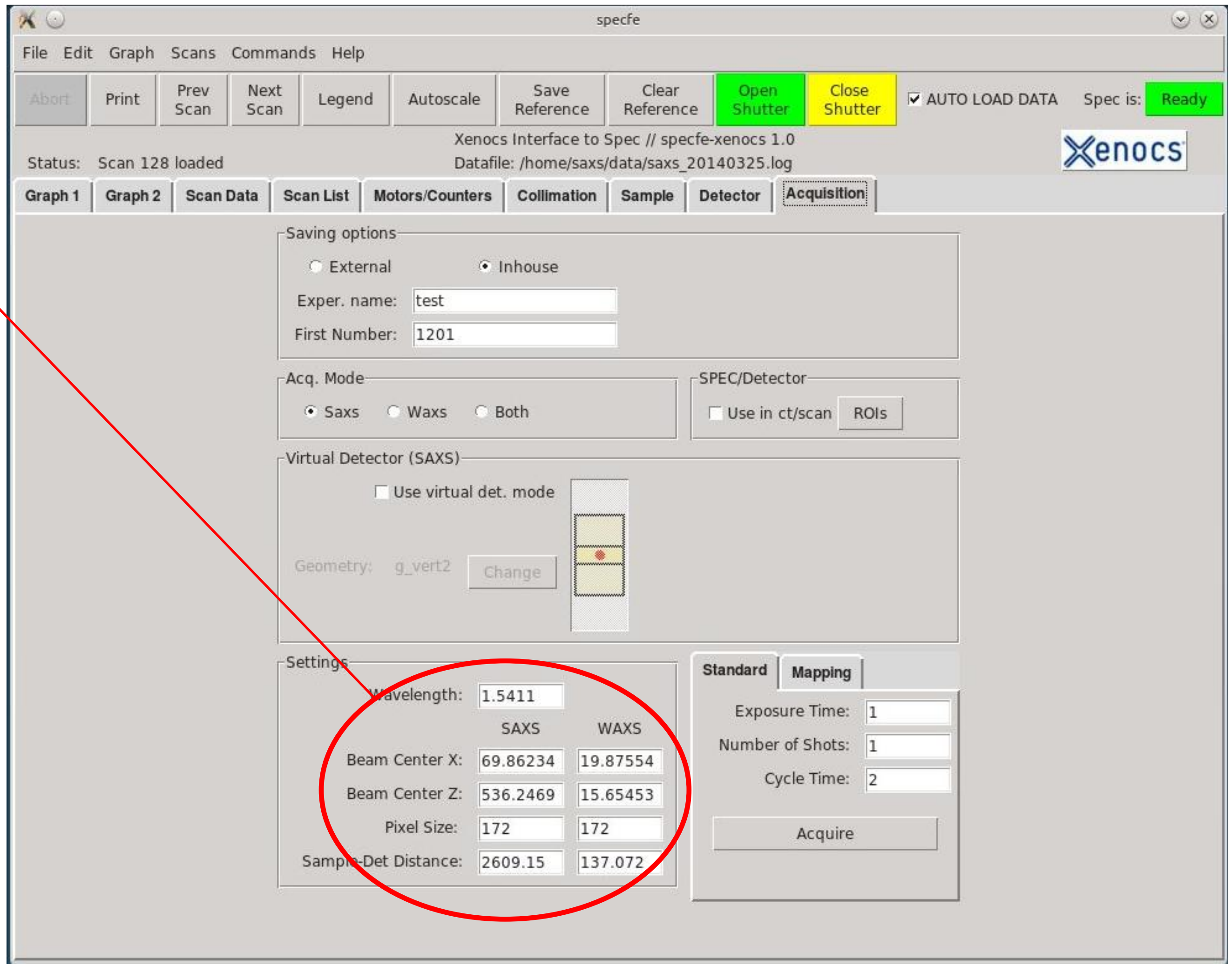
d_ = distance used to first integration

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

q_0 = q-value tabulated for the calibration peak
(AgBeh : q_o = 1.070 nm^{-1})

Experiment Info into the image header

Info for Heade r



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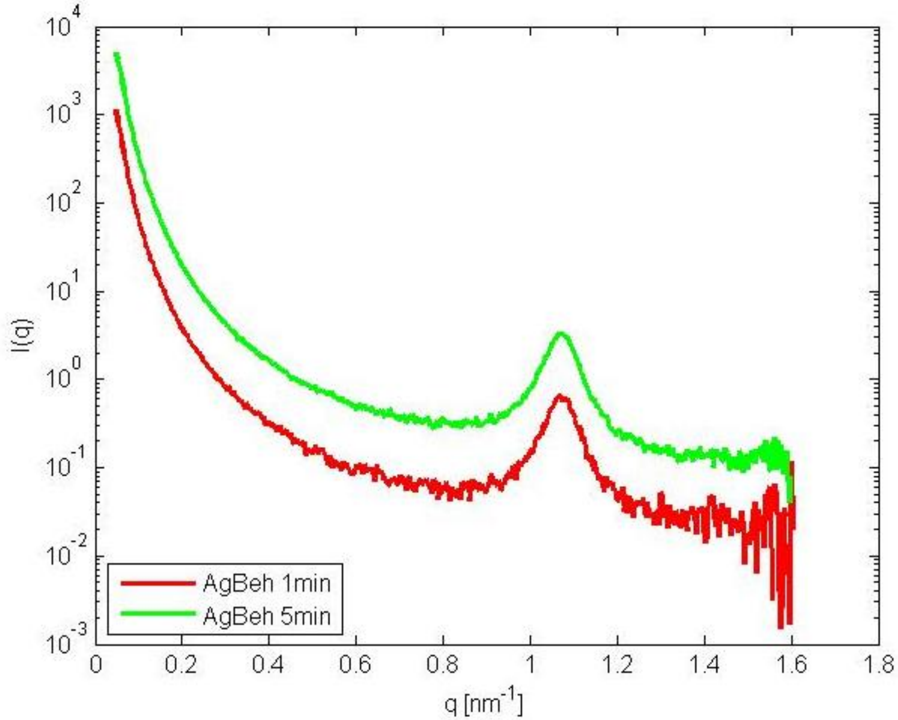
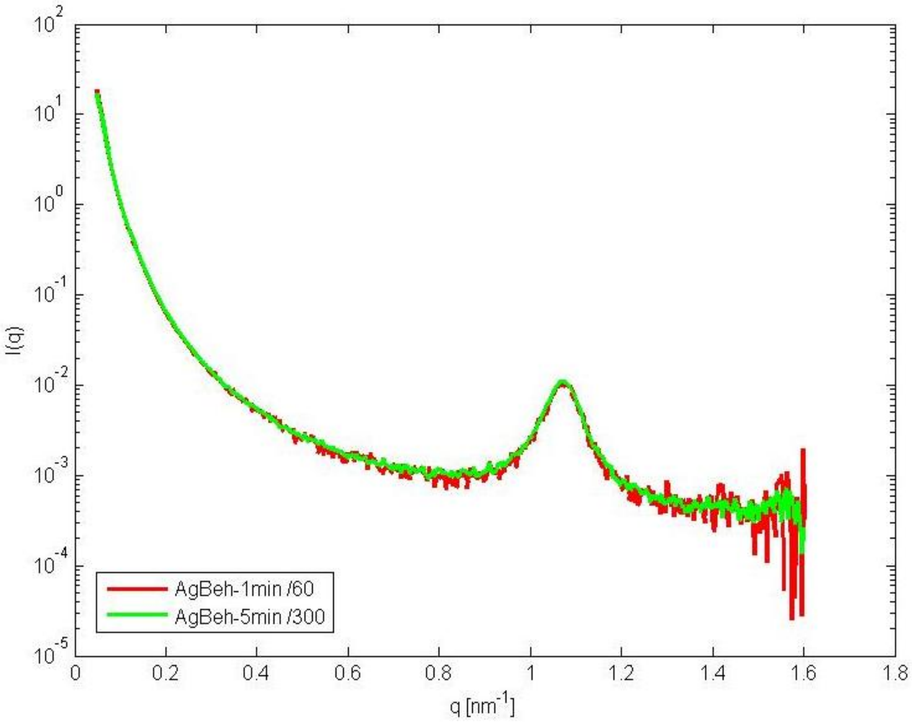
Data Normalization

Normalization to Exposure Time





Normalization to transmission

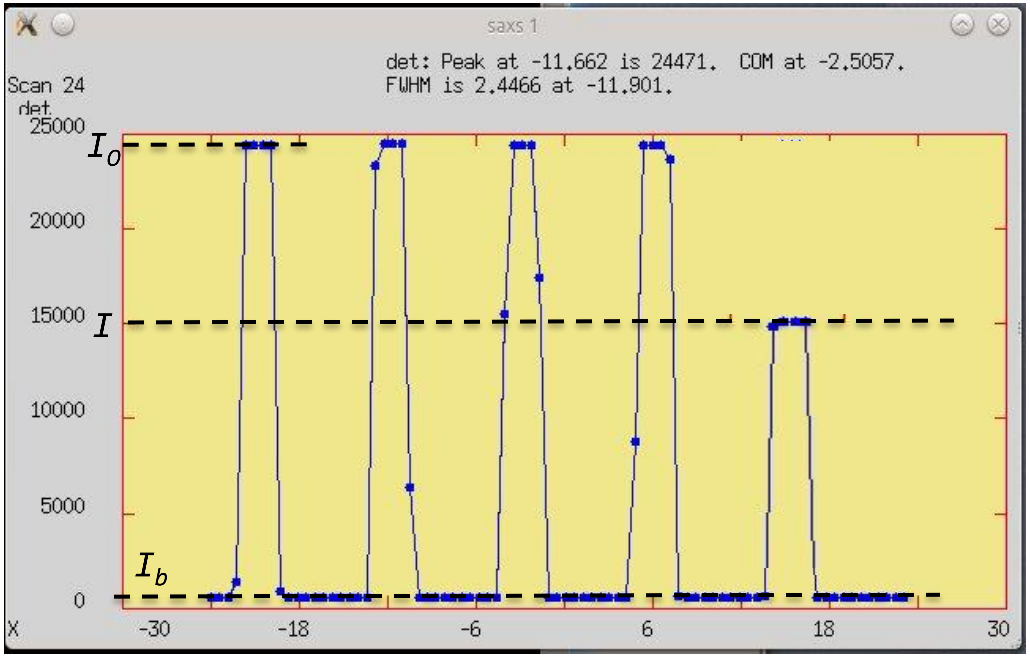
Use the values from a sample scan to get the transmission *T*,
ans then divide the scattering curves by the measured *T*

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

I_o = 24900
I = 15000
I_b = 400

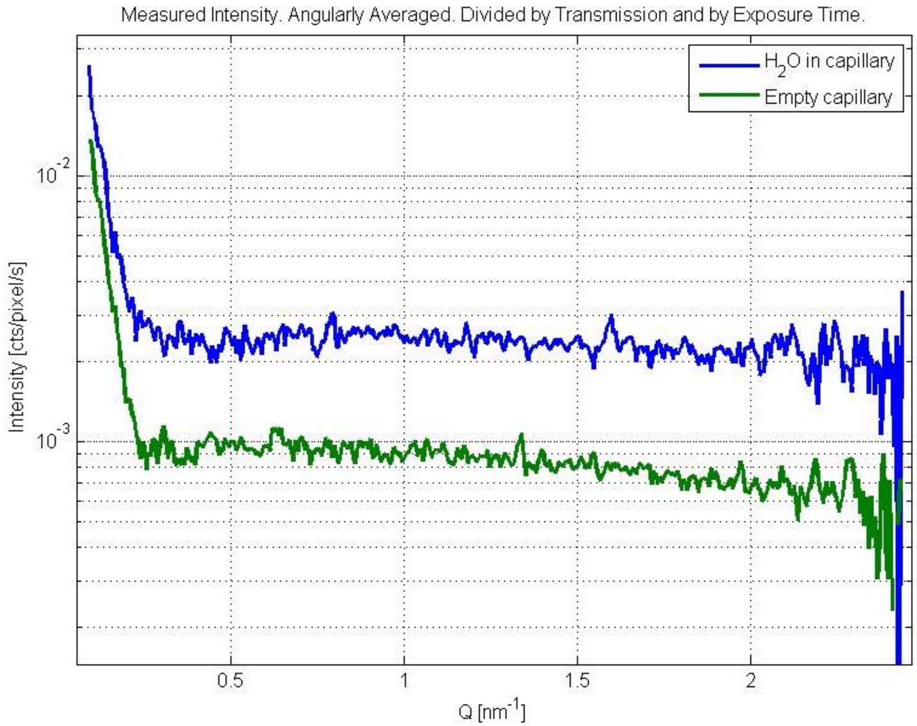
$T = (15000-400)/(24900-400)$

$T = 0.595918$ (~60%)



Absolute Intensity Calibration (with H_2O)

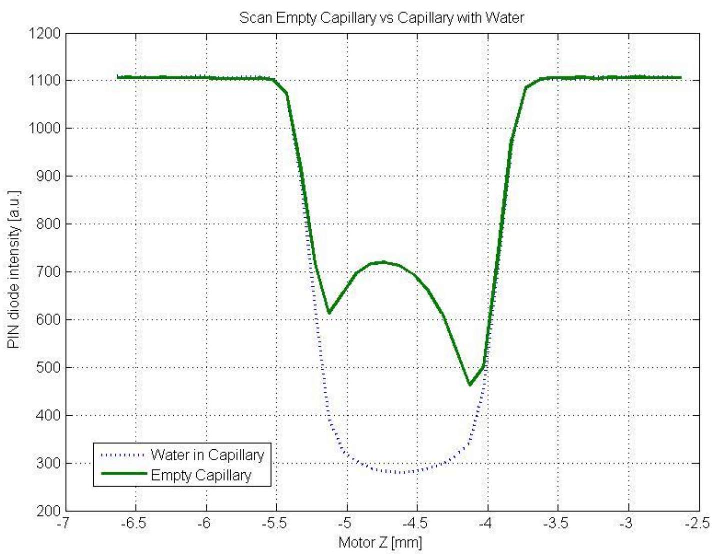
Measure scattering from empty capillary and capillary full of water



