



# Overview of APS SAXS Capabilities and Small-Angle Scattering Discussion Group

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*2004 APS User's Meeting, Thursday, 6 May 2004*

*Small-angle scattering is the premier technique for the size characterization of nanoscale objects.*

<http://small-angle.aps.anl.gov>



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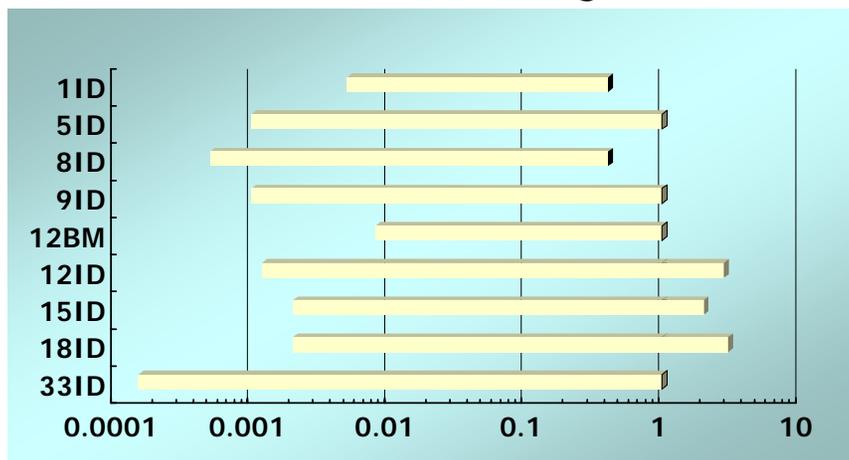
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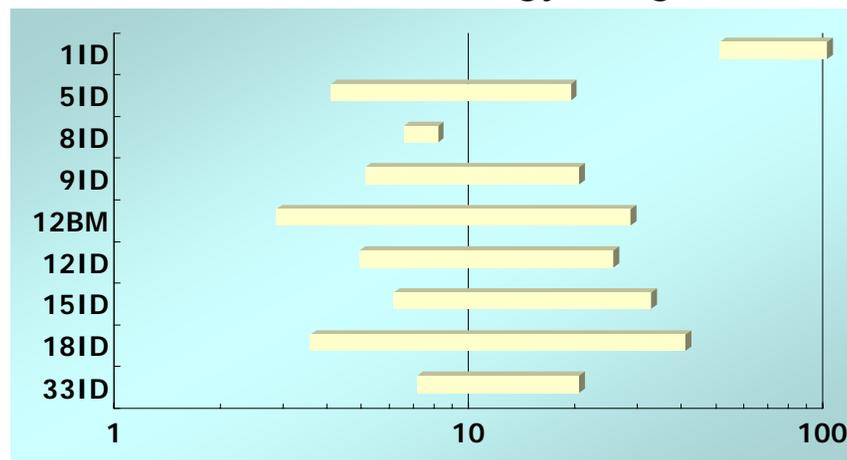
# Basic information

Nine different small-angle X-ray scattering (SAXS) beam lines are accessible to the APS general user. The combined capabilities of these beam lines span a broad range of reciprocal space and X-ray photon energy allowing for investigations from many disciplines of science including biology, materials science, environmental science, chemistry, medicine, and physics. Coupled with a high data throughput and fast time-resolved measurement capabilities, these instruments enable an efficient use of the high intensity and high brilliance APS X-ray source.

*accessible Q range*



*accessible energy range*



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# Beam Time Allocation Statistics

## (3 examples)

### 5ID DND-CAT

50% of 5ID beam time goes to SAXS (CAT and GU)

60% SAXS oversubscription (i.e. hours requested/hours schedules = 1.6)

25% of 5ID total beam time is given to GUs

50% of 5ID assigned GU time on the ID goes to SAXS

### 12ID BESSRC/XOR-CAT

42% of beam time went to SAXS/WAXS & grazing incidence in 2003

### 33ID UNICAT

45% of 33ID beam time in 2003 went to USAXS (CAT and GU)

33% of 33ID beam time in 2004 (to date) goes to USAXS (CAT and GU)

3.5x oversubscribed for USAXS in 2003

4.5x oversubscribed for USAXS in 2004-2 (2 out of 9)

