Jumpstart Processing and Liquid Surfaces (JPLS) Beamline Conceptual Design Review

Ben Ocko, BNL

4 October, 2017

Charge to the JPLS CDR Review Committee

1. Does the conceptual design support the JPLS scientific objectives?
2. Is the conceptual design mature enough to proceed to the next stage of engineering design development?
3. Have reasonable design options and alternative concepts been explored (Including detectors)?
4. Does the design present technical risks? Does the design present scientific risks?
5. Are there safety concerns related to the installation and operations of the JPLS concepts presented?

Outline of the Presentation

1. Introduction and Background
2. Science
3. Functional Requirements
4. Safety
5. Controls and DAMA
6. State of the Design
7. Verification and Validation
8. Budget and Schedules
9. How the conceptual design support the JPLS objectives

Introduction and Background

Objective

Scientific

Polymer Processing: to understand the basic physics and materials science of flexible electronics and additive manufacturing in environments approaching those in industrial settings.

Liquid interfaces: to understand the fundamental science of soft interfaces, applicable to issues in nano/bio-materials, environmental remediation, separations science for critical materials, and water purification.

Common Thread: Liquid interfaces play an important role in basic and applied sciences. Emphasis on static self-assembly (liquid interfaces) and dynamics of evaporation (polymer processing).

Facilities

To make excellent use of existing facilities, infrastructure and small beams that are well matched to the NSLS II source and where NSLS II can be world-class.

User Demand

Build facilities and infrastructure where there will be a large demand.

SMI/PLS Jumpstart (JPLS) Background

Located at 12-ID using existing hutch and infrastructure

Jump start: time shared (SMI)

Long term: canted undulator (PLS)

Time shared with SMI, expect ~30% of available beamtime.

Why Jump start?

Adds new capabilities (open environment and liquids)

Accomplished with limited resources

Leverages existing $1M investment in SMI

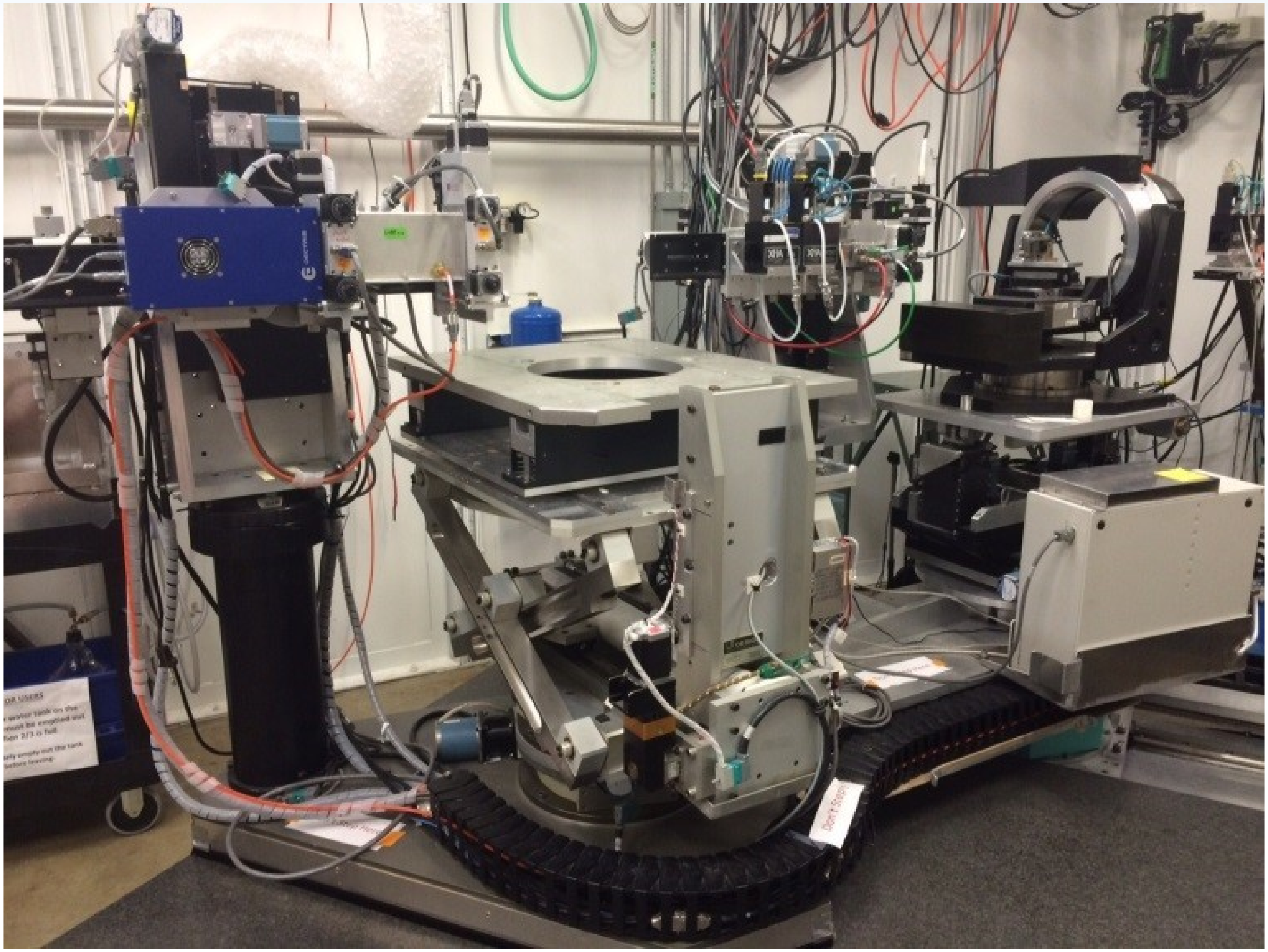
Expected cost is ~$1.5M, 18 month project duration