Data

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

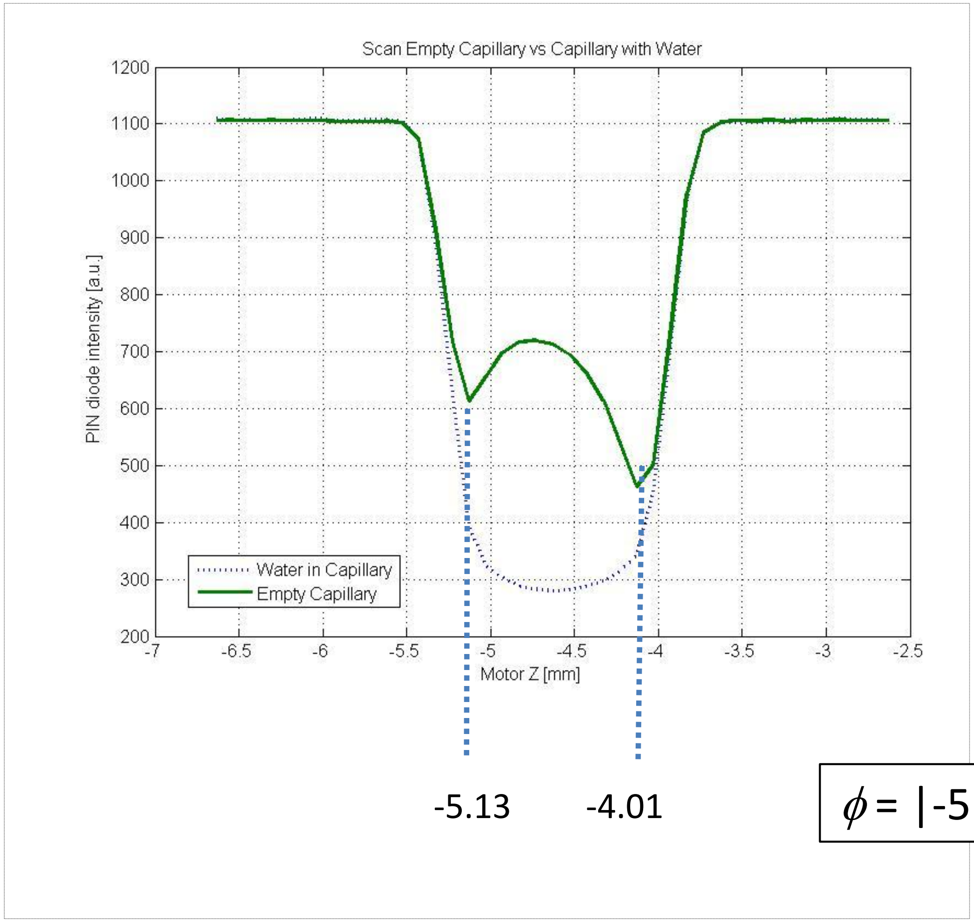
```
SAXS>dscan z -1.5 1.5 60 0.1
```



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

Absolute Intensity Calibration (with H_2O)

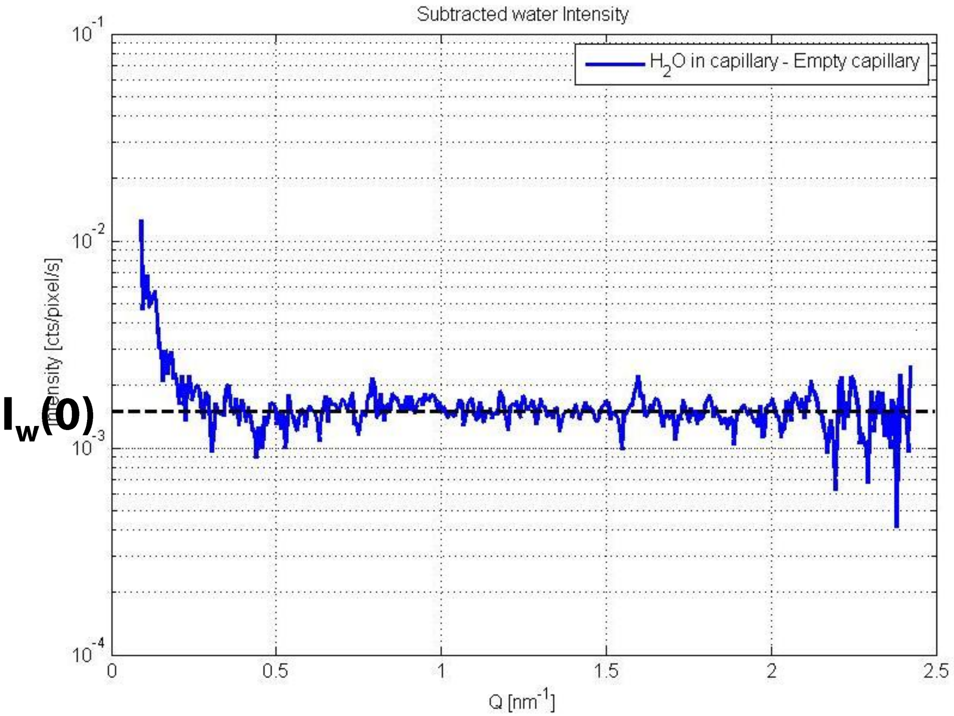
Measure of the diameter *f* of the capillary



ϕ = |-5.13| -| -4.01| = 1.12 mm

Absolute Intensity Calibration (with H_2O)

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

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

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

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

Correction factor

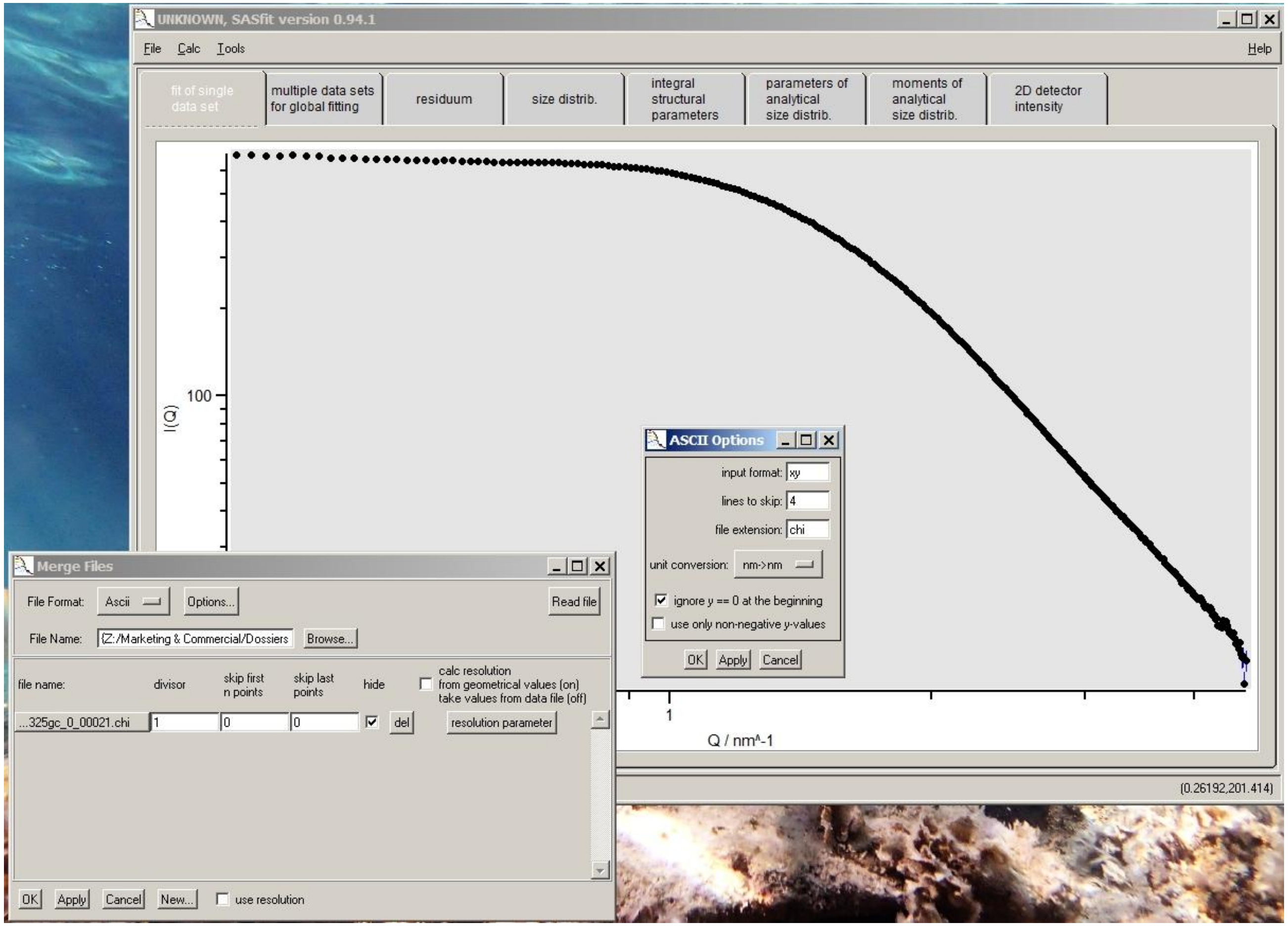
$\kappa = 1.66/1.79$

$\kappa = 0.927$

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)

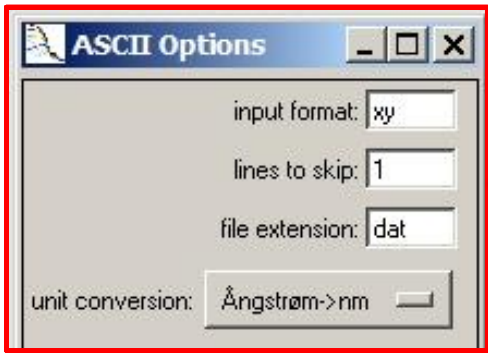
Load into SASfit (or other) the Glassy Carbon data from the calibration sample provided.

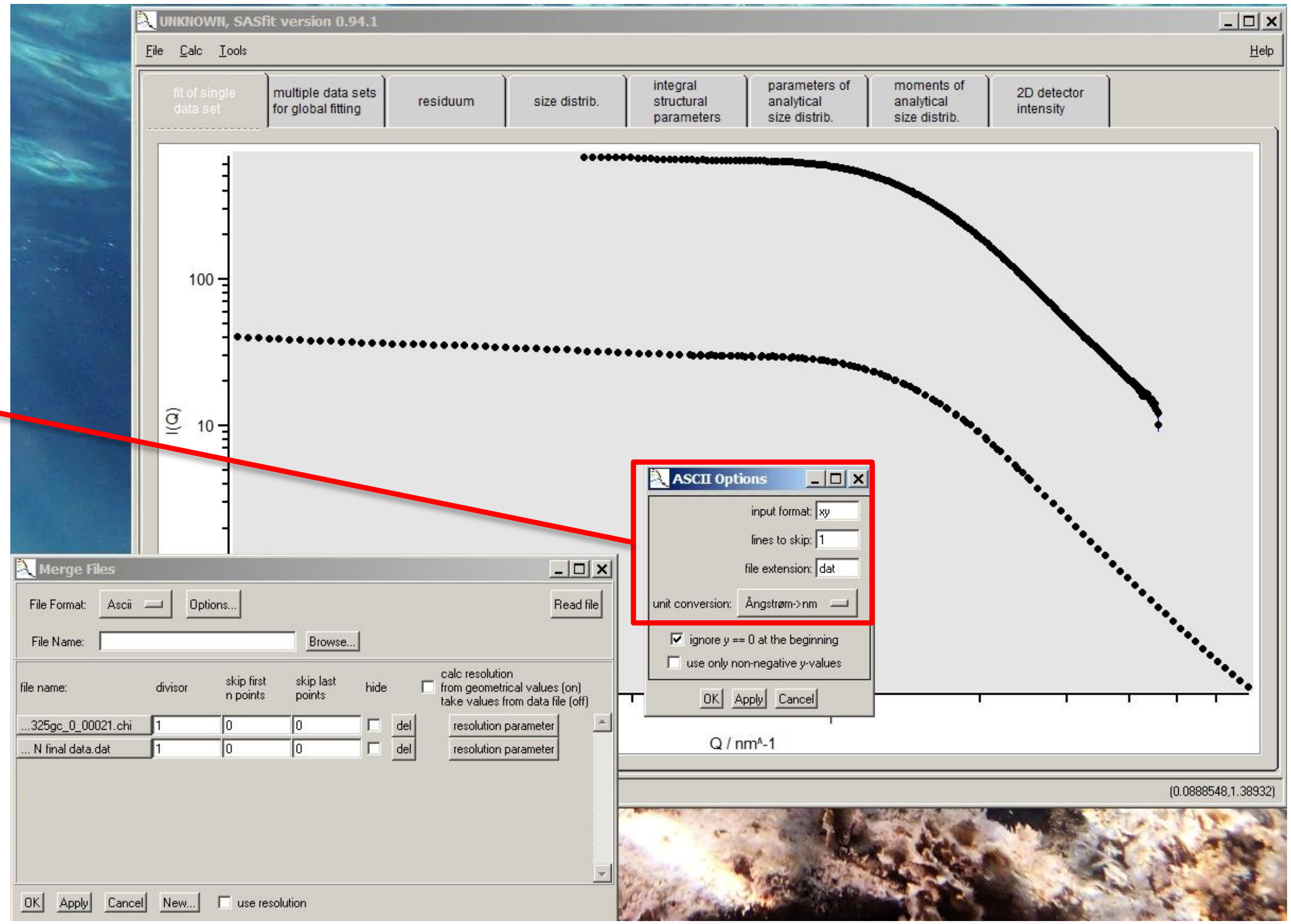


Absolute Intensity Calibration (with Glassy Carbon)