25% of total beam time is given to GUs



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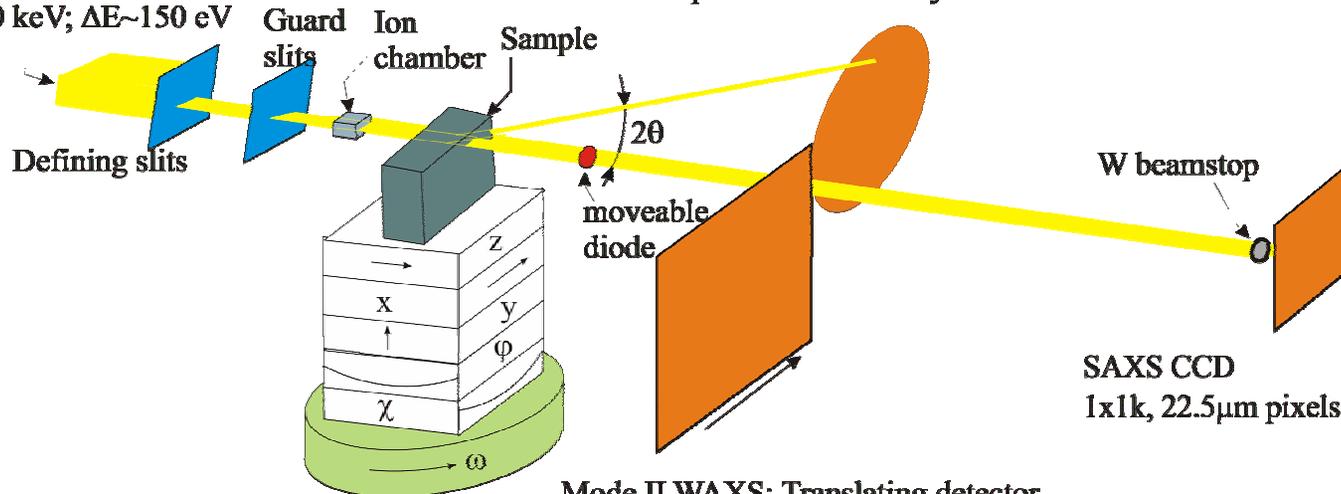
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# High-energy SAXS/WAXS at 1-ID

Beam from Undulator A  
and HE monochromator:  
 $E \sim 80 \text{ keV}$ ;  $\Delta E \sim 150 \text{ eV}$



Mode I WAXS: Fixed detector  
(CCD, 2x2k, 60 $\mu\text{m}$  pixels):  
capture  $\sim 1/2$  of Debye cones

Mode II WAXS: Translating detector  
(Image Plate 3.4x3.4 k, 100 $\mu\text{m}$  pixels):  
capture full Debye cones

## Typical q-ranges

- SAXS: 0.002-0.1 1/Å
- WAXS = 0.2-5 1/Å

## Features

- *Direct* depth resolution to  $\sim 5\mu\text{m}$  with current pinhole setup
- Strong penetration ability: comparable to neutrons
- Perform WAXS, SAXS (and imaging) with little change in setup
- $t_{\text{exposure}} \sim 1-100 \text{ sec}$  (both WAXS and SAXS)
- Plan to incorporate refractive-lens focusing optics (late 2004):
  - anticipate  $q(\text{min})$  to  $\sim 0.0005 \text{ 1/Å}$
  - 1-D spatial resolution to  $\sim 1\mu\text{m}$



