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Tutorial: SAXS data reduction and analysis

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SAXS-WAXS
USAXS
ID2 TIME RESOLVED

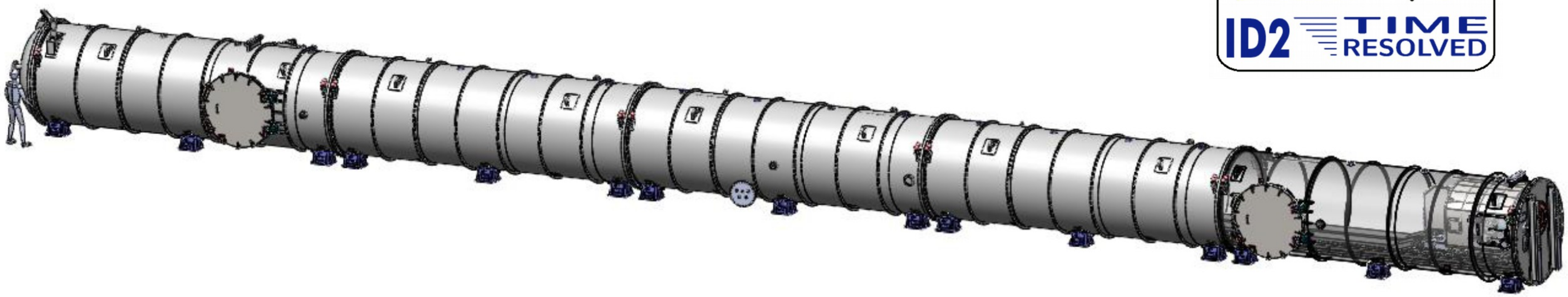
Outline

- ID02 data and metadata concept
- understanding of the data formats (EDF, HDF5)
- detector dependent corrections (non-linearity, dark current, flat field, spatial distortion, etc.) as well as the normalisation to an absolute intensity scale and the azimuthal integration to obtain one dimensional SAXS profiles
- data reduction schemes
  - + SPD (saxs programs)
  - + DAHU (PyFAI)

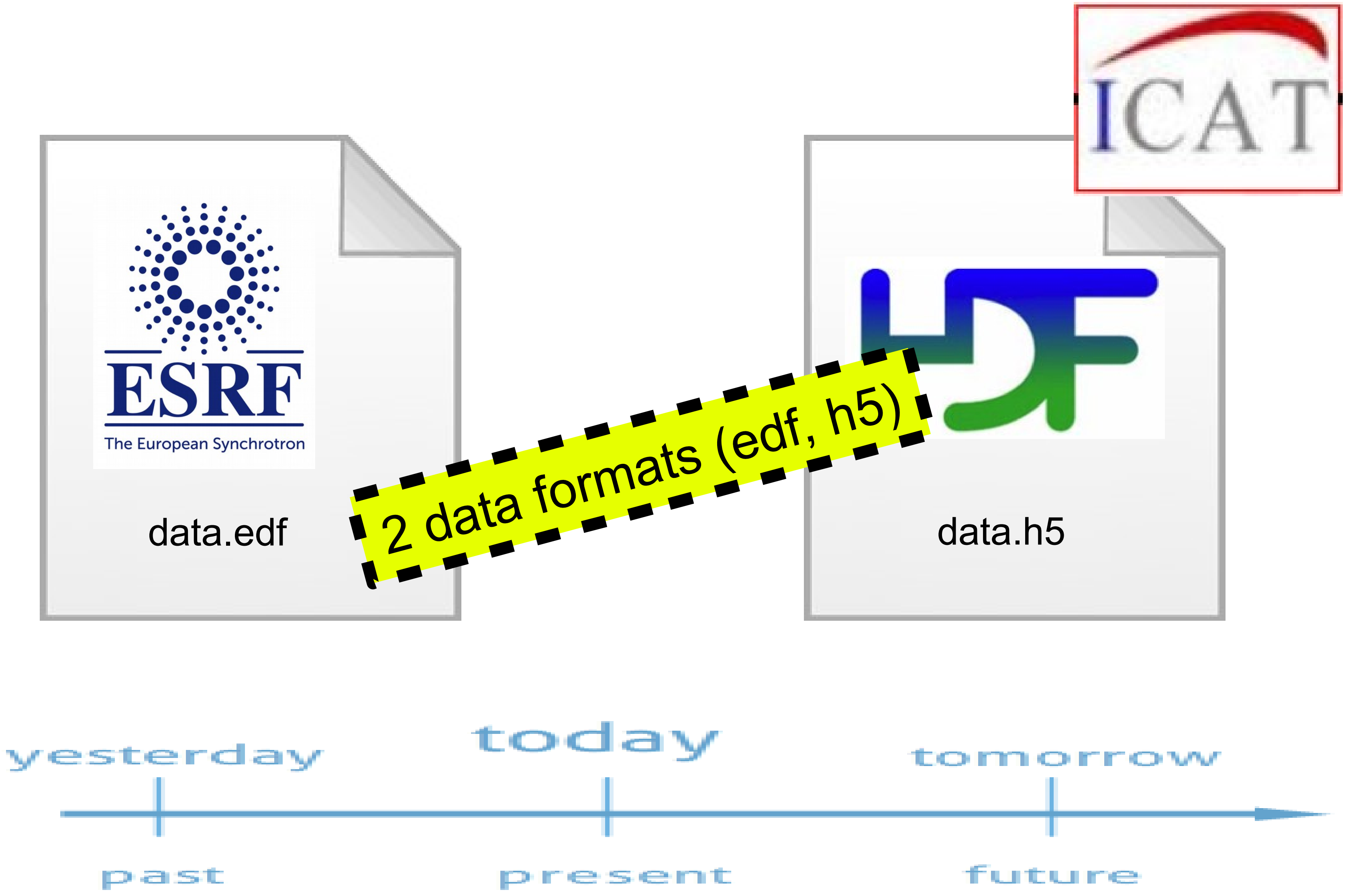
- SAXSutilities
  - + 1D, 2D visualization
  - + 1D, 2D data reduction
- More advanced features include direct modelling (standard models in small-angle scattering)

ID02 data and metadata concept

SAXS, WAXS and USAXS



ID02 data and metadata concept



ID02 data and metadata concept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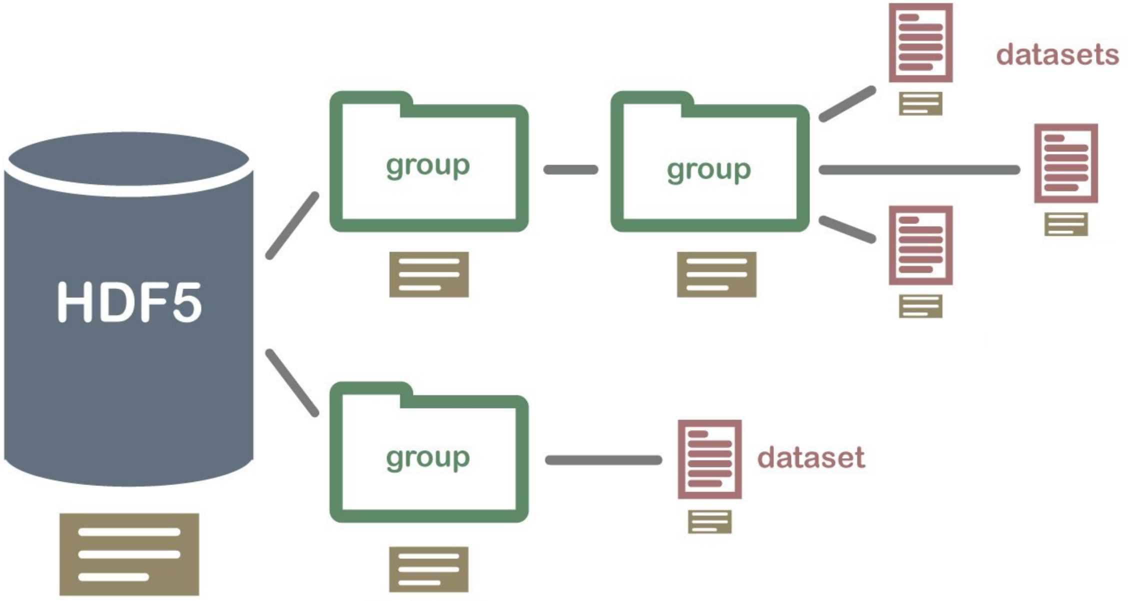
{
EDF_DataBlockID = 1.Image.Psd ;
EDF_BinarySize = 1440256 ;
EDF_HeaderSize = 8192 ;
ByteOrder = LowByteFirst ;
DataType = FloatValue ;
Dim_1 = 1000 ;
Dim_2 = 360 ;
Dummy = -10 ;
DDummy = 0.1 ;
Offset_1 = 0 ;
Offset_2 = 0 ;
History-1 = ""C:\\Program Files (x86)\\SAXSutilitie
    C:\\Data\\SAXSutilities_Testdata\\h5/og22_saxs_
HeaderID = EH:000001:000000:000000 ;
Compression = None ;
Image = 1 ;
SaxsDataVersion = 2.40 ;
Size = 1440256 ;






}
'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7-
    '7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7-
    '7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7-
```

ID02 data and metadata concept



ID02 data and metadata concept

understanding of the data formats (EDF, HDF5)

http://www.esrf.fr/files/live/sites/www/files/Instrumentation/software/data-analysis/OurSoftware/SAXS/SX_parametrization-ref-short_20130125.pdf

PETER BOESECKE SX_parametrization-ref-short_20130125.doc 2013-01-25

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Contents

understanding of the data formats (EDF, HDF5)

SX General Parameters

- *Dim_1, Dim_2*, ... (Dim_1 default is 0, all others are 1)
- *RasterOrientation* (1 to 8 possible, 9-16 for compatibility, needs to be tested, default 1)

SX Image Parameters

- *Offset_1, Offset_2*, ... (default: 0)
- *BSize_1, BSize_2*, ... (default: 1)

SX Scattering Parameters

- *PSize_1, PSize_2*, ... (no default) [m]
- *Center_1, Center_2*, ... (PoNI in image reference system including offsets, no default)
- *SampleDistance* (no default) [m]
- *WaveLength* (no default) [m]
- *DetectorRotation_1, DetectorRotation_2, DetectorRotation_3*, ... (default: 0) [rad]

Additional SX Scattering Parameters

- *ProjectionType* (Saxs|Waxs, default: Saxs: distances proportional to tan(2ϑ))
- *AxisType_1, AxisType_2*, ...(Distance, Angle, Numerator, default: Distance)

SX Intensity Calibration and Normalization Parameters

- *Intensity0* (no default), *Intensity1* (*Intensity0*)
- *NormalizationFactor* (default 1)
- *SampleThickness* (no default)

understanding of the data formats (EDF, HDF5)

special case ehf/edf files

```
{
EDF_DataBlockID = 1.Image.Psd ;
EDF_BinarySize = 1440256 ;
EDF_HeaderSize = 8192 ;
ByteOrder = LowByteFirst ;
DataType = FloatValue ;
Dim_1 = 1000 ;
Dim_2 = 360 ;
Dummy = -10 ;
DDummy = 0.1 ;
Offset_1 = 0 ;
Offset_2 = 0 ;
History-1 = ""C:\\Program Files (x86)\\SAXSuti
    C:\\Data\\SAXSutilities_Testdata\\h5/og22_
HeaderID = EH:000001:000000:000000 ;
Compression = None ;
Image = 1 ;
SaxsDataVersion = 2.40 ;
Size = 1440256 ;





}
'7+Á'7+Á'7+Á'7+Á'7+Á'7+Á'7
    '7+Á'7+Á'7+Á'7+Á'7+Á'7+
    '7+Á'7+Á'7+Á'7+Á'7+Á'7+
```

Peter Boesecke SaxsKeywords.doc 2013-01-25