World-class experts at NSLS II in liquid surface science

Will help move the PLS project forward

Better positioned to attract external funding for PLS

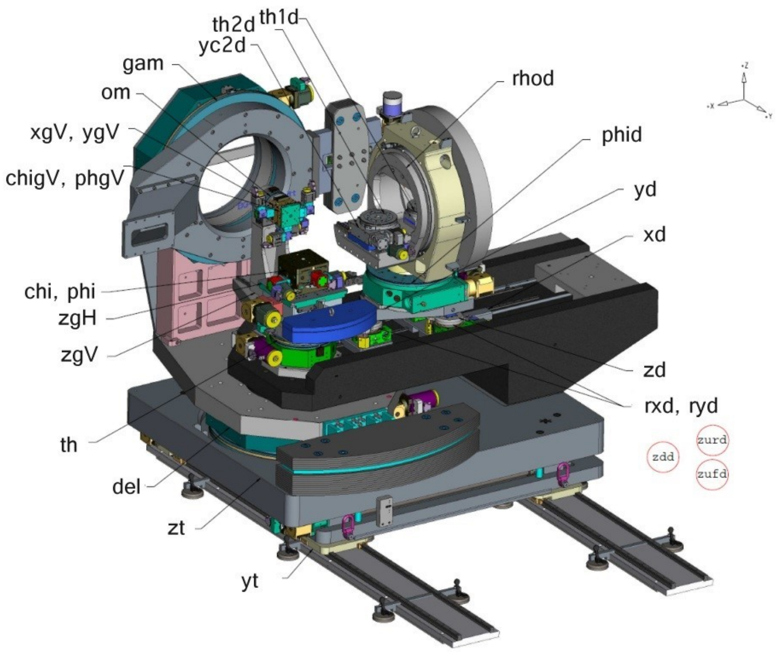
Perspective: Other liquid spectrometers in the US



The liquid surface spectrometer at the ChemMatCARS Sector 15 of the Advanced Photon Sources. Only US operational instrument.

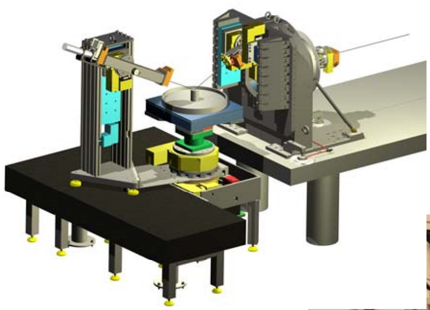
Perspective: liquid spectrometers of the world

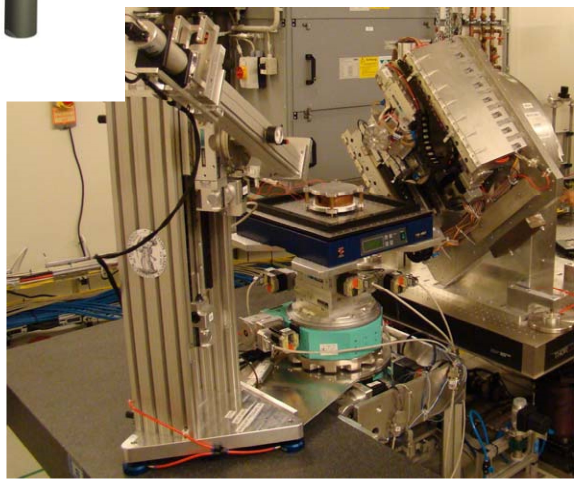
ESRF: Double Crystal Deflector (50%), 2014



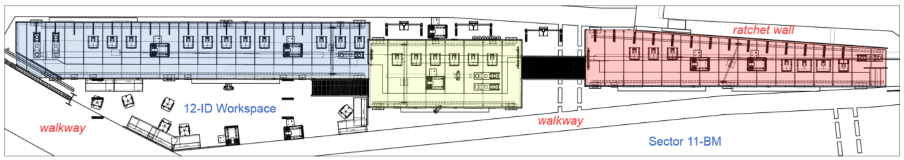
Petra III: Double Crystal Deflector (~33%) ~2009

The LISA spectrometer in place on P08, the high resolution X-ray diffraction beamline at PETRA III at DESY in Hamburg.