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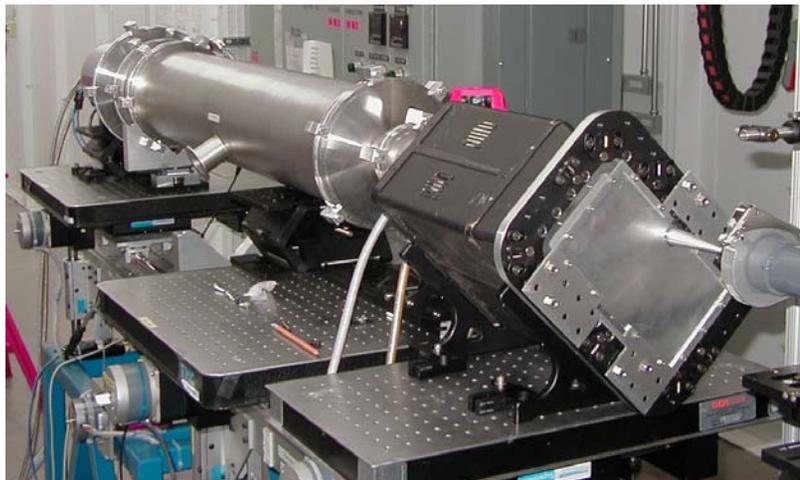
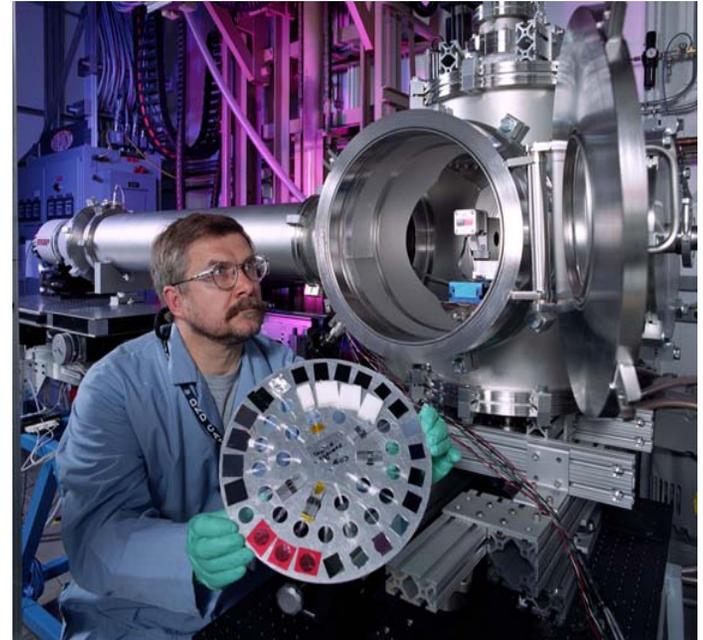
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# 5ID-D SAXS Capability Summary

- Ultra low background SAXS with an in vacuum sample changer. Which also allows for automated data collection on hundreds of static samples per day.
- Camera lengths up to 10m
- 7 – 17 keV energy range
- 4 sets of collimating slits
- Linkam Differential Scanning Calorimeter
- Linkam Hot Stage
- Primary detector is a Mar-USA 162mm CCD



- High speed (up to  $\sim 4$ Hz), simultaneous SAXS/WAXS on Roper CCD area detectors.



# 8-ID SAXS Capabilities

- X-Ray Photon Correlation Spectroscopy (XPCS) and Time-Resolved SAXS and Grazing-Incidence SAXS
- Monochromatic ( $\Delta E/E = 0.0003$ ) and wide-bandpass, high-intensity ( $\Delta E/E = 0.02$ ) “pink” incident radiation
- Length Scales from 5 nm to 900 nm
- Time scales from 5 ms to 1000 s
- In-vacuum measurements from -35 to 200 °C
- Direct-detection deep-depletion area detectors
- Sophisticated and easy-to-use data reduction algorithms



