

How should we build the biological SAXS beamline(s) at NSLS-II?

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**Bio-SAXS breakout session, NSLS-II workshop
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Overview

§ How does the high brightness of NSLS-II translate into better user experiments

Small beams: time-resolved, fibers

Membrane structures

§ Do we really want higher flux for every measurement

Limitation is radiation damage

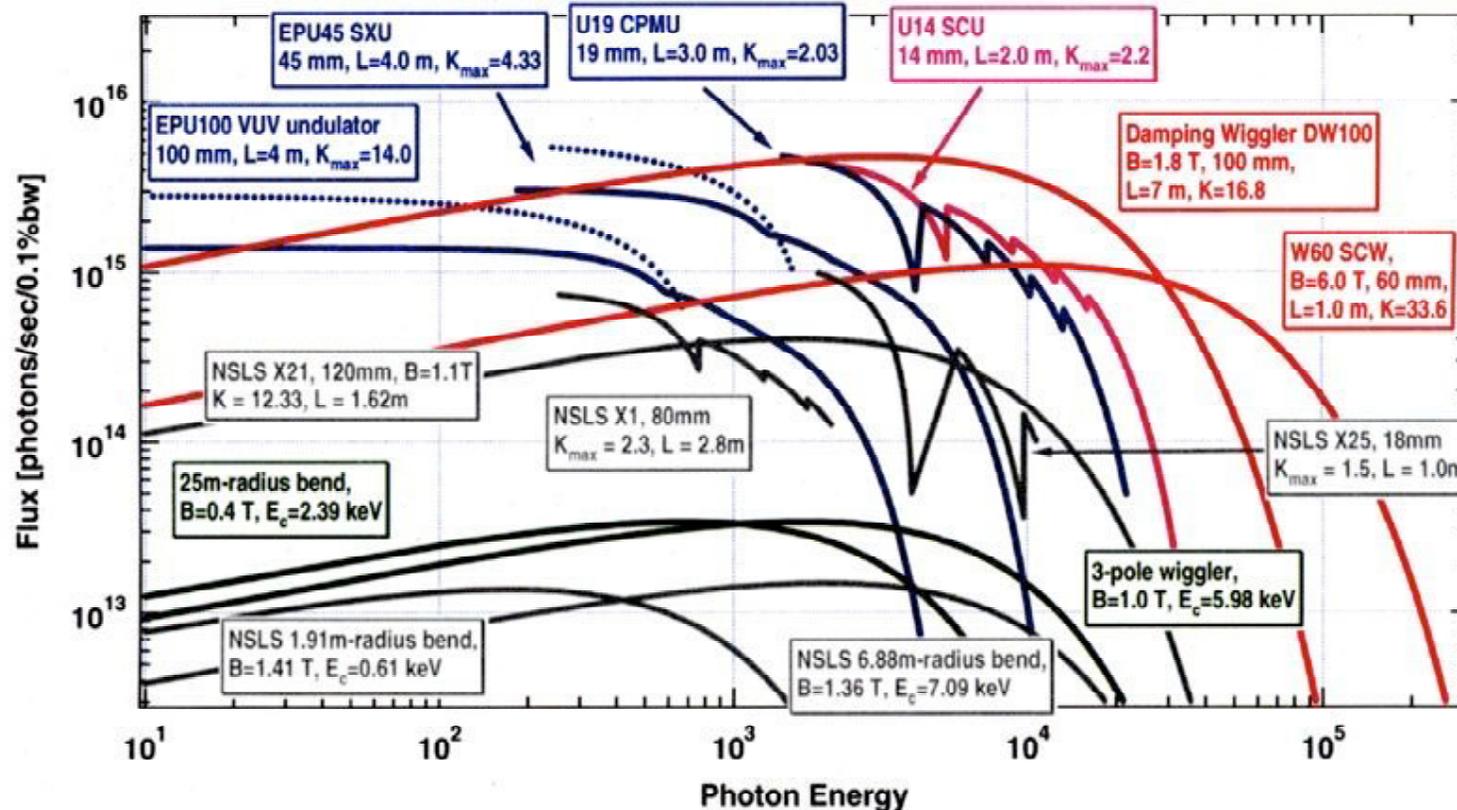
§ What else can the facility do for the user community

Multi-probe

Smaller sample consumption

User support: mail-in service, data analysis

NSLS-II source flux



Flux at the sample (assuming same SAXS setup as X21):
 BM: $\sim 10^{10}$ ph/s, ID: $\sim 3 \times 10^{13}$ ph/s
 Compared to $\sim 3 \times 10^{10}$ ph/s at X21 (wiggler, 10keV).

