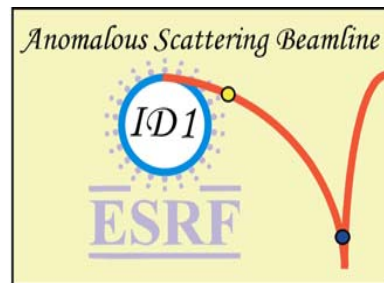# SAXS at the ESRF

Beamlines ID01 and ID02

Peter Boesecke

European Synchrotron Radiation Facility, Grenoble, France (boesecke@esrf.eu)

Contents

- History

- Current Situation

- Online/Offline Treatment (SAXS package/SPD program)

1 History

before 1994: Discussion of a common ESRF data format for all beam lines based on existing solutions,
e.g. hdf (->:not handy), ORACLE (->too expensive)

=> implementation of a home made "ESRF Data Format" (edf), allowing storage of metadata

1994: ID02 went into operation using a 2D gas-filled detector (1024x1024 pixels) using an ad hoc hardware format ("hm") using two files for data and SAXS metadata, but no easy way of data processing.

=> "ESRF Data Format" implemented + some simple programs

=> Offline conversion of "hm"-files to edf-files (including SAXS metadata)

1995: First version of "SAXS" data treatment programs for edf-files + SAXS metadata

1995 onwards: Ongoing discussions about metadata, improvements of the format.

~1999: To facilitate programming the edf-format is generally introduced at the ESRF, for **automatic data processing** the SAXS metadata becomes a standard at ID02.

since 1999: development of automatic offline/online data processing tools (spd, "SAXS"-programs)

# 2 Current Situation

ESRF SAXS Beamlines

- 🔴 **ID01** SAXS, WAXS, ASAXS, GISAXS, GID, XD, micro focusing, diffuse scattering, coherent scattering, imaging, reciprocal space mapping, (hard + soft matter)
- 🔴 **ID02** time-resolved SAXS/WAXS, ASAXS, USAXS (soft matter + biologic materials)
- **BM02** anomalous SAXS, WAXS, XD (hard + soft matter)
- 🔴 **ID09B** ps-time resolved protein crystallography (PX) (biologic materials)
- **ID10** SAXS, WAXS, GISAXS, XD, coherent scattering, XPCS (soft matter)
- 🔴 **ID11** high energy time-resolved XD, WAXS (material science)
- **ID13** micro focus, scanning diffractometry, SAXS, WAXS, GISAXS, PX (soft matter + biologic materials)
- **ID14/EH3** prospected SAXS station for biologic materials
- ⭕ **BM16** SAXS, WAXS, PX (soft matter + biologic materials)
- ⭕ **BM26** time-resolved SAXS/WAXS, GID, EXAFS, PX, ...

The list of beamlines and their experimental methods are not complete. There are more beamlines performing SAXS or WAXS experiments from time to time.

2 Current Situation

Beamline User Requirements

- The **productivity** of the beamline (#publications) increases when the data is processed immediately.

- Most of the users need **corrected data on-line** during acquisition to judge data quality.

- There is currently **no strong demand** by external users for a common data format.

- After the first experiment most of the users have found a way to analyze the data.

- The strongest demand for a common data format seems to come from **software developers**. Currently, most of the users seem to be happy with specific solutions.
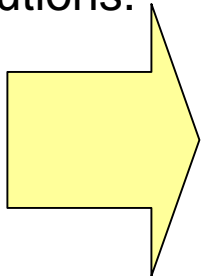
A homogenization of data formats can be triggered by standard programs, e.g. for online correction, visualization etc.

3 Online/Offline Treatment

Expected Result of Online Correction (Absolute Units)

No Pol. Corr.: Pixel intensities I normalized to:

$$I = \frac{1}{A}\frac{\partial \sigma}{\partial \Omega} = \frac{1}{T}\frac{\#scattered\_photons \Big/ sterad}{\#incident\_photons}$$

scattering intensity per scattering cross section [1/sterad] (normalized to T=1)

$$\frac{1}{V}\frac{\partial \Sigma}{\partial \Omega} = \frac{1}{t}\left(\frac{1}{A}\frac{\partial \sigma}{\partial \Omega}\right) = \frac{I}{t}$$

(*T and t can only be measured easily if sample is a flat plate*)

scattering intensity per scattering volume [1/sterad/m]

I: pixel intensity, A: sample cross section, t: sample thickness, V: scattering volume, Ω: spherical angle, T: sample transmission, σ: absolute scattering cross section, Σ: specific scattering cross section
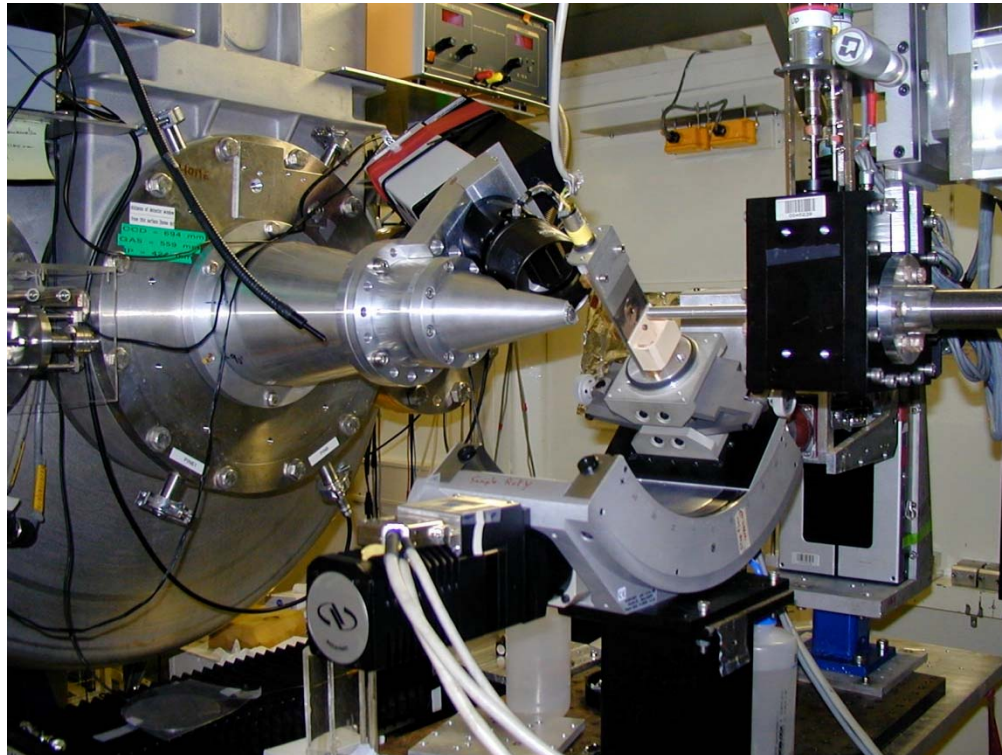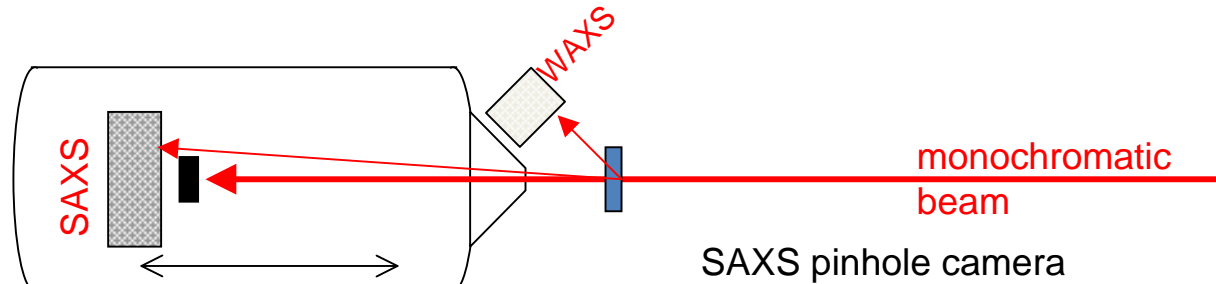
With Pol. Corr.: Pixel intensities I normalized to:

$$\frac{1}{t}\left(\frac{1}{A}\frac{\partial \sigma}{\partial \Omega}\Big/\frac{\partial \sigma_{electron}}{\partial \Omega}\right) = \frac{\#number\_of\_electrons}{scattering\_volume}$$

