Transmission SAXS/WAXS using the Pilatus3R 300K detector on the SAXSLAB instrument

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This SOP and others are available at http://prism.mit.edu/xray/education/downloads.html

The SAXSLAB instrument is setup to perform transmission small or wide angle X-ray scattering on freestanding samples, powders prepared in kapton tape, liquids in capillaries and gels in sandwich cells with mica windows. Standard training allows you to perform automated SAXS or WAXS measurements with a variable sample to detector distance dependent on the sizes that are intended to be probed; for SAXS (30-2300 Å) for WAXS (3-70 Å).

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# SAXSLAB Operation Checklist

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   a. Leave the tube power at full power level:  
     i. **45 kV and 0.66 mA** for the Cu microfocus tube  
     b. Vent SAXS system  
     c. Retrieve your sample  
     d. Evacuate SAXS system  
     e. Clean the sample stage and sample holders  
     f. Copy your data to a secure location  
     g. **Disengage** the SAXS in Coral | Ch VII  |

The Chapter number and section number on the right-hand side of the page indicate where you can find more detailed instructions in the SAXS SOP.
I. Startup Instrument

This section walks through starting the SAXS instrument and logging into Coral. The instructions below give some descriptions on how to use the Linux system. If you are not familiar with Linux, read Appendix A.

1. Assess Instrument Status and Safety
   a. Is the X-ray generator on?
      i. Look for indicator light on pole near X-ray source.
   b. What is the X-ray source power?
      i. The X-ray source is at full power (45 kV and .66 mA), and the shutter is remotely controlled. Shutter 1 will not open when pressed on the source controller.
      ii. If the source controller reads, (00 kV and .00 mA), please contact the X-ray lab staff.
      iii.

2. Engage the SAXS system in Coral.
   a. Navigate to the Terminal
   b. Type: cd /usr/local/coral-remote
   c. Type: ./coral-remote-cmse
   d. Login to Coral with your username and password
3. Start the programs Ganesha System Control

a. There is a toolbar displayed along the bottom of Workspace 1, with the programs that are used to control of the SAXSLAB system labeled SPEC, SL, DET, TVX, GEN, CAM1, GUI, and ED.

b. SPEC: software for instrument control and data acquisition - http://www.certif.com/
SL: server runs on the detector computer and handles processing of the data (cumulative files, includes headers, advanced noise-reduction).
DET: server talks directly to the Pilatus3R 300K detector
TVX: server for low level communication to DET server
GEN: server for communication from SPEC to the source generator for opening closing shutter, modifying voltage and current settings
CAM1: software to view samples in chamber via off axis camera
GUI: software for data visualization, image analysis operations and transformations, including batch mode data reduction and well as basic fitting - http://www.saxsgui.com/
ED: text editor

c. Blue indicates the service is active, Yellow indicates an optional service has been closed and can be reopened by clicking on the toolbar icon, and Red indicates that a critical service is not running.

d. SAXSGUI will indicate to open an initial image file.
   i. Open the file: /home/saxslab/SAXSShared/data/latest/latest_xxxxxxx_craw.tiff where xxxxxxx is the largest number possible in the directory.
   ii. Navigate to File > Open latest
   iii. Select the file: /home/saxslab/SAXSShare/data/latest/latest_xxxxxxx_craw.tiff
   iv. SAXSGUI now continuously updates as data is acquired. The indicator of this condition is the red Stop Monitoring button will be enabled at the bottom of the SAXSGUI data visualization screen.

4. Start the Ganesha GUI software (GICC = Ganesha Interactive Control Center).

a. The GICC software has 3 panels, left to right as can be seen below.
   Left: sample stage graphic control (opened by < button on left of main panel)
   Center: main control panel (always open)
   Right: macro editor panel (open by > button on right of main panel)
II. Sample loading procedure
This section describes the procedure for venting, loading the sample (freestanding or capillary) on the Ambient 8x8 stage and evacuating the system.

1. Vent chamber to load samples.
   a. In GICC, click the “Vent” button on the bottom right of the main control panel.
      i. Dialogue box: Please make sure the sample door is partially unlashed to prevent overpressure. Start venting? Click Start.
   b. After 120 seconds of venting the chamber door should be ajar. If this is not the case, click “Vent” button a second time and wait to see that the door opens.
   c. Click “Isolate” button to close the valves.