Small Beam + Small Angle

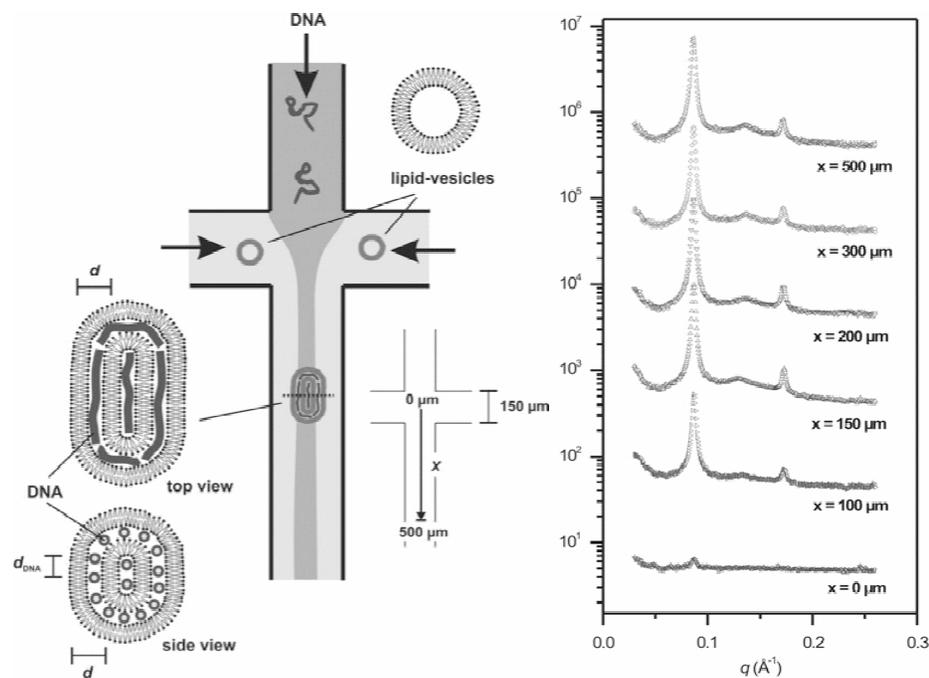
$$B \sim \frac{F}{\Delta\sigma\Delta\Omega} \quad q = \frac{4\pi}{\lambda} \sin \theta$$

Time-resolved SAXS using flowcells

hydrodynamic focusing, continuous flowcell
Austin group, Princeton, 1998

protein folding studies, Pollack group, Cornell, 1999

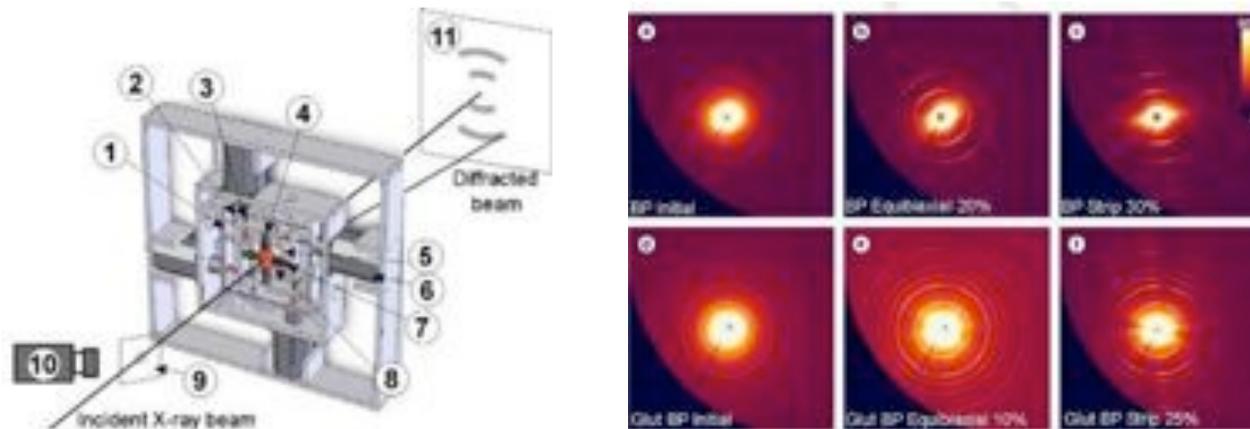
Self-assembly of DNA-intercalated lipid multi-layers
Otten et.al. J. Synch. Rad., 2005
ESRF, ID10B, CRL, $\sim 20\mu\text{m}$ spot size



