SAXS for biology optimized on the beamline SWING

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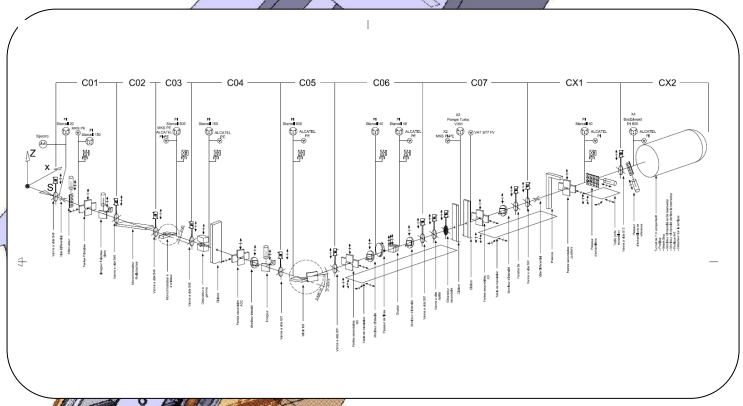
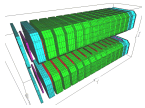
Abstract

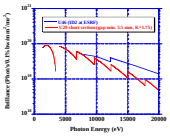
SWING is a beamline devoted to (A)SAXS, WAXS and (A)GISAXS at SOLEIL, opened to all scientific domains with no exclusion. The source is a U20 in-vacuum undulator, thus providing a beam with small size and low divergence. The energy range is 5-17 keV (Si111 Double Crystal Monochromator) with an expected flux of about 10^{13} photons/s. Focusing of the beam onto the detector plane is achieved by bending two perpendicular mirrors (Kirckpatrick Baez configuration), thus being insensitive to a change in energy. The size of the focused beam should be typically 400 µm x 100 µm (HWHM). A large motorized table allows for fine positioning of sample environments. A 17 x 17 cm² CCD detector is positioned on a three axes translation stage within a large chamber under primary vacuum. The sample to detector distance ranges from 0.6 to 8 m. A friendly graphical interface written in Java allows for collection and online data reduction, including automated determination of radius of gyration, peak maximum intensity and width, etc. Absolute intensity measurements will be achieved by using reference samples and calibrated monitors.
Despite its scientific versatility, a great effort has been made to provide biologists with optimized experimental conditions. An in vacuum thermostated capillary cell with online measurement of UV-Vis absorption will permit precise and reproducible measurements of proteins in solution. An HPLC system will be proposed for online purification. An automated solution sampler is presently under optimization for high throughput data collection. The complete biochemistry lab of SOLEIL, situated close to the beamline, is opened to those who need it.
SWING is at the moment under assembly and commissioning. We expect to welcome our first external users by the end of 2007.



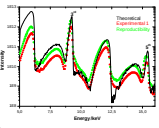


To deliver a high brilliance X-ray beam, an in-vacuum undulator is necessary: a gap value as small as 5.5 mm can be reached. The magnetic design of the U20 undulator was made by the insertion group of SOLEIL. The mechanical and vacuum design is close to that developed at ESRF.

σ_x (µm)	σ_z (µm)	σ'_x (µrd)	σ'_z (µrd)
388	8.1	14.5	4.6

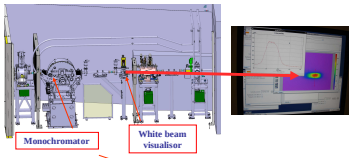


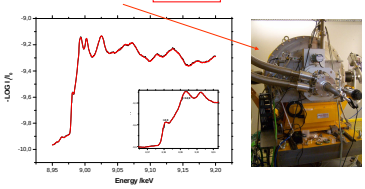
Theoretical calculations of U20 brilliance as a function of energy with constantly optimized gap.



Emission spectrum of SWING undulator kept at a fixed gap, g=12 mm, and I=100mA, compared to theory.
Measured with a diode after the first mirror by tuning the monochromator energy

The white beam was delivered for the first time on March, 15th, in the monochromator optics hutch

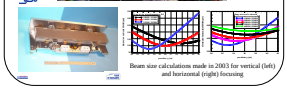




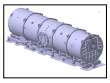
Absorption spectrum of a thin foil of Cu near its K-edge @ 8.979 keV, with mirrors inserted. The monochromator repeatability is better than 0.2 eV

The beam was focused at the detector position for the first time on July 12th, just before the summer shutdown !