Keywords for SAXS Data in EDF Files

EDF_DataFormatVersion = 2.42

Peter Boesecke

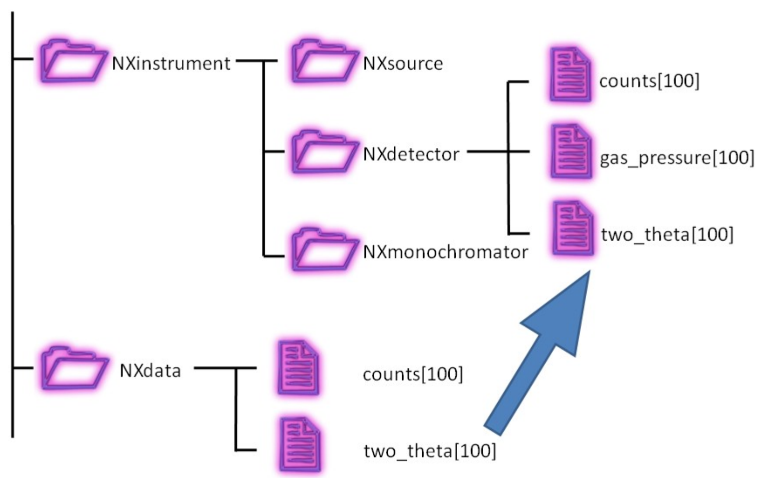
easily accessible metadata in edf

http://www.esrf.fr/files/live/sites/www/files/Instrumentation/software/data-analysis/OurSoftware/SAXS/SaxsKeywords-V2.459P2.033E2.229.pdf

understanding of the data formats (EDF, HDF5)

https://www.hdfgroup.org

http://www.nexusformat.org



understanding of the data formats (EDF, HDF5)

ESRF NeXus files

NXroot top level, one per file

NXentry one group per measurement

NXinstrument only one per NXentry

NXdetector

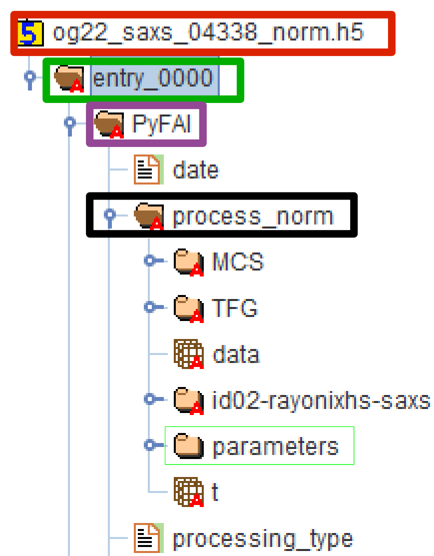
NXdata one NXdata group per plot

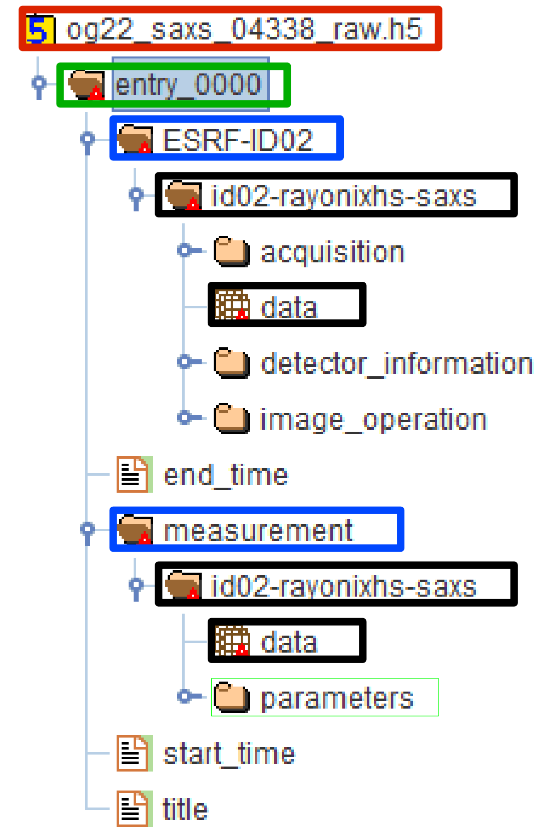
measurement (@NXcollection) flattened view of everything measured - only one per NXentry

NXsample

user (@NXuser)

NXprocess for analysis





raw data (*_raw.h5) [NXdata]:
/entry_0000/measurement/id02-XXXX/data

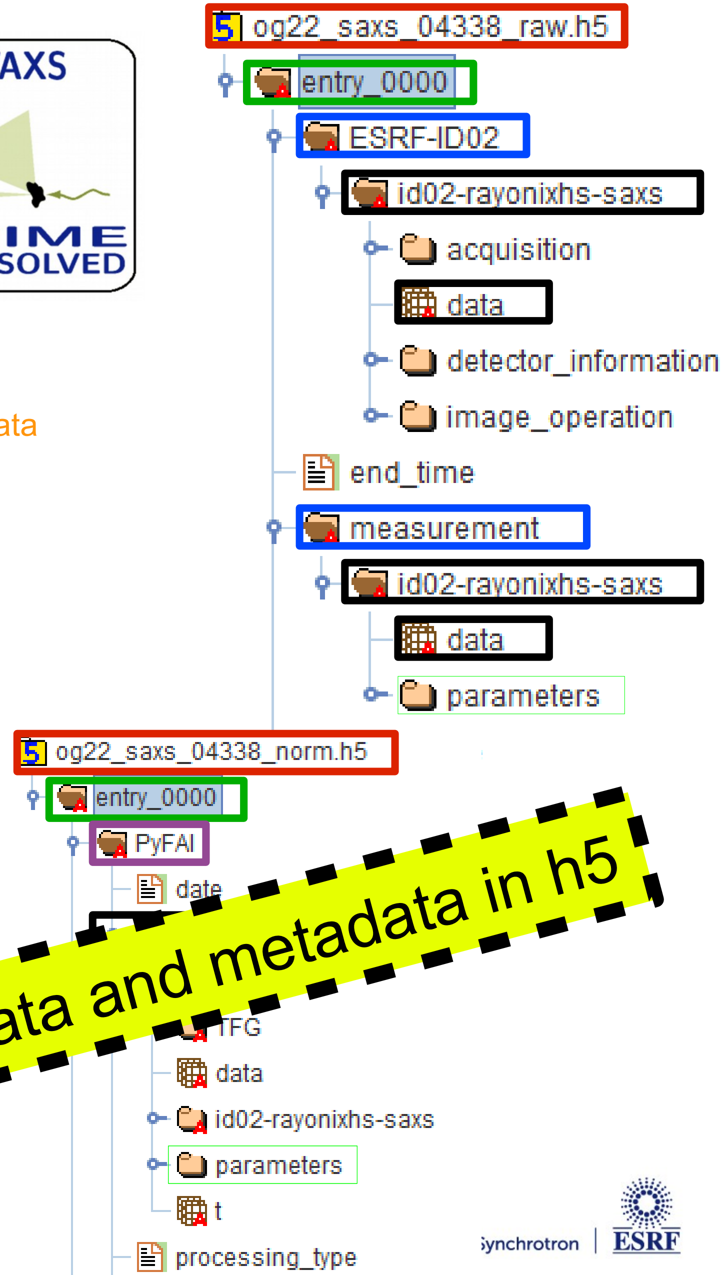
raw metadata (*_raw.h5)
/entry_0000/measurement/id02-XXXX/parameters

raw metadata (*_scalers_*_raw.h5)
/entry_0000/id02/MCS
/entry_0000/id02/TFG
/entry_0000/id02/parameters

Note: static and dynamic metadata are written in two different files

processed data [NXdata]:
/entry_0000/PyFAI/process_XXXX/data
/entry_0000/PyFAI/process_XXXX/q
/entry_0000/PyFAI/process_XXXX/t

processed metadata:
/entry_0000/PyFAI/process_XXXX/parameters
/entry_0000/PyFAI/process_XXXX/MCS
/entry_0000/PyFAI/process_XXXX/TFG



locating data and metadata in h5

(online) data reduction

(A) detector specific corrections

(B) scattering specific corrections

(C) sample and beam specific corrections

(A) detector specific corrections

CCD raw image [ADU]	i_{raw}	identical exposure times
CCD dark image [ADU]	i_{dark}	
Flat field image [photons/ADU]	f_2	(corrected for distortion)

1. Dark image subtraction [ADU] $\quad i_1 = i_{raw} - i_{dark}$
2. Spatial distortion correction [ADU] $\quad I_2 = SPD(I_1)$
3. Division by flatfield [photons] $\quad i_3 = i_1 / f_2$

- Subtraction and division are done pixel by pixel
- The spatial distortion correction consists of a horizontal and vertical displacement of each pixel

(B) scattering specific corrections

4. Normalization to I0 [photons] and conversion to scattering cross section [1/sterad]

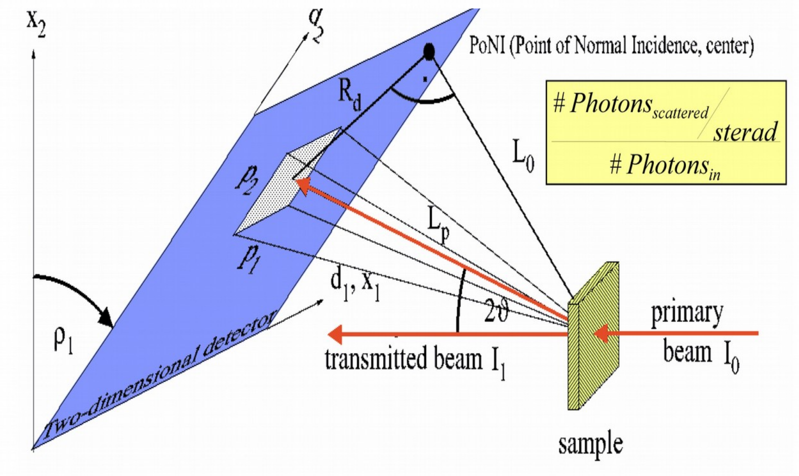
$$\left(\frac{1}{A}\right)\frac{\partial\sigma}{\partial\Omega} = \frac{\#Photons_{scattered}/sterad}{\#Photons_{in}} = i_4 = \frac{i_3}{I_0}\cdot\frac{L_p^{\,2}}{p_1\cdot p_2}\cdot\frac{L_p}{L_0}$$

$\frac{L_p}{L_0}$: inclined surface

$\frac{L_p^{\,2}}{p_1\cdot p_2}\cdot\frac{L_p}{L_0}$: $1/\Delta\Omega$

shortest sample to detector distance L_0
sample to pixel distance L_p
pixel size p_1, p_2

(see Bösecke, Diat, J. Appl. Cryst. (1997). 30, 867-871
and Narayanan, Diat, Bösecke, NIM A 467-468 (2001) 1005-1009)



(C) sample and beam specific corrections

5. Normalization to transmission and scattering volume, e.g. thin film: T=I1/I0, d: sample thickness

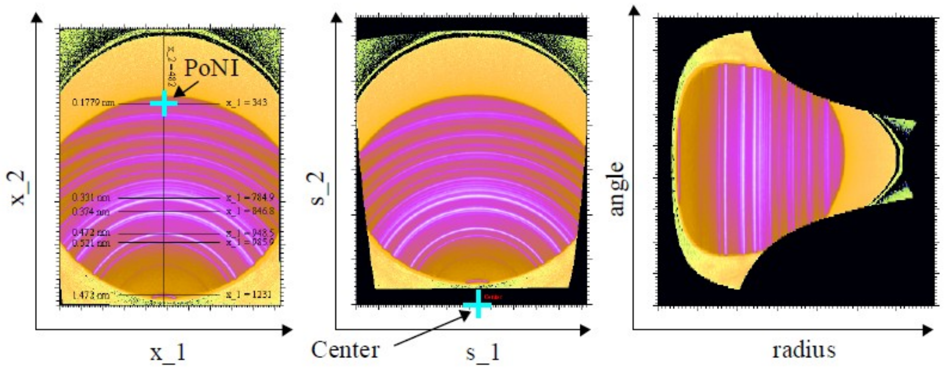
$$i_5 = \frac{i_3}{d \cdot I_1} \cdot \frac{L_p^{\ 2}}{p_1 \cdot p_2} \cdot \frac{L_p}{L_0}$$

6. Polarization correction (WAXS)

7. Reciprocal space mapping (WAXS) (Ewald sphere projections in reciprocal space, sample orientation required)

8. Azimuthal averaging

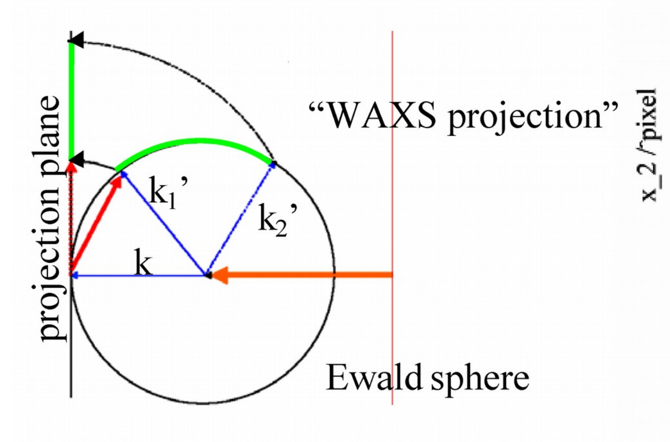
WAXS projection



raw WAXS pattern (inclination 32.5 degrees)

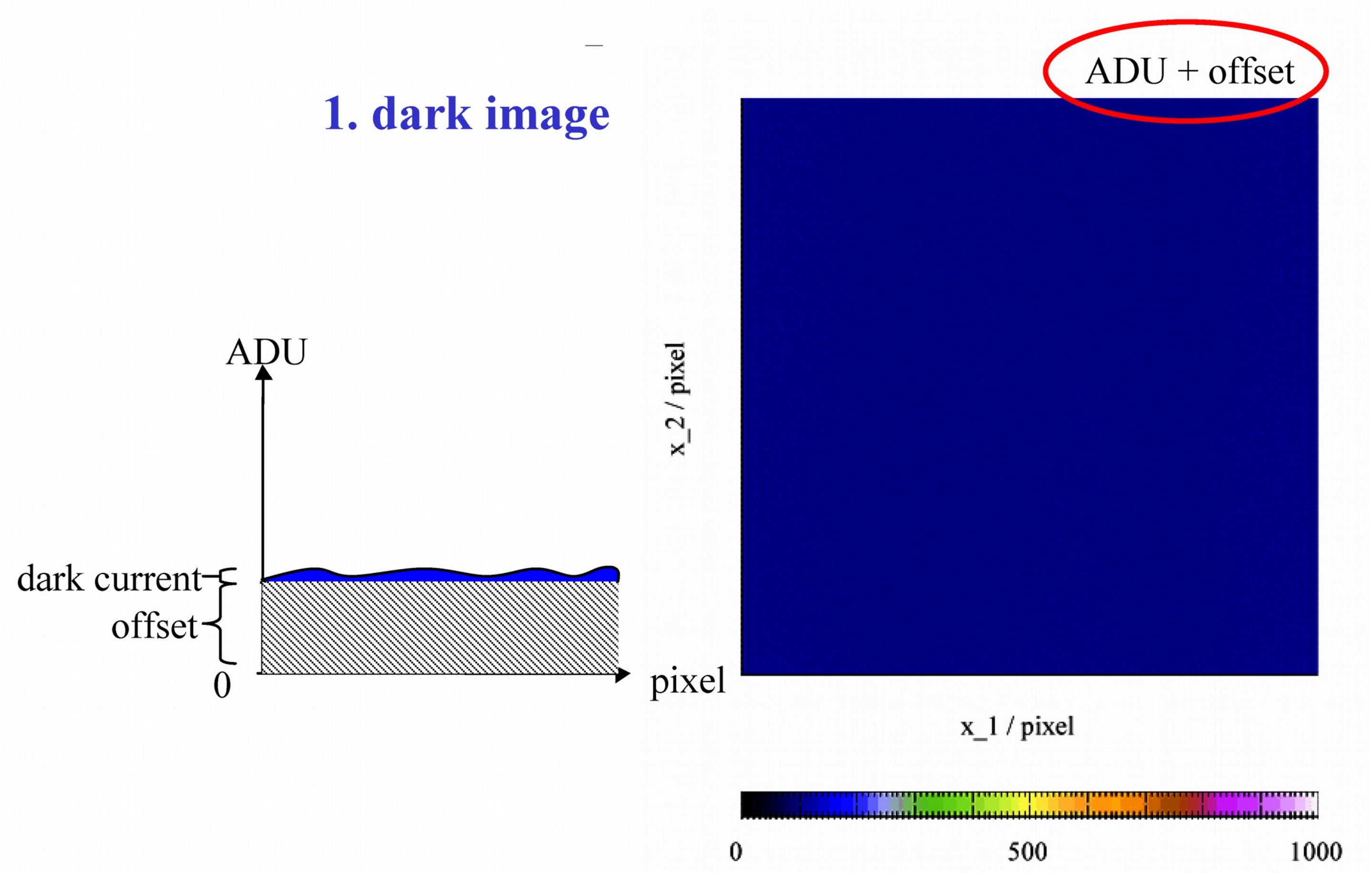
projected WAXS pattern

azimuthally regrouped WAXS pattern



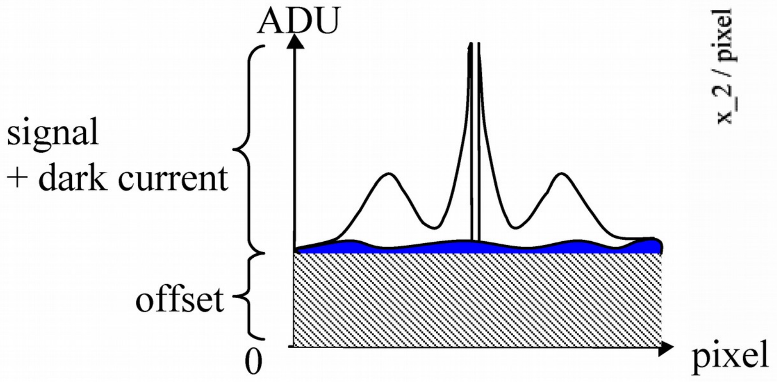
In Waxs projection the scattering pattern of a tilted detector can be geometrically analysed (azimuthal regrouping etc.) like a small angle scattering pattern.