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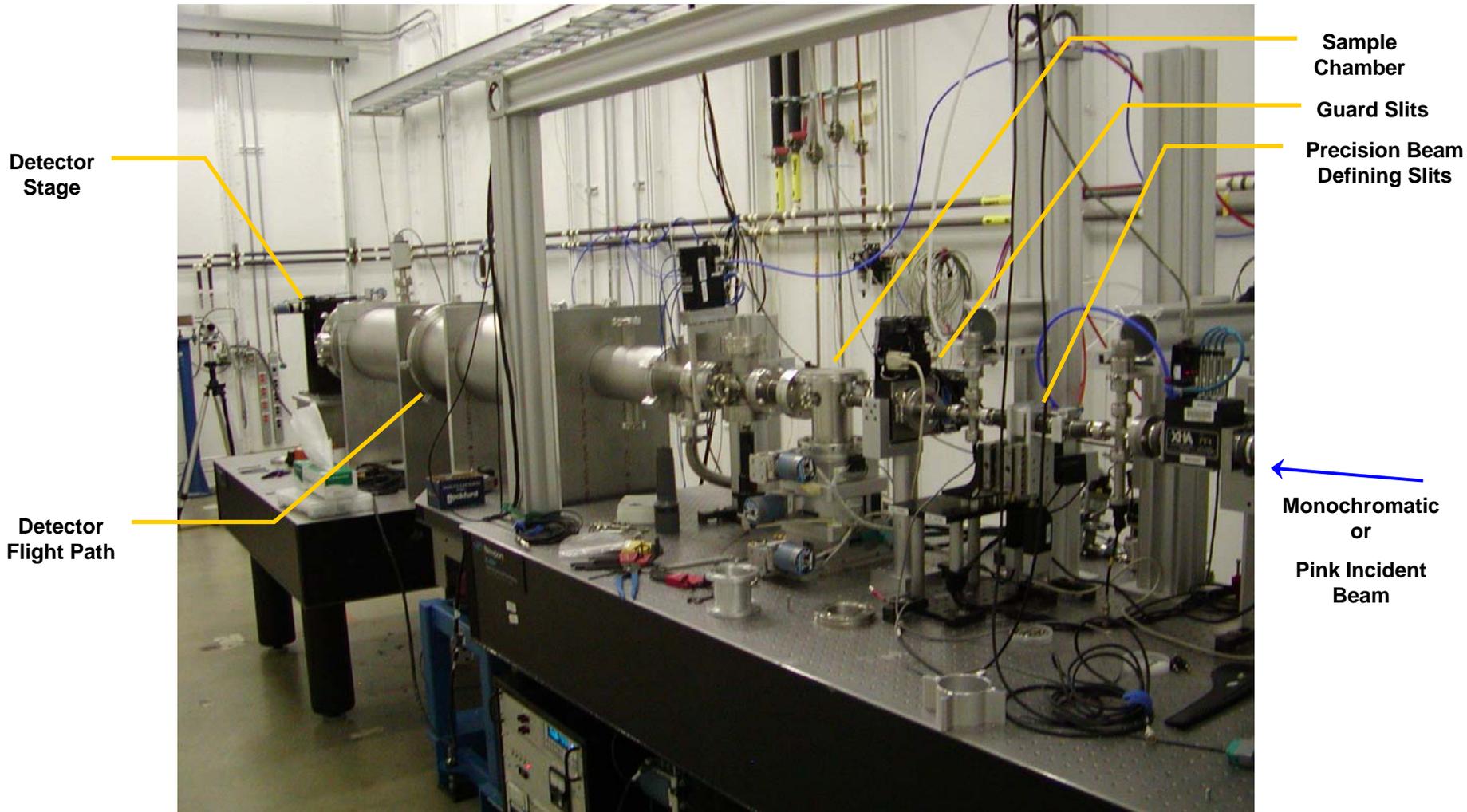
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# 8-ID SAXS Capabilities



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# Performance at 12ID-C

- Of the 215 days of beam time in 2003, 90 days or 42% was assigned to SAXS/WAXS and grazing incidence.
- 12ID-C SAXS station
  - regular and small angle X-ray scattering (SAXS)
  - anomalous small angle X-ray scattering (ASAXS)
  - grazing incidence small angle X-ray scattering (GSAXS)
- Systems being studied:
  - carbon and PAH from soot, fuel, coal, solvents, etc.
  - polymers, clay complex fluids
  - biological samples like proteins, RNA
  - Actinides, rare earth complexes
- The  $q$ -range of study varies from  $0.0015 \text{ \AA}^{-1}$  to  $2 \text{ \AA}^{-1}$ .