Hard X-ray + Small Angle

$$q = \frac{4\pi}{\lambda} \sin \theta$$

Hydrated biological tissues

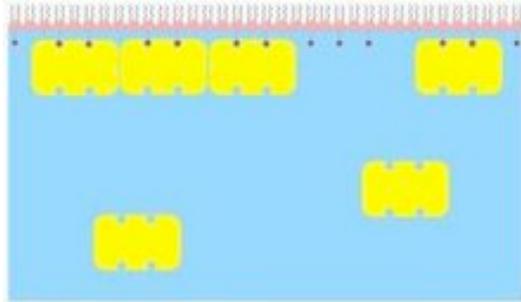


Diffraction from collagen fibers in connective tissues under biaxial stretch. The sample was submerged in an aqueous buffer, X-ray path length $\sim 7\text{mm}$

Liao et.al., Acta. Biomater., 2005

2D crystallography

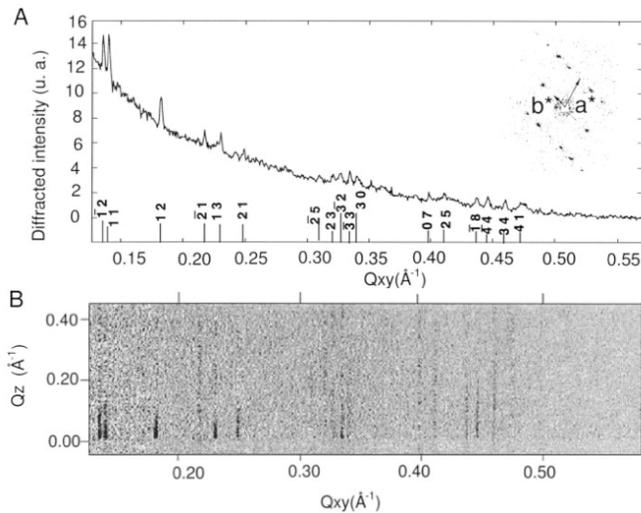
2D Streptavidin crystals at air-water interface



Vertically focused beam:
14 μ m FWHM, \sim 1" footprint

trough size is \sim 2" x 1.5"
(limited by beam footprint)

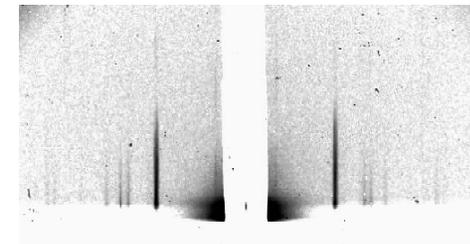
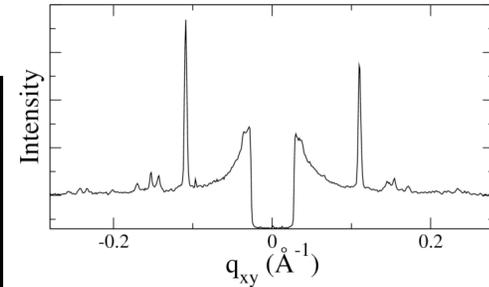
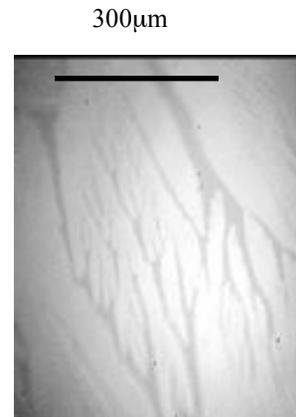
CCD for parallel detection.