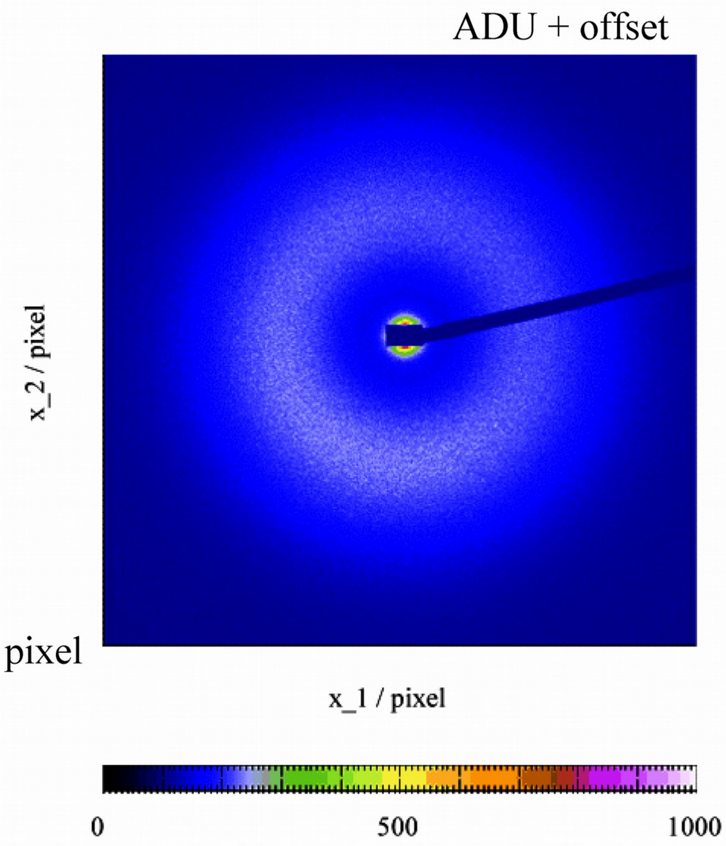
data reduction steps

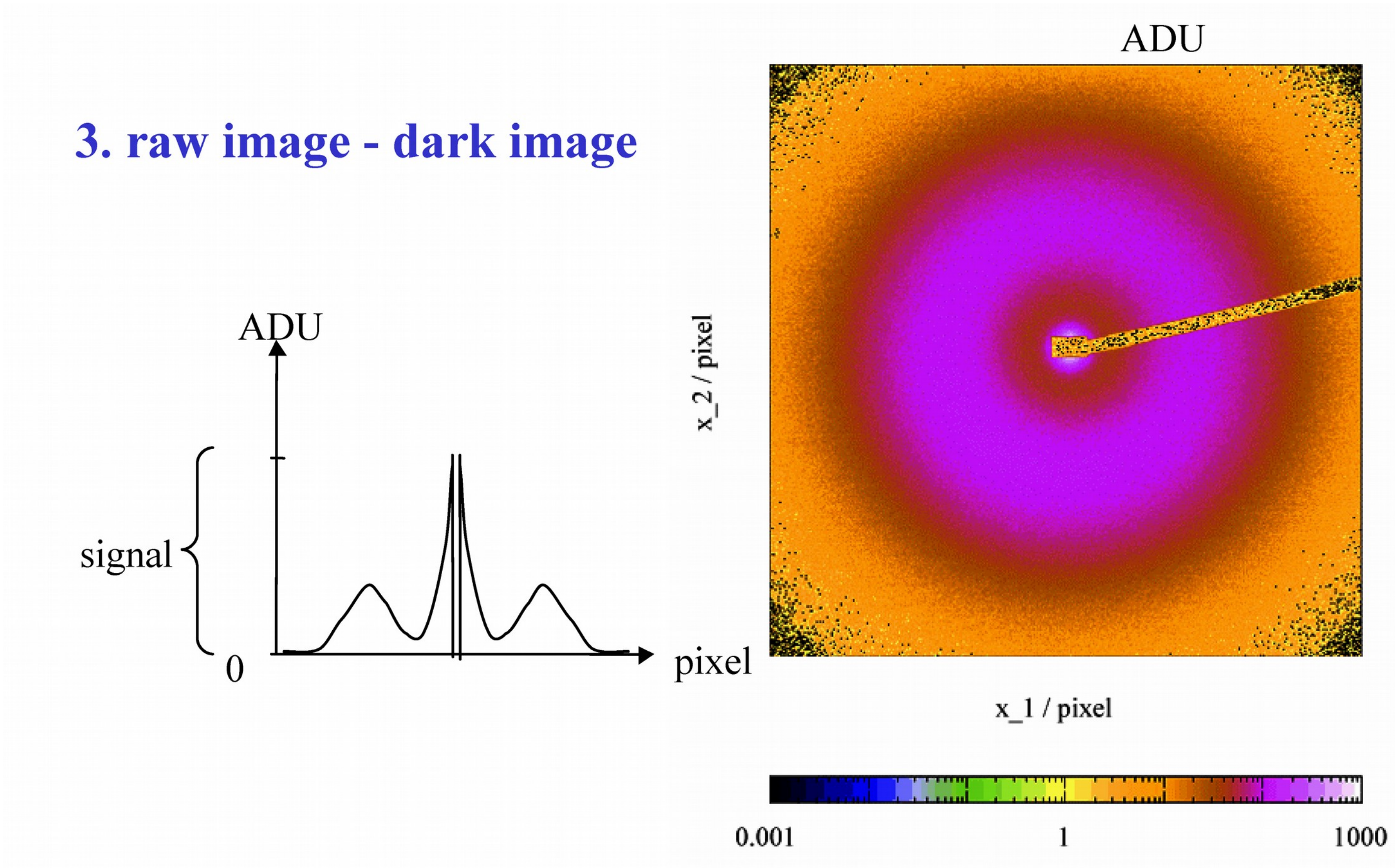


data reduction steps

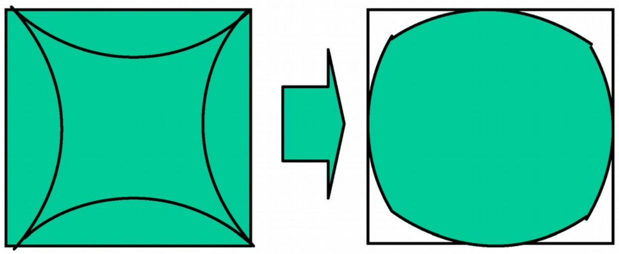
2. raw image

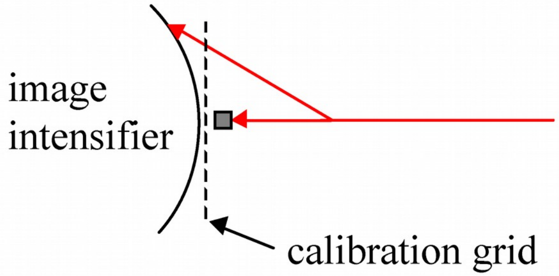


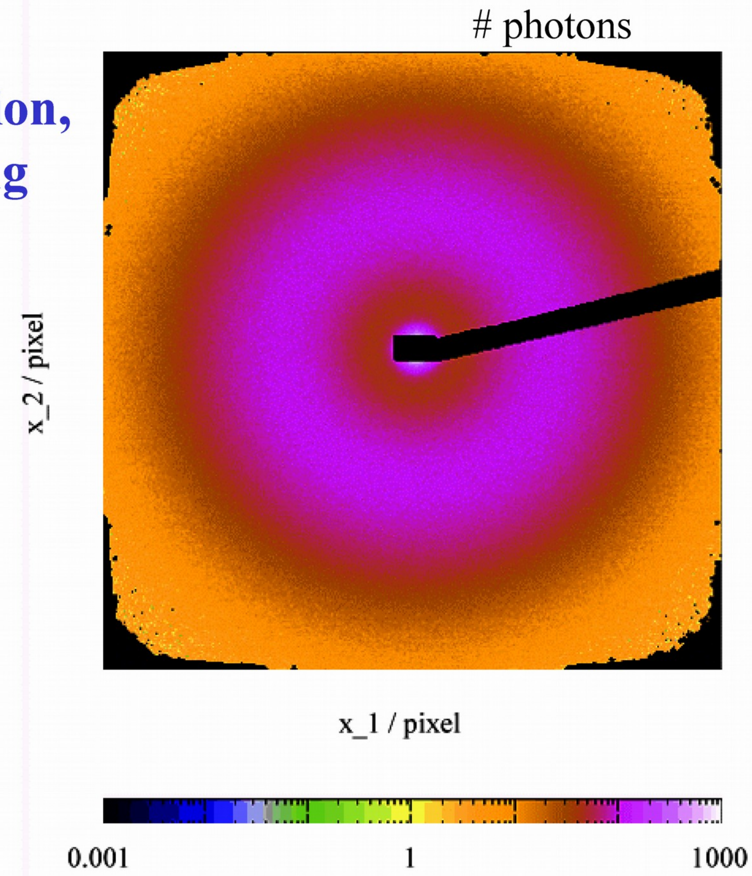



3. raw image - dark image
ADU
signal
0
pixel
ADU
x_2 / pixel
x_1 / pixel
0.001
1
1000

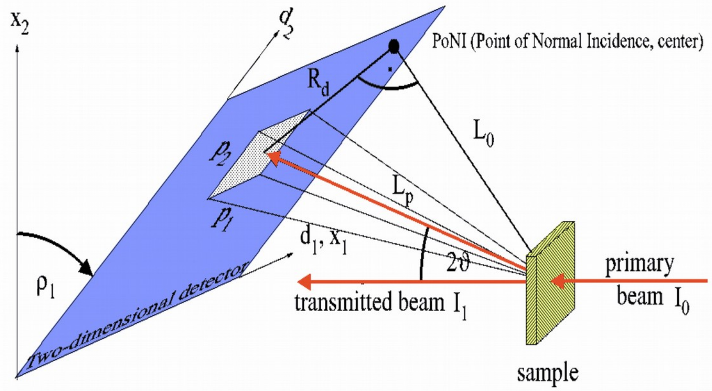
4. spatial distortion correction, division by flatfield, masking

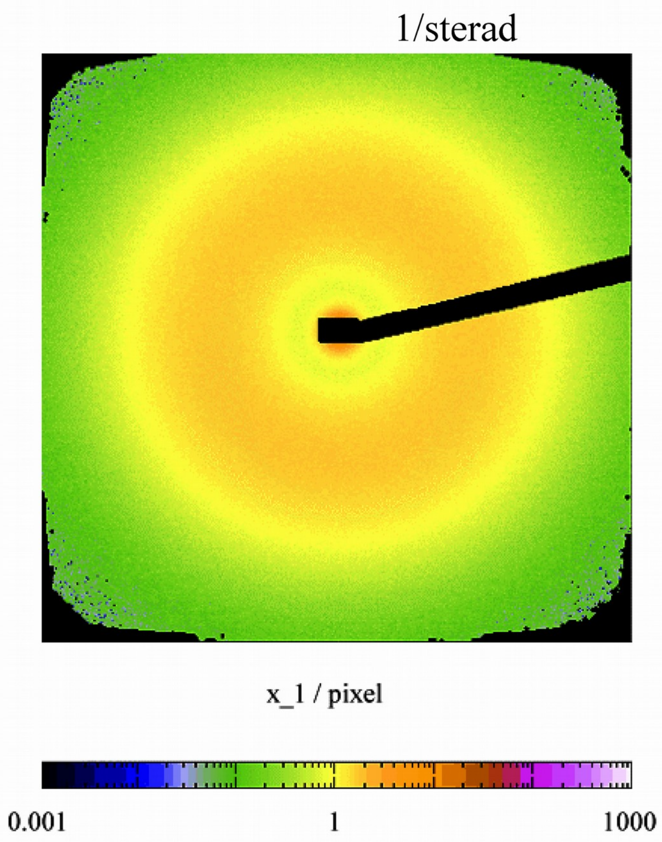




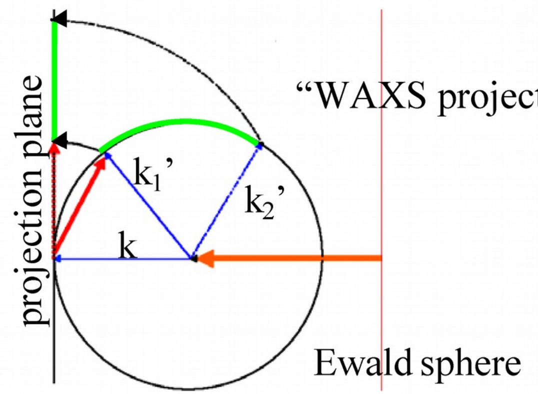


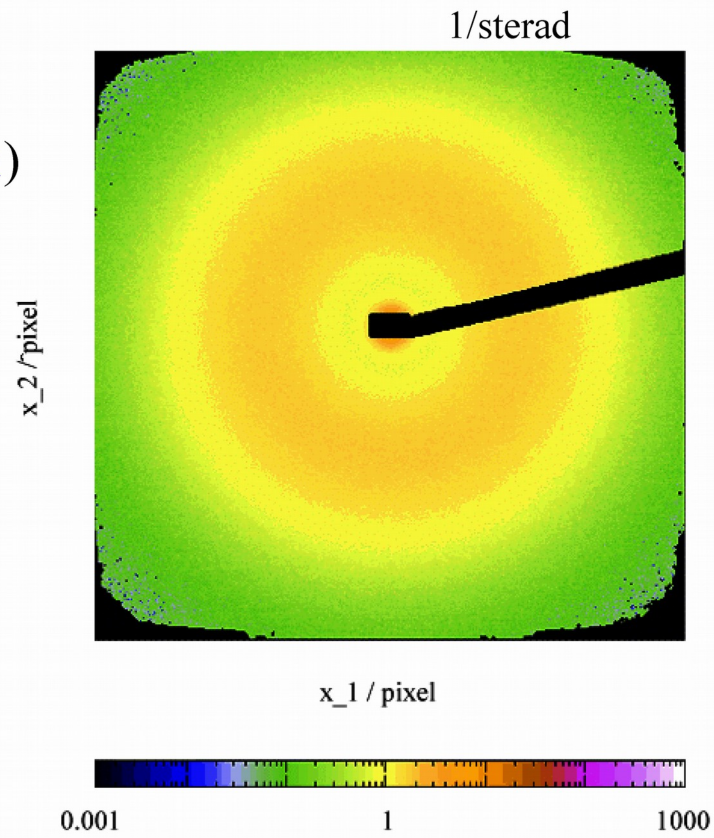
5. normalization to absolute scattering units



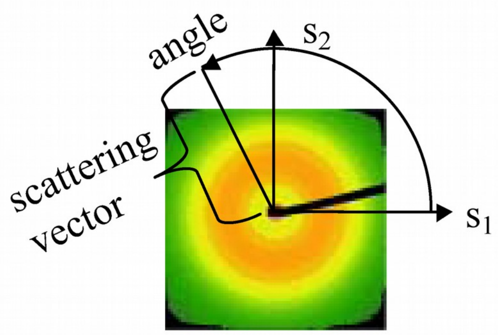


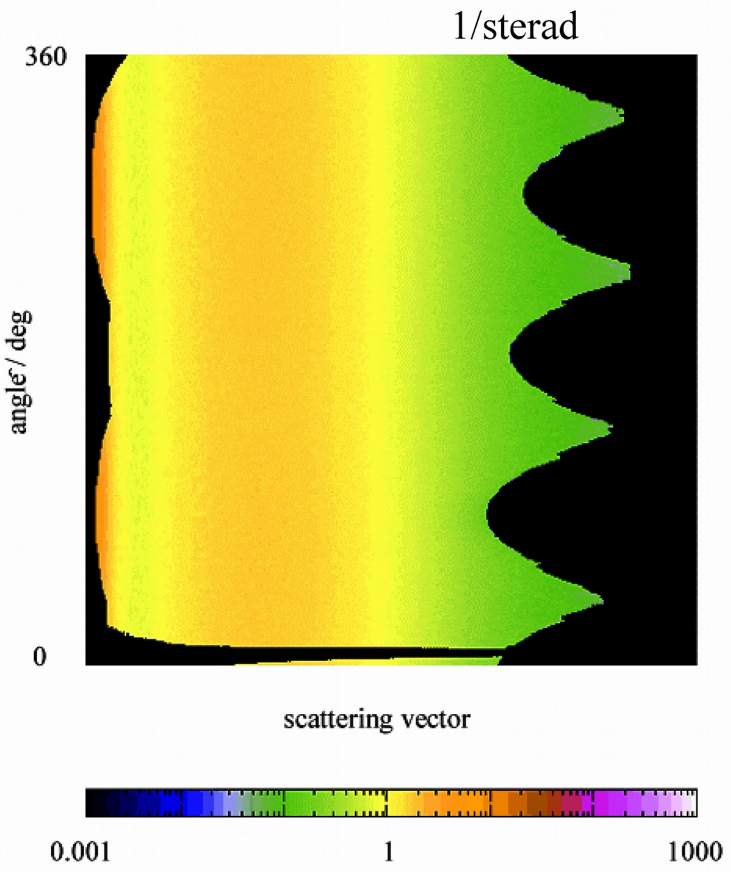
(6. Ewald sphere projection)





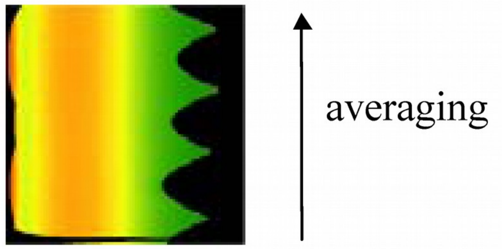
7. azimuthal regrouping

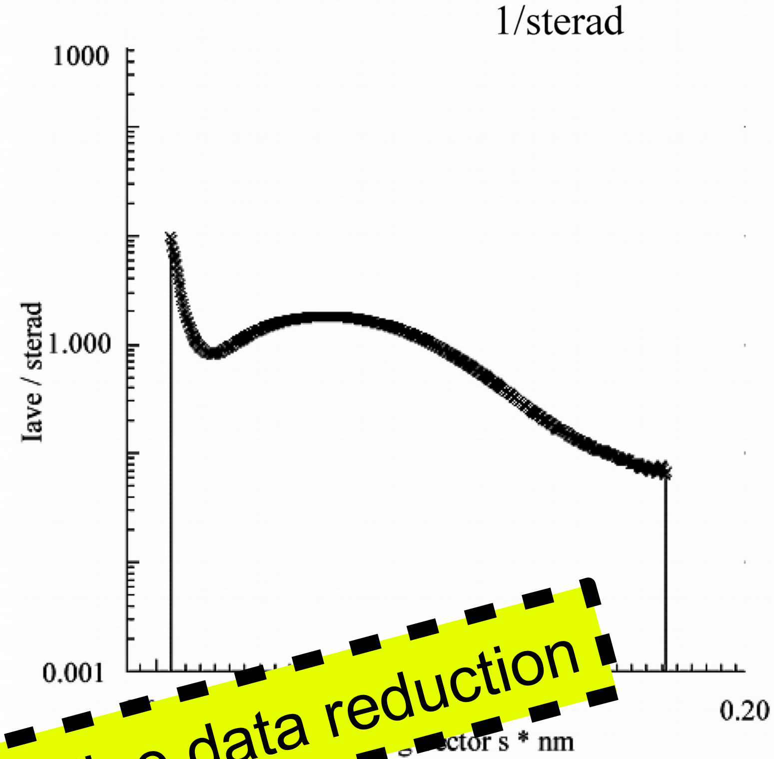