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# 12 ID-C design and set up

R. E. Winans, S. Seifert, D. Tiede, K. Carrado, P. Thiyagarajan, Chemistry Division, ANL

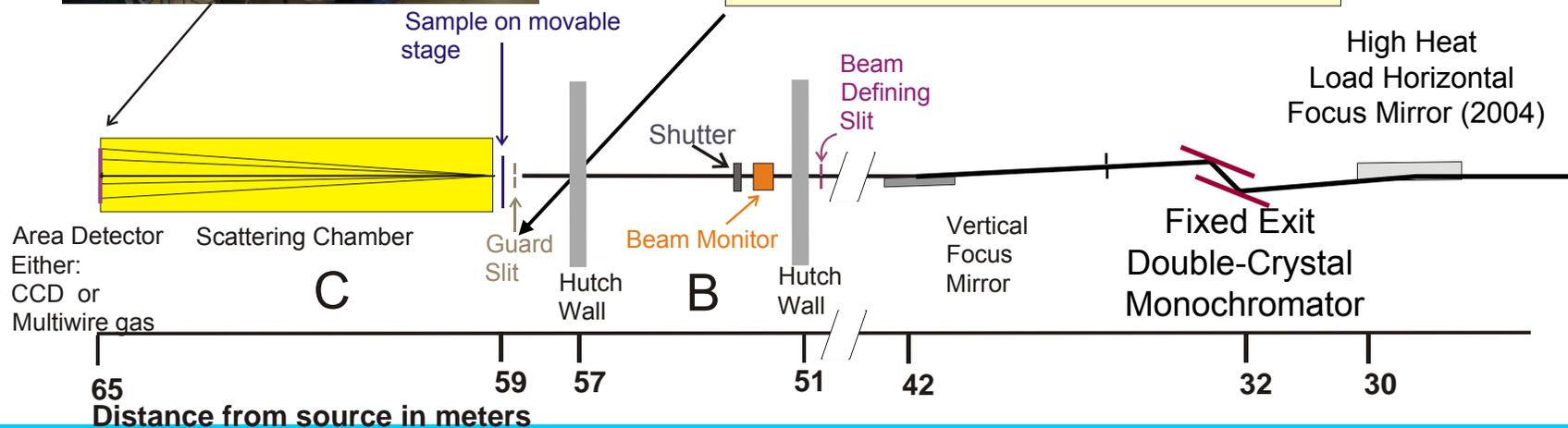
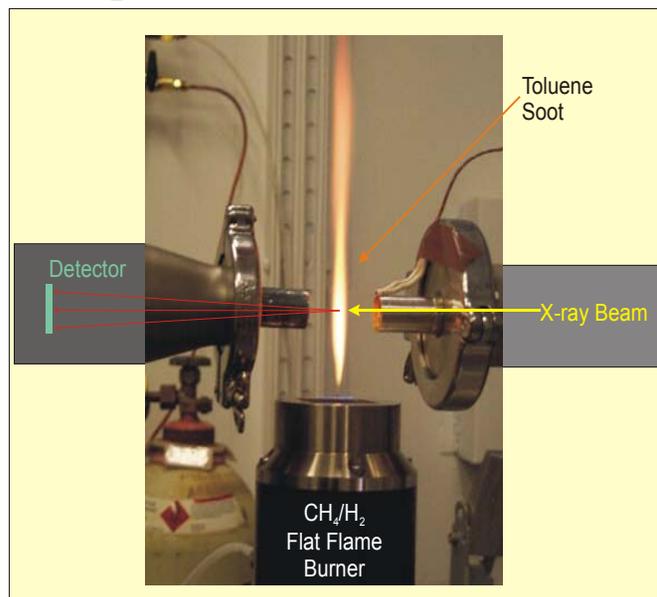
## MOSAIC CCD - GOLD DETECTOR

Size: 15 cm x 15 cm

Pixels: 3000 x 3000

Pixel size: 49 microns

Image readout: 1.7 sec



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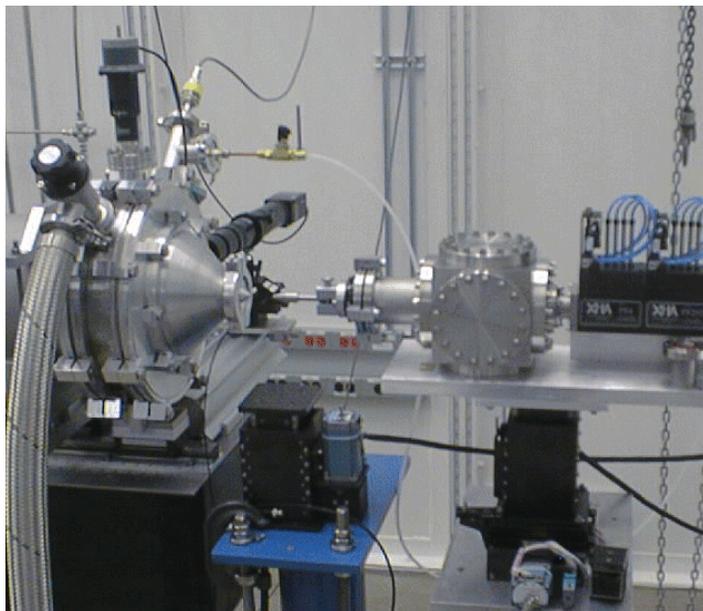
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# ChemMatCARS