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





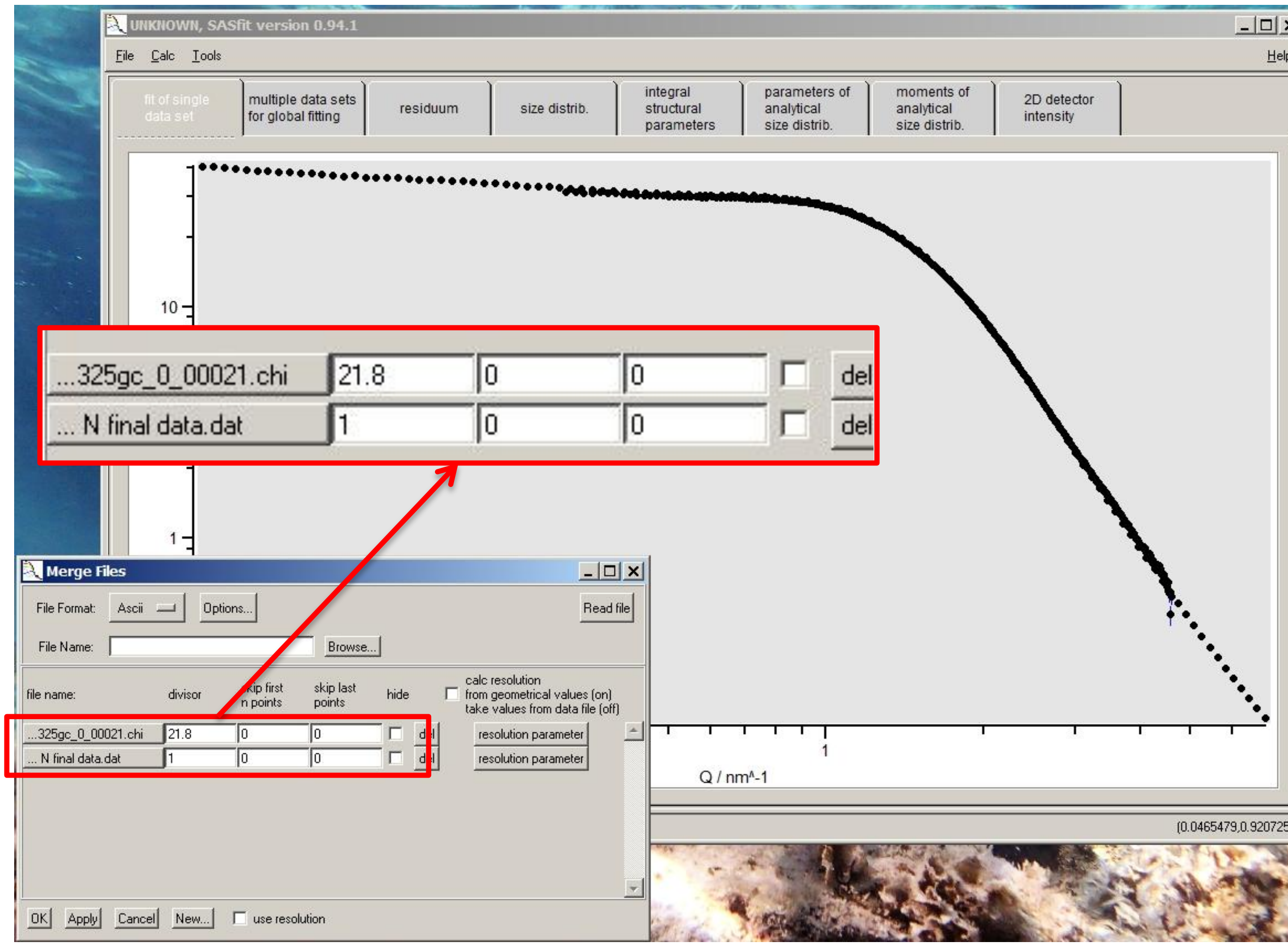
Absolute Intensity Calibration (with Glassy Carbon)

Calculate the factor to the 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.

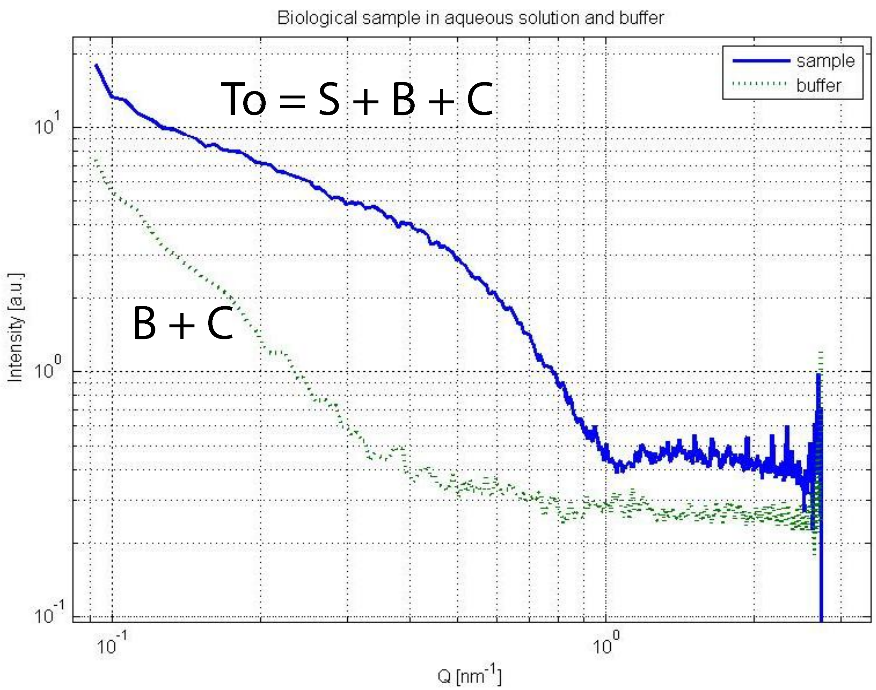
Unknown (and wanted) contribution to the total scattering coming from **the sample** may be extracted from the total scattering data by simple subtraction of the known contributions : camera and/or buffers or containers.

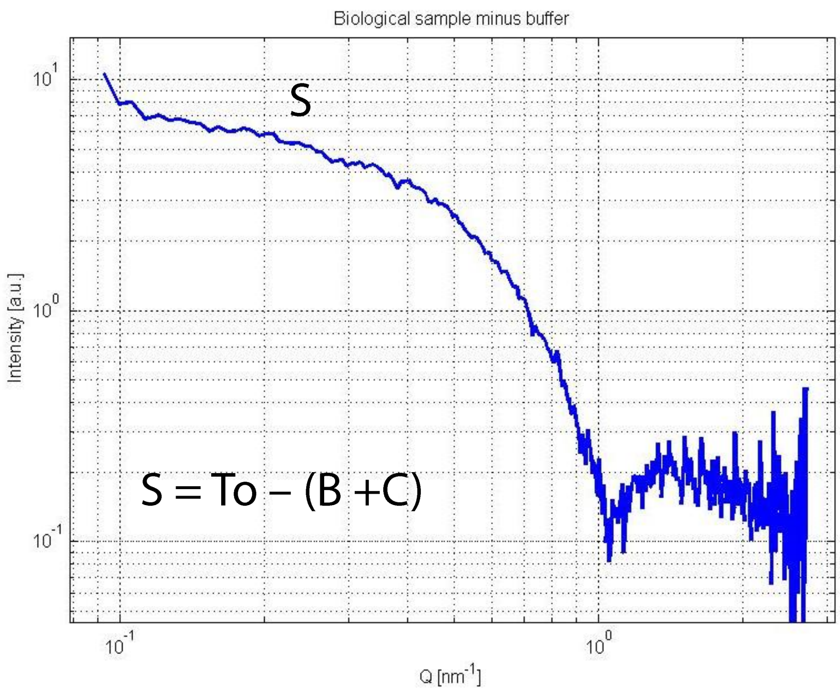
Subtraction can be done directly from the 2D images or from the 1D curves

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 preformed : data with sample minus data of buffer alone.



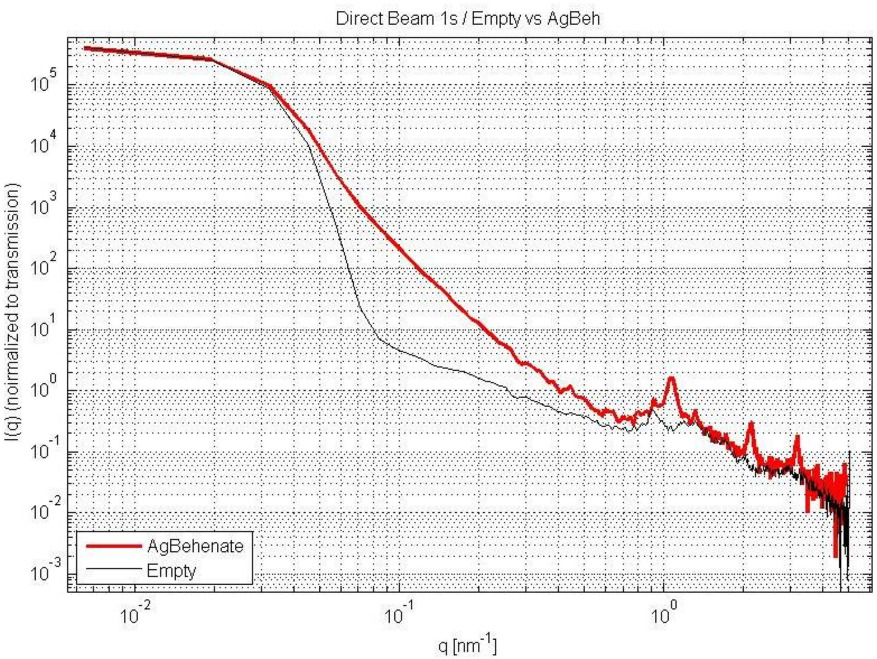


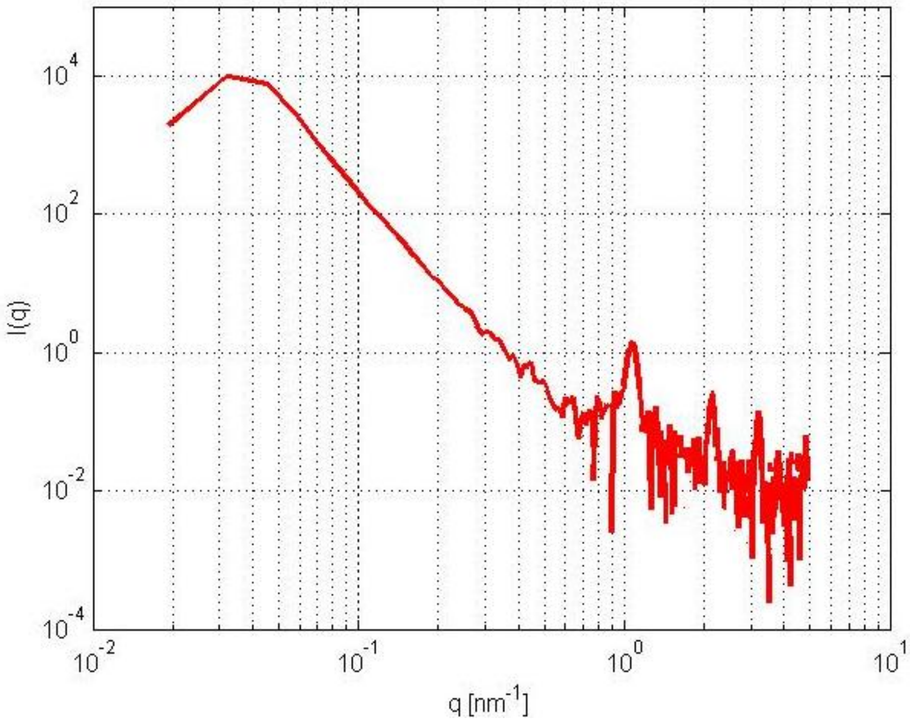
- Protein in its buffer (S+B)
- Buffer alone (B)