data reduction steps

8. azimuthal averaging

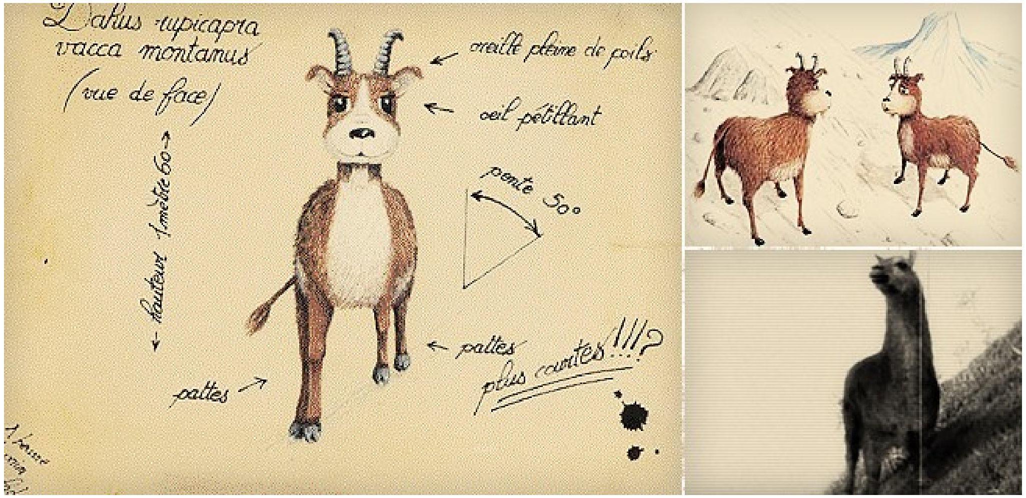




all automatised in online data reduction

+ SPD (saxs programs)

+ DAHU (PyFAI)



→ project for 2016: online variance calculation in PyFAI

http://www.esrf.fr/Instrumentation/software/data-analysis/OurSoftware/SAXS/SaxsDownloads.html

SPD – saxs programs

ID02 online data reduction (SPD) package was developed by P. Boesecke, A. Sole and R. Wilcke during 2001-2005

saxs programs are command line oriented programs (C) to perform certain tasks for data reduction

propagation of statistical errors

GUI is available for most common tasks in SAXSutilities

example:

➢ saxs_normn -p -ofac 1.8/2.75 -omod n +pass +flat -i3nam flat.msk -scal -i1nam .. stp3_%%%.edf,2,39 -onam stp3_%%%.nrm -obin 4 4

Program name	Short description
saxs_mac	standard operations on images, e.g.