Lenne et.al., Biophys. J., 2000

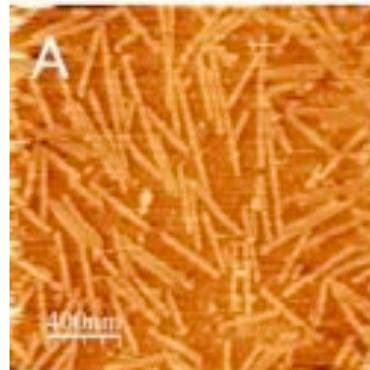
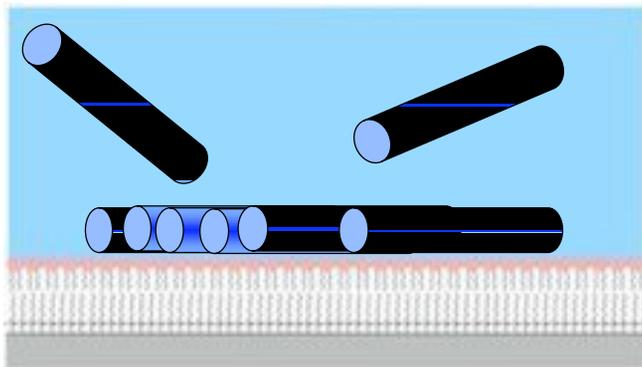
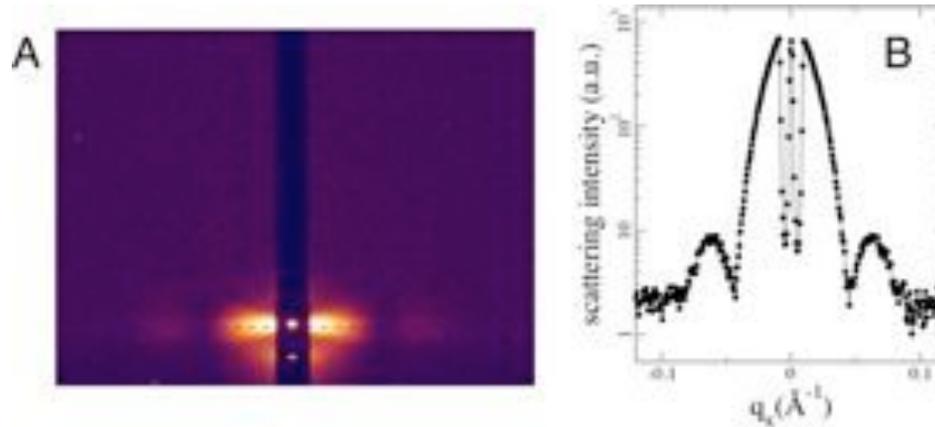
Normally requires large amount of sample (80mm x 120mm)

Linear detector + Soller slits for high resolution: Must scan



Fukuto, Wang, Yang on-going

2D solution scattering



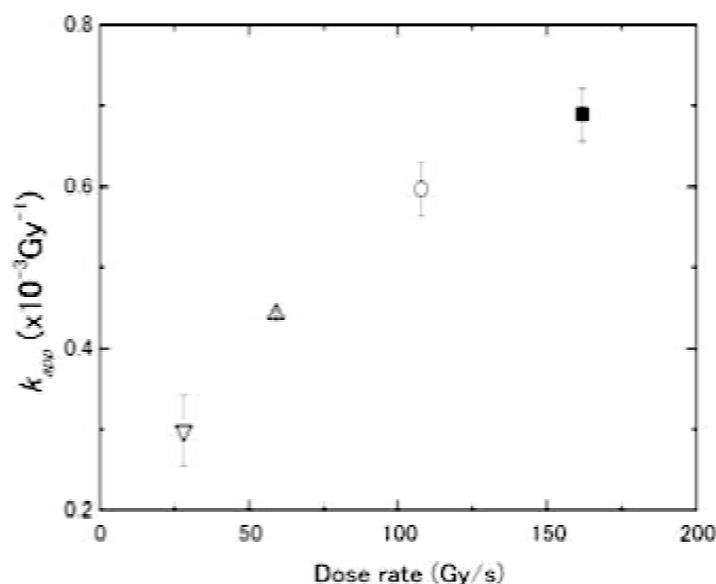
Tobacco Mosaic Virus (TMV) adsorbed to a substrate submerged in a buffer solution

Yang, Wang, Fukuto, Checco on-going

Radiation damage

Radiation-induced aggregation in lysozyme solution

Total dose limitation: Henderson/Garbin limit for protein crystals ($\sim 3 \times 10^7$ Gy).
400 Gy for radiation-induced aggregation to appear.