## SAXS/WAXS instrument

Scientist in charge : David Cookson



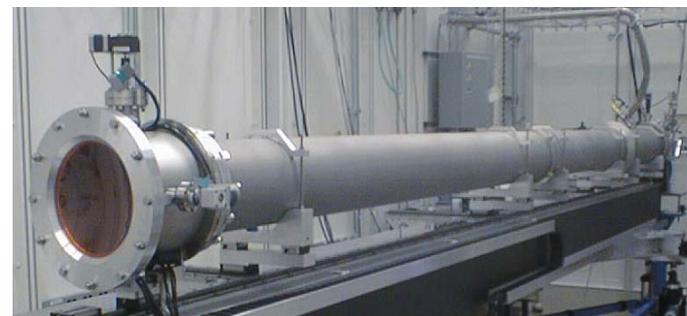
### Facility Scientists

- David Cookson
- Myungae Lee
- David Schultz



- |                                     |            |
|-------------------------------------|------------|
| First light through instrument      | - Dec 2000 |
| First signal to noise trials        | - Feb 2001 |
| First sample in-vacuum trials       | - Aug 2001 |
| First dilute solution scattering    | - Aug 2002 |
| First <i>in-situ</i> fiber spinning | - Apr 2003 |
| First temp crystallization studies  | - Apr 2003 |

# SAXS at ChemMatCARS (Sector 15)

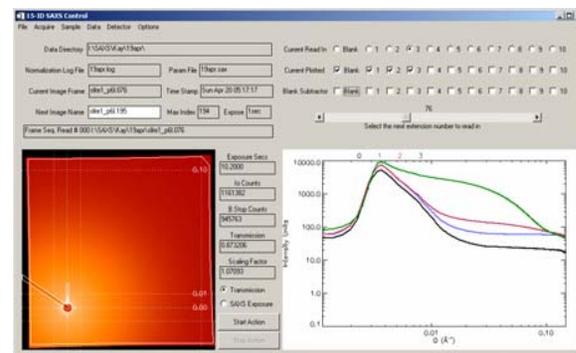


Sample view and alignment

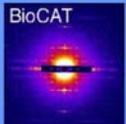
200um spot on phosphor



- Camera lengths of 0.5, 2 and 7 meters
- Length changeover takes ~ 20min
- Energy range 6 – 32keV
- Minimum Q ~ 0.0015 (1Å, 7metres)
- In-line optical sample alignment
- Integrated control & data processing
- 1.5meters of floor space for sample apparatus



BioCAT

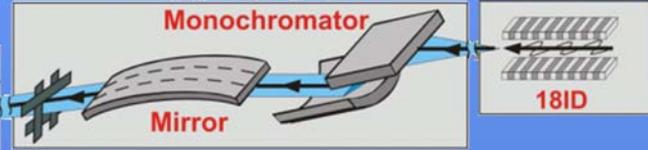
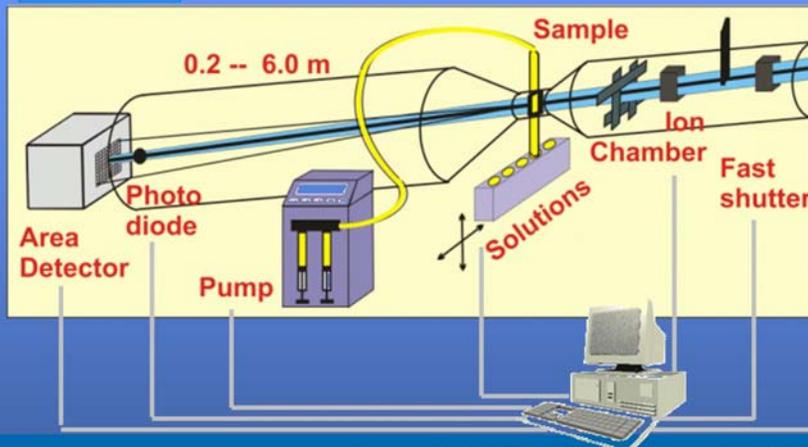


18ID

# SAXS FROM BIOLOGICAL SOLUTIONS

## From High-Throughput Automation to Microsecond-Time Resolution

Contact: Tom Irving, Elena Kondrashkina  
[www.bio.aps.anl.gov](http://www.bio.aps.anl.gov)