	multiply by factor and add constant
saxs_add,	add, subtract, multiply, divide
saxs_sub,	several images
saxs_mul,	
saxs_div	
saxs_normn	flatfield correction and
	normalization of scattering patterns
	to absolute units in 1/sterad
saxs_waxs	waxs projection and backprojection
saxs_angle	transformation to polar coordinates
saxs_row,	row, column projection of images
saxs_col	
saxs_ascii	convert images to ascii
saxs_patch	patch images to other images
saxs_stat	calculate image statistics

P. Bösecke, "Reduction of two-dimensional small- and wide-angle X-ray scattering data," J. Appl. Cryst. 40, 423-427 (2007)

saxs programs

further info:

http://www.esrf.fr/files/live/sites/www/files/Instrumentation/software/data-analysis/OurSoftware/SAXS/SaxsPrograms-V2.459P2.033E2.229.pdf

PETER BOESECKE SaxsPrograms.doc 2013/01/25

P. Bösecke, "Reduction of two-dimensional small- and wide-angle X-ray scattering data," J. Appl. Cryst. 40, 423-427 (2007)

PyFAI - Fast Azimuthal Integration in Python

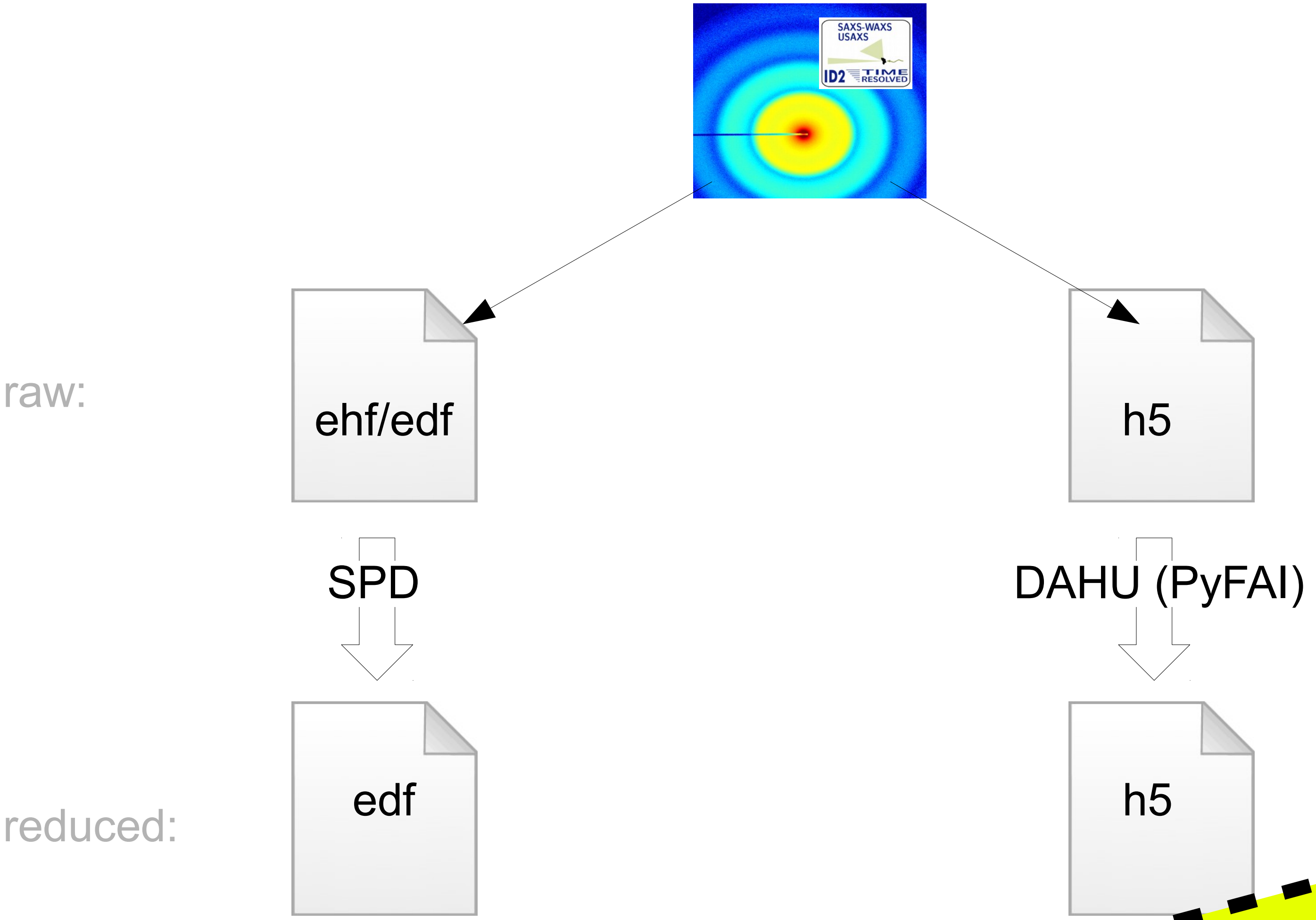
PyFAI is a novel library for azimuthal integration which already provides geometric equivalence with SPD (Bösecke, 2007) and Fit2D (Hammersley et al., 1996).

- designed to reduce SAXS, WAXS and XRPD images into 1D curves (azimuthal integration)
- as a library, the aim of pyFAI is to be integrated into other tools with a clean pythonic interface (e.g. beamline control systems)
- both local and total intensities are conserved in order to obtain accurate quantitative results during the transformation from cartesian space to polar space
- parallelized on graphic cards (GPU)
- shares the geometry definition of SPD
- features also command line tools for batch processing (however not directly compatible with ID02 metadata concept)

[1] J. Kieffer and D. Karkoulis, PyFAI, a versatile library for azimuthal regrouping, Journal of Physics: Conference Series 425, 202012 (2013)

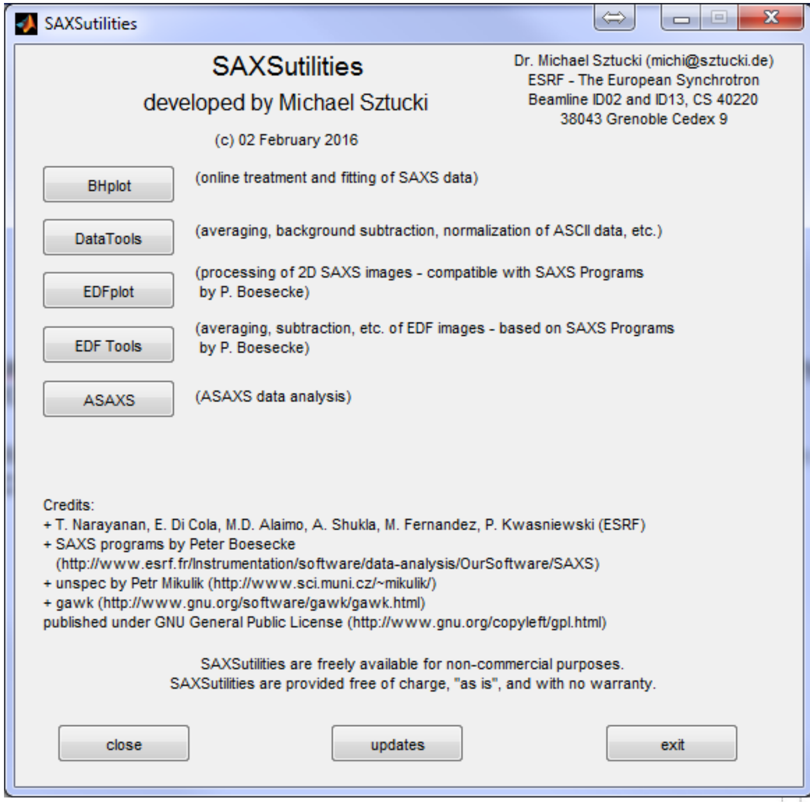
[2] J. Kieffer and J.P. Wright, PyFAI: a Python library for high performance azimuthal integration on GPU, Powder Diffraction 28, S339 (2013)

ID02 online data processing

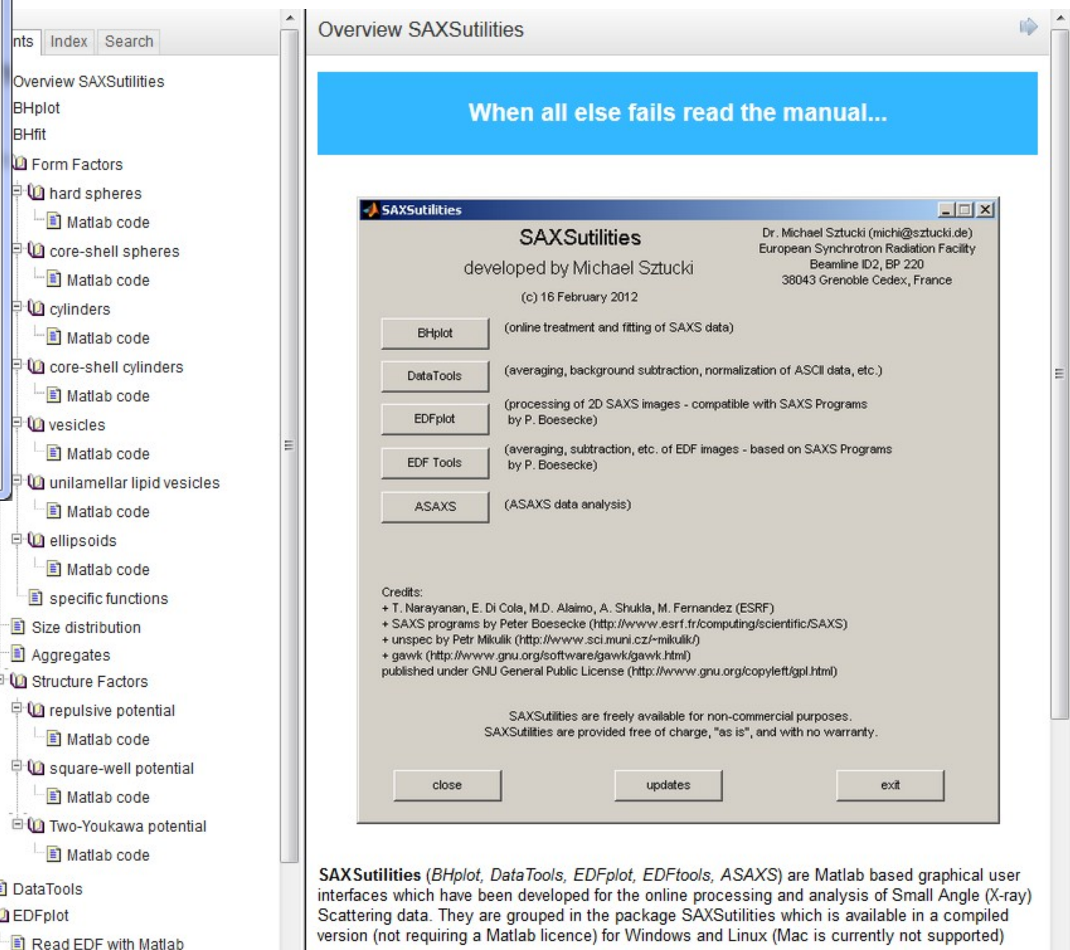


two routes for online data reduction

SAXSutilities



http://www.saxsutilities.eu



SAXSutilities

Get latest Windows version (WindowsXP, Vista, Windows7, Windows8) :

Download and install program files using the installer (64bit): SAXSutilitiesSetup64.exe
Do not forget to install the Matlab runtime environment!!
You will be asked at the end of the installation routine to download and install this runtime environment. The installation has to be done only once.

Download 32bit version: SAXSutilitiesSetup32.exe
This version might not support all features of the 64bit version!!

Get latest Linux version *(64-bit only!)* (developed under Debian6):

New procedure since October 2013 (Matlab R2013b) !!!

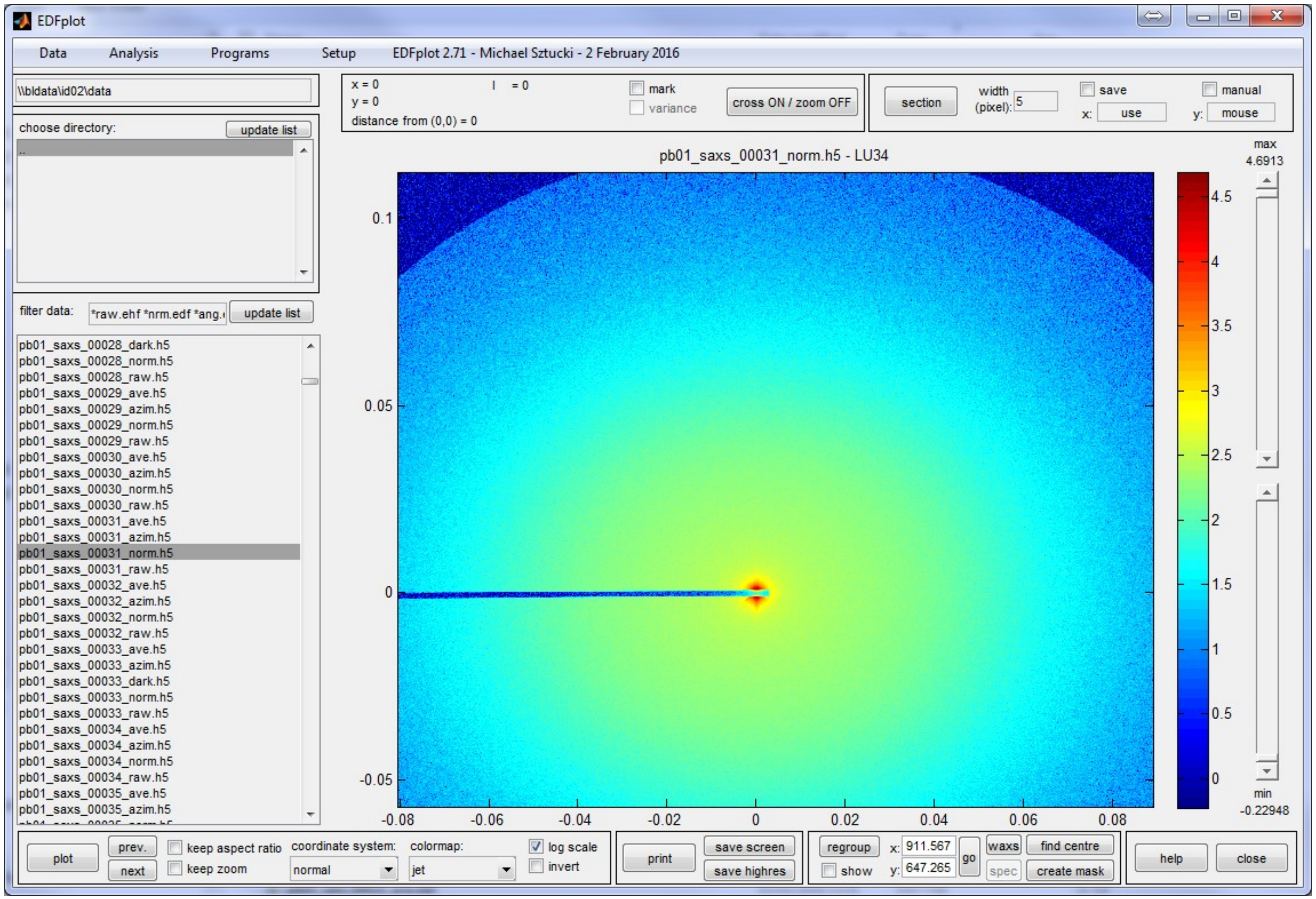
1. Download and unpack to a folder of your choice: saxsutilities.zip
   `> unzip saxsutilities.zip`
2. Download and unzip: MCRInstaller2013b_glnxa64.zip
   `> unzip MCRInstaller2013b_glnxa64.zip`
   Then, run the MCR Installer script from the directory where you unzipped the package file by entering:
   `> ./install`
   Add the environment variables **LD_LIBRARY_PATH** and **XAPPLRESDIR** to your system as indicated in the last step of the installation procedure.