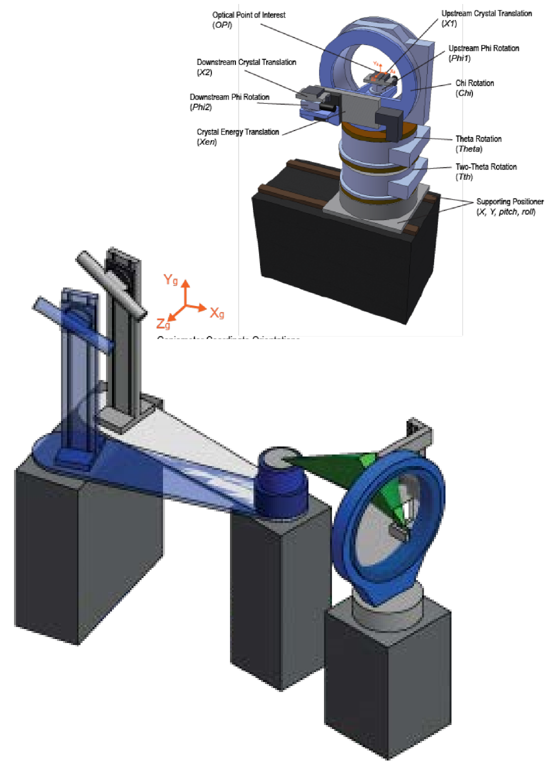




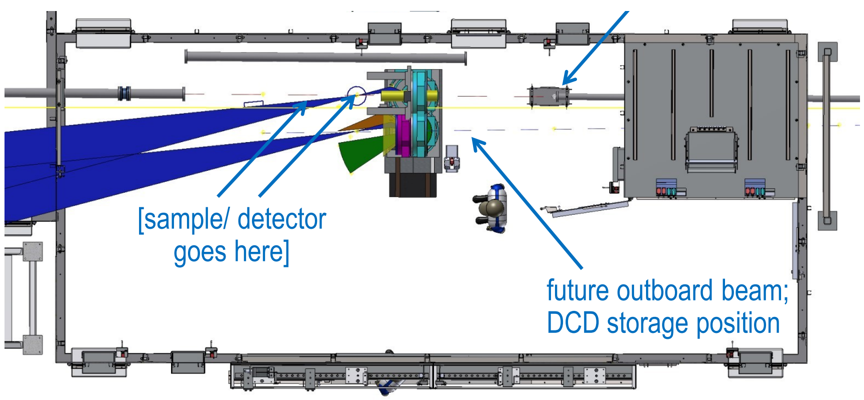
Historical 1: SMI Project (shared beamline)



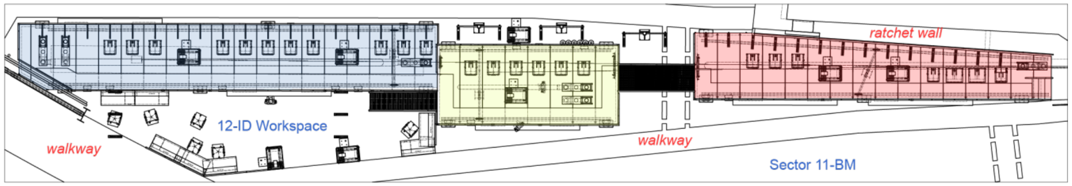
Shared beam with SMI



SMI originally budgeted for a new double crystal deflector, using much existing X22B NSLS.
Very high bid, specifications were difficult to achieve.
SMI project approved in 2011, liquid surface aspects of SMI curtailed in 2015.

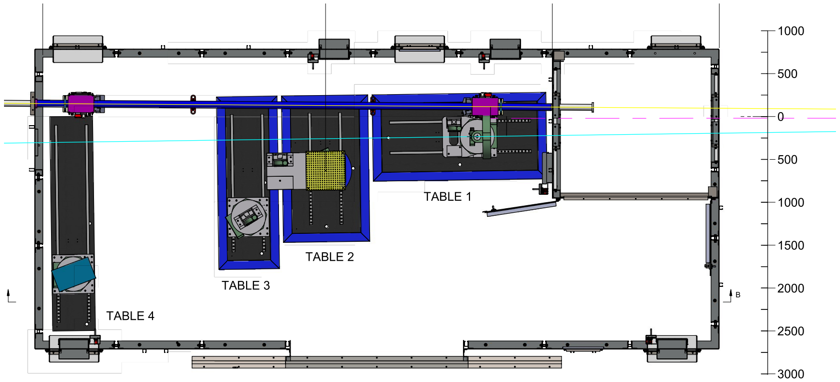


Historical 2 (PLS: independent/canted beamline)