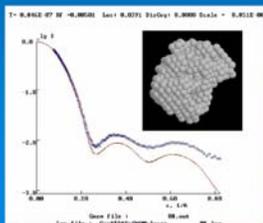


- $10^{13}$  X-ray photons on sample
- 30 – 150  $\mu\text{m}$  beam size
- < 1 mg/ml min. solution concentration
- 100 ms – 1 s standard exposure time
- 3 ms time resolution, stopped-flow mixer
- 150  $\mu\text{s}$  resolution, continuous-flow mixer

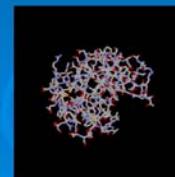
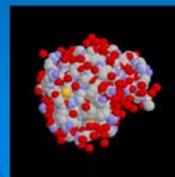
Automated Data Acquisition: CCD, EPICS, BioCAT-Scan software

Initial Data Processing, Rg, Dmax, P(r): BioCAT-IgorPro macros

Molecular shape: D.Svergun's programs [www.embl-hamburg.de](http://www.embl-hamburg.de)



Lysozyme-molecule shape simulations



Lysozyme PDB data, 6lyz

APS User Meeting, May 2004



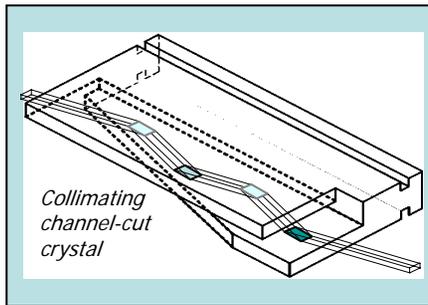
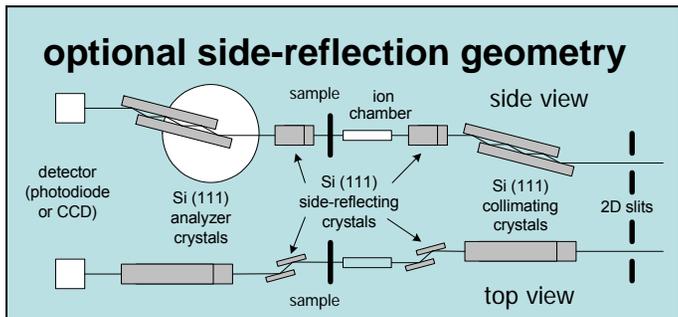
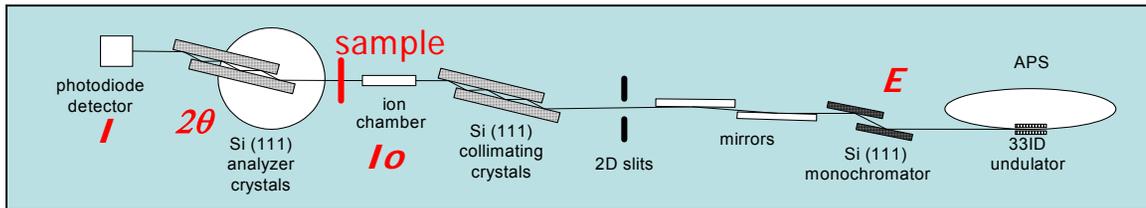
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## Versatile USAXS (Bonse-Hart) facility for advanced materials research at APS Beam Line 33ID-D



- Q range:  $0.00015 \leq Q, \text{Å}^{-1} \leq 1$
- X-ray energy tunability: 7 – 20 keV
- energy resolution:  $\Delta E/E \approx 0.00015$
- energy harmonic content  $< 10^{-6}$
- 0.5 x 2 mm<sup>2</sup> beam size, adjustable
- $I_0 > 10^{12}$  ph/s (10 keV) delivered to sample
- sample in air (ca. 1/4 m from detector)
- automatic sample changer/translation stage
- absolute determination of  $d\Sigma/d\Omega(Q)$
- USAXS imaging

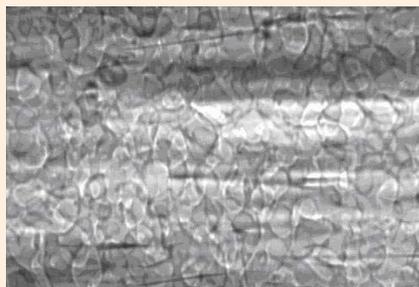


# Example USAXS data

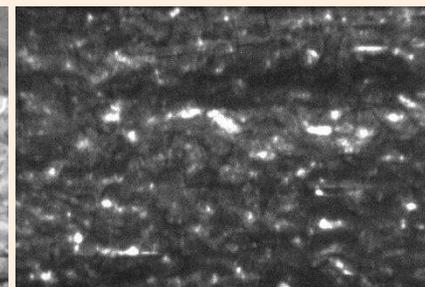


[www.uni.aps.anl.gov/usaxs](http://www.uni.aps.anl.gov/usaxs)