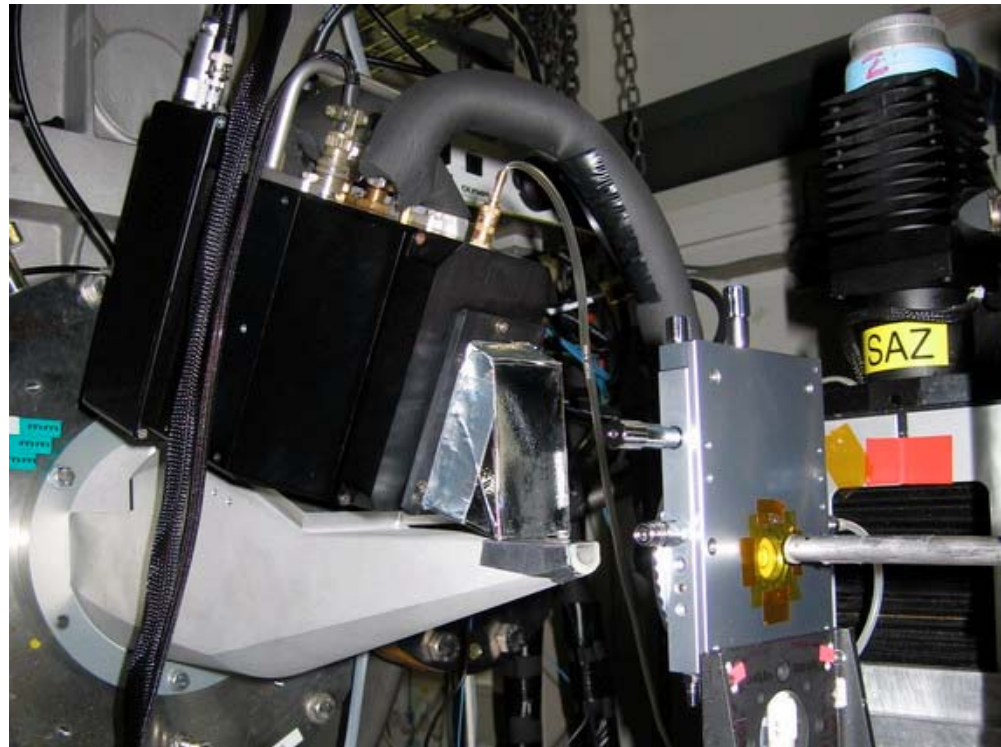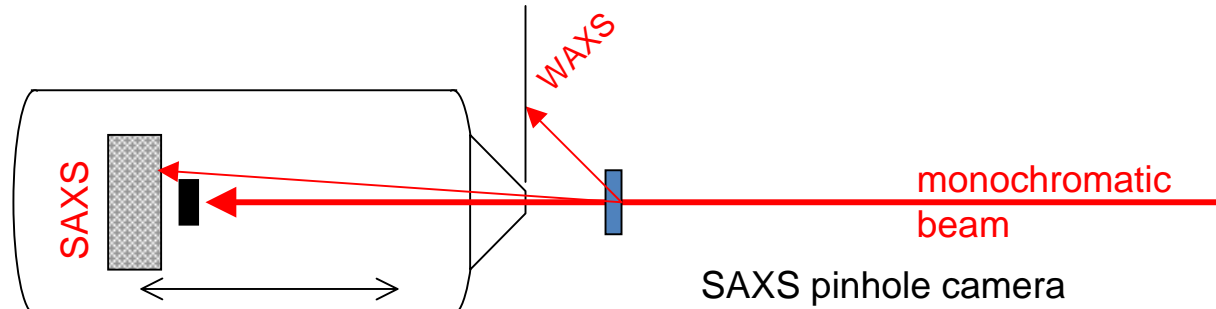number of electrons per sample volume [#e⁻/nm³] (normalized to T=1)