All new components,
canted, independent beamline

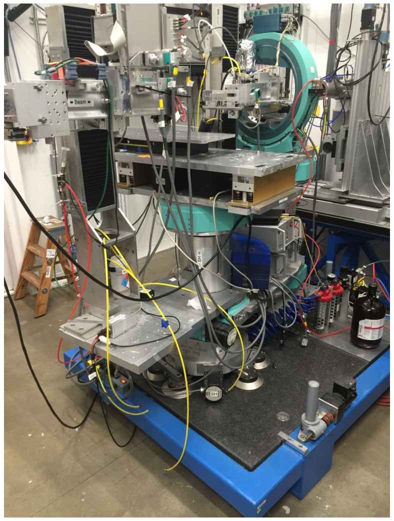
Detailed plans, calculations, and budgeting for beamline and endstation
Well received by the community in 2016
Cost ~$9M to complete, endstation alone is about >$1M without detectors

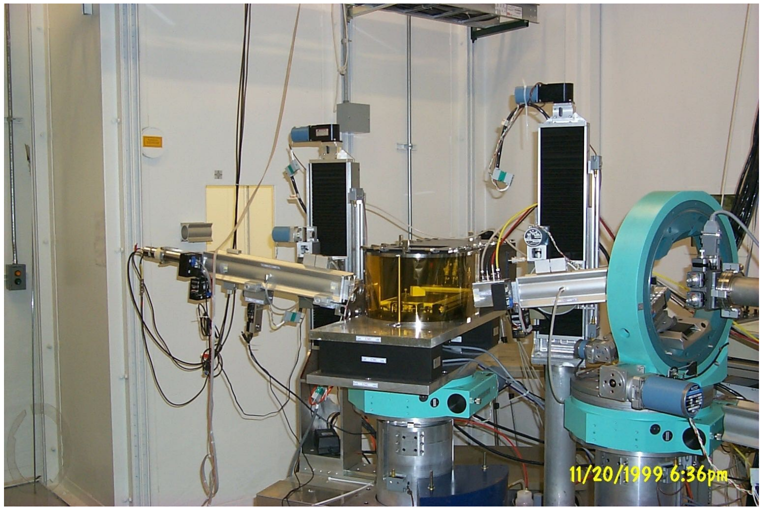


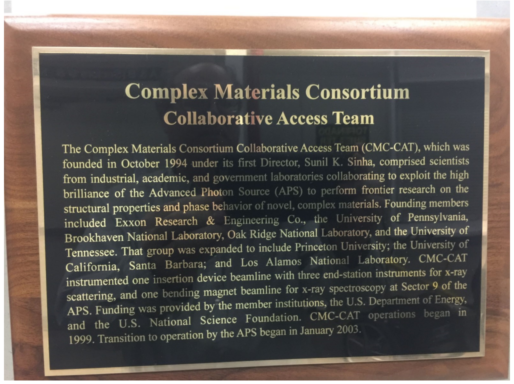
U.S. DEPARTMENT OF ENERGY | Office of Science

BROOKHAVEN NATIONAL LABORATORY | National Synchrotron Light Source II

Historical 3: APS/9ID shutters







ANL/APS/CMC (9ID)
Liquid Spectrometer
1999-2015

BNL/NSLS/X22B Liquid Spectrometer
1985-2015

Both instruments are now at NSLS II. JPLS will use these to construct a new and improved instrument.

Detailed Objectives

World class instrument for studies of liquid surfaces and processing

- Open design for high versatility
- Uses much/most of existing hardware from previous BNL and APS instruments
- Fit into existing hutch with minimal modifications
- Single Crystal Deflector Design, capable of upgrade to Double Crystal Deflector

Methods: x-ray reflectivity, grazing angle fluorescence, grazing incident small and large angle incident scattering

Liquids sample environments/conditions: Langmuir troughs, liquid metals UHV chambers, small liquid/liquid and liquid vapor chambers, low vibrations (both passive anD active), appropriate ventilation, sample weights, up to 200 kg

Processing sample environments/conditions: Roll-to-Roll Processing, UHV and Vapor Phase Deposition Chambers, these require complementary characterization instrumentation.

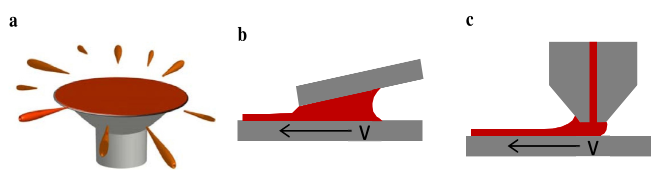
Beam characteristics (not part of this review): small beams ~ 5 microns (with slits), energy (8-24 keV), high intensity.