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



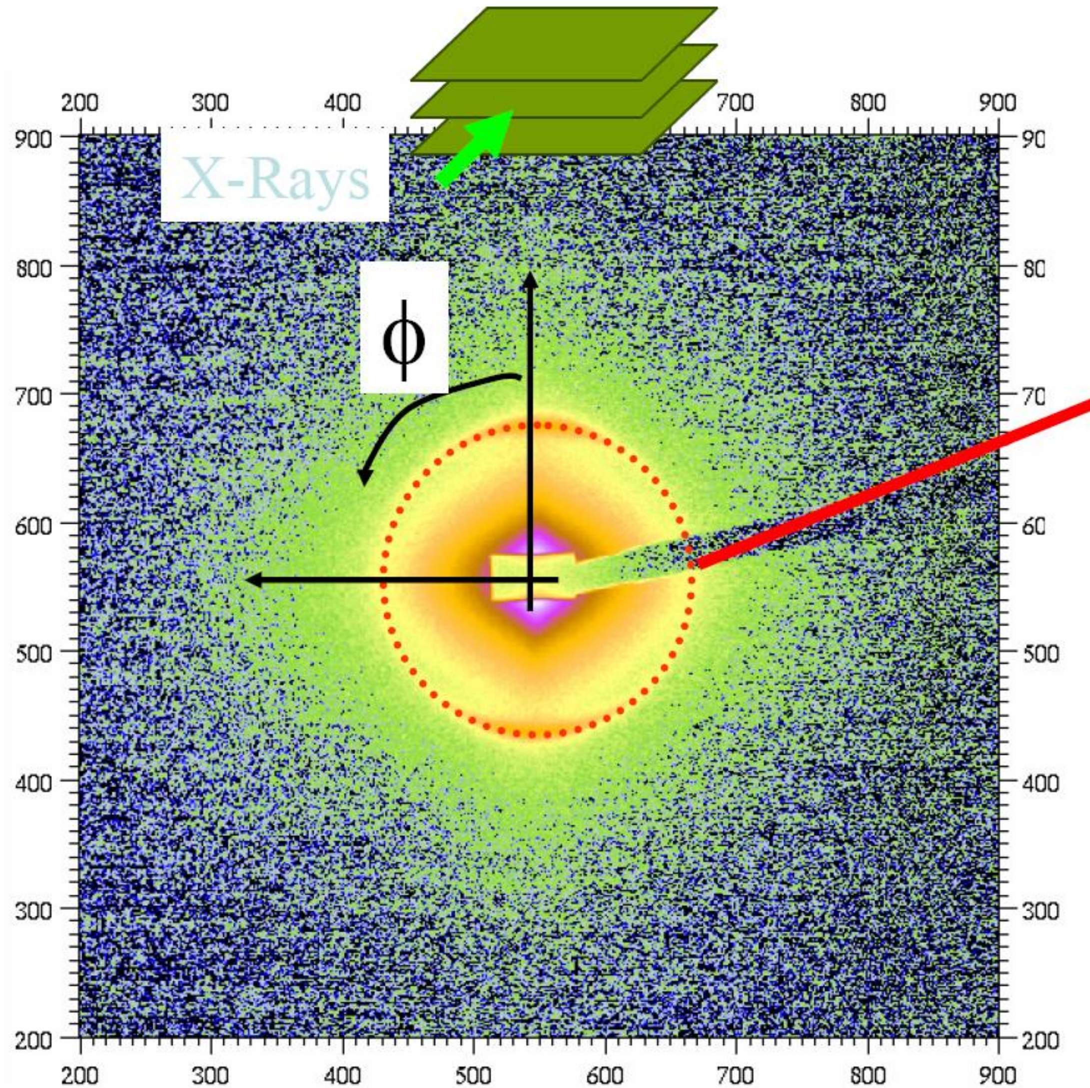


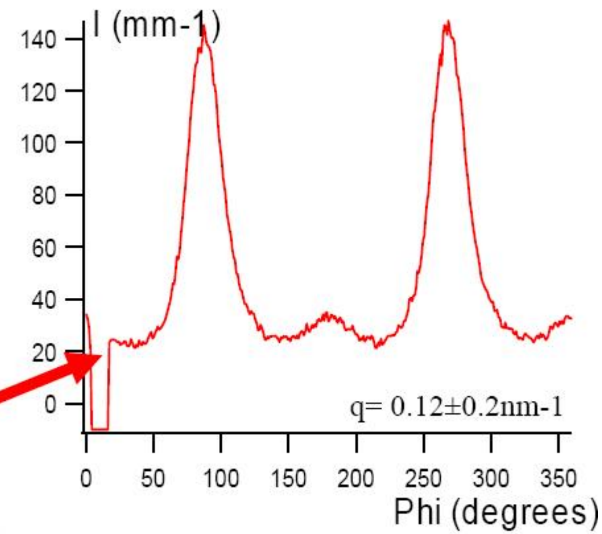
xenocs

Exploring the very small

Especial Cases / Aligment

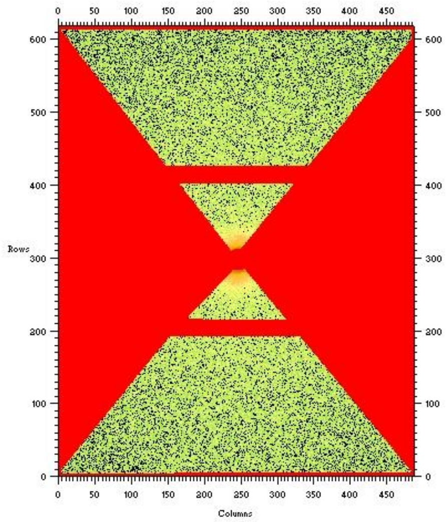
Anisotropy analysis

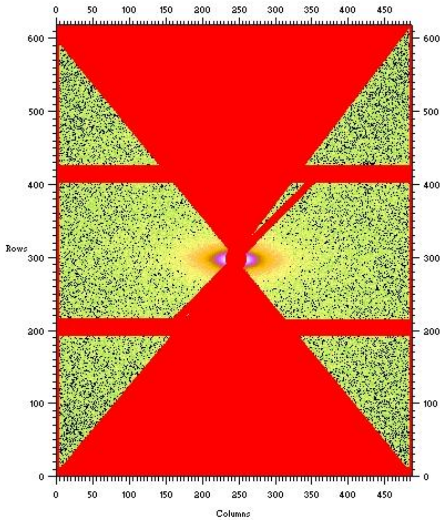


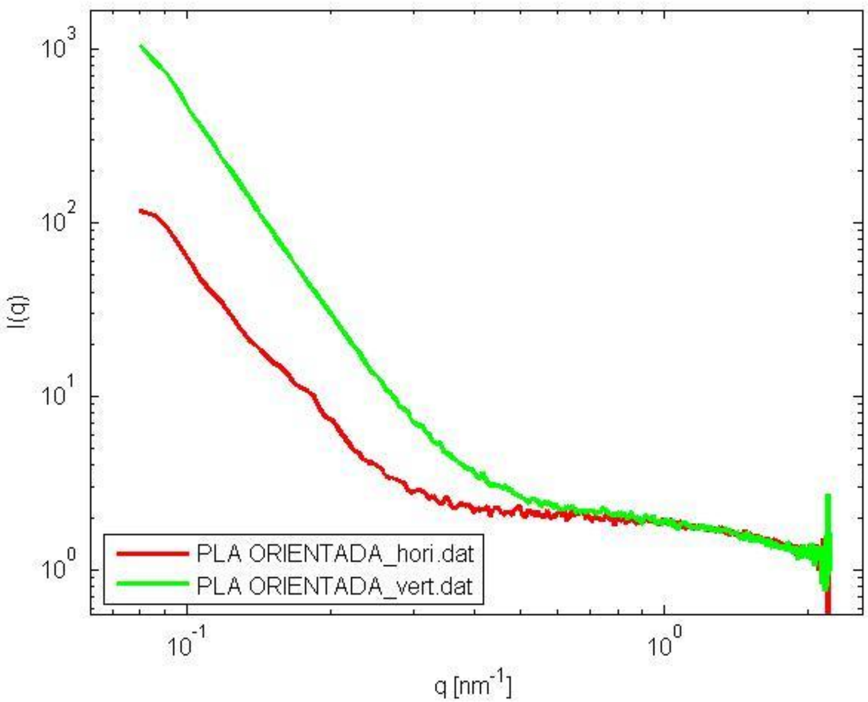


Anisotropy analysis from intensity plot at given angular position.

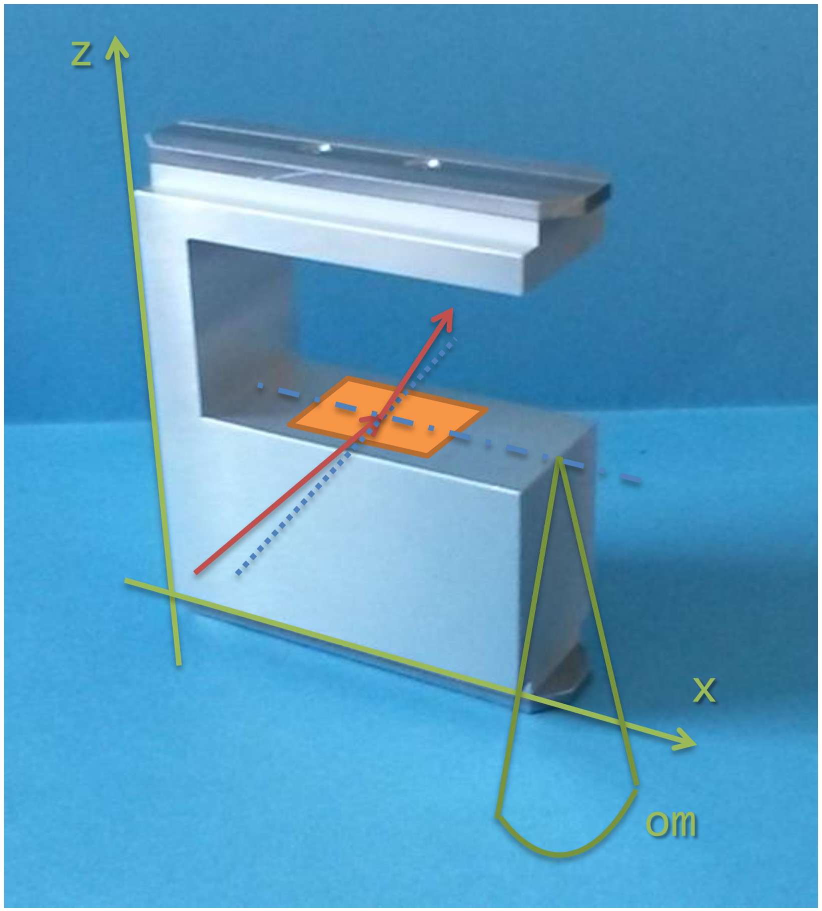
Anisotropy analysis

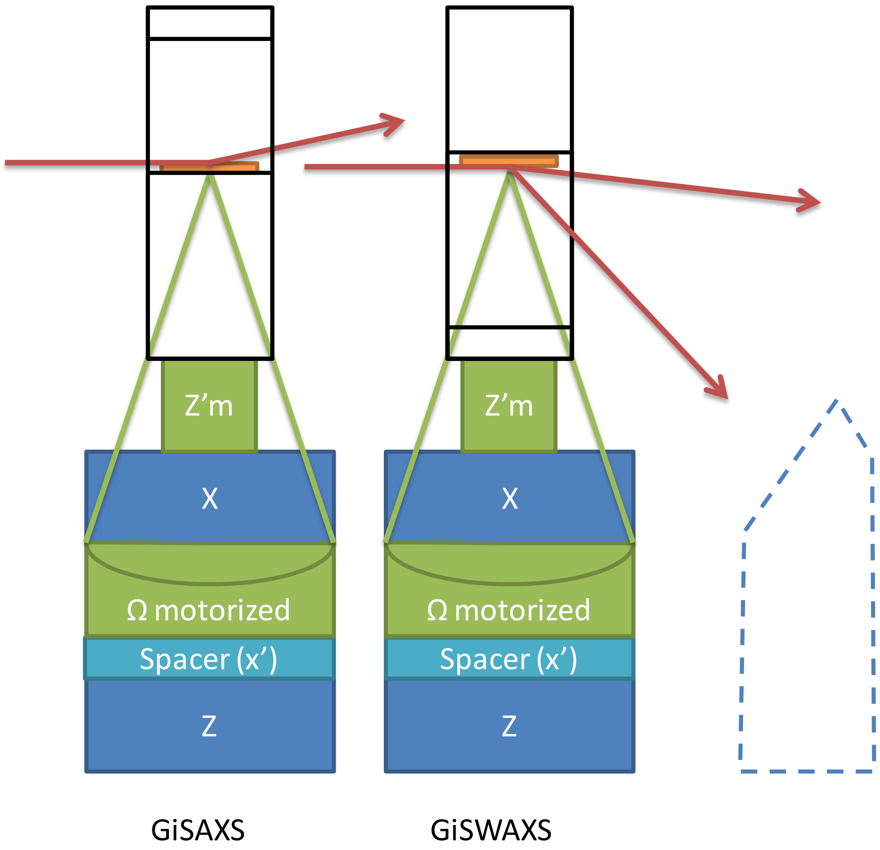






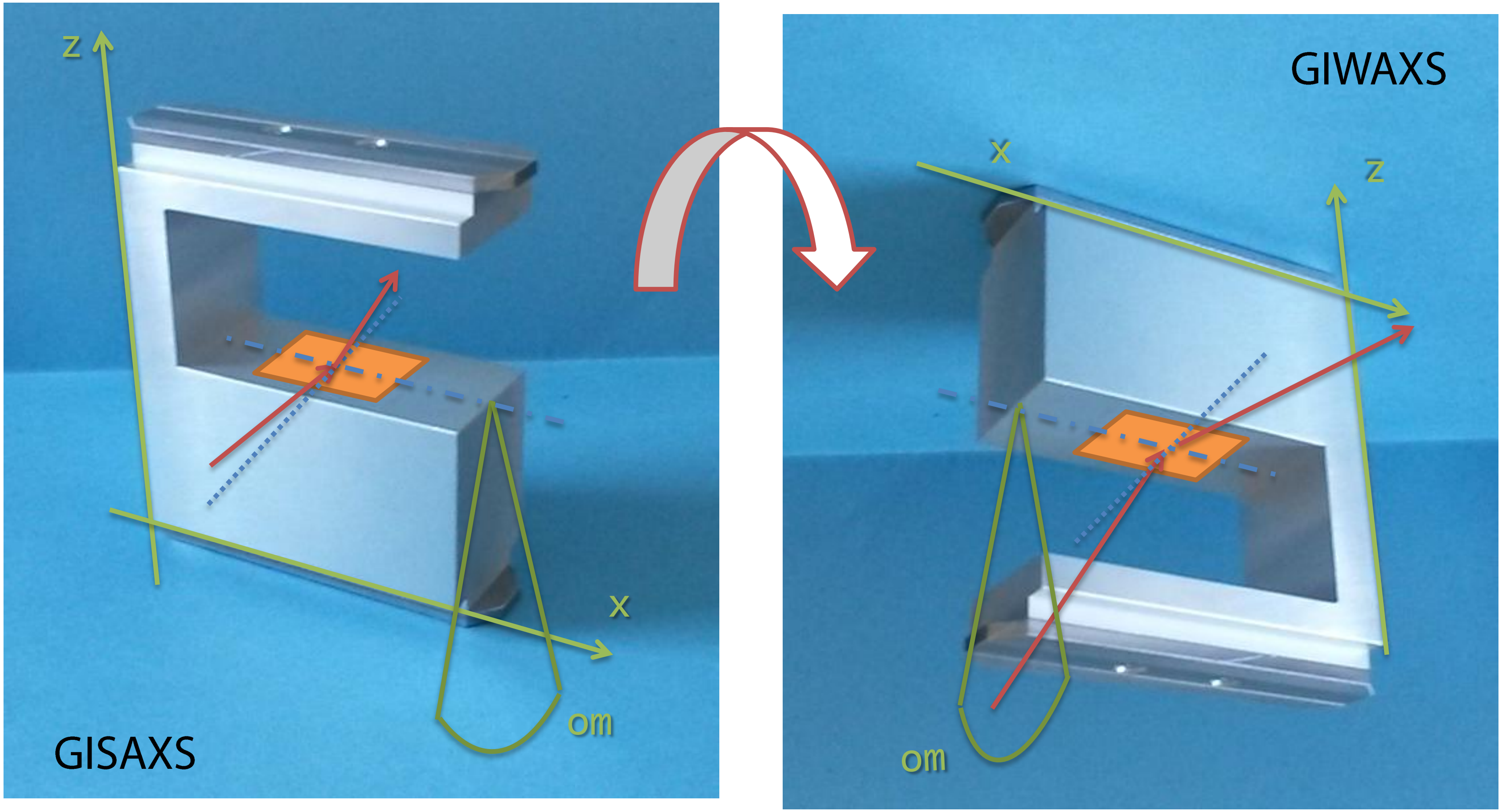
Simple GISAXS stage





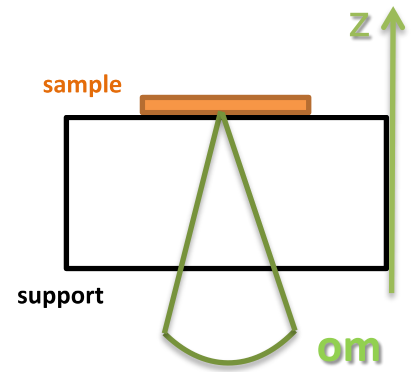
GISAXS / GIWAXS

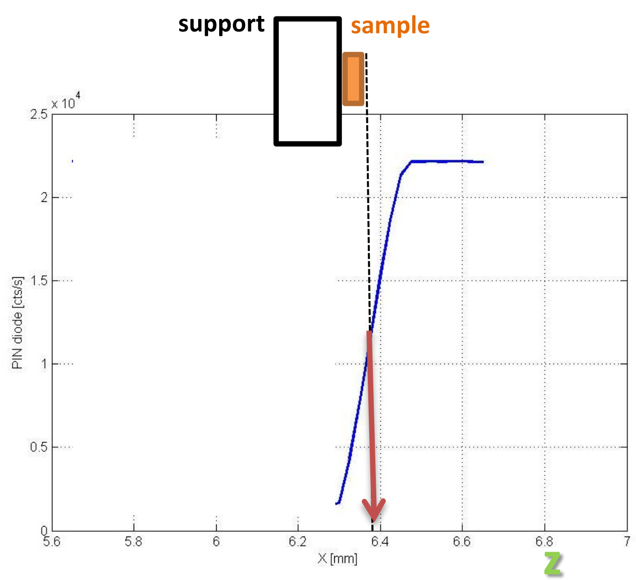
Flip-flop the stage, not removing the sample