3 Online/Offline Treatment
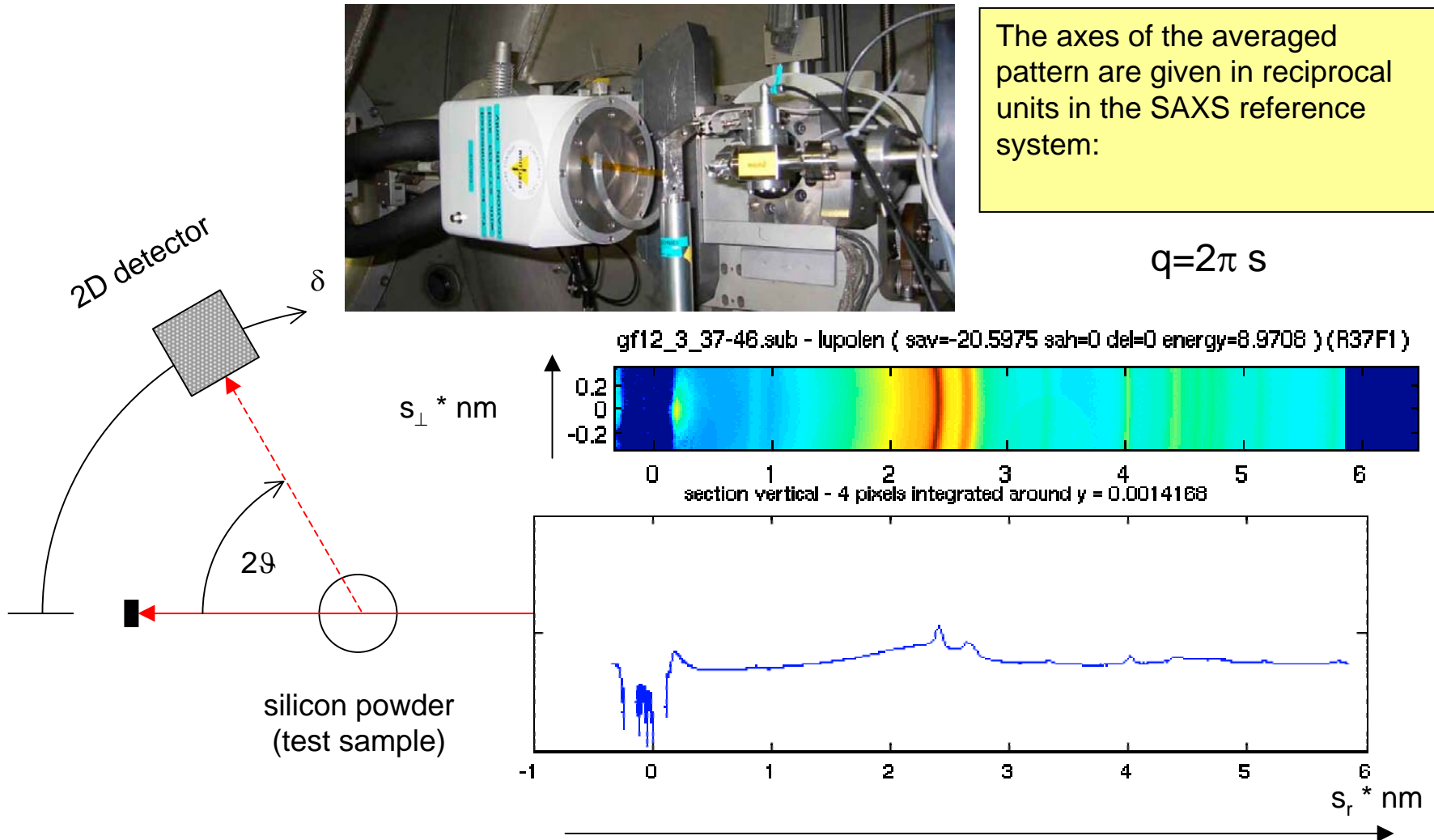
General SAXS/WAXS Scattering Geometry

SAXS pinhole camera

SAXS-WAXS
USAXS

ID2 TIME RESOLVED

ESRF

3 Online/Offline Treatment

General SAXS/WAXS Scattering Geometry

WAXS

SAXS

monochromatic beam

SAXS pinhole camera

SAXS-WAXS
USAXS

ID2 TIME RESOLVED

SAZ

ESRF

3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (WAXD Example)

The axes of the averaged pattern are given in reciprocal units in the SAXS reference system:

$$q=2\pi\,s$$

2D detector δ

$s_\perp$ * nm

2ϑ

silicon powder
(test sample)

gf12_3_37-46.sub - lupolen (sav=-20.5975 sah=0 del=0 energy=8.9708)(R37F1)

section vertical - 4 pixels integrated around y = 0.0014168

s_r * nm

3 Online/Offline Treatment

General SAXS/WAXS Scattering Geometry

A, B, C: X-ray detectors

WAXS

C

B

A at

large   small

distance

additional detectors
(e.g. microscopes, fluorescence detectors,
thermocouples, strain gauges, spectrometer etc.)

I1-monitor

sample

I0-monitor

monochromatic
X-ray beam

beam stop

SAXS

frame number

experiment control
(SPEC)

name_0_0001.edf   (SAXS)
name_1_0001.edf   (WAXS)
...
name_0_0999.edf
name_1_0999.edf
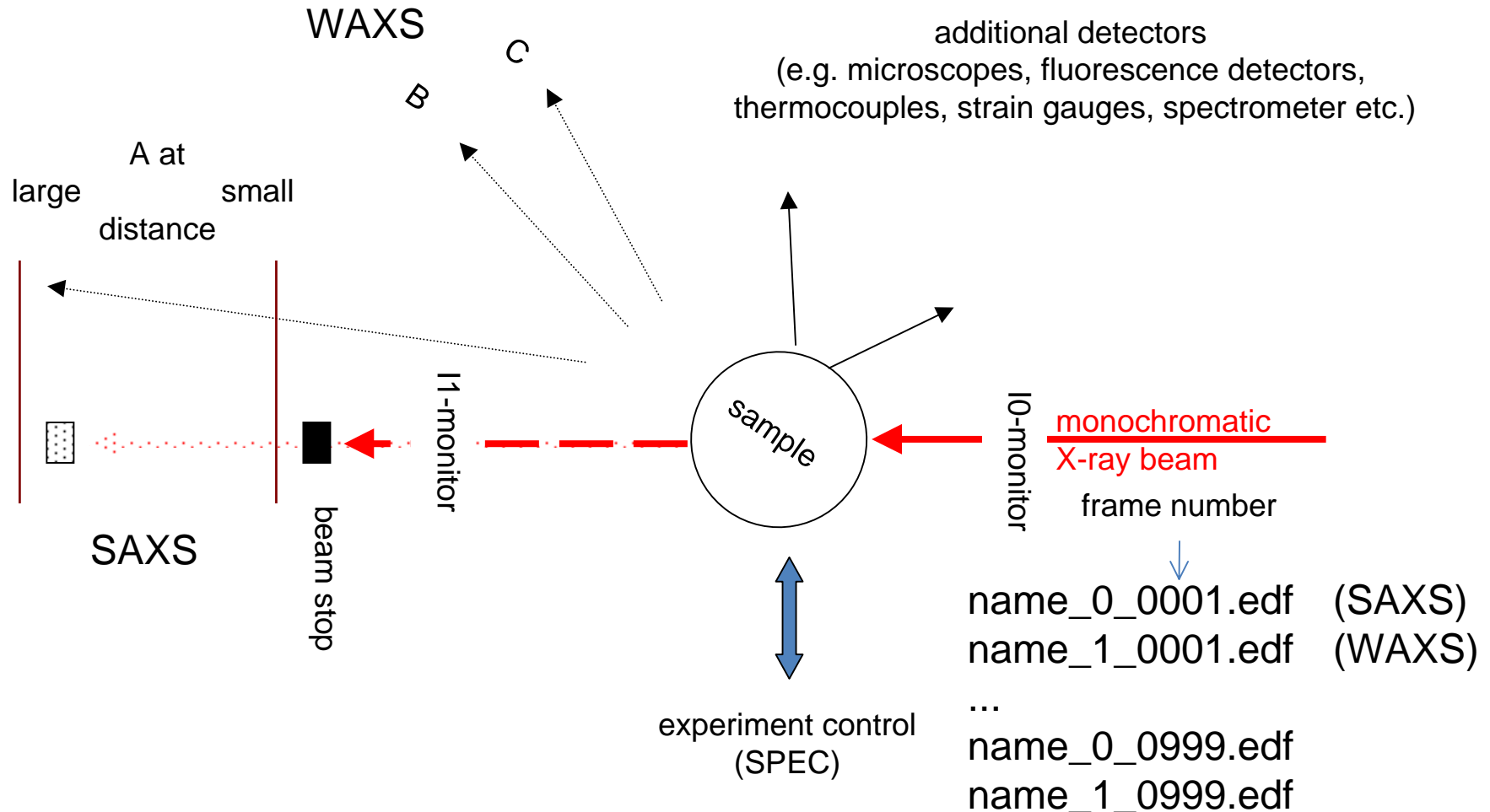
3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (General Considerations)

- Time resolved 2d data from SAXS and WAXS detectors
- Peak data rates -> 40 Megabytes/s
- Increasing amount of data (GB -> TB)

- Detector corrections are only obvious to specialists
- Data quality can only be estimated after basic corrections
- Data must be reduced before it can be used
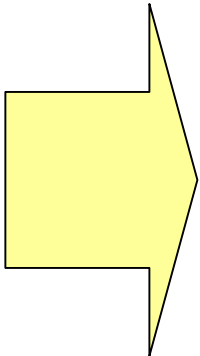
- Raw data is saved with all experimental information
- Data is automatically reduced after an exposure
- History of data treatment is saved
- Corrected data is given in absolute units (1/sterad)