Science

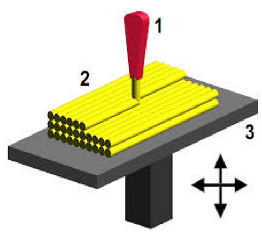
Polymer Processing

Polymer processing modifies mechanical, structural, and electronic properties.
Operando structural studies essential to understand the relevant processing mechanisms.
Often Involves solvent evaporation, melting and stretching.

Examples of Polymer Processing



Spin coating | Blade coating | Slot-die-coating



Additive Manufacturing, i.e. Fused Deposition Molding (FDM)

Other Examples

- Fatigue testing
- Nanoimprint
- Gravure (engraving)
- Laser ablation
- Blow molding
- Fiber spinning
- Interfacial polymerization processes

Roadblocks to progress in polymer processing, and the opportunities for transformational advances in this field are well aligned with the DOE Grand Challenges:

1. *How do remarkable properties of matter emerge from complex correlations of the atomic or electronic constituents and how can we control these properties?*
2. *How do we characterize and control matter away - especially very far away - from equilibrium?*

Processing challenges for Printed Electronics

Low temperature processing almost invariably means structures are path dependent, meta-stable/ non-equilibrium, multiple polymorphs.

Optimal film morphology may not be the equilibrium structure

particularly true for Organic Photovoltaic Devices and Organic Electronics

Provide scientific understanding so as to 'dial-in' optimal structure

blend composition, loading, solvent, additive, temperature, …

Inform & validate multi-scale physics based simulations of the structural evolution.

Process optimization is purely empirical: process 'rules' are non-existent

JPLS will investigate underlying science.

Processing chamber requirements:

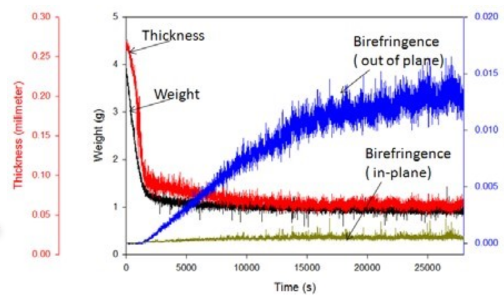
1.Need to use multiple fields (UV, magnetic, electric)

2.Correlate x-ray results with optical characterization methods →

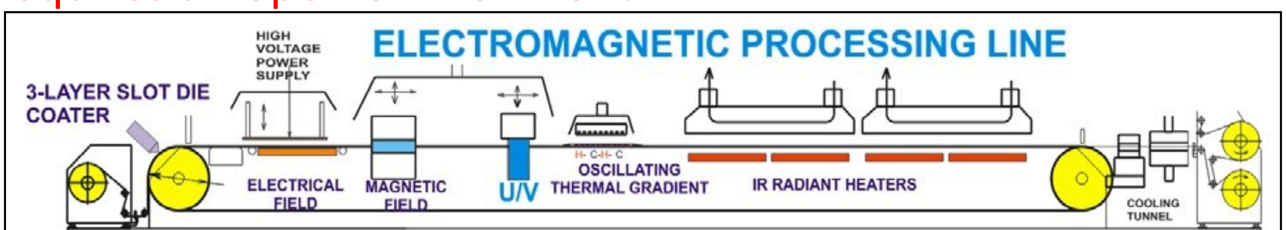
→ Both require reasonably large chambers

Objective: Include many features/diagnostics of R2R on a beamline?

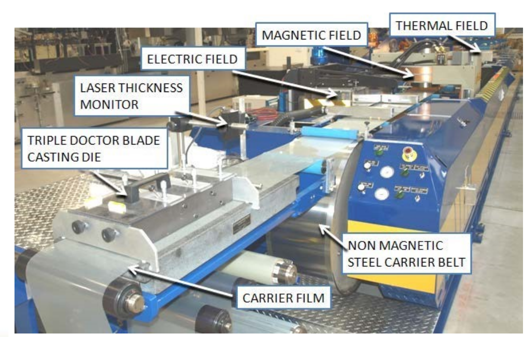
Requires an open environment.



Example of real time monitoring data on Polyamic acid solution (*Cakmak).*



R2R at the University of Akron, 70 feet in length



Overview: Liquid Surface/Interface Beamline

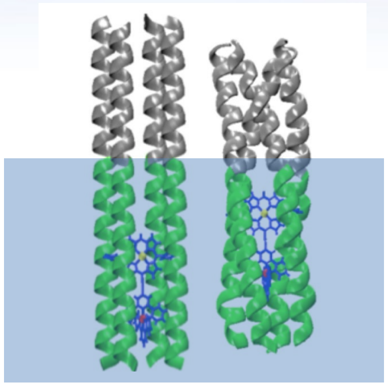
X-ray Scattering is the leading technique for the study of structure at liquid surfaces and interfaces

Techniques for World-Leading Research:

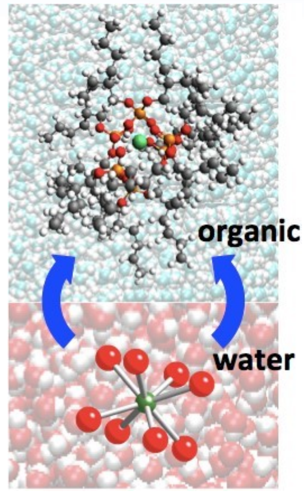
- High resolution scattering
  X-ray reflectivity not available elsewhere at NSLS II for liquid surfaces
- Element-specific studies
- Temporal resolution (sub-second to minutes)
- Optimized use of 2D detector

Overview: Liquid Surface/Interface Science

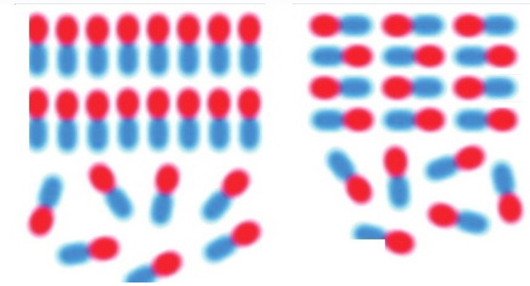
Overview of scientific scope



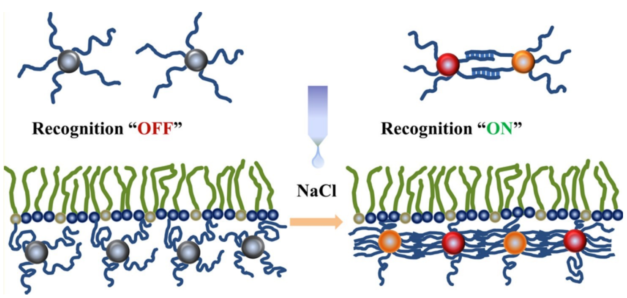
Directed Assembly
for
Functional Interfaces



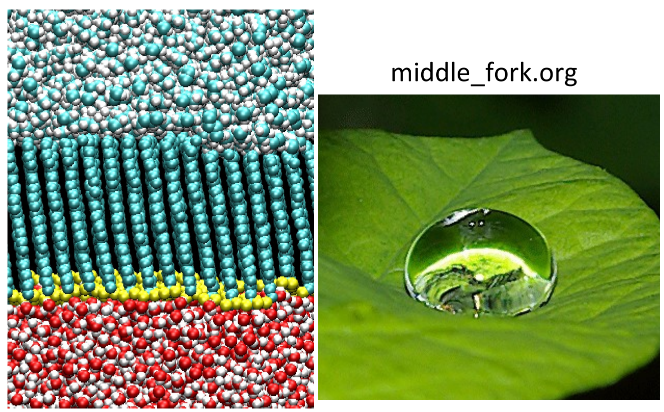
Ion Extraction
for
Environmental
Remediation
and
Metal
Production



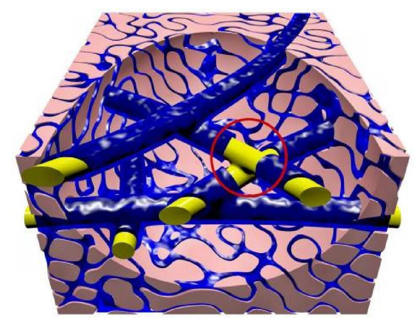
Ionic Liquid Interfaces
for
Energy Applications



Nanoparticle
Growth and Ordering
For
Nanotechnology



Interfacial Interactions
for
Coatings, Solders, Nano-layers



Desalination
Technologies

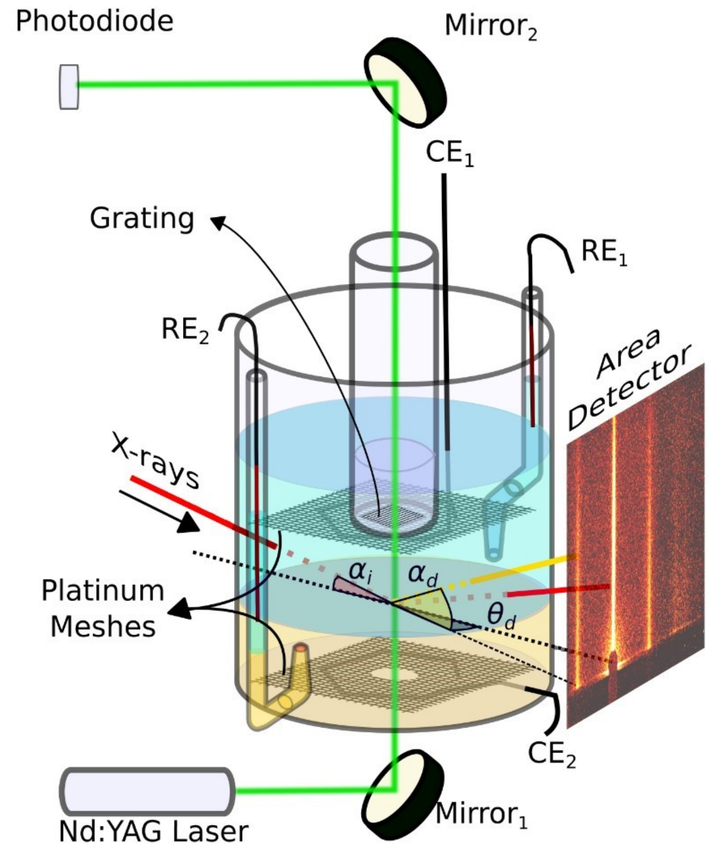
Science Focus: Electrostatic Control of Interfacial Processes