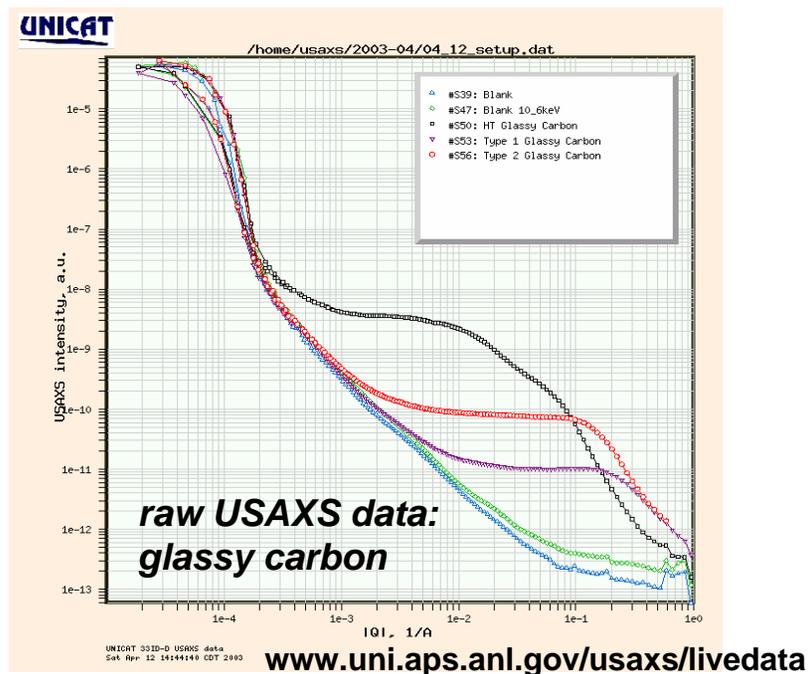
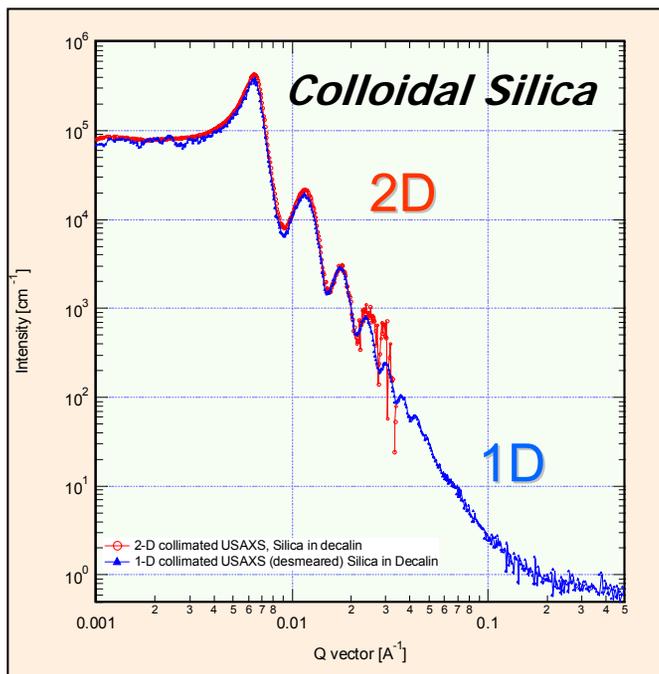
## USAXS image: tissue engineered sample



radiograph (Q=0)



USAXS image (Q=0.0005 1/A)



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# ANL Small-Angle Scattering Special Interest Group

## Our Mission

The Small-Angle Scattering special interest group at Argonne National Laboratory has been created to promote awareness of the small-angle scattering facilities at the APS, IPNS, and elsewhere on the ANL campus and to foster communications between the various research groups. Through this group, we believe we can build a strong user community for small-angle scattering at the APS, IPNS, and throughout ANL.

- Cooperation
- Collaboration
- Correlation
- Calibration
- Chow (cholesterol)

## Membership

- Open to all
- Email list server
- Monthly meetings

## Sponsorship

- WWW site hosted by UNICAT

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