   Note *that this has to be done only once for each new release of Matlab.*
3. Add an environment variable **SAXSUTILITIESPATH** to your system which points to the directory created in step 1.
4. Note that unspec by P. Mikulík has to be installed.
5. Note that saxsprograms by P. Bösecke have to be installed.
6. Note that roca by P. Bösecke has to be installed. (Since version saxs_V2.461P2.043E2.236R1.500 included in saxsprograms)
7. Start SAXSutilities from the folder created in step 1.

   You can also use a start script like:

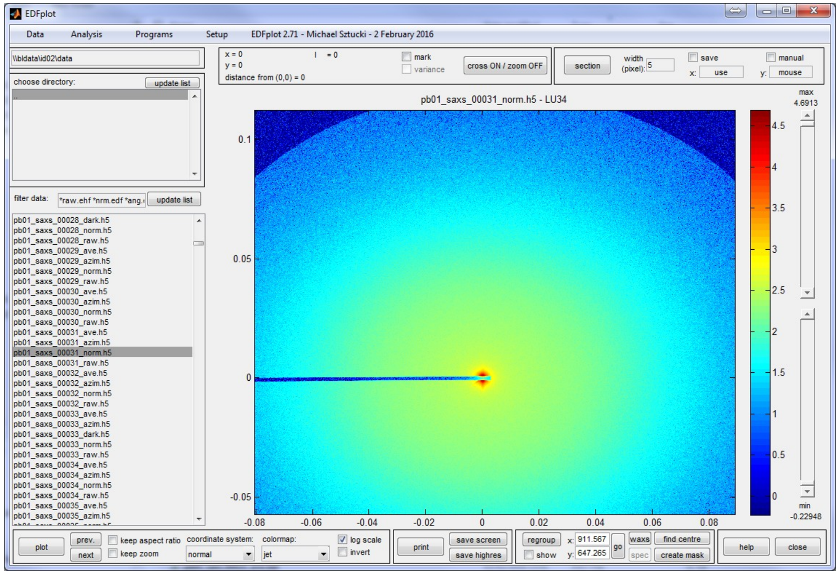
```
#!/bin/csh
setenv LD_LIBRARY_PATH [...as indicated by MCRInstaller...]
setenv XAPPLRESDIR [...as indicated by MCRInstaller...]
setenv SAXSUTILITIESPATH [/path/to/directory/of/SAXSutilities]
[/path/to/directory/of/SAXSutilities]/SAXSutilities
```

http://www.saxsutilities.eu

SAXSutilities – 2D visualisation (EDFplot)



SAXSutilities – 2D visualisation (EDFplot)



Saxs COORDINATES < ↯ > Saxs PROJECTION

Saxs PROJECTION (scattering pattern on a flat 2D detector)

```
                  (distance of pixel from poni on detector)  wavelength0
SAXS coordinate = ----------------------------------------* -----------
                  (distance of poni from sample)            wavelength
                                                  where wavelength0 is 1e-9 m
```

SAXS(Saxs) coordinate ~ s*nm for 2Theta<<1

Waxs PROJECTION (projection of the scattering pattern from the EWALD sphere to a plane perpendicular to the incident beam)

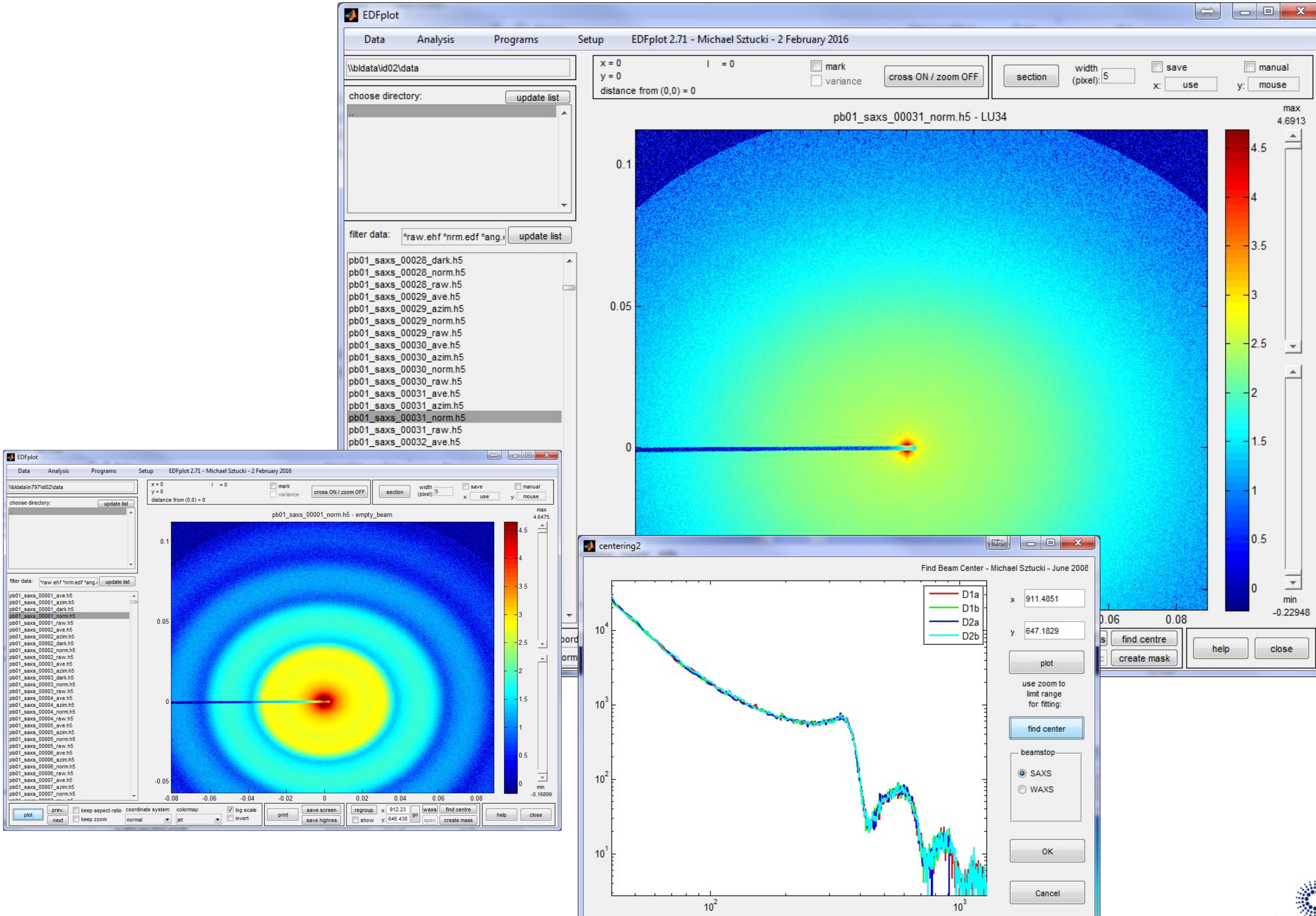
In the Waxs projection, the scattering pattern of the detector is distorted in such a way that the SAXS coordinate of the new pattern is equal to s:

SAXS(Waxs) coordinate = s*nm

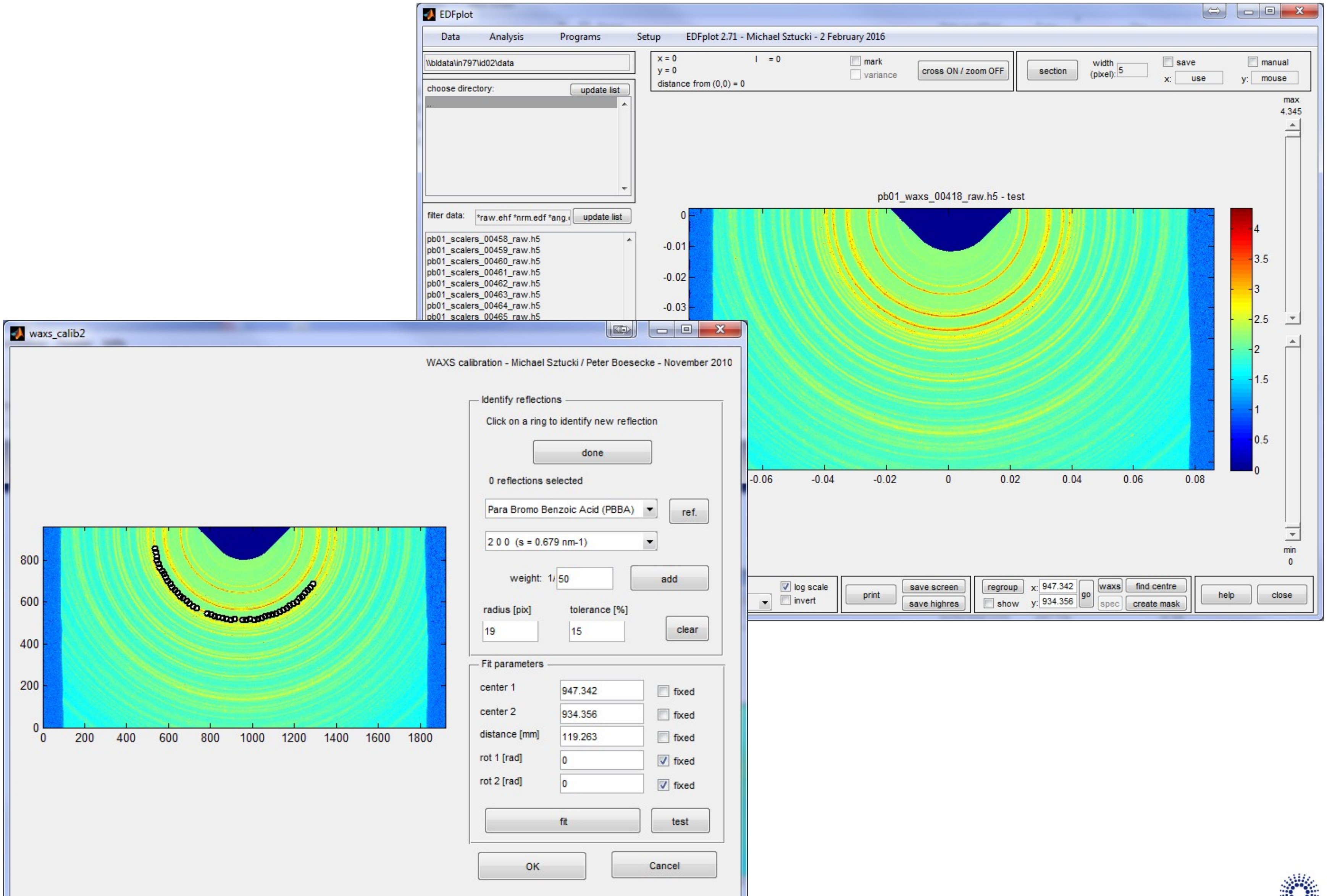
In Waxs PROJECTION the scattering pattern of a tilted detector can be geometrically analysed (azimuthal regrouping etc.) like a small angle scattering pattern.