Electrified Interfaces

Significance:

• Voltage-tunable control of interfacial processes – nanoparticle ordering, membrane/protein interactions

• Many energy, complex fluid, and biomaterials processes controlled by electrostatics at interfaces



Nanoletters 14, 6816 (2014)

Challenges:

- Atomic level characterization of ion distributions at buried interfaces
- Heterogeneous, multi-component interfaces

Role of Liquid Interfaces:

• Fluid platform for assembling and reacting soft matter under intense electric fields

• Interactions of voltage-controlled interfacial assembly with either aqueous or organic components in the subphases – opportunities for sensing, filtering, enhanced reactivity

Science Focus: Directed Assembly for Tailored Functionality

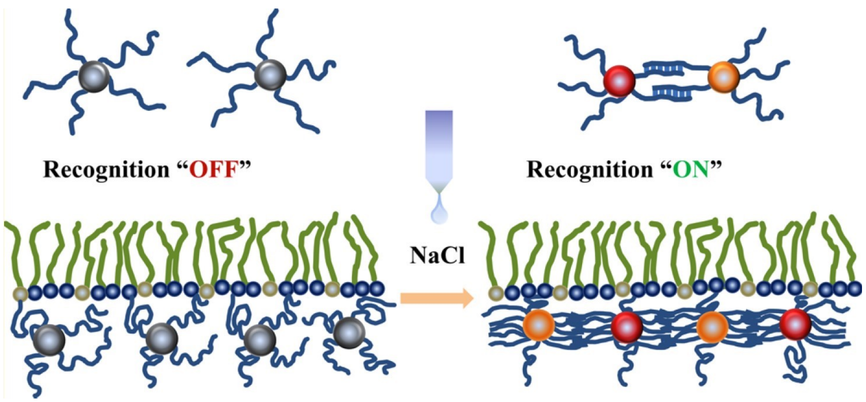
Nanoparticle Materials

DOE Grand Challenges:

- "master energy and information on the nanoscale to create new technologies with capabilities rivaling those of living things"
- "perfect … synthesis of new forms of matter with tailored properties"

Significance:

- Chem- and bio-inspired pathways to create nanoscale ordering for specific functionality (switches, amplifiers, charge transport, molecular recognition, materials synthesis)



JACS 136, 8323 (2014)

DNA Programmable Assembly of Nanoparticles (Oleg Gang)