Kuwamoto et.al.,
J. Synch. Rad.,
2004

Dose rate limitation: No aggregation below 10 Gy/s. Faster aggregation at higher dose rate.

- ⌘ @10 keV, 1 mm² beam size, 1 mm path length (40% absorption)
must flow the sample if the flux exceeds 1.5×10^{10} ph/sec

Instrumentation Strategy

For radiation-hard samples and time-resolved studies

ID beamline, modelled after X9 (in construction)
shared with non-biological SAXS

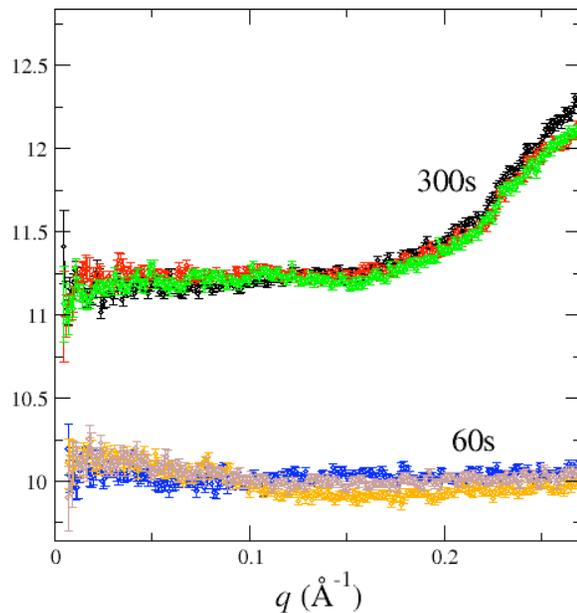
For protein solution scattering

bending magnet / 3-pole wiggler source
modelled after X21
ML monochromator + toroidal mirror
dedicated to solution scattering, with support for data analysis
partner with PX beamline

Detectors

CCDs are most commonly used today

- readout noise
- dark current (affected by temperature stability)
- zingers
- intrinsically low QE



Averaged dark current from a Mar CCD

Other options are becoming available

Pixel array detector

PILATUS, SLS
79.4 x 34.1mm²
0.217mm pixel
photon-counting
20bit dynamic range
100frames/sec