Reference system	Applied affine coordinate transformation
Array	$f_A = f$
Image	$f_I = \text{Offset} + f$
Region	$f_B = (\text{Offset} + f) \times \text{BSize}$
Real	$f_R = (\text{Offset} + f) \times \text{PSize}$
Center	$f_C = \text{Offset} - \text{Center} + f$
Normal	$f_N = (\text{Offset} - \text{Center} + f) \times \text{PSize}$
Saxs	$f_S = \text{Normal}(\text{Offset},\text{Center},\text{PSize}, f)/\text{SampleDistance}/(\text{WaveLength}/\text{nm})$

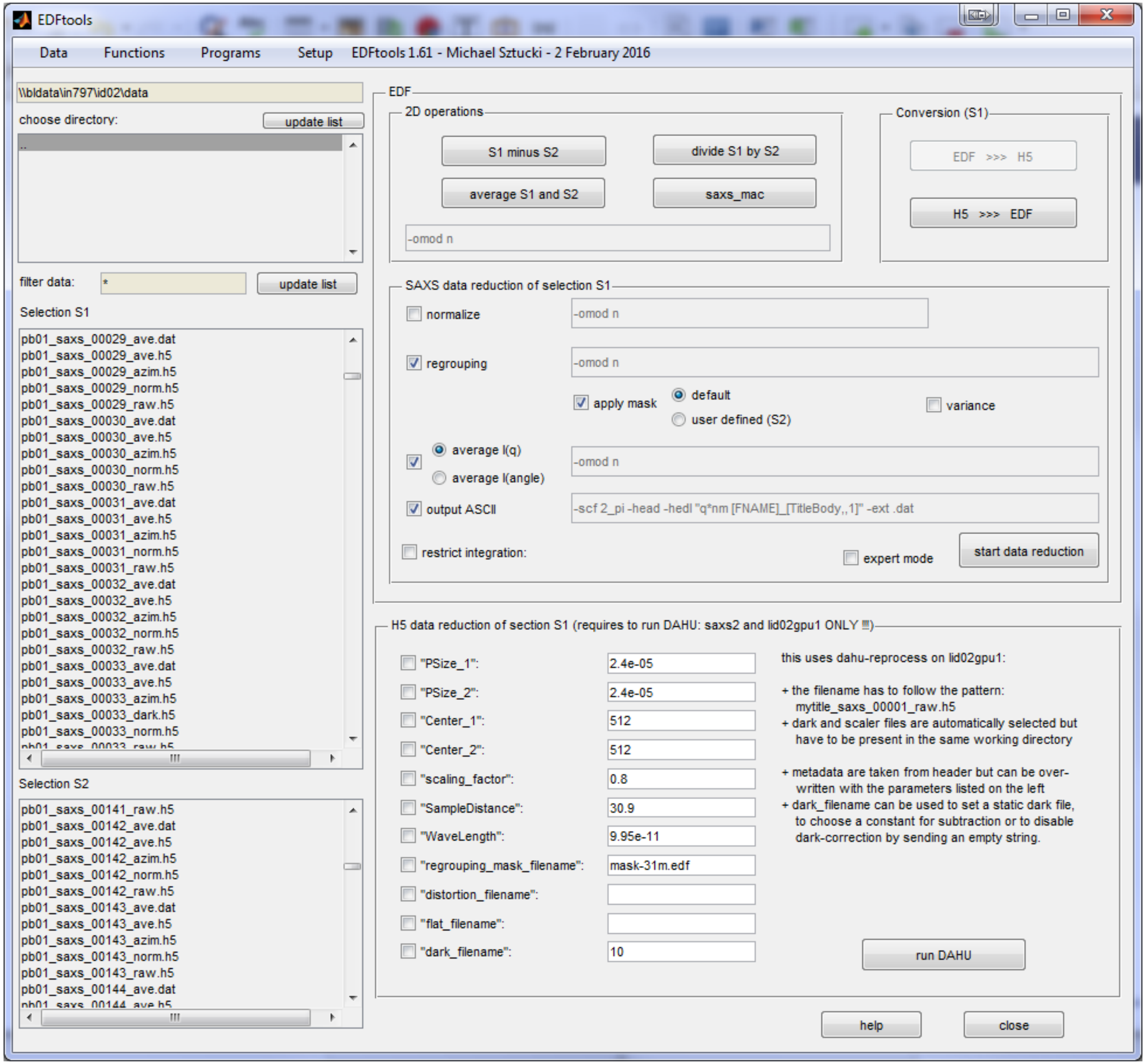
SAXSutilities – 2D visualisation (EDFplot)



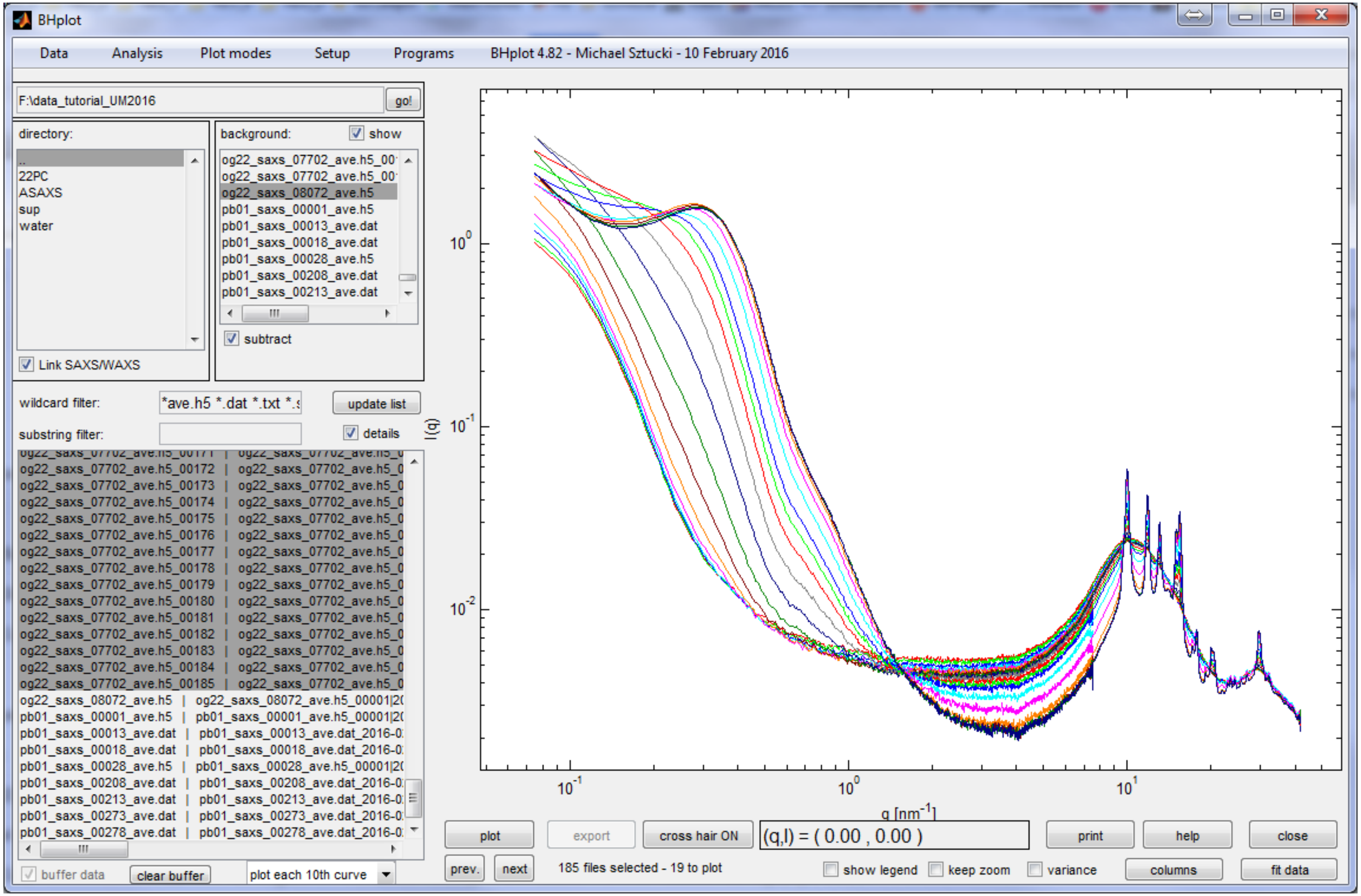
SAXSutilities – 2D visualisation (EDFplot)



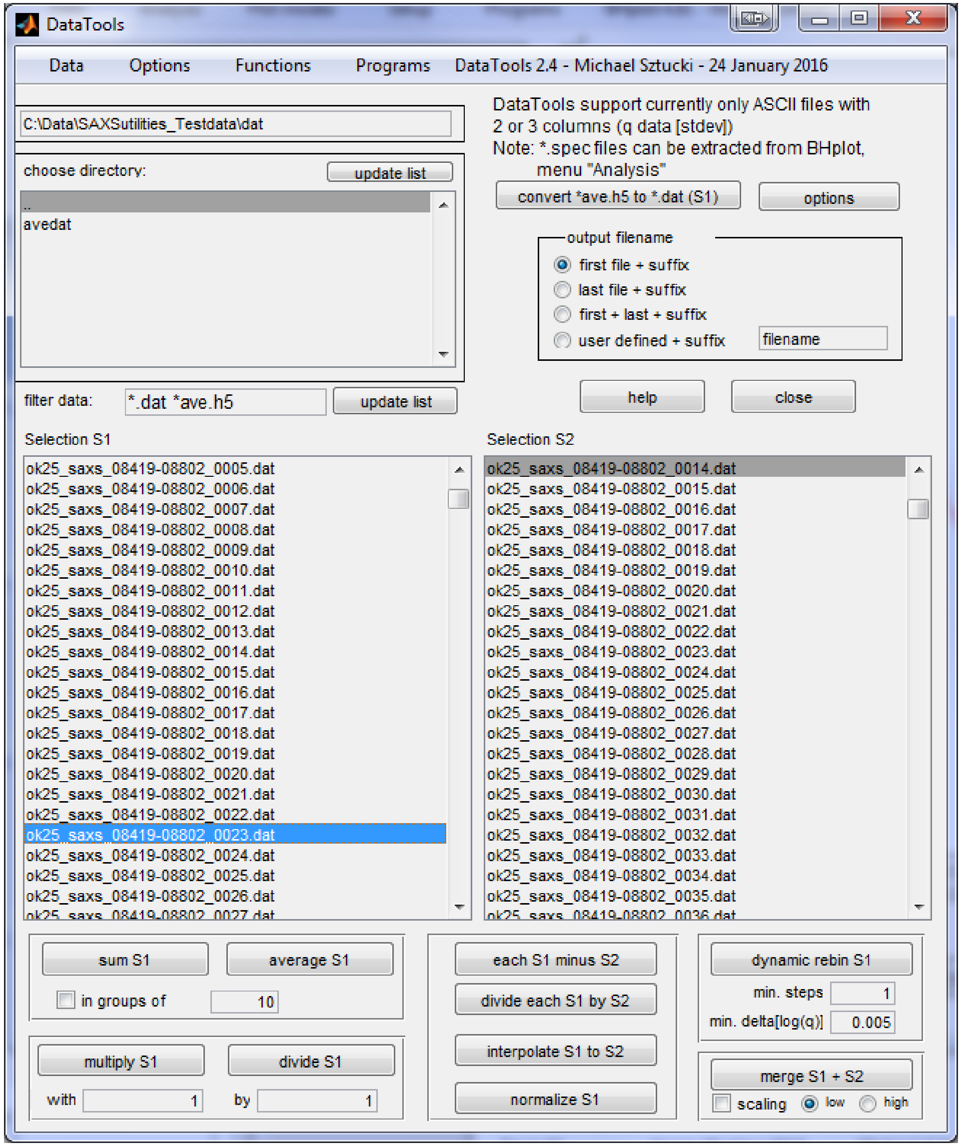
SAXSutilities – 2D tools (EDFtools)



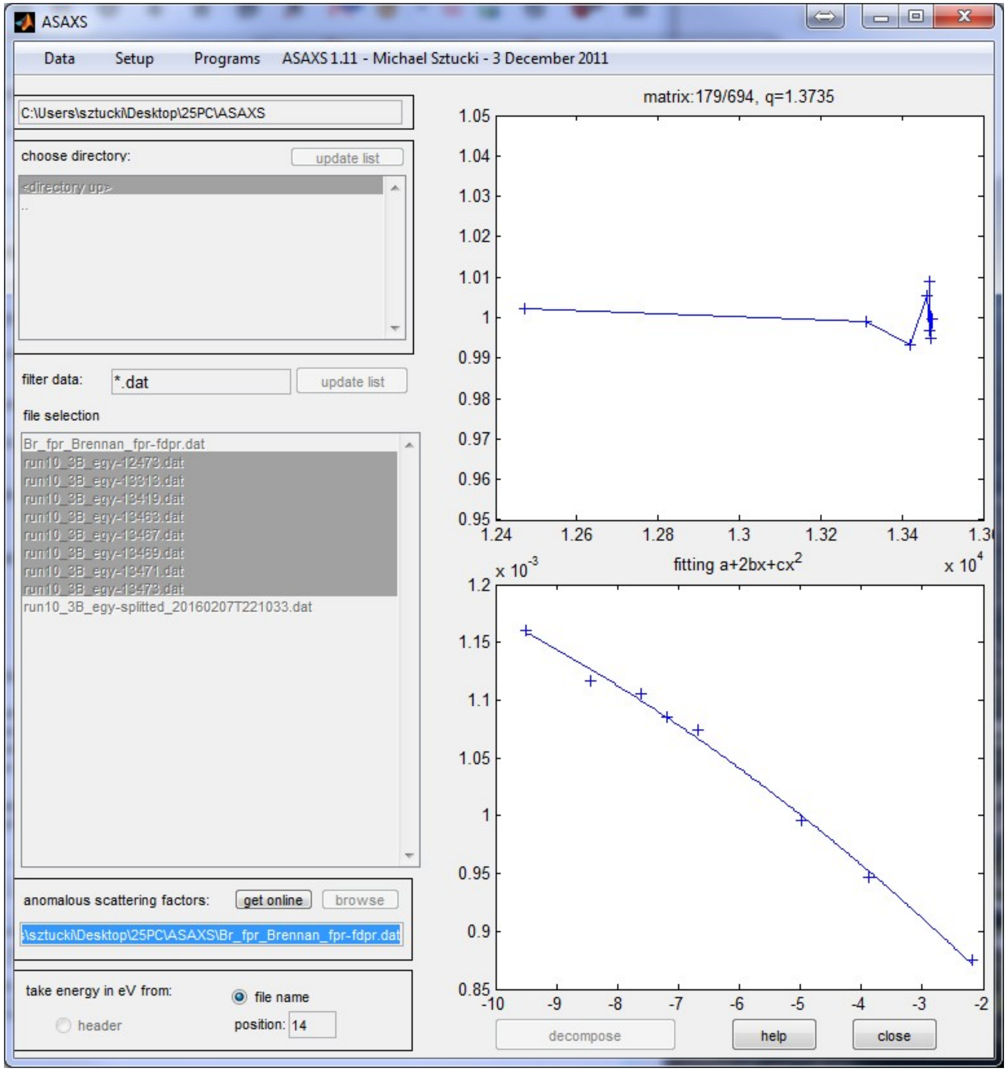
SAXSutilities – 1D visualisation (BHplot)

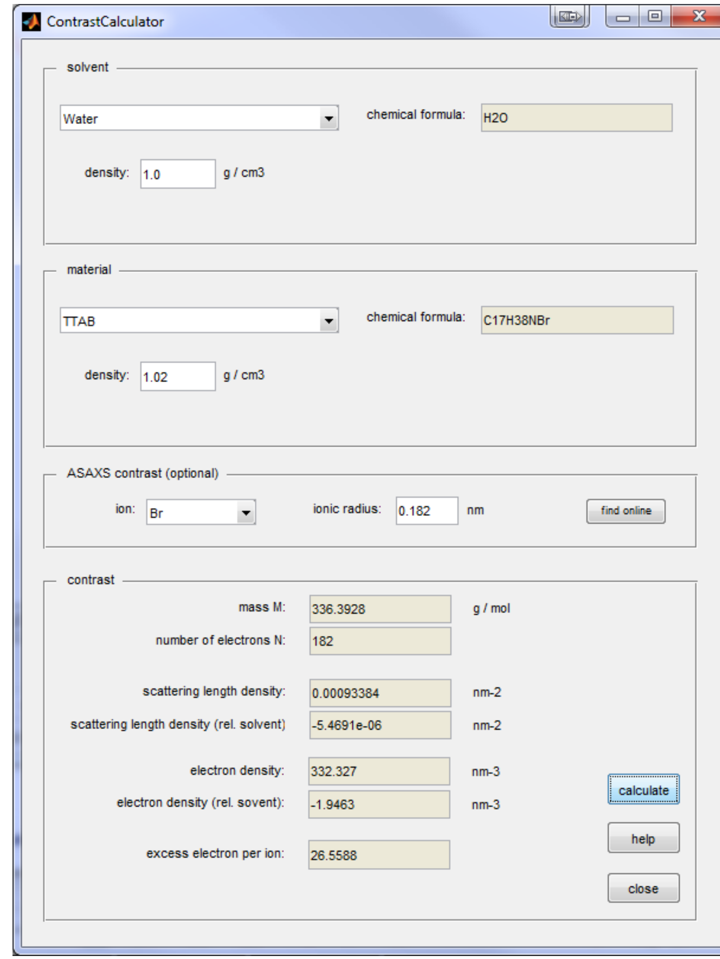


SAXSutilities – 1D tools (DataTools)



SAXSutilities – special routines (ASAXS)





X-ray scattering factor of a single counterion:

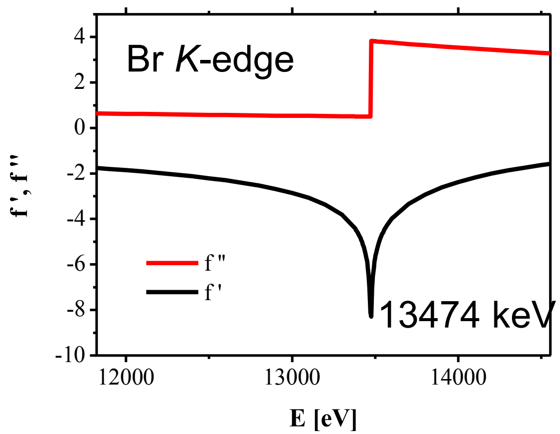
$$\Delta f(E) = \Delta f_0 + f'(E) + if''(E)$$

Energy dependent scattering length density profile:

$$\Delta\rho(r,E) = v(r) r_e \Delta f(E)$$

Scattering amplitude:

$$F(q,E) = F_0(q) + [f'(E) + if''(E)] v(q)$$



$$I(q,E) = F_0^2(q) + 2f'(E)F_0(q)v(q) + [f'^2(E) + f''^2(E)] v^2(q)$$

Total Intensity

Intensity measured far below the edge ≈ SAXS-Intensity

“Cross-term”

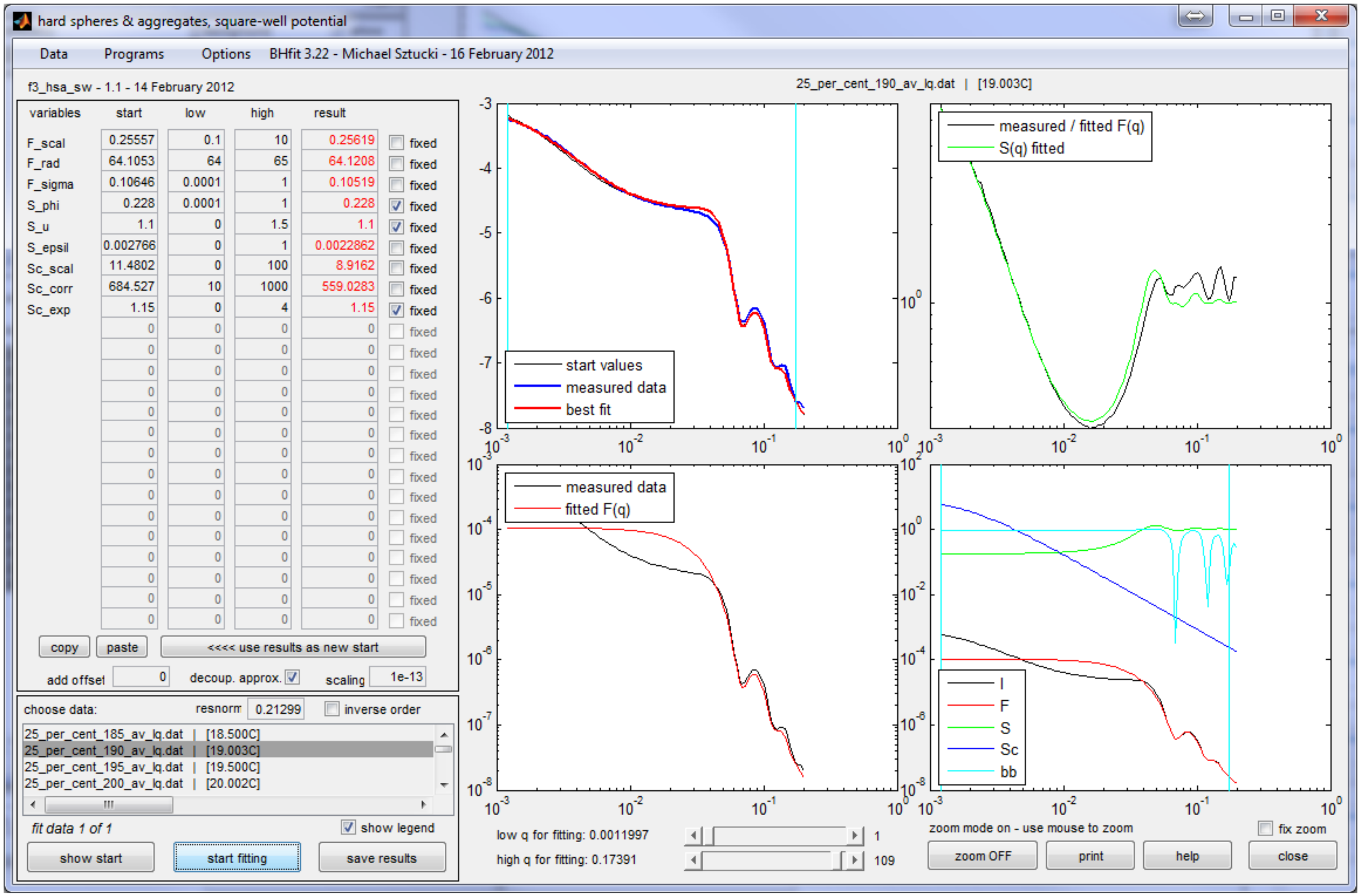
“Self term”of the counterions

⏩⏩ radial distribution of

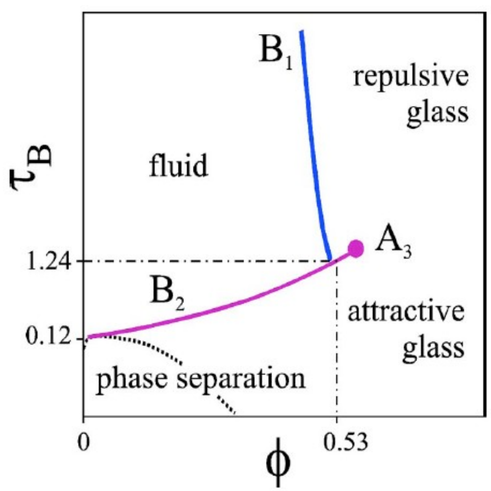
$F_0(r)$: non-resonant scattering units (macroion + constant part of counterions

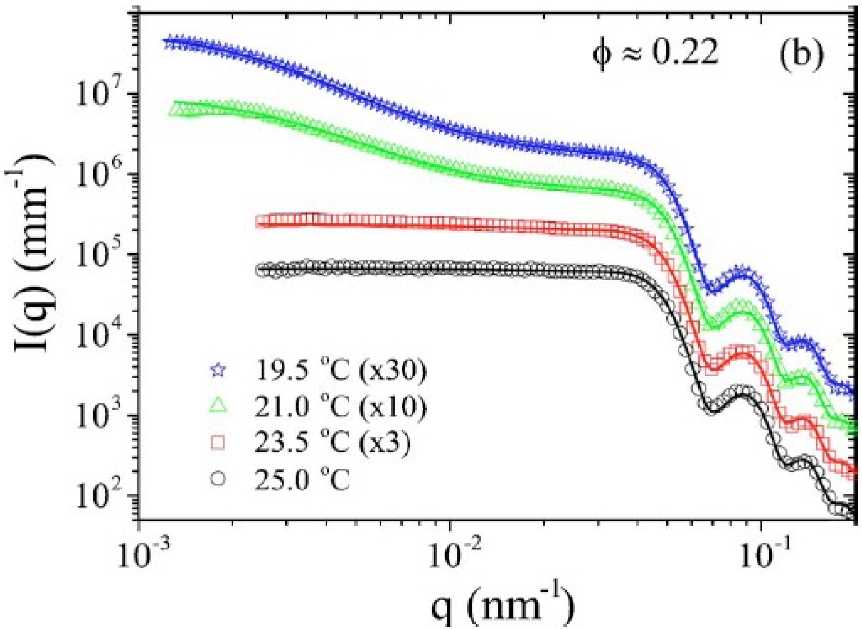
$v(r)$: resonant scattering units (radial distribution of counterions)