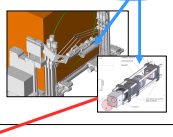
KB mirrors vacuum chamber

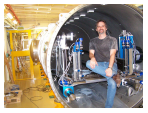


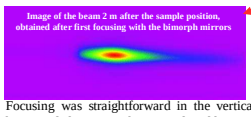
Detector vacuum chamber (10^{-2} mbar)

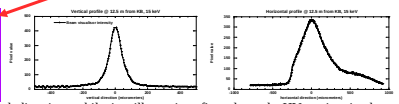


- longitudinal displacement : 6 m
- transversal displacement (x_d) : 40 cm
- easy opening of back part
- 3 beam-stops + 2 centered attenuators
- 1 beam visualisor





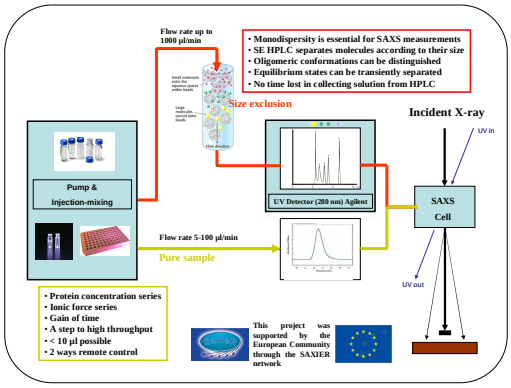




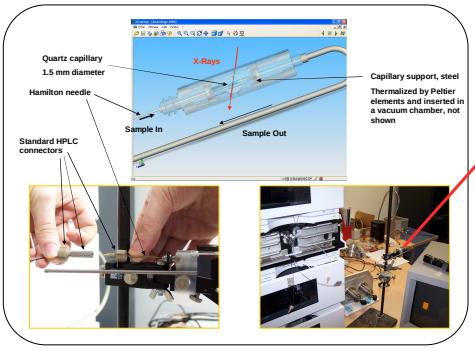
Focusing was straightforward in the vertical direction, while it still requires fine electrode HV tuning in the horizontal direction. The vertical width was checked to remain around 100 µm whatever the position within the detector vacuum chamber.

A complete system of purification and automated solution sampler will be available for SAXS measurements on proteins in solution

Principle of the online purification / autosampler



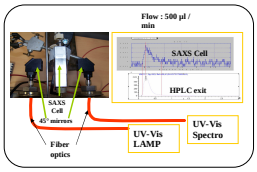
Connection of the HPLC system to the SAXS measurement cell



A step to high throughput

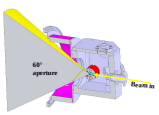
3 µl of water between two air bubbles of controlled volume can be inserted with high repeatability.

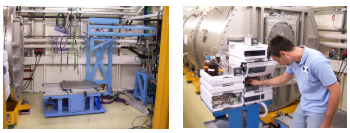
The injection system is presently being optimized to get the same behaviour with protein solutions.



The first tests performed on the online SE-HPLC with a solution of BSA showed that no dilution occurs between the HPLC exit and the SAXS measurement cell.

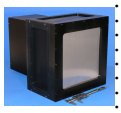
The SAXS measurement cell is inserted in a vacuum chamber with a large angle exit which can be directly connected to the front of the detector chamber, thus eliminating scattering by air or windows. Fiber optics allow for on-line UV-Vis absorption spectroscopy. A video-camera constantly points towards the sample capillary.





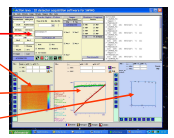
The insertion of the set-up within the X-ray beam path is facilitated by the large amount of space available around the motorized table for sample environments.

PCCD170 detector made specially for SWING by AVIEX



- Stable Offset.....Offset directly read on hidden pixels
- Low noise.....< 0.8 X Pb rms (12 keV), with M = 1.7
- Large area.....170 x 170 mm² → (Ω_{min}/Ω_{max} > 80)
- High Dynamics....17000
- High saturation....14000 X Pb / Pixel
- Fast.....0.5 s (full frame), 0.12s (bin : 8 x 8)
- Small pixels.....43 µm
- Narrow PSF.....FW 1% < 300 µm, FW 1%< 1 mm

Java graphical interface for on-line data reduction



Software donwloadable by users