3 Online/Offline Treatment

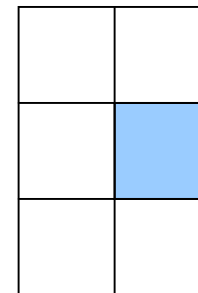Detector Orientations and Geometries

Orientations
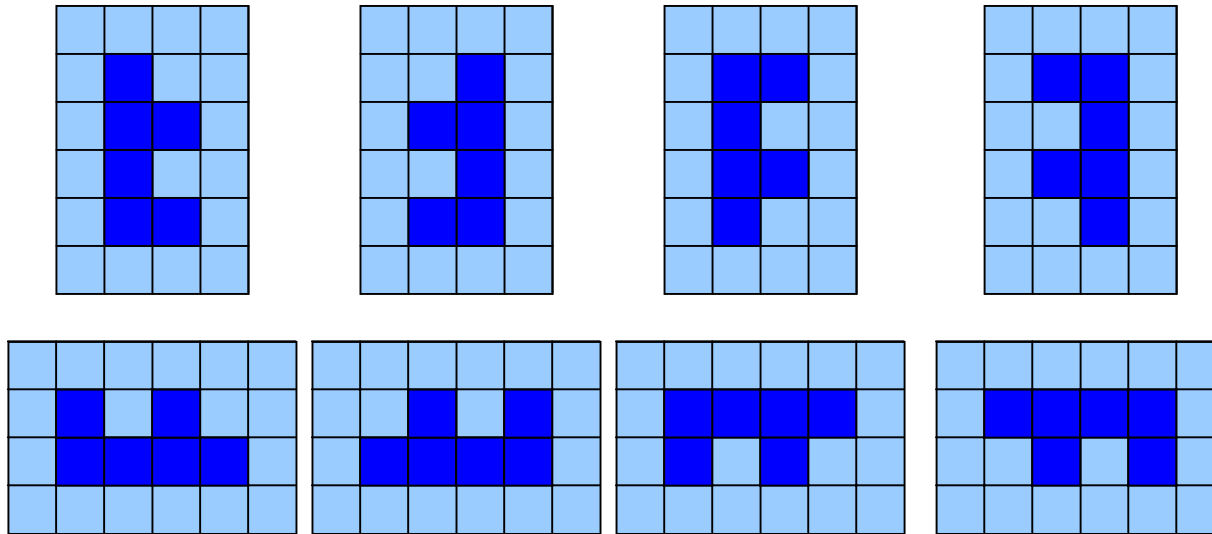
Geometries
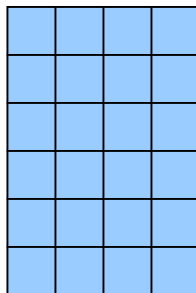
a) full unbinned detector   b) region of interest   c) binned
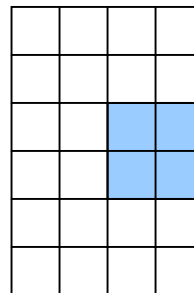
3 Online/Offline Treatment

Detector Specific Corrections

Corrections

dark-image

flat-field

spatial distortion

subtraction of
zero level

normalization of
quantum efficiency
of each pixel

spatial distortion
correction

after these corrections

Each corrected pixel contains the
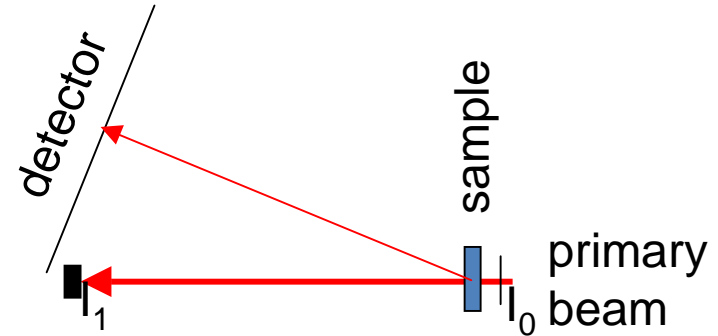number of incident photons (+statistical error)

-> all steps done online during data acquisition (SPD)
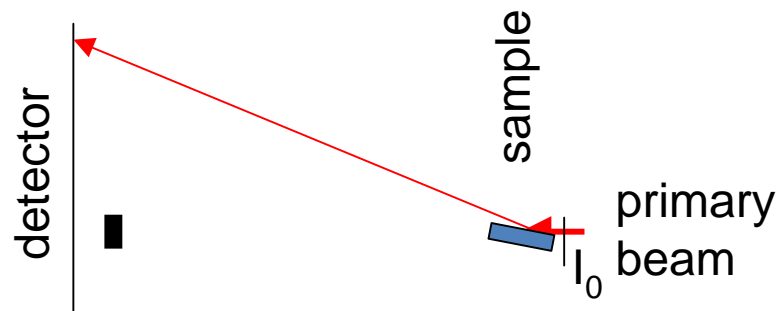
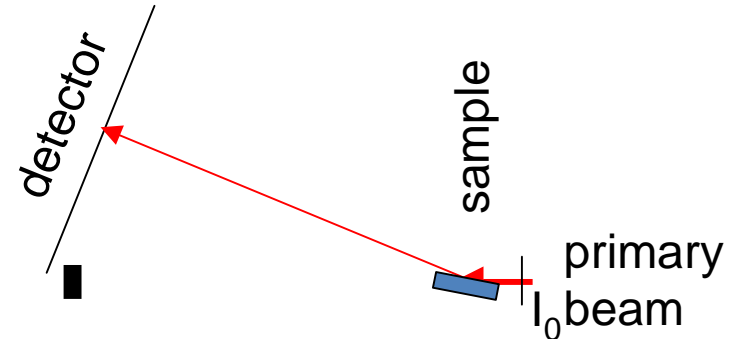# 3 Online/Offline Treatment

SAXS/WAXS Scattering Geometries
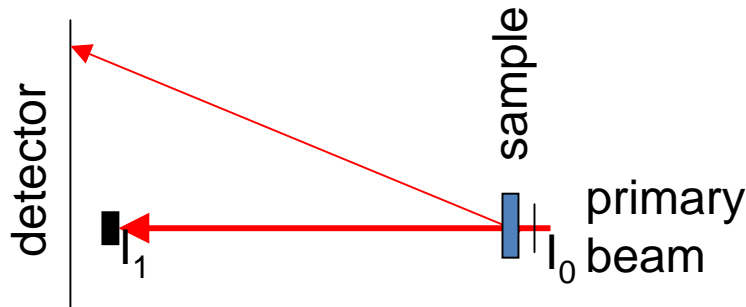
a) (A)SAXS/WAXS/WAXD
detector perpendicular
to the incident beam

