Tutorial: SAXS analysis strategies and softwares

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Grenoble (France)
Outline

- SAXS instrumentation and theory
- SAXS data reduction and calibration
- Good practice for data recording
- SAXS data formats
- Tools for (on-line) data reduction and visualization: SPD, saxis programs, PyFAI, SAXSutilities

Data Interpretation / modelling
- Form factor, Guinier / Porod law, Unified fitting, size distributions, structure factor
- Programs: SAXSutilities, IRENA, SASview, SASfit
scattering of X-rays at small-angles originate from spatial fluctuations of the electron density within the material

\[ q = \frac{4\pi}{\lambda} \sin(\theta/2) \]

**Measured Intensity:** \[ I_S = i_0 T_r \varepsilon \Delta \Omega \frac{d\sigma}{d\Omega} \]

- \( i_0 \) - incident flux
- \( T_r \) - transmission
- \( \varepsilon \) - efficiency
- \( \Delta \Omega \) - solid angle

**Differential scattering cross-section**

\[ I(q) = \frac{d\Sigma}{d\Omega} = \frac{1}{V_{Scat}} \frac{d\sigma}{d\Omega} \]
SAXS instrumentation

SAXS, WAXS and USAXS

µ-beam SAXS, WAXS
SAXS instrumentation

SAXS, WAXS and USAXS

µ-beam SAXS, WAXS
(A) detector specific corrections

(B) scattering specific corrections

(C) sample and beam specific corrections
(A) detector specific corrections

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

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

- 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 10 [photons] and conversion to scattering cross section [1/sterad]

\[
\left( \frac{1}{A} \right) \frac{\partial \sigma}{\partial \Omega} = \frac{\text{\# Photons}_{\text{scattered}}}{\text{\# Photons}_{\text{in}}} \text{ sterad} = \frac{i_4}{I_0} \cdot \frac{L_p^2}{p_1 \cdot p_2} \cdot \frac{L_p}{L_0} \cdot \frac{1}{\Delta \Omega}
\]

shortest sample to detector distance
sample to pixel distance
pixel size

and Narayanan, Diat, Bösecke, NIM A 467-468 (2001) 1005-1009)

normalizing a two dimensional scattering pattern measured with a plane two-dimensional detector to absolute intensities in units of scattered photons per steradian and per incident photon, i.e. scattering cross section d\sigma/d\Omega per sample cross section A;
to correct for absorption the normalized pattern is divided by the sample transmission T
5. Normalization to transmission and scattering volume, e.g. thin film:
\[ i_5 = \frac{i_3}{d \cdot I_1 \cdot p_1 \cdot p_2} \cdot \frac{L_p^2}{L_0} \]

T=I/I_0, d: sample thickness

6. Polarization correction (WAXS)

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

8. Azimuthal averaging
WAXS projection

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

azimuthal regrouping

azimuthal averaging

averaging

angle / deg

scattering vector

l/sterad

l/sterad

scattering vector s * nm

0.001 1 1000

0.001 0.10 0.2

0.001 1 1000

0.001 1 1000

angle / deg

scattering vector
Good practice of data recording

background subtraction
flow-through cells
radiation damage
Absolute intensity calibration

using secondary standards like water, lupolen, glassy carbon, …
Absolute intensity calibration

using secondary standards like water, lupolen, glassy carbon, …

scattering of 1mm of water at RT: 0.00164
ID02 data and metadata concept

**data.edf**

```plaintext
{
  EDF_DataBlockID = 1.Image.Psd;
  EDF_BinarySize = 1440256;
  EDF_ReaderSize = 8192;
  ByteOrder = LowByteFirst;
  DataType = FloatValue;
  Dim_1 = 1000;
  Dim_2 = 360;
  Dummy = -10;
  Dummy = 0.1;
  Offset_1 = 0;
  Offset_2 = 0;
  History-1 = "C:\Program Files (x86)\SAXSutilities\C:\Data\SAXSutilities_Testdata\h5\og22_saxs."
  HeaderID = "EH:000001:000000:000000;"
  Compression = None;
  Image = 1;
  SaxsDataVersion = 2.40;
  Size = 1440256;
}
```

**data.h5**

**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

**NXdetector**

**NXdata** one NXdata group per plot

**data**

**metadata**

**NXsample**

user (@NXuser)