Flat panel imager

Hamamatsu, Rad-Icon
very compact
good for SAXS/WAXS
very high dark current



Simultaneous SAXS/WAXS

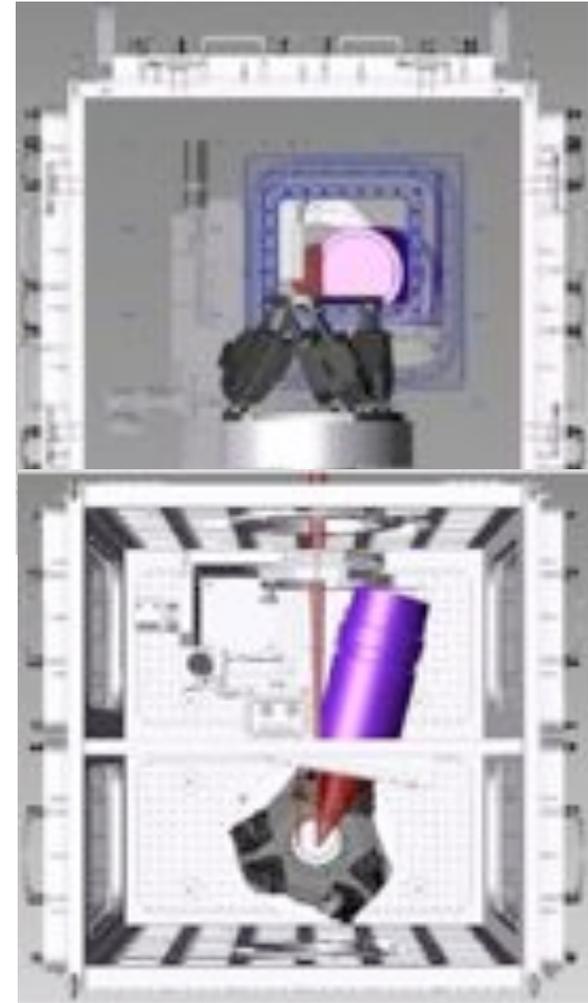


WAXS detector custom made by Photonic Science



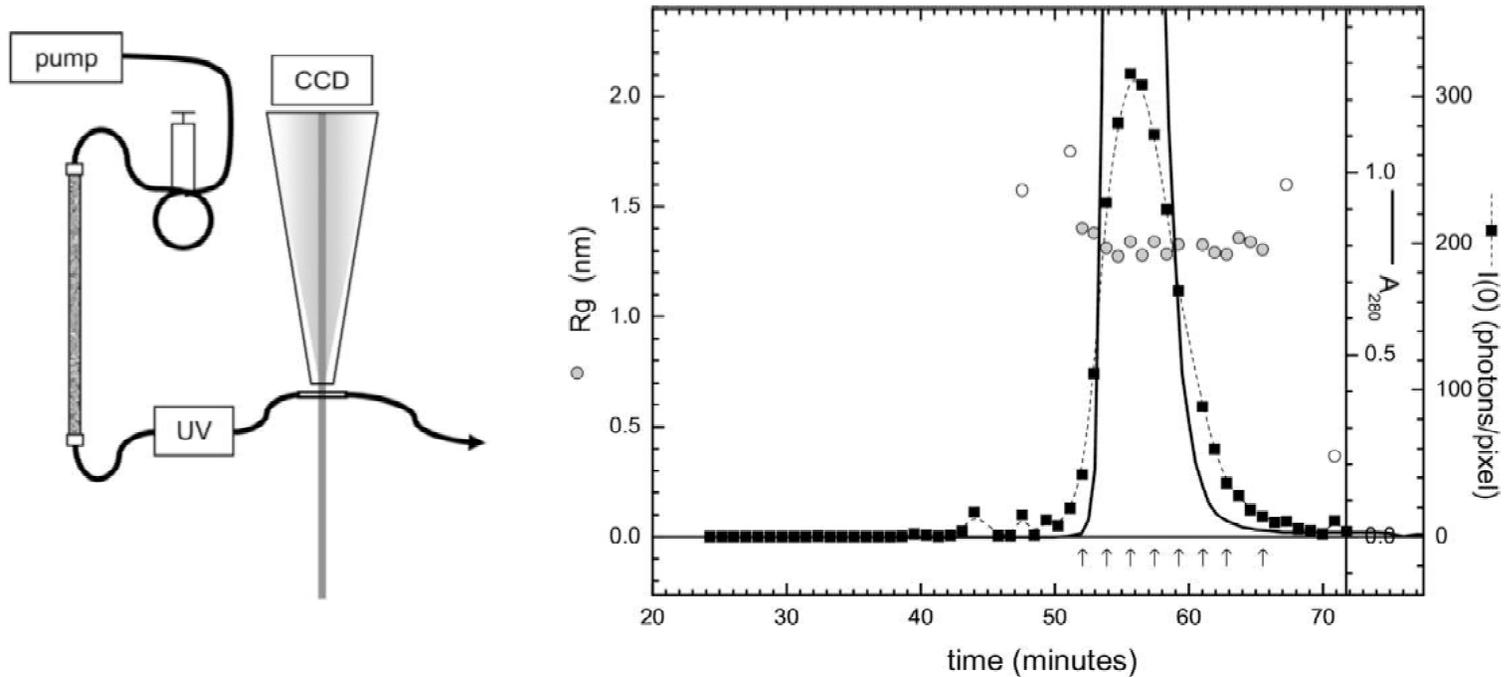
Roper SAXS/WAXS pair at APS DND-CAT

The detector is no longer in production



X9, construction in progress

Multi-Probe



Mathew et.al., J. Synch. Rad., 2004

Chromatography detectors (UV-Vis, CD, DLS, ...) provide more information on the sample.

OR

SAXS/WAXS is just another chromatography detector.