b) (A) WAXS/WAXD
detector (strongly) inclined with
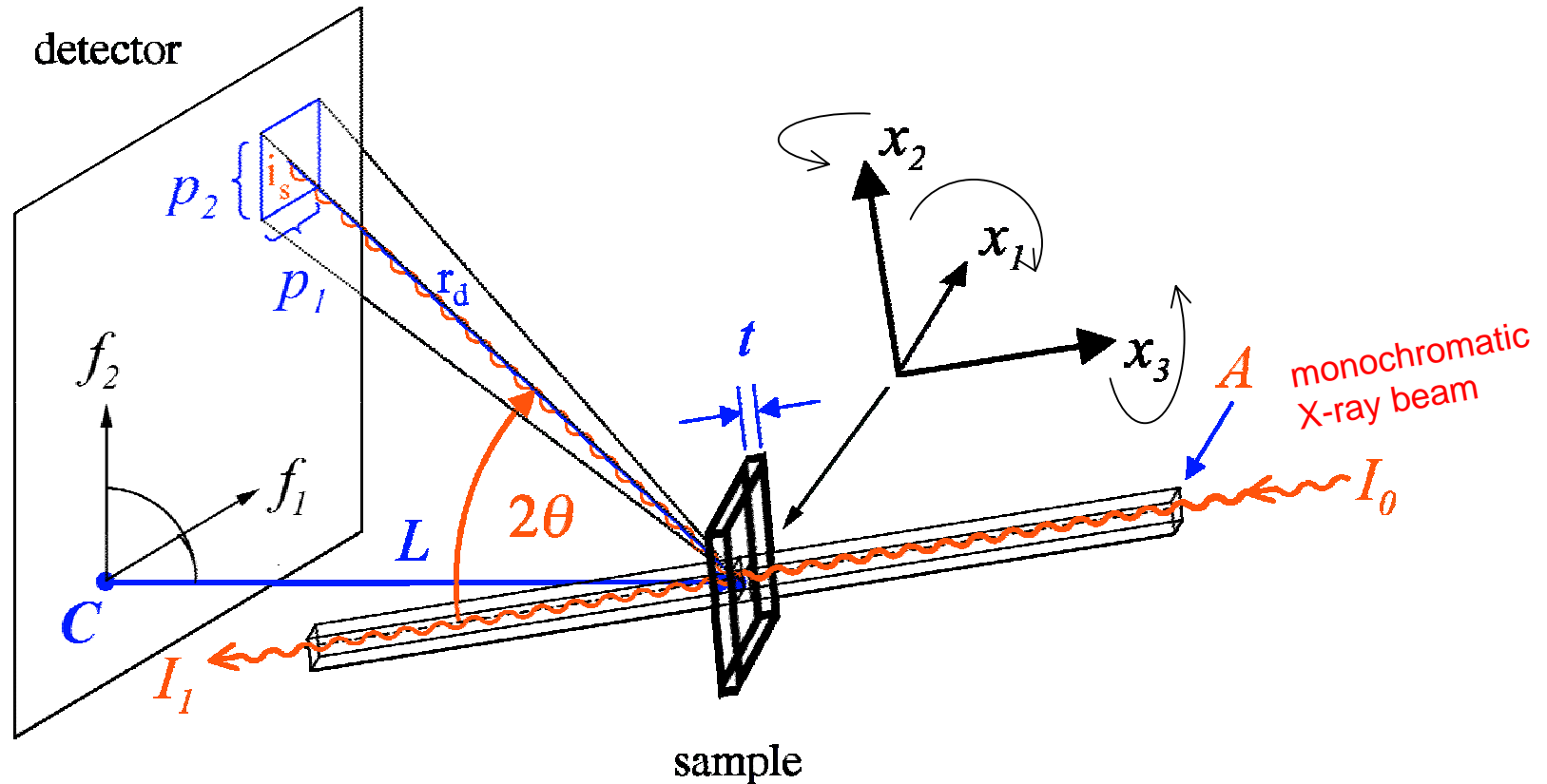respect to the incident beam

c) (A) GISAXS

d) (A) GID

3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (General Parametrization)

C = Center $\equiv$ Point$O$fNormal$I$ncidence, L = SampleDistance

3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (General Keywords)

Keywords describing the scattering geometry. Default values in brackets

Keyword	Unit	Comment	Keyword	Unit	Comment
Dim_1, Dim_2	pixel	Array dimension [0] [1]	PSize_1, PSize_2	m	Pixel size in meters
RasterOrientation	-	Raster orientation [1]	SampleDistance	m	Distance PoNI to sample
Offset_1, Offset_2	pixel	Spatial array offsets [0]	WaveLength	m	X-ray wavelength
BSize_1, BSize_2	pixel	Pixel size relative to size of unbinned pixel [1]	DetectorRotation_1, DetectorRotation_2, DetectorRotation_3	rad	Rotations of the detector plane in the laboratory system [0]
Center_1, Center_2	pixel	Point of normal incidence (PoNI)	ProjectionType	-	"Saxs" or "Waxs" ["Saxs"]

Keywords describing intensity and exposure time.

Keyword	Unit	Comment
Intensity0	(*)	Integrated beam intensity before the sample during the exposure time
Intensity1	(*)	Integrated beam intensity after the sample during the exposure time
ExposureTime	s	exposure time

(*)   Ideally, the intensities should be measured in number of photons integrated during the exposure time.

3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (WAXS Projection)

The SAXS Reference system is not appropriate for large scattering angles. To compare different scattering patterns taken at large scattering angles non-affine transformations are necessary.
The parameter ProjectionType=Waxs indicates that the data is represented in the Waxs projection (default: ProjectionType=Saxs)
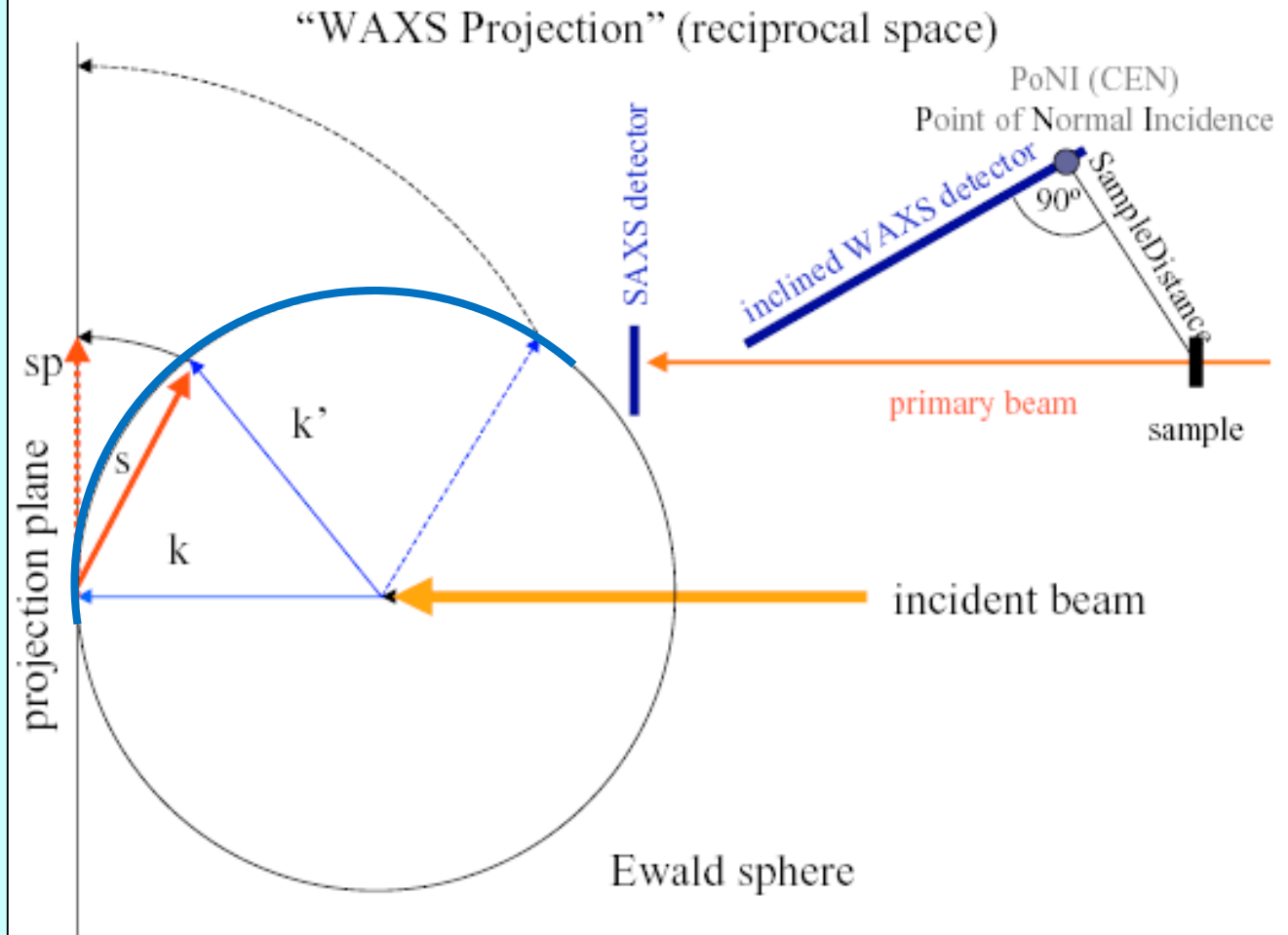
3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (Data Header)

```
ByteOrder = LowByteFirst ;
DataType = UnsignedShort ;
Dim_1 = 1242 ;
Dim_2 = 1152 ;
Title = test ( nu=0 mu=0 chi=90 phi=0 del=0.0124 energy=5.0002 ) (R1F1) ;
Time = 2007-09-25T11:27:39.212245+0200 ;
Intensity0 = 1.23937e+12 photons (Mon5) ;
Intensity1 = 9.27289e+11 photons (saxs1) ;
ExposureTime = 1 s (Seconds) ;
Dummy = 0 ;
DDummy = 0.1 ;
Offset_1 = 0 pixel ;
Offset_2 = 0 pixel ;
Center_1 = 597.019 pixel ;
Center_2 = 1214.3 pixel ;
BSize_1 = 1 ;
BSize_2 = 1 ;
PSize_1 = 5.5e-05 m ;
PSize_2 = 5.55e-05 m ;
SampleDistance = 3.13572 m ;
WaveLength = 2.47963e-10 m ;
RasterOrientation = 1 ;
```