2. Choose the appropriate sample holder.
   a. For liquid samples use quartz capillaries 1.5 mm diameter.
      i. Prepare capillaries by drawing liquid into syringe
      ii. Place syringe all the way to bottom of capillary.
      iii. Mix epoxy resin and hardener on weigh paper using wooden stick.
      iv. Allow epoxy to dry for a minimum of 30 minutes prior to loading into SAXS system.
      **Be patient. Capillaries with wet epoxy may cause the epoxy and your sample to coat the detector window upon evacuating the chamber.**
v. Load capillaries into Ambient 8x8 holder with 1.5 mm bracket. Gently screw down bracket, be careful to not break the capillaries in the holder.

vi. Load holder in to chamber with SAXSLAB logo visible. Be sure to push holder in to back reference plate. Tighten thumbscrews.

***Only certain columns and rows of holes are available sample stage y and z motor limits.***

Stage Z,Y coordinate system: Holes are 4mm in diameter, 8mm center to center

- Z limits: holes 03 to 05
- Y limits: holes 02 to 09

b. For freestanding samples use the hole array called “Ambient 8x8”.
   - i. Measure the thickness of sample with digital caliper.
   - ii. Adhere sample to holder with kapton tape or double sided tape.
   - iii. Hole 0306 is the reference position.
   - iv. Make sure your sample is in the hole column and row boundaries referenced above.
   - v. Prepare a silver behenate standard sample in kapton tape for calibration.
   - vi. Load holder in to chamber with SAXSLAB logo visible. Be sure to push holder in to back reference plate. Tighten thumbscrews.

c. Close chamber door and make sure that the O-ring seal is compressed by pulling up the handle.

3. Record the row and column positions of your samples on the Ambient 8x8 holder.

4. Evacuate SAXS system
   a. In GICC, click “Evacuate” button on the bottom right of the main control panel.
      - i. Expect a loud noise as the pump starts.
   b. The vacuum gauge should read 0.08 mbar after 5 minutes of the chamber evacuating.
III. Alignment of sample to X-ray beam

This section describes how to plan measurements, change configurations, align the beamstop, position the sample into the beam and collect calibration measurements of silver behenate prior to automated data acquisition. Specific details on how to create and run an automated batch program are covered in sections IV and V.

1. What is the intended size you would like to probe?
   a. SAXS/WAXS measurements are taken as a function on scattering angle, 2θ, from the forward direction of the radiation. A plot of relative intensity as a function of scattering angle, 2θ, assumes the radiation wavelength is known (CuK\textsubscript{α1} = 1.5409 Å). To normalize the scattering profile to a wavelength independent scale the conversion to, \( q \), the momentum transfer is

   \[
   q = 4 \pi \sin \theta / \lambda,
   \]
   where \( \theta \) is \( 1/2 \) the scattering angle and is the \( \lambda \) wavelength in Ångstroms.

   \[
   q = 2\pi/d,
   \]
   where \( d \) is real space length in units of Å and \( q \) is in units of Å\(^{-1}\).

2. In GICC main control panel, select an appropriate Configuration to observe a specific length scale. Options are 2 Apertures WAXS, MAXS, SAXS and ESAXS. Click Go button.

| Configuration | Name          | Description                  | detx | Sample-detector distance | Aperture Sizes | Aperture | q\textsubscript{max} | q\textsubscript{min} | d\textsubscript{max} | d\textsubscript{min} |
|---------------|---------------|------------------------------|------|--------------------------|----------------|----------|-----------------|-----------------|----------------||-----------------|
| 0             | Open          | Open Apertures               | mm   | mm                       |                | I/A      | I/A             | A               | A              |                 |
| 1             | WAXS          | Wide Angle 2mm beamstop      | 0    | 109.1                    | 0.9-0.9        | 0.037    | 2.06            | 168             | 3.05           |                 |
| 2             | MAXS          | Medium Angle 2mm beamstop    | 350  | 459.1                    | 0.9-0.4        | 0.009    | 0.50            | 706             | 12.5           |                 |
| 3             | SAXS          | Small Angle 2mm beamstop     | 950  | 1059.1                   | 0.7-0.3        | 0.0039   | 0.22            | 1629            | 28.7           |                 |
| 4             | ESAXS         | Extreme Small Angle 2 mm beamstop | 1400 | 1509.1                   | 0.45-0.2       | 0.0027   | 0.18            | 2321            | 41             |                 |


3. In the SPEC window, type the command
   a. SPEC> calib_trans
   b. This command moves the stage to the limit switch in Y and Z and center at hole 03,06.

4. In GICC, open Current Holder panel with “>” button.
   a. In the dropdown menu, choose “Ambient 8x8” holder.
   b. Middle 03,06 reference position?
Click Go.

c. Current Holder graphic is now accurately portraying the actual hole positions.

d. Actual position of hole 03,06 is **Horizontal: 29.580 mm** and **Vertical 38.768 mm**.