User Support

What makes SAXS/WAXS just another detector:

software to provide simple (R_g , I_0 , ...)

staff support for more advanced data analysis

automatic sample handling

Automatic Sample Handling, Mail-in Service

Multiple sample cells

simple, fast
path length, scattering background not reproducible; can't flow sample

Liquid sample handler w/ single flow-through cell

solution processing possible
slow; air bubble



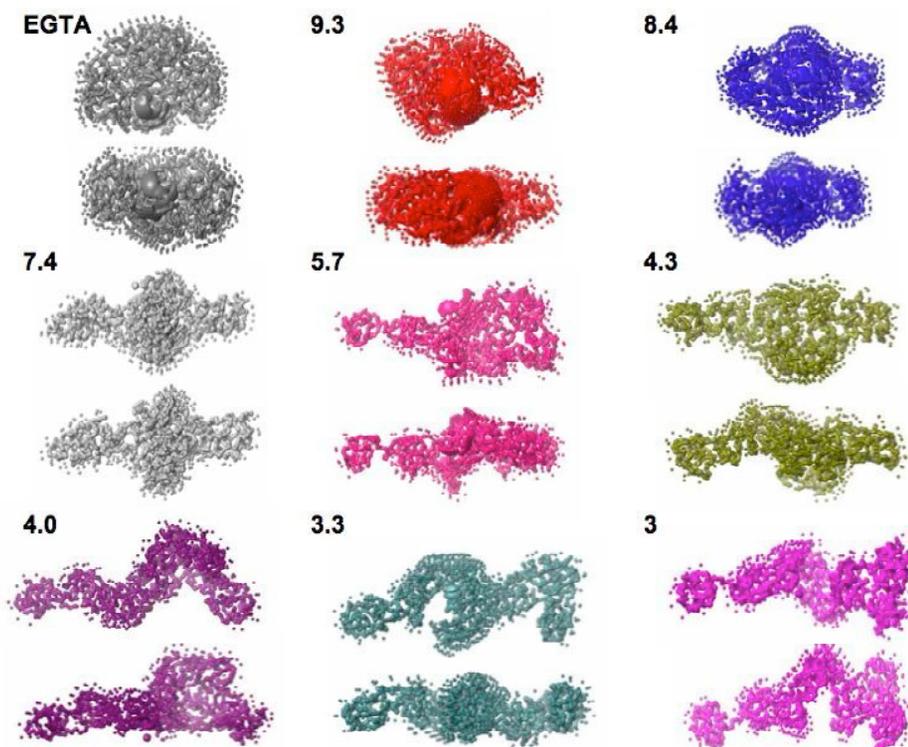
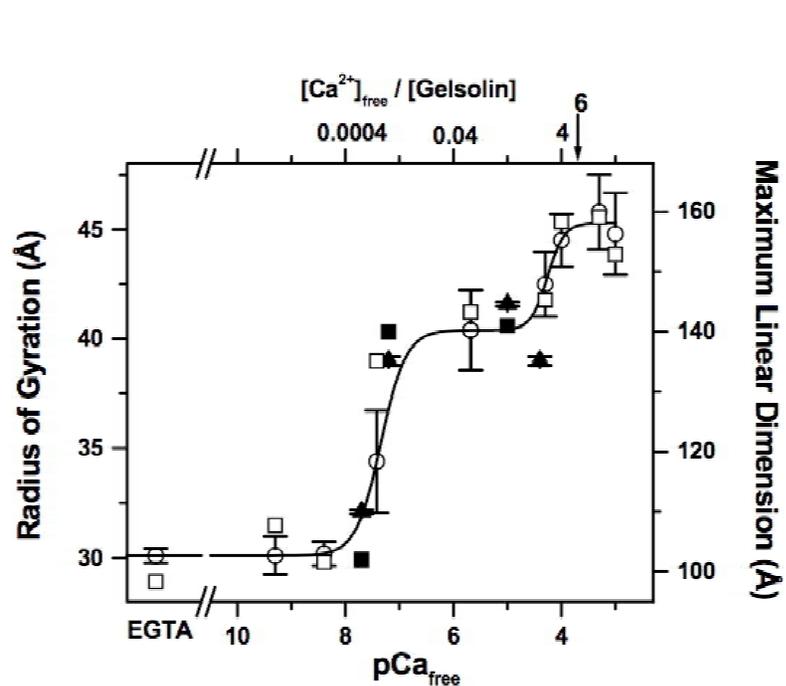
Gilson 215, single loop



home made, two loops

Benefits of Automatic Solution Processing

Chemical assays with SAXS



Conformation change of Gelsolin as a function of free Ca concentration

Ashish et.al., J. Biol. Chem., 2007

Summary

§ 1 ID + 1 BM

ID SAXS beamline shared with other experiments

BM-based SAXS beamline dedicated to solution scattering

§ Build up the user base today

Very few user groups now (limited by beamline capacity)

X9

Requested post-doc to support data analysis

§ Develop capability for user support and multi-probe