3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (Tools)

SAXS program package

Peter Boesecke (http://www.esrf.eu/computing/scientific/SAXS)
- SAXS Package Manual
- SAXS Format Manual
- SAXS package download

SAXSutilities

Michael Sztucki (http://www.sztucki.de/physik/physik.php)
- BHplot (online treating and fitting of 1D SAXS data)
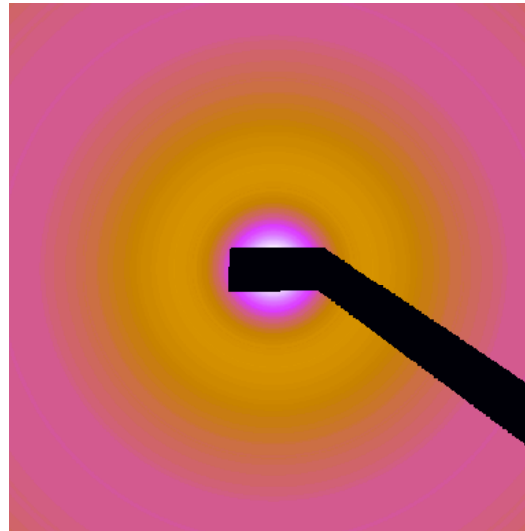- DataTools (1D SAXS data analysis)
- EDFplot (processing of 2D SAXS images)

3 Online/Offline Treatment

General SAXS/WAXS Scattering Geometry (Reference Systems)

SAXS L=1 m	SAXS L=3 m	combined L=1 m & L=3 m
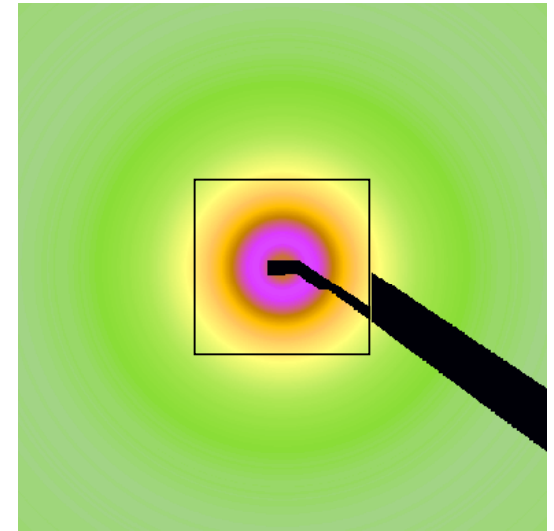
SAXS reference system (affine transformation of detector coordinates):

S=Normal/SampleDistance/(WaveLength/nm)=
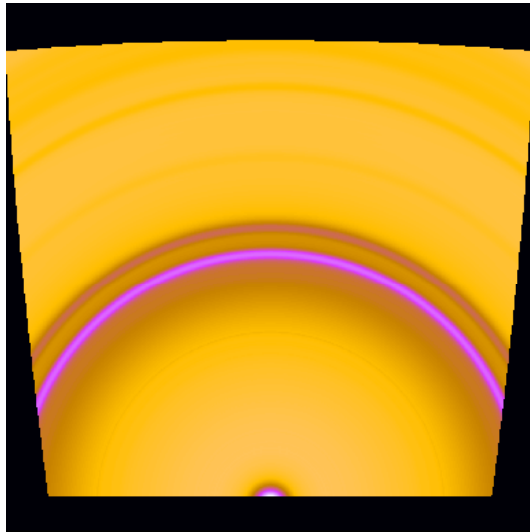(Offset-Center +A)*PSize/SampleDistance/(WaveLength/nm)

saxs_ave -rsys saxs lupolen_1m.msk lupolen_3m.msk lupolen_1m.ave

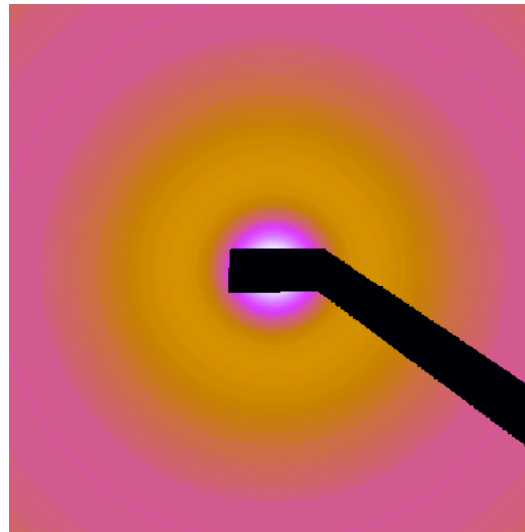In the same way saxs and waxs images can be combined