**NXprocess** for analysis
understanding of the data formats (EDF, HDF5)

http://www.esrf.eu/home/UsersAndScience/Experiments/CBS/ID02/available_software.html
→ SAXS programs → SX_parametrization

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understanding of the data formats (EDF, HDF5)

SX General Parameters
- \textit{Dim}_1, \textit{Dim}_2, \ldots \text{ (Dim}_1\text{ default is 0, all others are 1)}
- \textit{RasterOrientation} \text{ (1 to 8 possible, 9-16 for compatibility, needs to be tested, default 1)}

SX Image Parameters
- \textit{Offset}_1, \textit{Offset}_2, \ldots \text{ (default: 0)}
- \textit{BSIZE}_1, \textit{BSIZE}_2, \ldots \text{ (default: 1)}

SX Scattering Parameters
- \textit{PSIZE}_1, \textit{PSIZE}_2, \ldots \text{ (no default) [m]}
- \textit{Center}_1, \textit{Center}_2, \ldots \text{ (PoNI in image reference system including offsets, no default)}
- \textit{SampleDistance} \text{ (no default) [m]}
- \textit{WaveLength} \text{ (no default) [m]}
- \textit{DetectorRotation}_1, \textit{DetectorRotation}_2, \textit{DetectorRotation}_3, \ldots \text{ (default: 0) [rad]}

Additional SX Scattering Parameters
- \textit{ProjectionType} (Saxs|Waxs, default: Saxs: distances proportional to tan(2\theta))
- \textit{AxisType}_1, \textit{AxisType}_2, \ldots \text{(Distance, Angle, Numerator, default: Distance)}

SX Intensity Calibration and Normalization Parameters
- \textit{Intensity0} \text{ (no default), Intensity1 (Intensity0)}
- \textit{NormalizationFactor} \text{ (default 1)}
- \textit{SampleThickness} \text{ (no default)}
Keywords for SAXS Data in EDF Files

EDF_DataFormatVersion = 2.42

Keywords for SAXS Data in EDF Files
EDF_DataFormatVersion = 2.42
Peter Boesecke

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http://www.esrf.eu/home/UsersAndScience/Experiments/CBS/ID02/available_software.html
→ SAXS programs → Saxs Format Manual
understanding of the data formats (EDF, HDF5)

raw data
- rk28_saxes_01006_raw.h5
- entry_0000
  - end_time
  - instrument
  - measurement
  - id02-rayonixhs-saxes
    - data
    - array
    - header
    - start_time
    - title

pyFAI (normalized)
- rk28_saxes_01006_norm.h5
- entry_0000
  - PyFAI
    - MCS
    - date
  - id02-rayonixhs-saxes
    - parameters
    - processing_type
    - program
    - result_norm
      - data
      - data_errors
      - t
    - version
    - detector_name
    - input
    - plugin_name
    - program_name
    - start_time
    - title

pyFAI (regrouped)
- rk28_saxes_01006_azim.h5
- entry_0000
  - PyFAI
    - MCS
    - TFG
    - date
  - id02-rayonixhs-saxes
    - parameters
    - processing_type
    - program
    - result_azim
    - chi
    - data
    - data_errors
    - q
    - t
    - version
    - detector_name
    - input

pyFAI (1D)
- rk28_saxes_01006_ave.h5
- entry_0000
  - PyFAI
    - MCS
    - TFG
    - data
  - id02-rayonixhs-saxes
    - parameters
    - processing_type
    - program
    - result_ave
    - data
    - data_errors
    - q
    - t
    - version
    - detector_name
    - input

SAXSutilities/programs
- sym_2V4_end.h5
- entry_0000
  - input
  - program_name
  - saxesutilities
    - data
    - array
    - array_errors
    - header
    - header_array
    - header_array_errors
    - start_time
    - title

HDF viewer: www.hdfgroup.org

NXDATA:
- data/data_errors (pyFAI), array/array_errors (beamline)

metadata:
- parameters (pyFAI), header or header_array/header_array_errors

NOTE: "default" attributes tell you where to find data

NOTE: only "header" describes NXDATA according SX_parametrization
"parameters" are only a copy of header of raw data
**ID02 tools for data reduction and visualization**