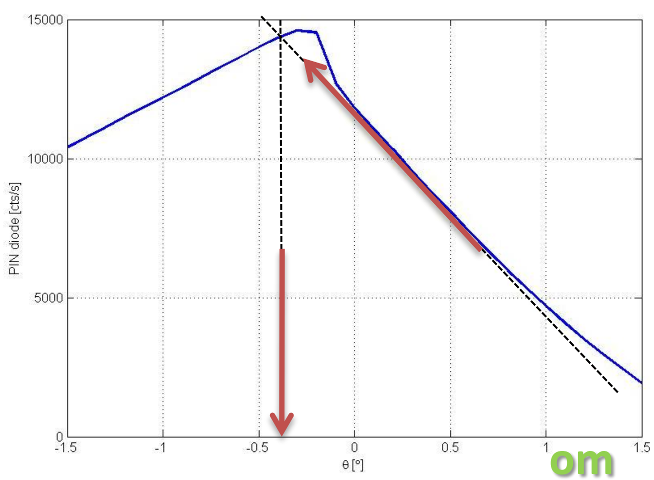


GISAXS alignment

Simple Stage

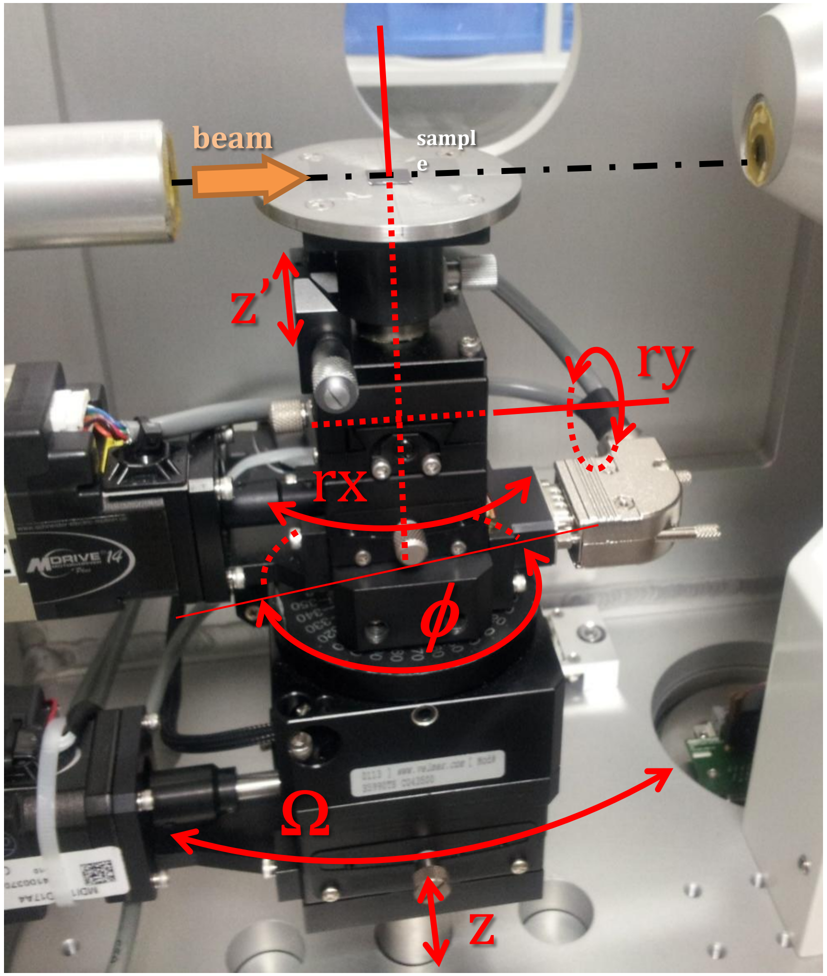


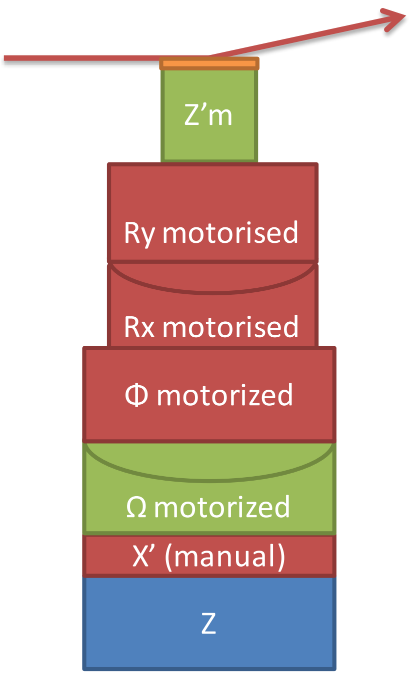




Advanced GISAXS stage

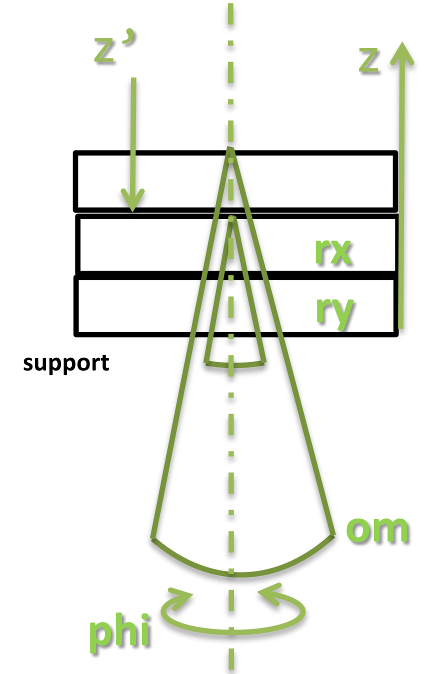
z : vertical translation
om : omega, reflection tilt
phi : vertiocal rotation
rx : fine tilt
ry : fine reflection tilt
z’: manual vertical translation





GiSAXS

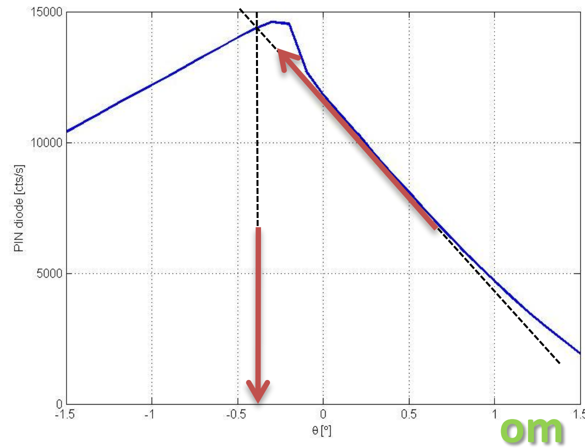
GISAXS alignment

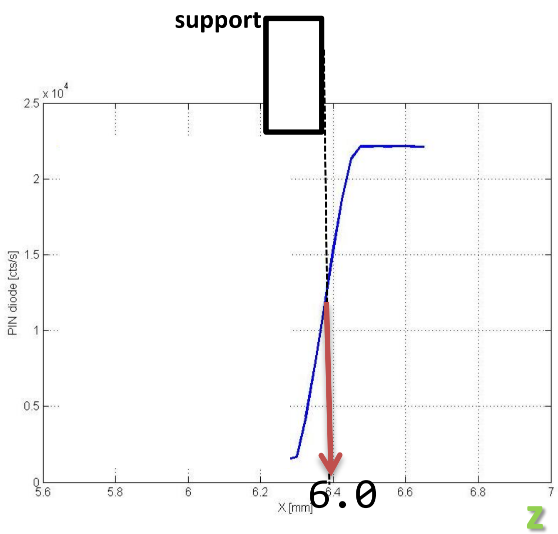


Advanced Stage

Align first the sample support into the beam.

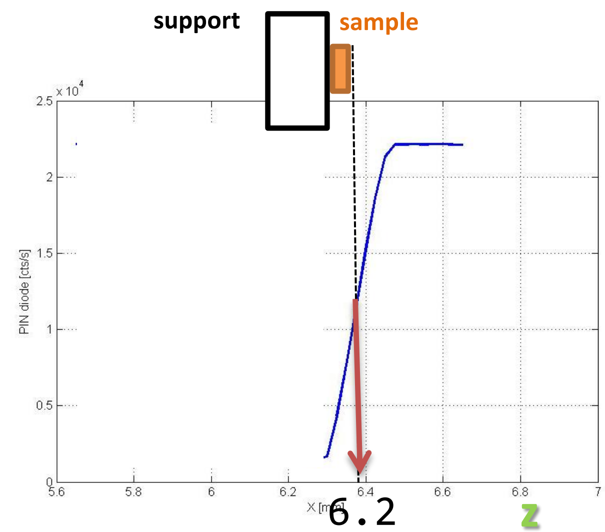
1) check for edge
2) check for horizontality





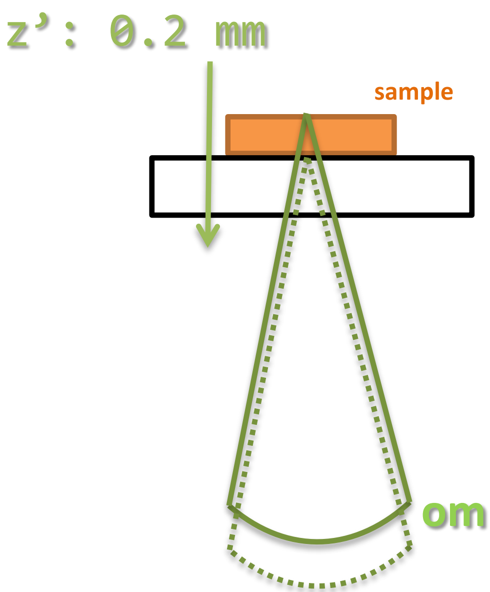
Install the sample and repeat the edge scan. This will give the sample thickness.

Sample thickness :
6.2 – 6.0 = 0.2 mm

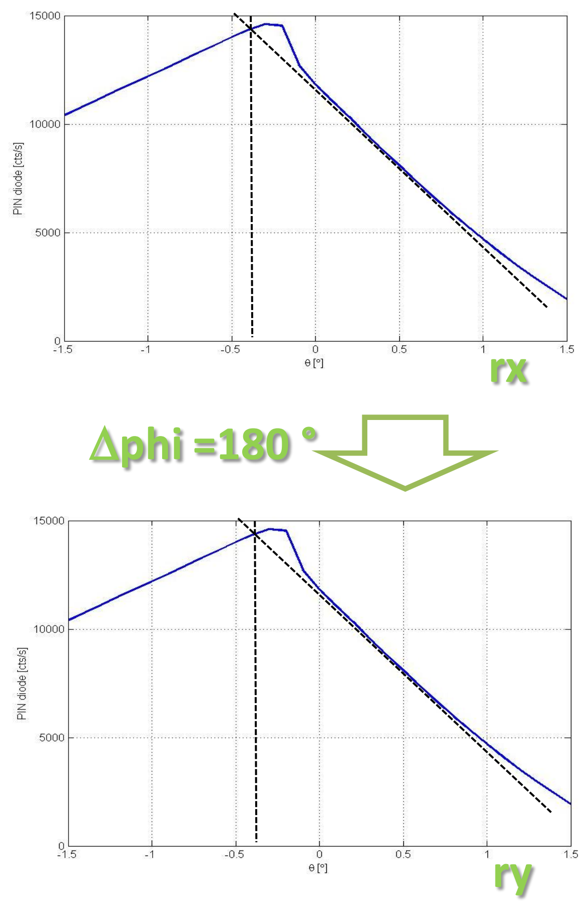


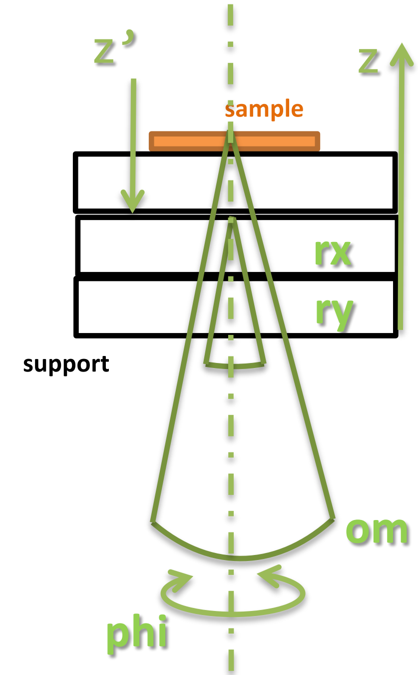
GISAXS alignment

Advanced Stage



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





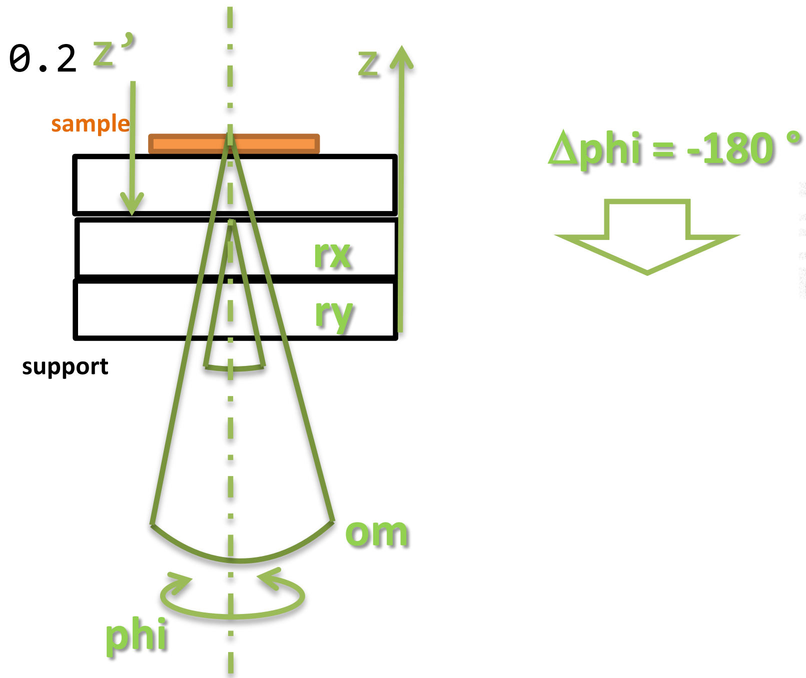
Now align the sample for horizontality in both directions