3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (SAXS/WAXS Example)
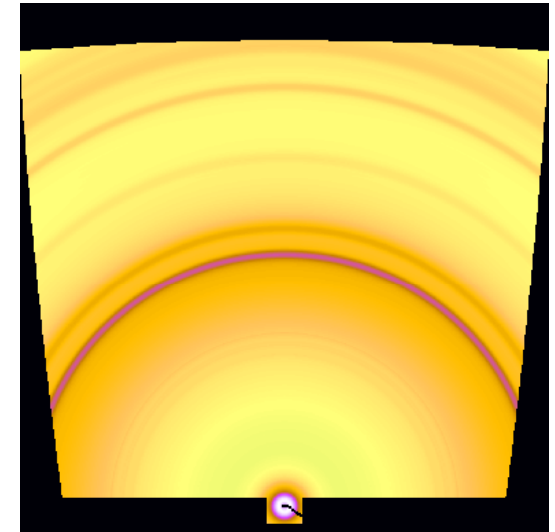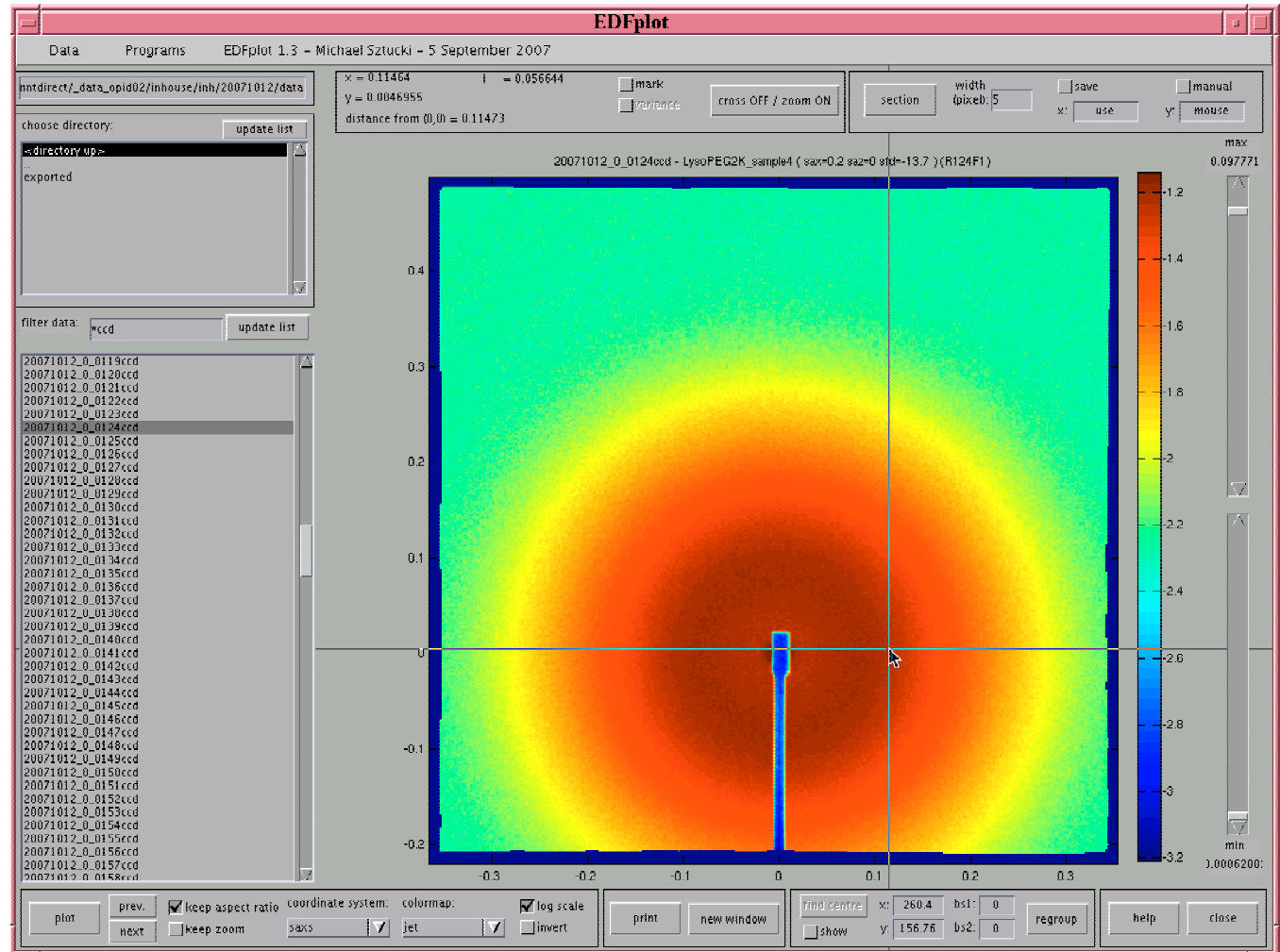
WAXS

SAXS

combined

saxs_ave -rsys saxs lupolen.wax lupolen_3m.msk lupolen.ave

3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (EDFplot)

MatLab based GUI
by Michael Sztucki
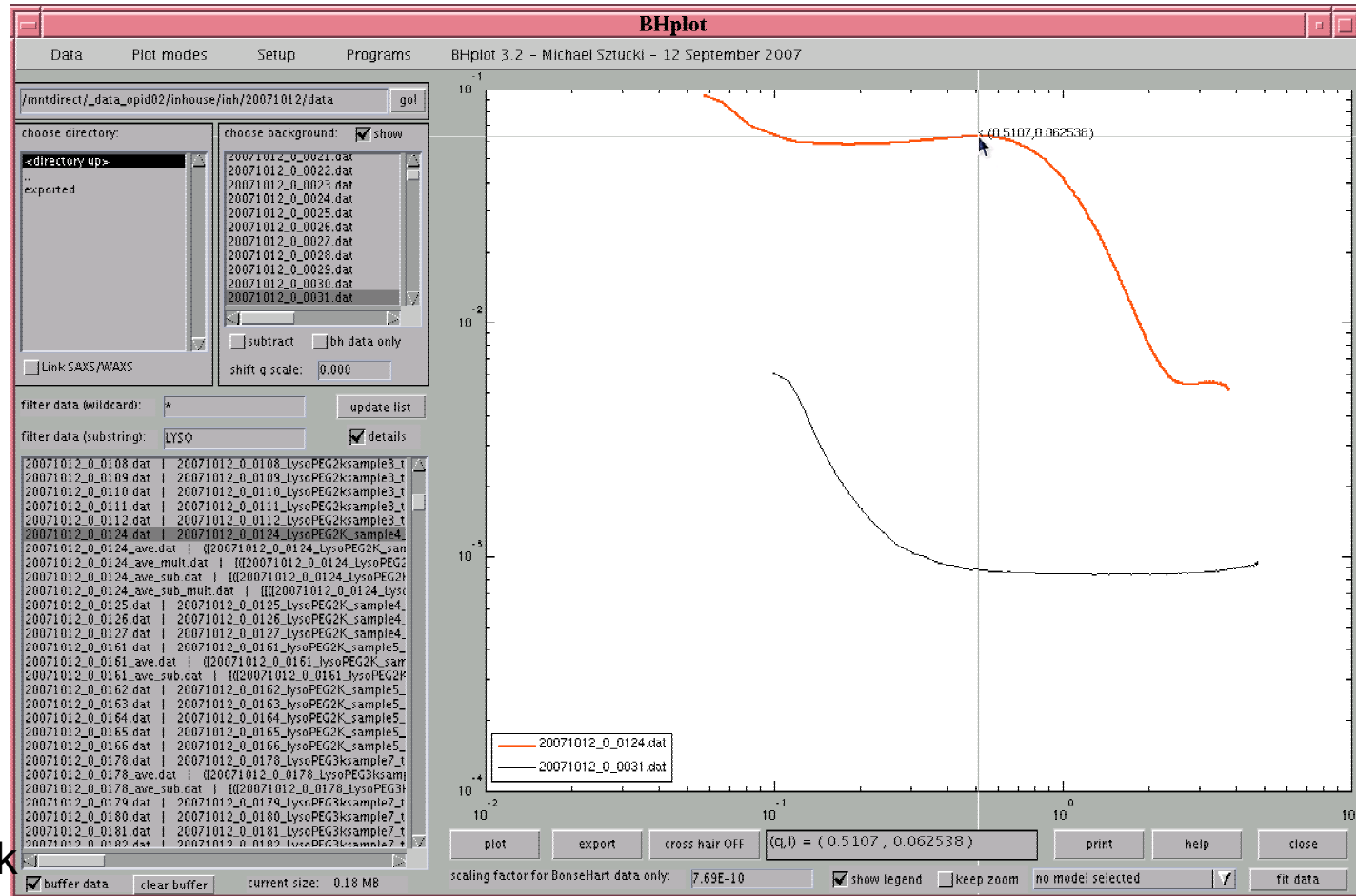www.sztucki.de/Physik