**available programs:**
- spd
- saxs programs
- SAXSutilities
- PyFAI

→ on-line and offline ←

demonstration:
data reduction in 2D and 1D:
e.g. partial integration, averaging, subtraction, merging

typical artefacts

http://www.esrf.fr/home/UsersAndScience/Experiments/CBS/ID02/BeamlineDescription/DataReduction/SoftwareIntroduction.html
new ESRF data policy
https://www.esrf.eu/datapolicy

ID02 online data processing

raw:
- edf/edf
- SPD / saxes programs

reduced:
- edf
- DAHU (PyFAI) / SPD / saxes programs
- h5
- h5/edf
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

**SPD – s axs programs**

`saxs_arc`
- `omod n`
- `i1nam ra26_saxs_%%%nrm.edf,1,500`
+ `mask`
- `i2nam mask-2m5.edf`
- `onam ra26_saxs_%%%nrm_azim.edf`
+ `pass`
- `p`

`saxs_curve`
- `alf1 80_deg`
- `alf2 100_deg`
- `scf 2_pi`
- `head`
- `hedl "q*nm [TitleBody,,1]"
- `ext .dat`
- `i1nam ra26_saxs_%%%nrm_azim.edf,1,500`
- `onam ra26_saxs_%%%nrm_80-100_ave.dat`
- `p`

**Note:** double the number of ‘%’ in windows bat scripts...
saxs programs

further info:

http://www.esrf.eu/home/UsersAndScience/Experiments/CBS/ID02/available_software.html

→ SAXS programs → SaxsPrograms Package Manual

NEW in spring 2020:
SAXSutilities2 developed in Python 3.6/3.7 package with Windows / Linux installer

http://www.saxsutilities.eu
SAXSutilities – SAXSutilities2 ready for restart of beamlines in spring 2020

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: sasxutilities.zip
   > unzip sasxutilities.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 SXSUTILITIESPATH to your system which points to the directory created in step 1.

4. Note that unpec by P. Mikulik 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 SXSUTILITIESPATH [/path/to/directory/of/SAXSutilities]
   [/path/to/directory/of/SAXSutilities]/SAXSutilities

http://www.saxsutilities.eu
SAXSutilities(2) – 2D visualisation (EDFplot / plot2D)
**Saxs COORDINATES < ✈ > Saxs PROJECTION**

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

(distance of pixel from poni on detector) \( \text{wavelength}_0 \)

\[
\text{SAXS coordinate} = \frac{\text{distance of poni from sample}}{\text{wavelength}}
\]

where \( \text{wavelength}_0 \) is \( 1 \times 10^{-9} \text{ m} \)

SAXS(Saxs) coordinate \( \sim s \times \text{nm} \) for \( 2\Theta < 1 \)

**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 \( s \):

\[
\text{SAXS(Waxs) coordinate} = s \times \text{nm}
\]

In Waxs PROJECTION the scattering pattern of a tilted detector can be geometrically analysed (azimuthal regrouping etc.) like a small angle scattering pattern.
SAXSutilities(2) – 2D visualisation (EDFplot / plot2D)
SAXSutilities – 2D visualisation (EDFplot)
SAXSutilities(2) – 1D visualisation (BHplot / plot1D)
→ SAXS instrumentation and theory
→ SAXS data reduction and calibration
→ Good practice for data recording
→ SAXS data formats
→ Tools for (on-line) data reduction and visualization: SPD, saxes programs, PyFAI, SAXSutilities

→ Data Interpretation / modelling
→ Form factor, Guinier / Porod law, Unified fitting, size distributions, structure factor
→ Programs: SAXSutilities, IRENA, SASview, SASfit
Form factor (dilute case)

\[ I(q) = N |F(q)|^2 \]

scattering amplitude:

\[ F(q) = \int_V \rho(r) e^{iqr} dV \]

in case of isotropic particles:

\[ F(q) = 4\pi \int_0^\infty \Delta \rho(r) \frac{\sin(qr)}{qr} r^2 dr \]

in case of isotropic spherical particles:

\[ |F(q)|^2 = V_s^2 \Delta \rho^2 \left( \frac{3[\sin(qR_s) - qR_s \cos(qR_s)]}{(qR_s)^3} \right)^2 = V_s^2 \Delta \rho^2 P(q, R_s) \]
Form factor (dilute case)