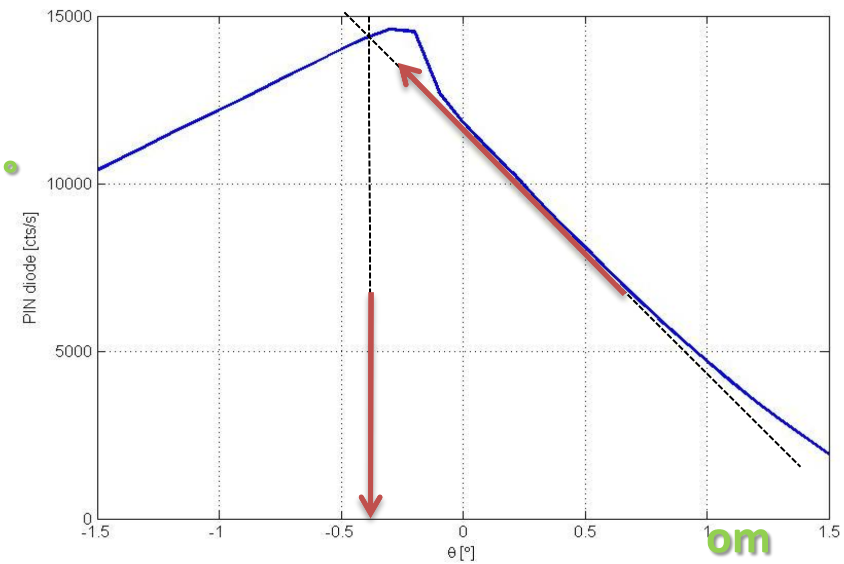
1) Direction ‘rx’
2) Turn ‘phi’ motor 180°
3) Direction ‘ry’

GISAXS alignment

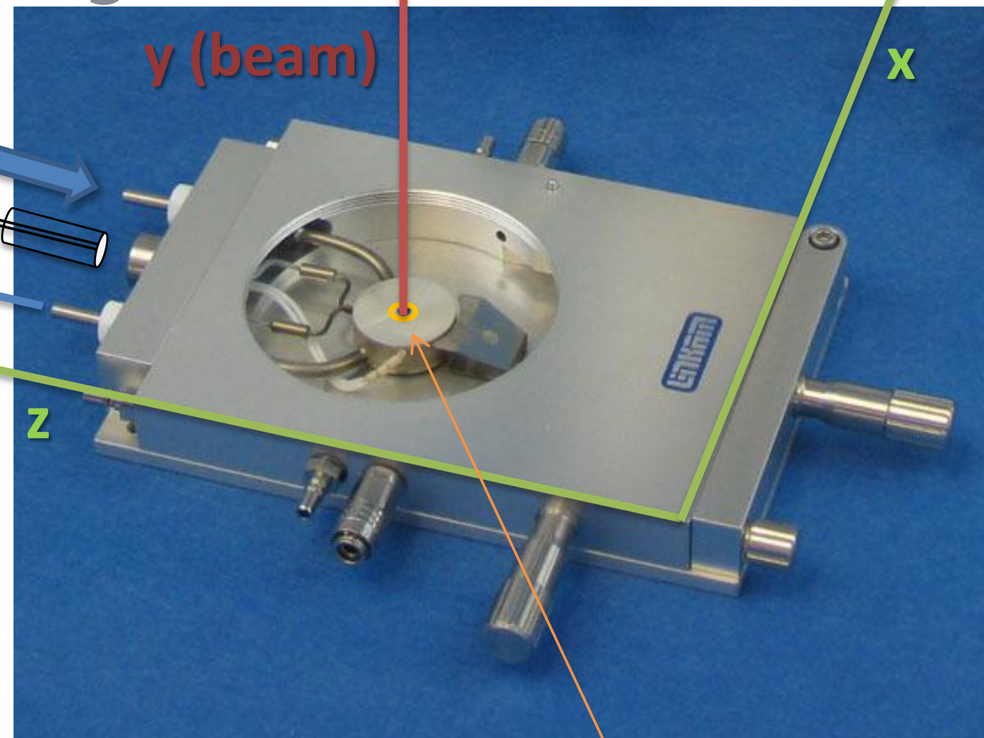
Advanced stage

6.2 - 6.0 = 0.2





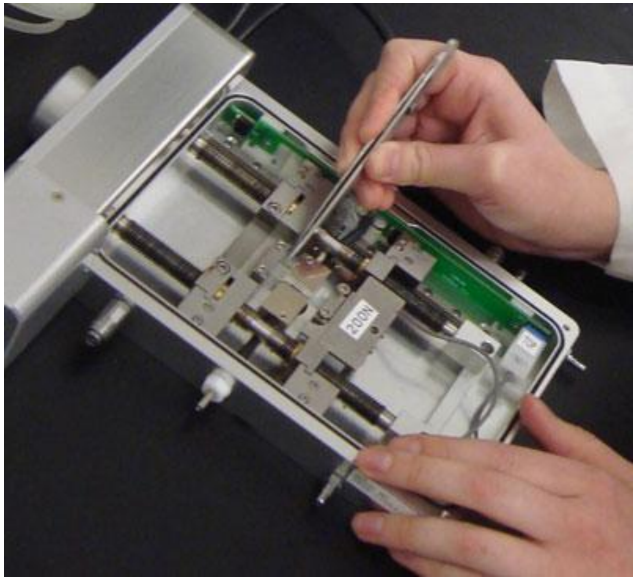
Linkam Temperature Stage



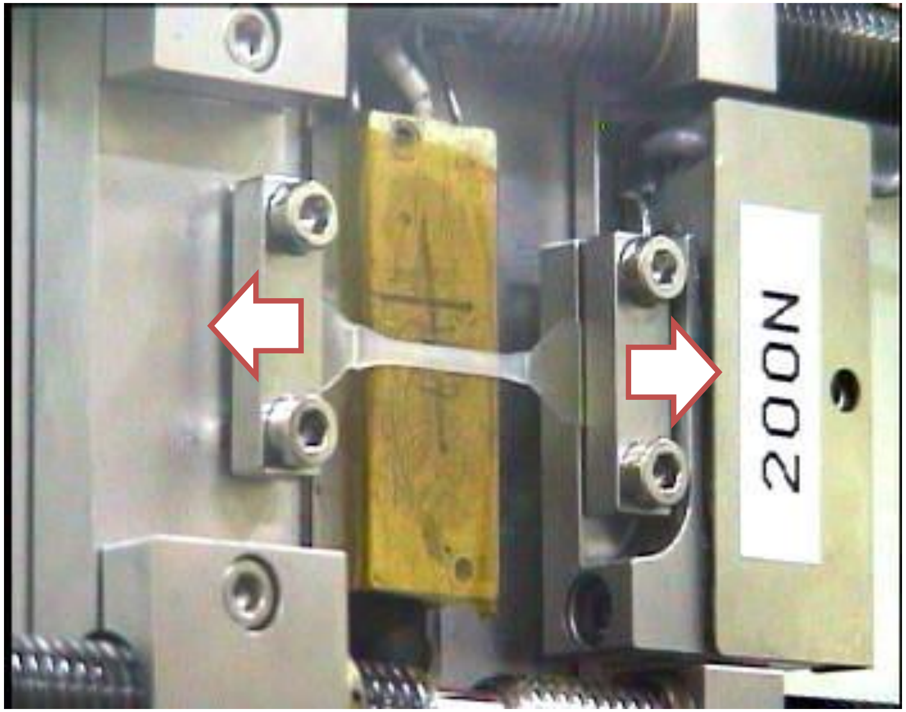
Temperature range :
-196 °C to 350 °C

Single sample : self-standing or capillary

Linkam Tensile Stage



Single sample : self-standing

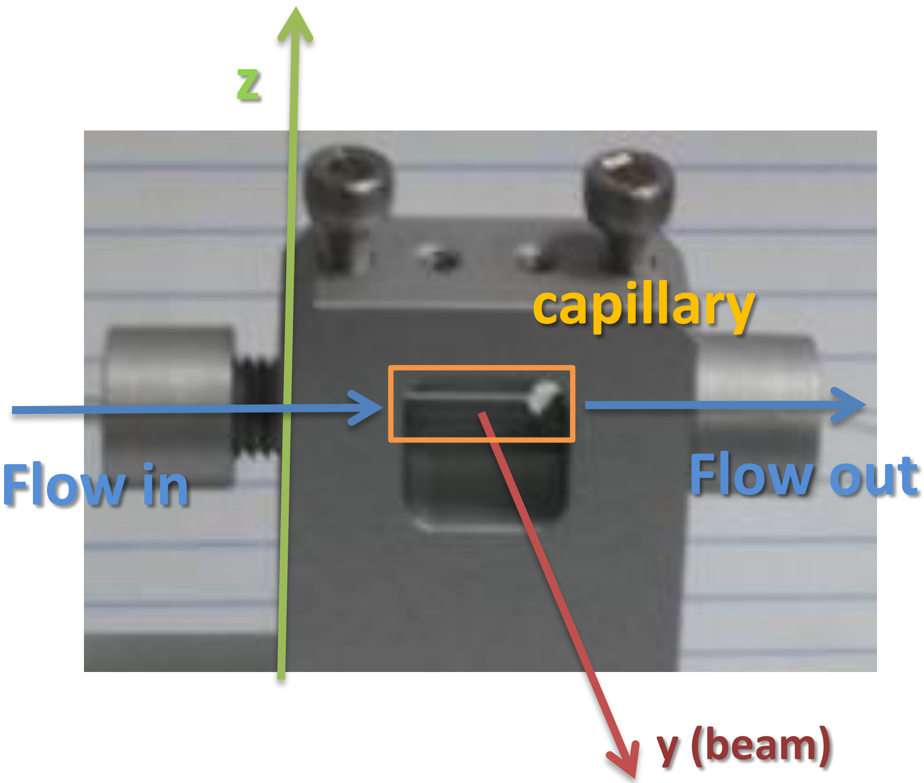


Temperature range :
-196 °C to 350 °C

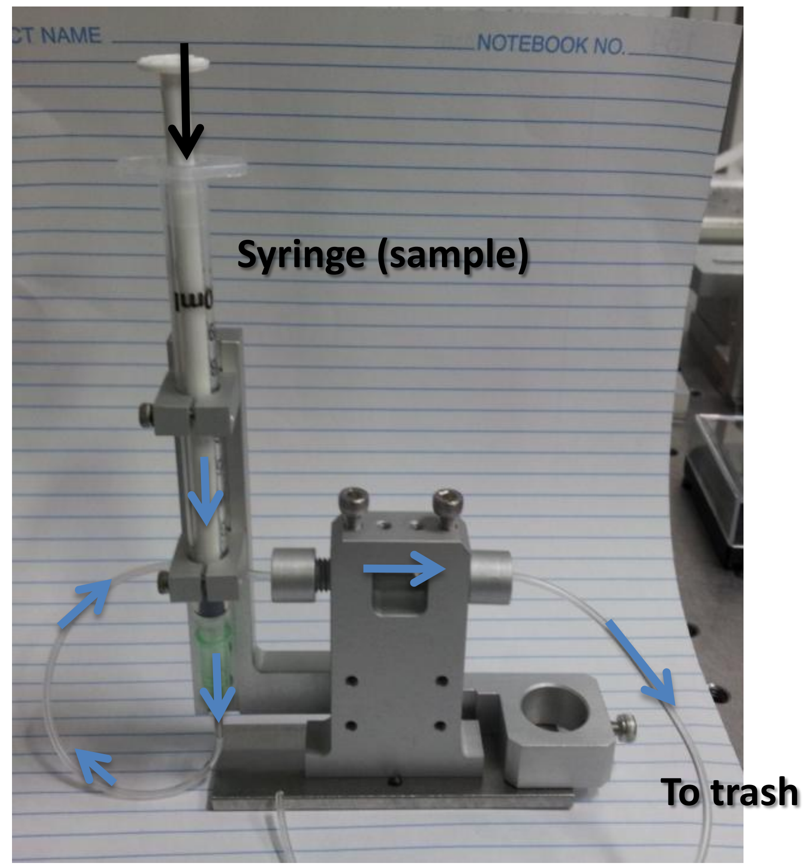
Flow-through

No temperature controlled.