3 Online/Offline Treatment
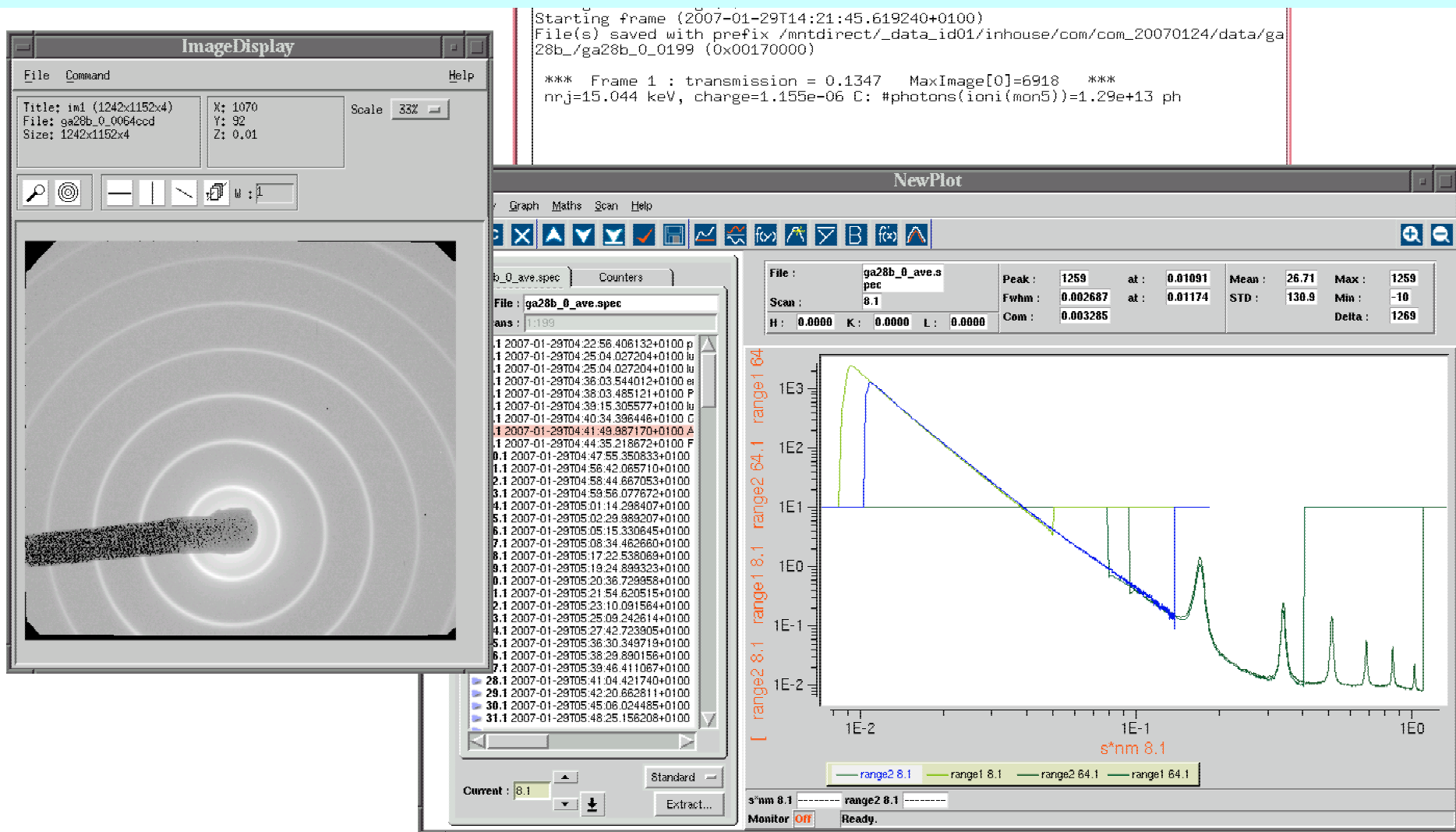
SAXS/WAXS Scattering Geometry (BHplot)

MatLab based GUI
by Michael Sztucki
www.sztucki.de/Physik

3 Online/Offline Treatment

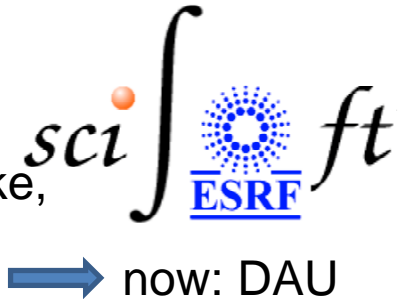## SAXS/WAXS Scattering Geometry (SPEC interface)

3 Online/Offline Treatment

SAXS/WAXS Scattering Geometry (SPD program)

Spatial **D**istortion Correction **P**rogram

(credits: Joerg Klora, Andy Hammersley, Rainer Wilcke,
Armando Sole, Claudio Ferrero, Andy Goetz
Jerome Kieffer,...)

$sci\int$ ESRF ft

➡ now: DAU

The program SPD exists in several versions:
- offline program (command line driven)
- data pipe version (spec data pipe driven)
- server version (spd is running as a server) (A. Goetz, under test)

SPD is used online for **basic detector corrections** (dark image, flat field, distortion) and **absolute intensity normalization**. The speed of **azimuthal regrouping** is going to be optimized.
The calculation time is typically 1 s per image. Azimuthal regrouping can be longer, depending on the chosen integration range.

4 Summary

Parametrization (if necessary, extendable to higher dimensions)

Data Specific
DATA ARRAY, VARIANCE ARRAY (optional),
ByteOrder, DataType, Dim_1, Dim_2, DataRasterOrientation,
Dummy, DDummy (to mark invalid pixels)

Detector Specific
Offset_1, Offset_2, RasterOrientation, BSize_1, BSize_2

Geometry Specific
PSize_1, PSize_2, SampleDistance, Center_1, Center_2,
DetectorRotation_1, DetectorRotation_2, DetectorRotation_3

Scattering Specific
WaveLength

Projection Specific
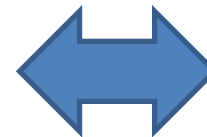Projection Type

Intensity Specific
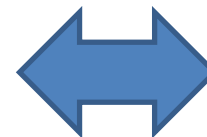Intensity0, Intensity1, ExposureTime

Time Specific
Time
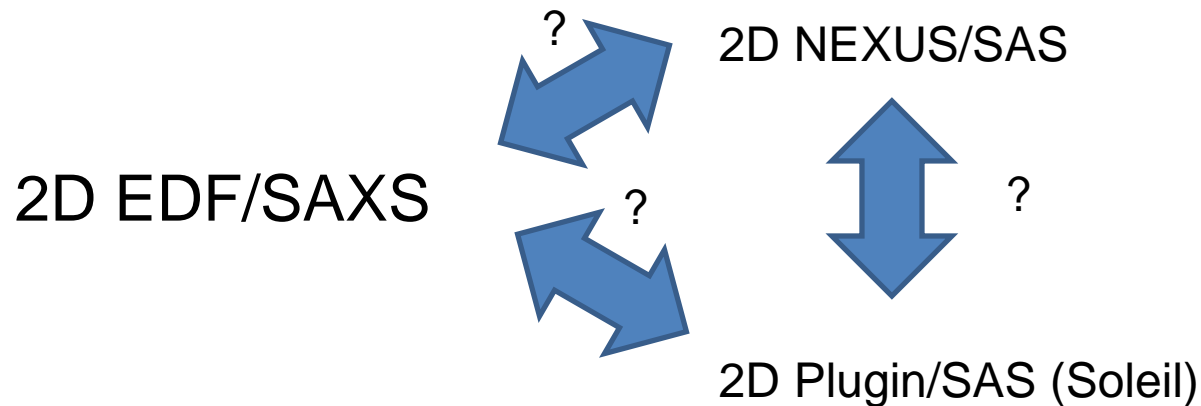
Informational
Title, History

2D NEXUS SAS

2D Plugin (Soleil)

4 Summary

SAXS/WAXS Scattering Geometry (General Keywords)

What is the meaning of the parameters in NEXUS/SAS or at Soleil/SAS?

?   2D NEXUS/SAS

2D EDF/SAXS   ?   ?

2D Plugin/SAS (Soleil)

Parameters are not obvious, even if they are as simple as detector distance!

A strict definition is needed!!!

detector   sample

detector   this   or   that?