5. Move to the Blank Position (Horizontal, Vertical) at (1mm, 1mm) by clicking the **Go To** button.

6. In main control panel, click the button **“Fine Tune BS”** to realign the beamstop ensuring that the sample is not in the way as the automated alignment process occurs (30 seconds).

7. In Current Holders panel, hover over the positions of the sample to see the Z,Y coordinate.
   a. Select a hole where the silver behenate calibrant is positioned.
   b. At the bottom of the Current Holder panel, click the **“Sample to Beam”** button to move the sample into the beam path.
   c. In the Current Holder graphic, the green O should move to the desired position confirming the translation of the stage.

8. In Measurement Details, take a test measurement of silver behenate powder
   a. Description: SAXS Sample name 60sec
   b. Time: 60 sec
   c. Note down Image number and click **Measure** button.

9. In SAXSGUI, navigate to **File > Open latest**
   a. Select the file: `/home/saxslab/SAXSShare/data/latest/latest_xxxxxx_craw.tif` where `xxxxxxx` is the largest number possible in the directory.
      i. craw files are not corrected for cosmic background radiation.
      ii. caz files are corrected for cosmic background radiation (in corrected folder).
   b. SAXSGUI now continuously updates as data is acquired. The indicator is the Stop Monitoring button will be enabled.

10. Move to the sample of interest in position Z,Y by clicking on it in the Current Holder panel. Click **Sample to Beam** button.

11. In Measurement Details, take a test measurement of the sample of interest.
    a. Description: sample name, exposure time, hole position, sample thickness
    b. The exposure time is dependent on the following factors:
       1. Configuration:
a. **ESAXS** → X-ray beam traverses 1.5 m with slit sizes of S1: **0.45** and S2: **0.2** mm
b. **SAXS** → X-ray beam traverses 1.0 m with slit sizes of S1: **0.7** and S2: **0.3** mm
c. **MAXS** → X-ray beam traverses 0.46 m with slit sizes of S1: **0.9** and S2: **0.4** mm
d. **WAXS** → X-ray beam traverses 0.11 m with slit sizes of S1: **0.9** and S2: **0.9** mm

2. The square of the *scattering length density contrast* of a material system:

\[
\left( \rho_{\text{region}_1} - \rho_{\text{region}_2} \right)^2
\]

For a single element or compound, the scattering length density is calculated by the equation,

\[
\rho_{\text{XRAY}} = \frac{\rho_m N_A}{M} \sum_i n_i b_e z_i,
\]

where \( \rho_m \) is the mass density, \( N_A \) is Avagadro’s number, \( M \) is molecular weight, \( n_i \) is the number of atoms in molecule type \( i \), \( z_i \) is the atomic number of atom \( i \), \( b_e \) is the scattering length of an electron. The x-ray scattering length is calculated by

\[
\frac{\mu_0 e^2}{4 \pi m} = 2.85 \times 10^{-5} \text{Å},
\]

where \( \mu_0 \) is the permeability of free space (\( 4\pi \times 10^{-7} \text{ N·Å}^{-2} \)), \( e \) is the elementary charge (\( 1.602 \times 10^{-19} \text{ C} \)) and \( m \) is the mass of an electron, (\( 9.109 \times 10^{-31} \text{ kg} \)). Conveniently, \( b_e = r_f f_i \) the radius of the electron multiplied by the real part of the atomic scattering factor.

If the sample is a mixture, the contrast is governed by

\[
\rho_{\text{mix}} = \frac{V_1 \rho_1 + V_2 \rho_2}{V_1 + V_2},
\]

where \( V_1 \) and \( V_2 \) are the volume fractions of the mixture and \( \rho_1 \) and \( \rho_2 \) are the scattering length densities calculated from \( \rho_{\text{XRAY}} \) equation above.