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

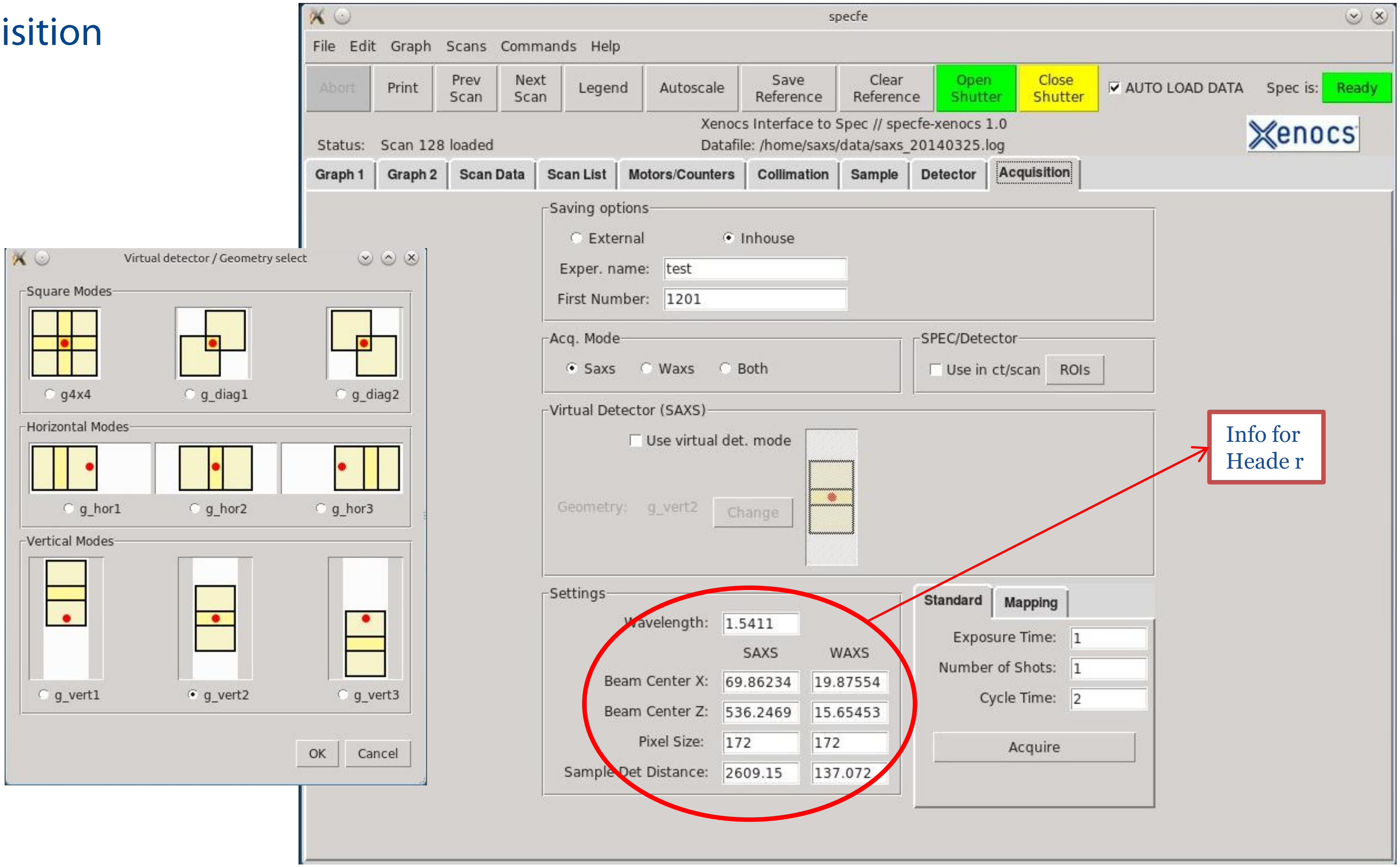


Push piston for flow in



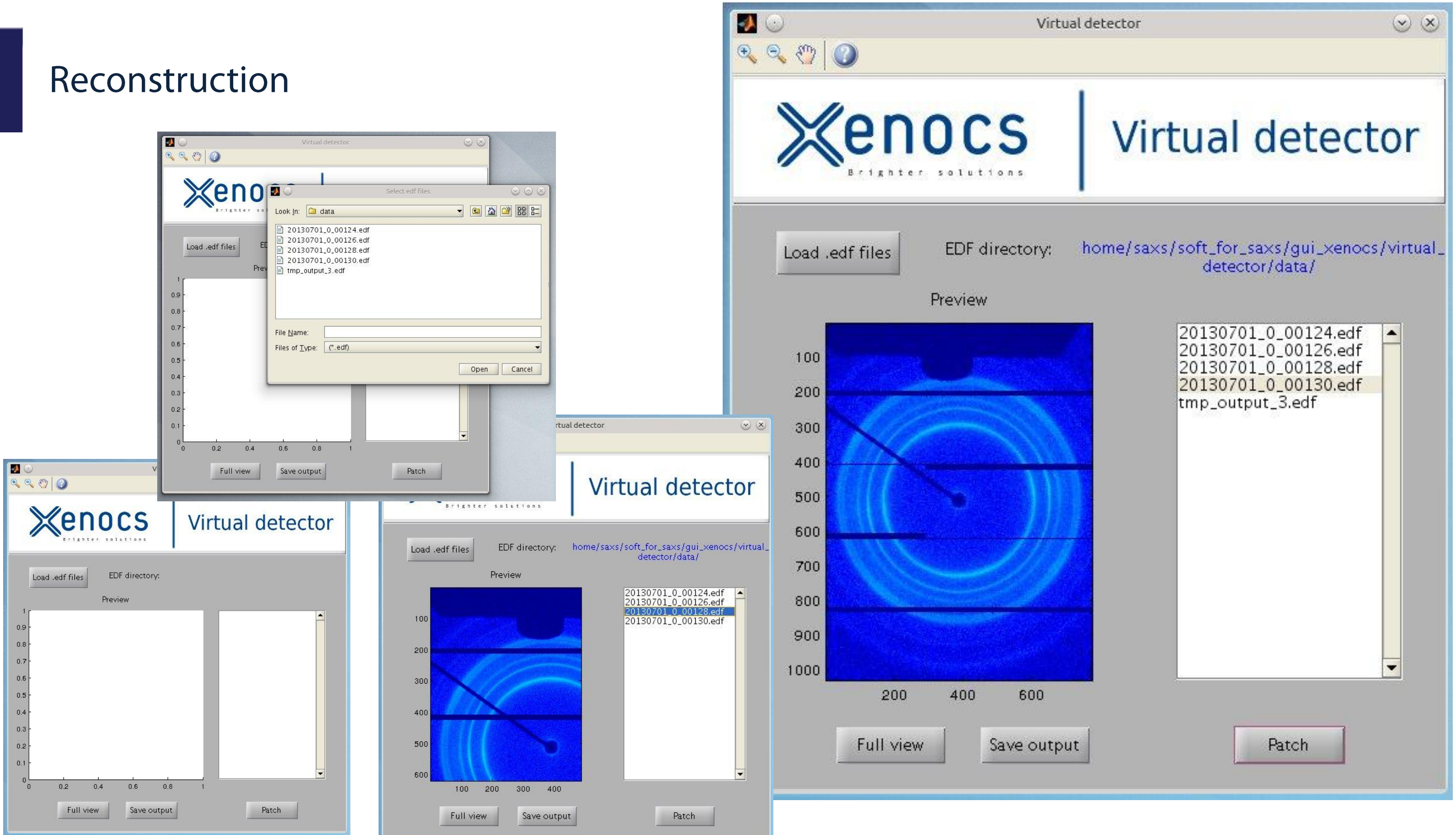
Virtual Detector

Acquisition

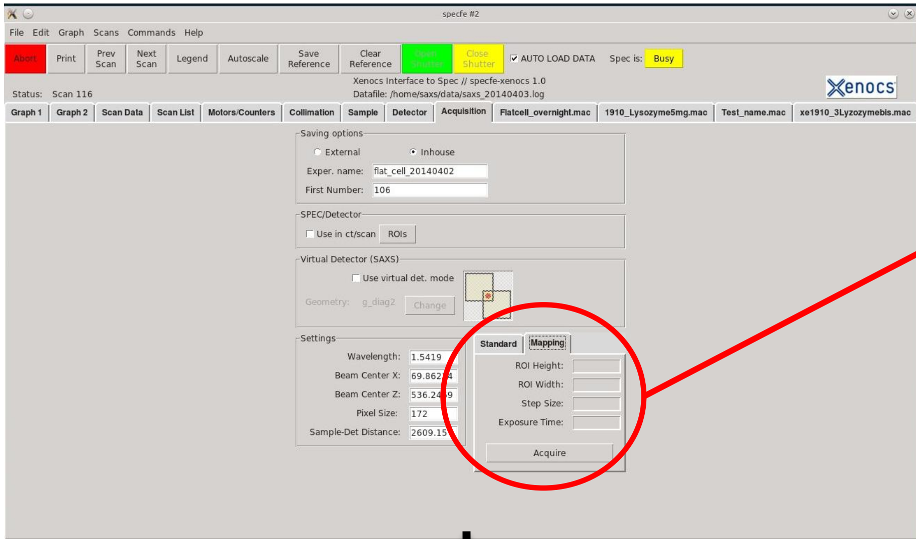


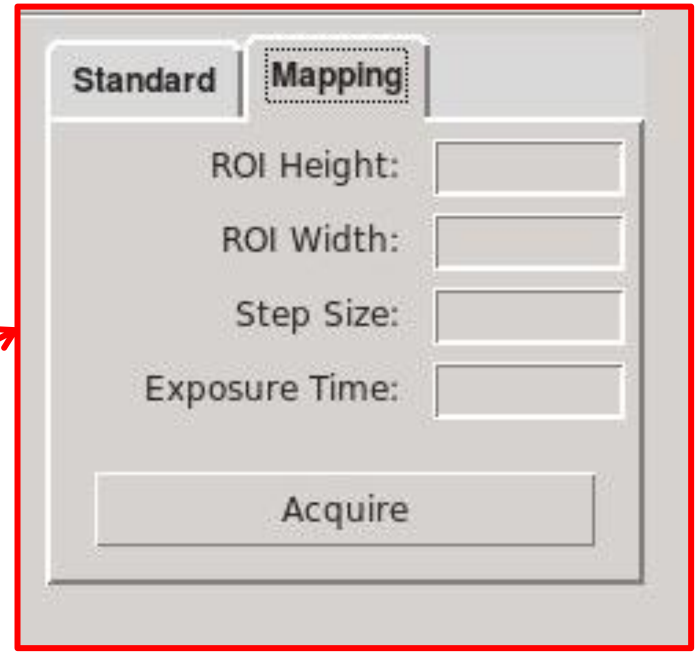
Virtual Detector

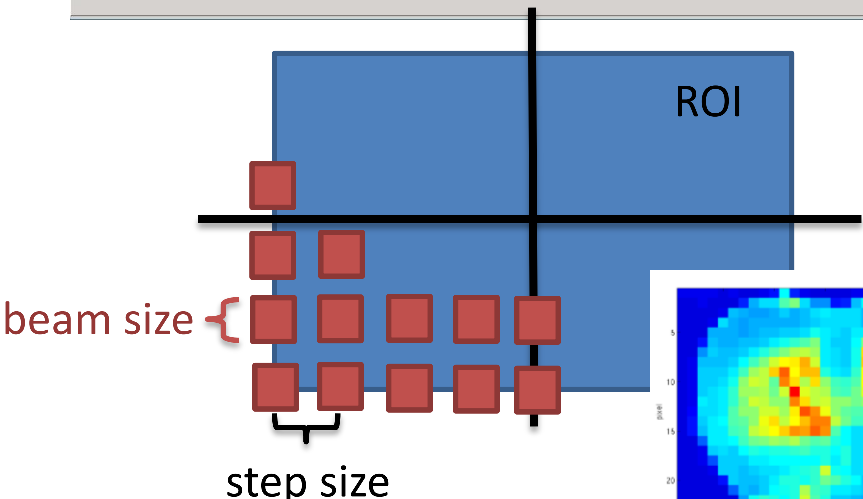
Reconstruction

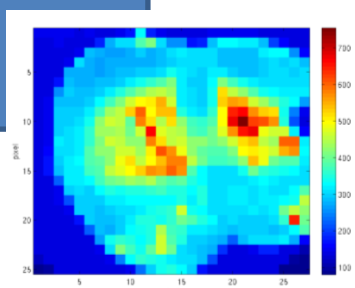


Mapping Acquisition







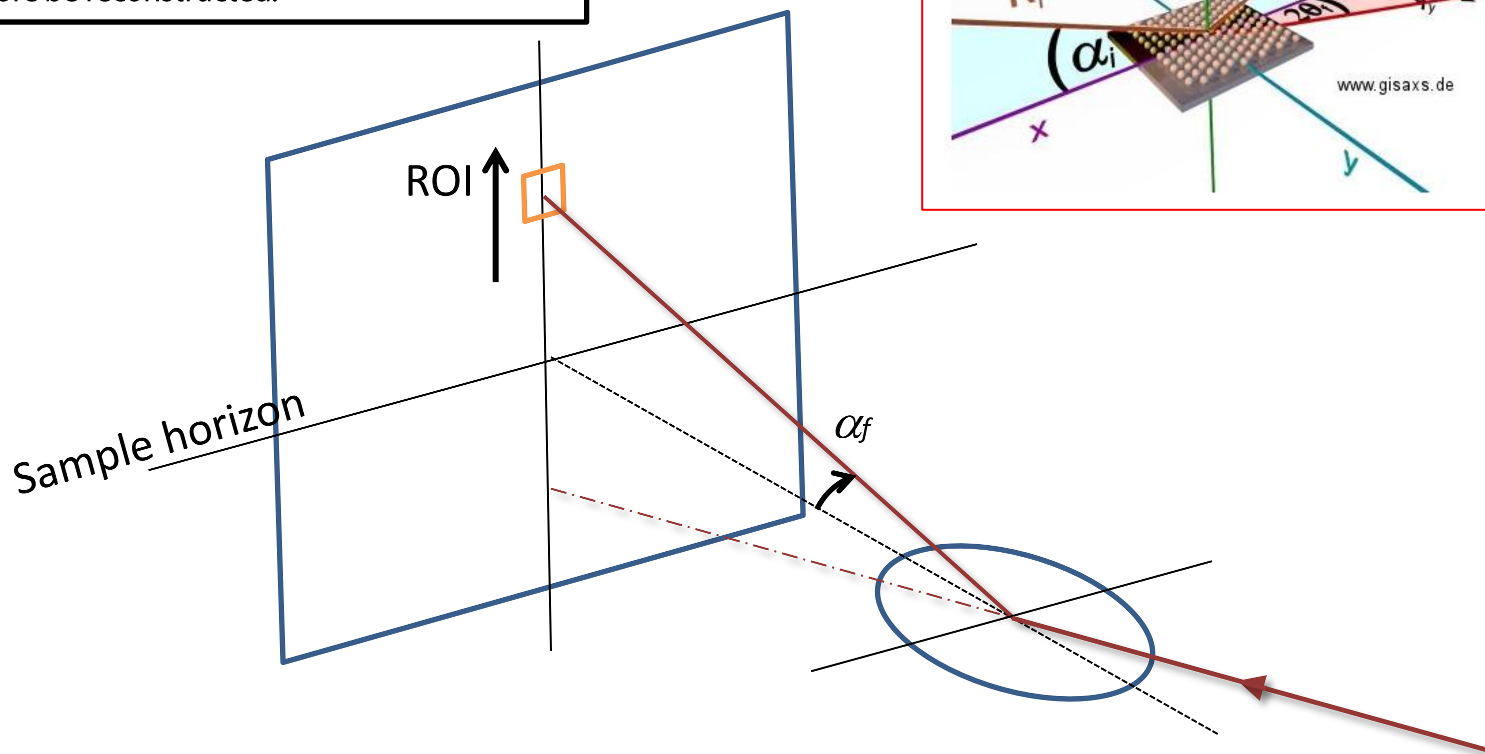


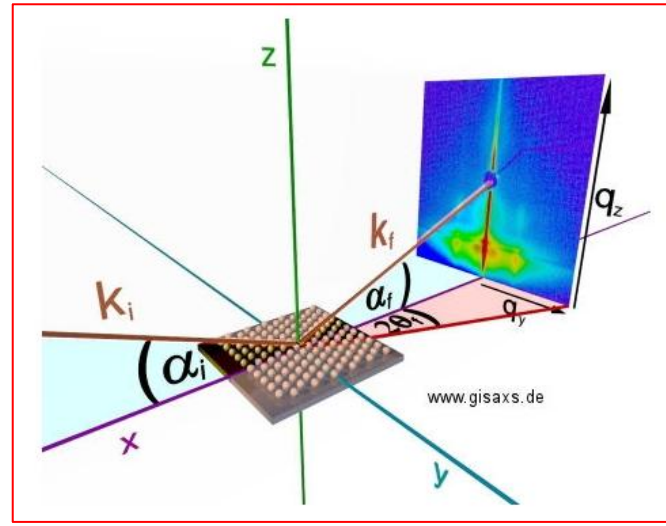
RASTER SCAN:
- The acquisition program calculates the number of measured points as function of the size of the ROI and the size of the beam, and calculates the motor positions for the sample stage to move.
-Takes one SAXS/WAXS exposure for every position (moves automatically) of the scan starting from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

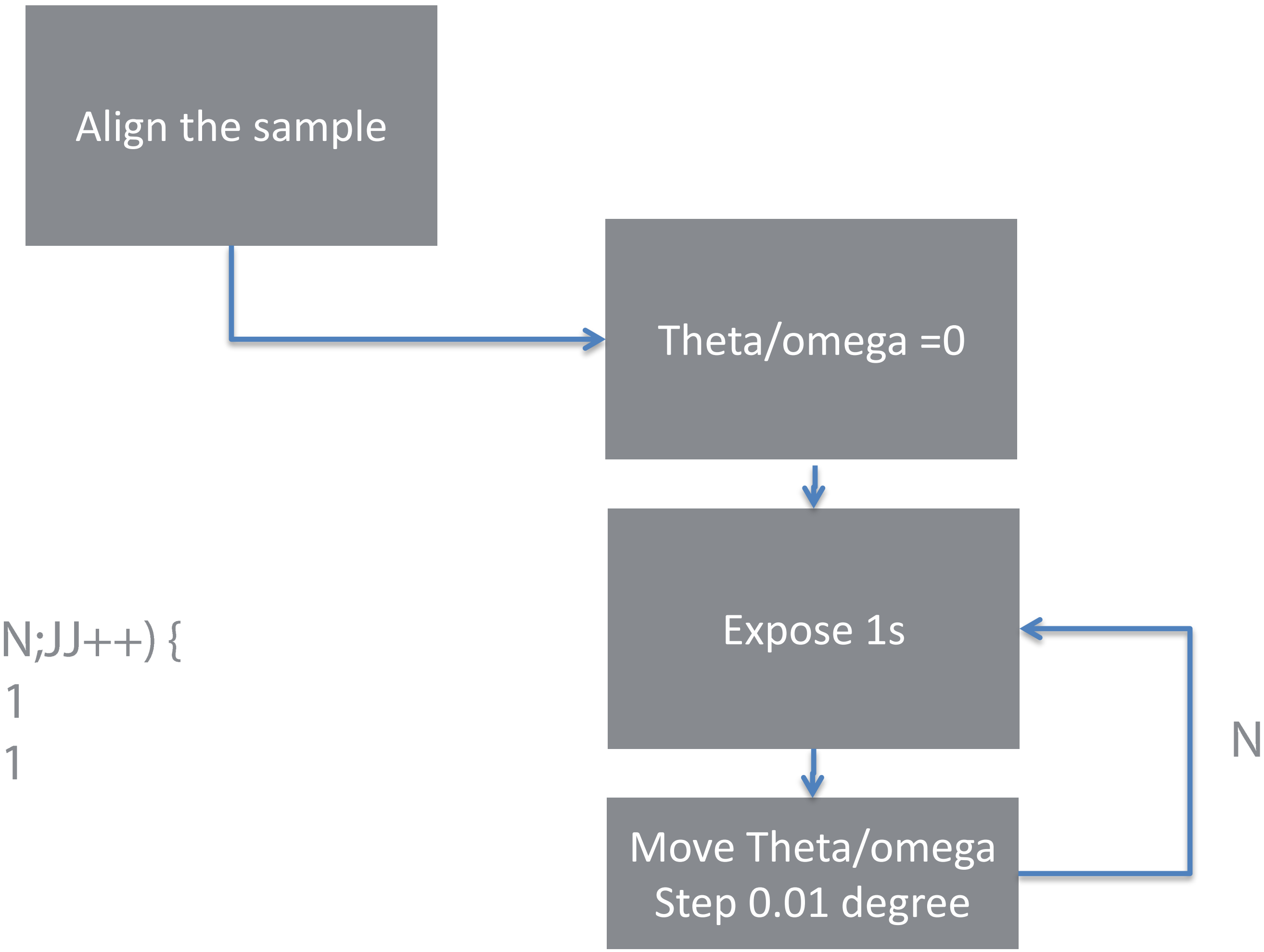




Reflectometry

Acquisition

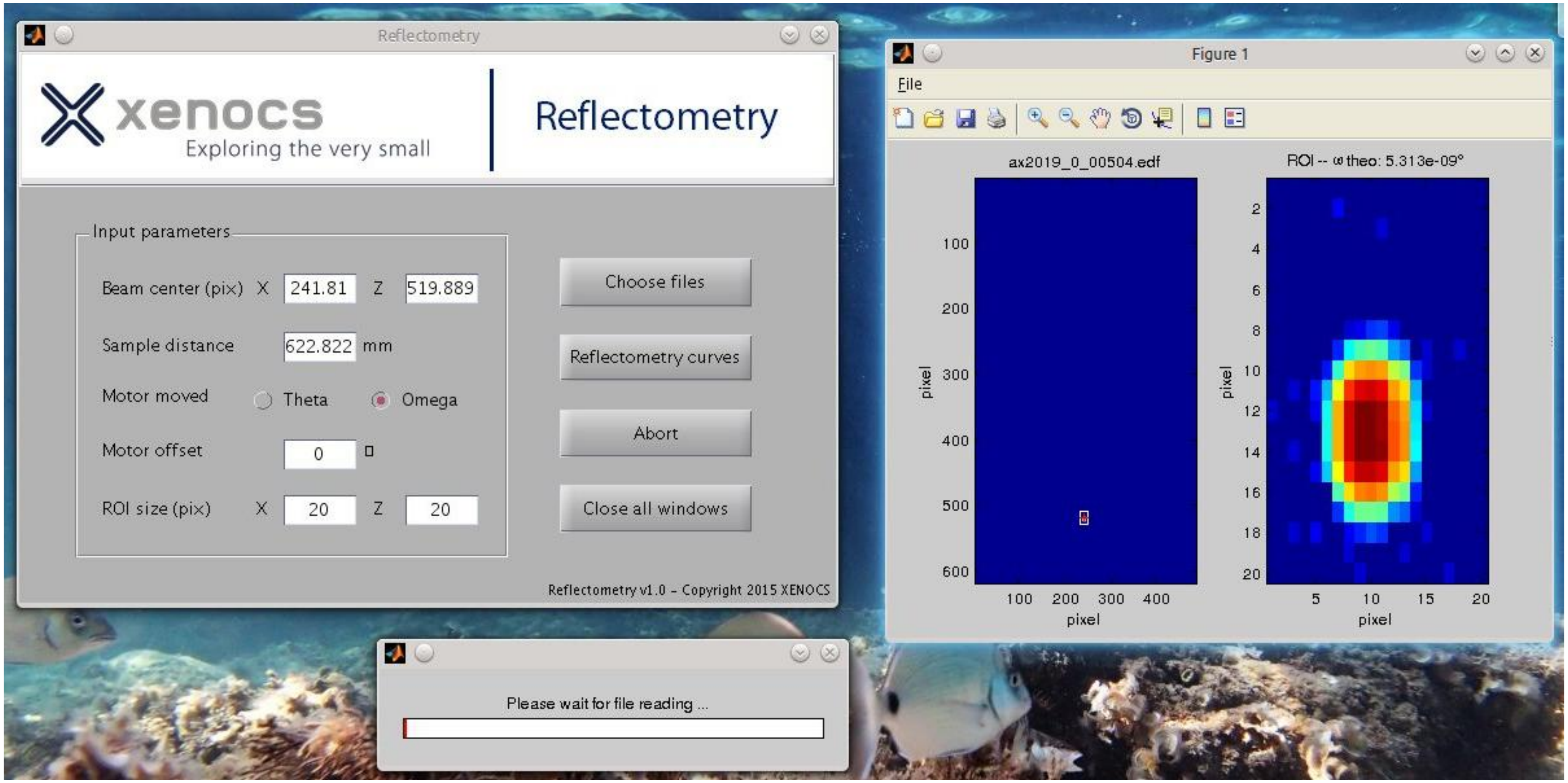
- SPEC macro



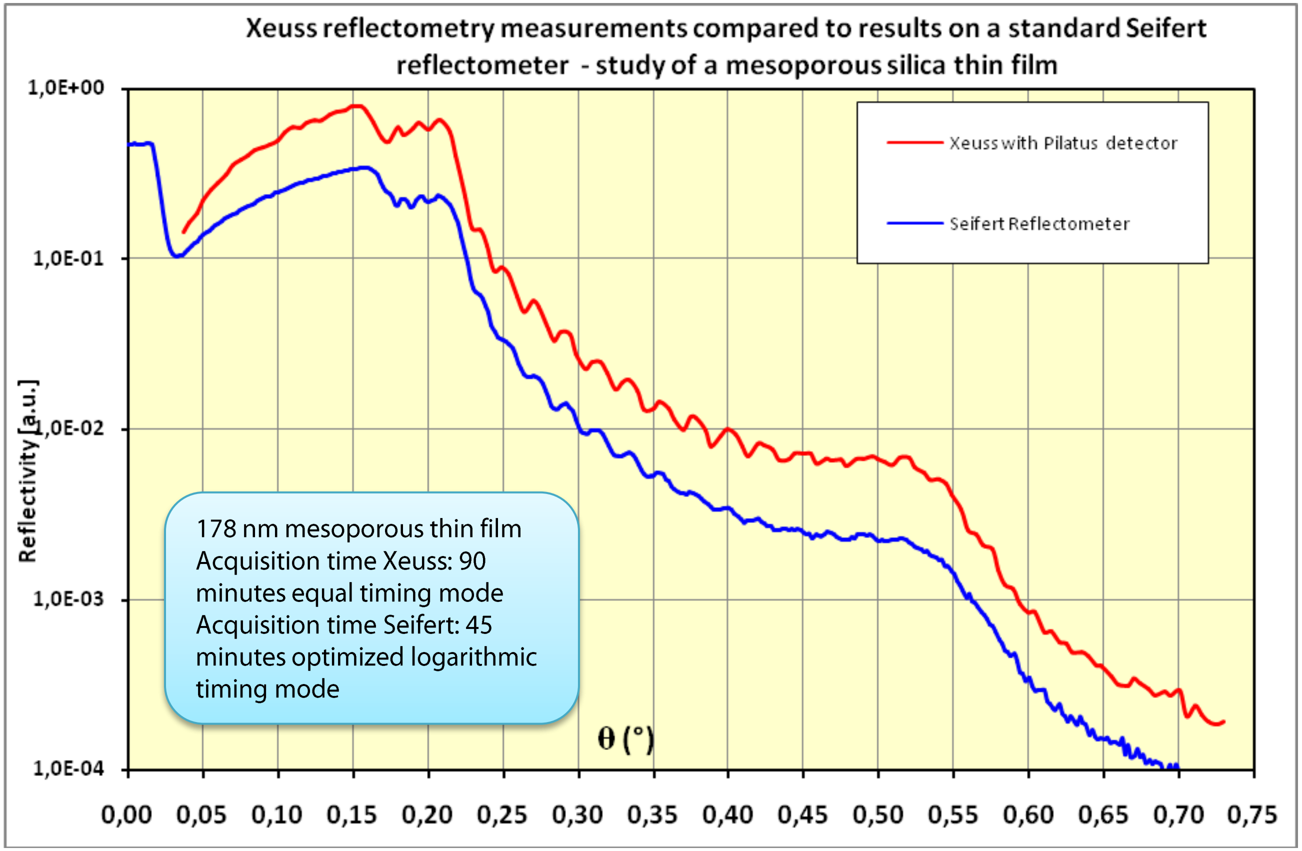

```
umv  th 0

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!