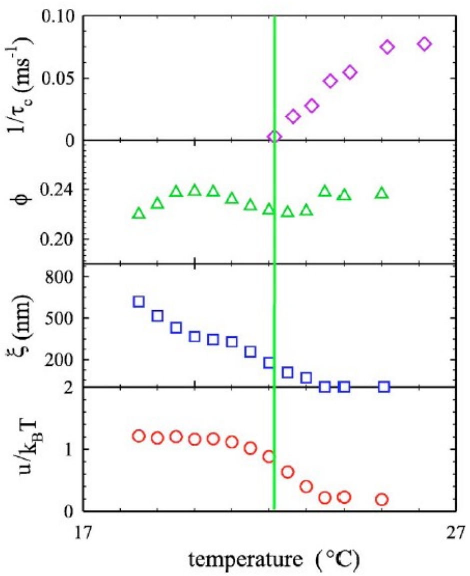
SAXSutilities - modelling



SAXSutilities - modelling







PHYSICAL REVIEW E **74**, 051504 (2006)

Kinetic arrest and glass-glass transition in short-ranged attractive colloids

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(Received 6 July 2006; published 17 November 2006)

A thermally reversible repulsive hard-sphere to sticky-sphere transition was studied in a model colloidal system over a wide volume fraction range. The static microstructure was obtained from high resolution small angle x-ray scattering, the colloid dynamics was probed by dynamic x-ray and light scattering, and supplementary mechanical properties were derived from bulk rheology. At low concentration, the system shows features of gas-liquid type phase separation. The bulk phase separation is presumably interrupted by a gelation transition at the intermediate volume fraction range. At high volume fractions, fluid-attractive glass and repul-

Conclusions

Online data reduction and analysis are crucial for the productivity of any Small-Angle X-ray Scattering instrument especially when dealing with large amount of data or taking decision during a time-resolved experiment or radiation damage testing. The developed programs and tools do not only work at the ESRF beamline, but can also be installed at the home laboratory for more detailed data analysis.

http://www.esrf.fr/Instrumentation/software/data-analysis/OurSoftware/SAXS/SaxsDownloads.html

Acknowledgements

P. Boesecke, A. Sole, R. Wilcke, J. Kieffer

T. Narayanan and all present and former ID02 staff

BCU (beamline control unit), DAU (data analysis unit)

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