Uniform sphere of radius, $R_S$

$$P(q, R_S) = \left( \frac{3 \sin(qR_S) - qR_S \cos(qR_S))}{(qR_S)^3} \right)^2 = F_0^2(qR_S)$$

Randomly oriented cylinder of radius $R_C$ and height $H$

$$P(q, R_C, H) = \int_0^{\pi/2} \left\{ \frac{2J_1(qR_C \sin \varphi)}{qR_C \sin \varphi} \left[ \frac{\sin((qH/2) \cos \varphi)}{(qH/2) \cos \varphi} \right] \right\}^2 \sin \varphi \, d\varphi$$

$J_1$ – first order Bessel function and $\varphi$ – orientation angle.

Spherical shell of inner and outer radii $R_1$ and $R_2$

$$V^2 P(q, R_1, R_2) = \frac{16\pi^2}{9} \left[ R_2^3 F_0(qR_2) - R_1^3 F_0(qR_1) \right]^2$$

$F_0(q, R)$ – sphere function given above.

Spherical core-shell of core and shell radii $R_1$ and $R_2$

$$F^2(q, R_1, R_2) = \left[ V_2 \Delta \rho_2 F_0(qR_2) - V_1 \Delta \rho_1 F_0(qR_1) \right]^2$$

$V_1$ and $V_2$ are volumes of inner and outer spheres, $\Delta \rho_1$ and $\Delta \rho_2$ are contrast between shell and core, and shell and medium, respectively.
Form factor (dilute case)
Limiting form of $I(q)$: Guinier and Porod law

**Guinier plot:**

$$I(q) = N V^2 \Delta \rho^2 \exp\left(\frac{-q^2 R_g^2}{3}\right)$$

valid at low concentration and small values of $q$, $qR_g < 1$

plot of $\ln(I)$ against $q^2$ has slope $-R_g^2/3$

radius of Gyration depends on particle shape

$R_g^2$ is the average squared distance of the scatterers from the centre of the object

- **Sphere** $R_g^2 = \frac{3}{5} R^2$
- **Ellipse** $R_g^2 = \frac{a^2 + b^2}{4}$
- **Cylinder** $R_g^2 = \frac{R^2}{2} + \frac{h}{12}$

**Porod law:**

for $qR_g >> 1$

$$I(q) = 2\pi N \Delta \rho^2 S q^{-4}$$

$I(q) \propto q^{-p}$

$$\begin{cases} p = 4 & \Rightarrow \text{sharp interface} \\ 3 \leq p < 4 & \Rightarrow \text{surface fractal} \\ p < 3 & \Rightarrow \text{mass fractal} \\ p \approx 2 & \Rightarrow \text{gaussian polymer chain} \end{cases}$$
Unified fitting (G. Beaucage)

for polydisperse systems consisting of multiple structural levels

\[ I(q) = G \exp\left(-\frac{q^2 R_G^2}{3}\right) + B \left\{ \frac{\text{erf}\left(\frac{q R_G}{\sqrt{6}}\right)}{q} \right\}^p \]

\[ G = N \Delta \rho^2 V^2, \quad B = 2\pi N \Delta \rho^2 S \]

\[ BR_G^{4/1.62} = \text{polydispersity index} \]
Unified fitting (G. Beaucage)


using Irena package by Jan Ilavsky in Igor Pro: https://usaxs.xray.aps.anl.gov/software/irena

- Unified fit
- Modeling
- Size distribution
- Guinier-Porod model
- Fractal model
- Reflectivity
- PDDF
- Peak fitting tool
- ...

![Graph showing data points and fitted curves with labels for parameters and data like Q, Intensity, LoglogPlot, and Fit controls.