3. Thickness of sample:
   a. The optimum thickness is one divided by the linear absorption coefficient of the material, \( \mu \).
      \[
      t = \frac{1}{\mu}, \quad \text{where} \quad \mu \text{ is the thickness at which the incident intensity is reduced by 1/e. [http://henke.lbl.gov/optical_constants/](http://henke.lbl.gov/optical_constants/)}
      \]
   c. To perform measurements on an *absolute scale* the thickness of the sample must be known.
      i. In Measurement Details, *type in the sample thickness* in centimeters.
      ii. Check boxes for “Transmission + 10” and “BS mask”
         i. The volume normalized differential scattering cross section is calculated by,
            \[
            \frac{1}{V} \left[ \frac{d\sigma}{d\Omega} (q) \right] = \frac{I(q) - B(q)}{I_0 T(t)(\Delta \Omega)}
            \]
where $V$ is the volume of the sample, $I(q)$ is the scattered intensity, $B(q)$ is the background intensity, $I_0$ is the intensity in the forward scattering direction, $T$ is transmission factor, $t$ is thickness of the sample and $(\Delta \Omega)$ is the collection solid angle. By collecting data as a function of the differential scattering cross section, one can quantify certain values (number density of scatterers, molecular weight, volume fraction, specific surface area, surface-to-volume ratio, volume weighted or number weighted mean size).

d. Note down Image number and click Measure button.

IV. Writing and Running an automated measurement macro

This section covers making a macro to automate the data collection at multiple sample positions, detector configurations and, when enabled, will display scattering data on an absolute scale. The y-axis is differential scattering cross section in units of cm$^{-1}$ and the x-axis is photon momentum transfer, q, in Å$^{-1}$.

1. Open the right panel for the macro editor with the “>” button.
   Notice: When clicking on any button in the GICC, the action is not performed but instead, written into the macro list.
   a. At the top of the macro editor, click the “New” button
   b. Move to the sample of interest in position (Z,Y) by clicking on it in the Current Holder panel. Click Sample to Beam button.
   e. In Measurement Details, select a measurement
      i. Add a description including: sample name, exposure time, hole position, sample thickness
      ii. Choose a measurement time in seconds.
      iii. Click Measure button.
   f. Repeat for all samples in holder.
   g. Save macros written in the GICC panel with the .gicc extension.

2. If you’d like to insert an instruction in a macro ahead of another without deleting all of the instructions, right click on the instruction and select “Insert before”. The instruction clicked has an anchor icon next to it.
   a. Perform mouse clicks on GICC to insert intended action.
   b. When done adding actions, right click and “Disable insert before”
   c. Save macros written in the GICC panel with the .gicc extension.

3. Check the sequence of the measurement macro.
4. Click the “Run” button to watch automated collection of the SAXS/WAXS patterns for each sample specified in the macro.
V. Data reduction with SAXSGUI

This section walks you through the steps of data reduction with SAXSGUI software from 2D image data to 1D profiles that can then be loaded into standard SAXS data analysis software (SASview).

1. SAXSGUI Basics for output of data:
   a. Processing > AP with Metadata
      1. Select the *cosmically corrected*.caz files from `/home/saxslab/SAXSShared/data/latest`
      2. The shared folder to save files: `/home/saxslab/SAXSShared/Your folder`
   ii. Data will be outputted in the follow file extensions
      1. .grad file (ASCII file with header details)
         a. 3 column format ($q$, $I$, $\sqrt{I}$) or
         b. 3 column format ($q$, $\frac{d\sigma}{d\Omega}$, $\sqrt{d\sigma}$)
      2. .jpg of 1D data from azimuthally averaged 2D data
      3. .jpg of 2D image with colorbar
      4. .jpg screenshot of SAXSGUI screen
   iii. If the SAXSGUI program fails to Autoprocess, Close it and reopen via the Toolbar.

Appendix A. Linux info
The instrument computer operating system is Ubuntu version 14.04 LTS. Tutorials available at http://linuxsurvival.com/

Appendix B. Additional instrument info
This section provides additional information about the controlling the drives on the instrument via SPEC commands and Pilatus3R 300K detector information.

1. The Scans section of the main control panel provides the ability to optimize on the center of holes or capillaries.
2. Click on the position of the sample you intend to align.
3. For fine alignment of a capillary sample, click “Align Capillary” button or for hole with sample, click “Align Hole”.

   a. Select Vertical or Horizontal alignment radio button.

   b. Select a half range value appropriate to the size of the object that is to be aligned. Example: holes are 4mm in diameter and refillable capillaries have 2mm outer diameter so 3mm on each side of the current position is appropriate, 6mm in total will be scanned.

   c. In this mode, the beamstop will move out of the way and the entire Pilatus detector will integrate all counts and plot as a function of ysam. The specplot software will be automatically initiated.

   ![Filled capillary scan](image1) ![Empty capillary scan](image2)

Note: the beamstop does not re-position itself back into the same spot after the scan has finished. To move the beamstop back to the previous position, in the Beamstop menu on the bottom left of the main graphics menu, click the button “In”.

Detector info:

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### Detector info:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
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<tr>
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<tr>
<td>Total number [pixels]</td>
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<td>Gap widths [µm x 2]</td>
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