Relevant references include:

Size distributions

Size distribution – using Maximum Entropy, Total Non-negative least square (TNNLS) & Regularization methods for evaluation of small-angle scattering from scatterers represented by number of different form factors.
Size distributions
Interaction → Structure factor

Form Factor = scattering from within same particle
⇒ depends on particle shape

Structure Factor = scattering from different particles
⇒ depends on interactions between particles

\[ I(q) = N V^2 \Delta \rho^2 P(q) S(q) \]

\[ S(q) = 1 + 4\pi N \int_0^\infty (g(r) - 1) \frac{\sin(qr)}{qr} r^2 dr \]

>> structure factors for monodisperse systems

+ hard sphere repulsive potential,
+ short range attractive square-well potential
+ 2Youkawa potential
Form (and structure) factor fitting

\[ \tau_B \]

\[ B_1 \text{ repulsive glass} \]

\[ B_2 \text{ attractive glass} \]

\[ \phi \]

\[ \phi \approx 0.22 \]

\[ I(q) \text{ (mm)} \]

\[ 1.24 \]

\[ 19.5 \, ^\circ \text{C (x30)} \]

\[ 21.0 \, ^\circ \text{C (x10)} \]

\[ 23.5 \, ^\circ \text{C (x3)} \]

\[ 25.0 \, ^\circ \text{C} \]

\[ q \text{ (nm}^{-1}) \]

---

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

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H. Hoekstra

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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 repulsive glass states exist. The phase separation admits a kinetic arrest regime at intermediate volume fractions.
SAXSutilities – contrast calculation

Contrast Calculator

Solvent:
- Water
  - Density: 1.0 g/cm³

Material:
- TTAB
  - Density: 1.02 g/cm³

ASAXS contrast (optional):
- Ion: Na⁺
  - Ionic radius: 0.182 nm

Contrast:
- Mass M: 336.3928 g/mol
- Number of electrons N: 182
- Scattering length density: 0.00093384 nm⁻²
- Electron density: 332.327 nm⁻³
- Excess electron per ion: 26.8588

Calculate
indexing and identifying different crystal structures

<table>
<thead>
<tr>
<th>Crystal Structure</th>
<th>Indices</th>
</tr>
</thead>
<tbody>
<tr>
<td>LAM</td>
<td>1:2:3:4:5:6:7</td>
</tr>
<tr>
<td>HCP</td>
<td>1: $\sqrt{3}$:2: $\sqrt{3}$:3: $\sqrt{4}$: $\sqrt{3}$:4</td>
</tr>
<tr>
<td>PC</td>
<td>1: $\sqrt{2}$: $\sqrt{3}$:2: $\sqrt{5}$: $\sqrt{6}$: $\sqrt{3}$:3</td>
</tr>
<tr>
<td>BCC</td>
<td>1: $\sqrt{2}$: $\sqrt{3}$:2: $\sqrt{3}$: $\sqrt{6}$: $\sqrt{3}$:3</td>
</tr>
<tr>
<td>FCC</td>
<td>$\sqrt{3}$:2: $\sqrt{3}$: $\sqrt{6}$: $\sqrt{12}$:4: $\sqrt{3}$</td>
</tr>
<tr>
<td>HCPm</td>
<td>$\sqrt{32}$:6: $\sqrt{4}$: $\sqrt{68}$: $\sqrt{96}$: $\sqrt{14}$</td>
</tr>
<tr>
<td>DD</td>
<td>$\sqrt{5}$: $\sqrt{6}$:2: $\sqrt{5}$: $\sqrt{3}$: $\sqrt{15}$: $\sqrt{11}$</td>
</tr>
<tr>
<td>Ia3d</td>
<td>$\sqrt{3}$:2: $\sqrt{7}$: $\sqrt{8}$: $\sqrt{10}$: $\sqrt{11}$: $\sqrt{13}$</td>
</tr>
<tr>
<td>Pm3n</td>
<td>$\sqrt{2}$:2: $\sqrt{5}$: $\sqrt{6}$: $\sqrt{8}$: $\sqrt{10}$: $\sqrt{12}$</td>
</tr>
</tbody>
</table>
http://www.sasview.org/
http://www.sasview.org/
SASview 5
Conclusions

Saxs Programs:
http://www.esrf.eu/home/UsersAndScience/Experiments/CBS/ID02/available_software.html
→ SAXS programs
Or directly: http://www.esrf.eu/Instrumentation/software/data-analysis/OurSoftware/SAXS

SAXSutilities:
http://www.saxsutilities.eu
--- there you will find soon this presentation ---

Irena:
https://usaxs.xray.aps.anl.gov/software/irena

SASfit:
https://kur.web.psi.ch/sans1/SANSSoft/sasfit.html

SASview:
http://www.sasview.org
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