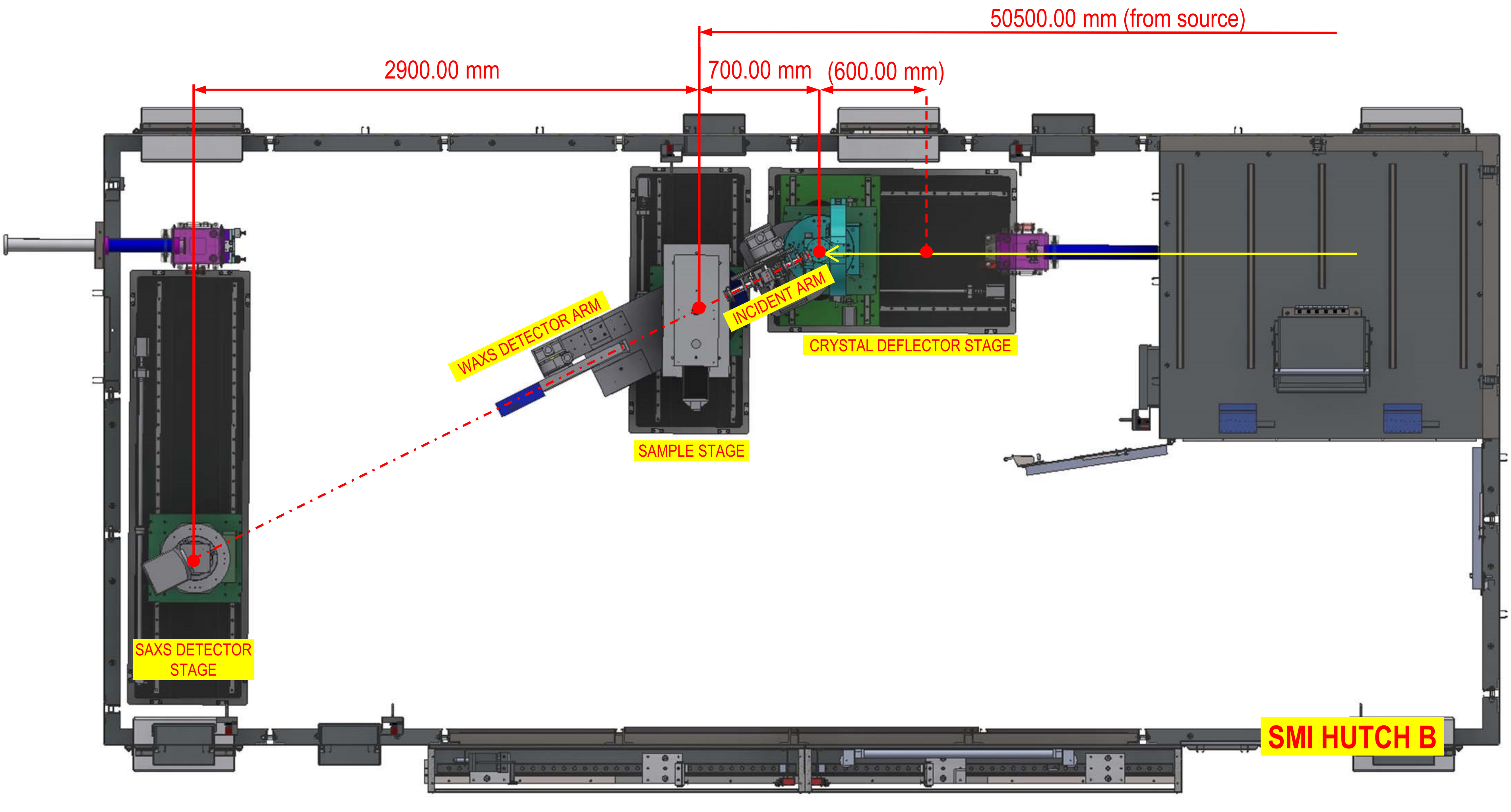
Role of Liquid Interfaces:

- Dynamic platform for the assembly of nanoparticle materials
- Control of interfacial environment (pH, ionic content, temperature, viscosity, electric potential) and nearby molecular species that bind to or chemically interact with assembly

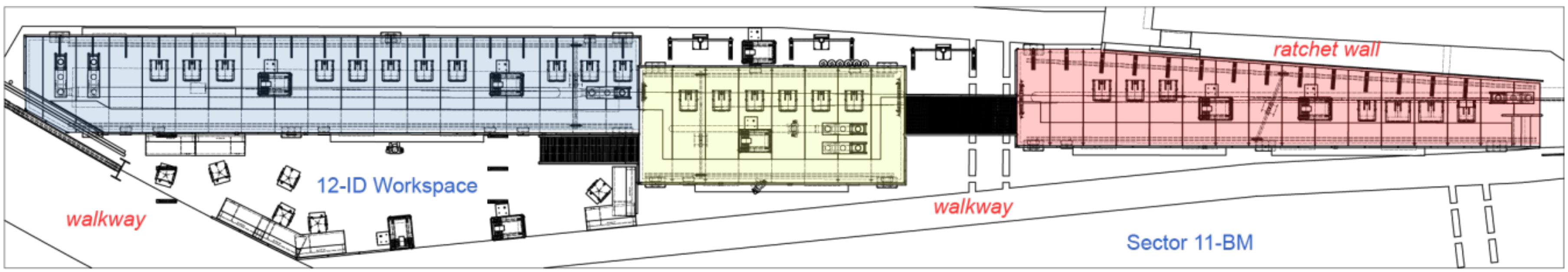
Functional Requirements

Functional Requirements

The present design is motivated by many functional requirements and practical considerations
hutch size, proximity to walls, beam characteristics, cost, ….

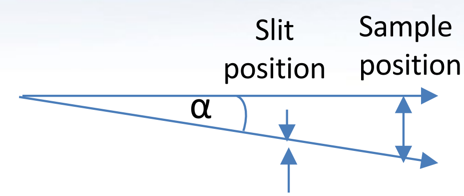


Beamline Layout



Requirements 1: Spatial Resolution

Beam must impinge in the center of a small sample at grazing angles.
With vertical beams as small as 5 µm, incident beam and sample should have ~1 µm resolution. Accuracy over the entire range should be ~2 µm. Also depends on beam stability (not a subject here).



y – sample displacement
Δy– sample displacement error

(1) Crystal deflector stage accuracy:
Δy =2 µm corresponds to Δα=2e-6 rads at 1 m.
Corresponds to $\Delta\chi$ = 0.0005° (8 keV) and $\Delta\chi$ = 0.001° (16 keV)
Huber 512.1 χ expected accuracy is 0.008° over the entire range, off by an order of magnitude.
Requires validation over more limited range.
Key finding: Additional investigations required to find best solution. Possibilities include

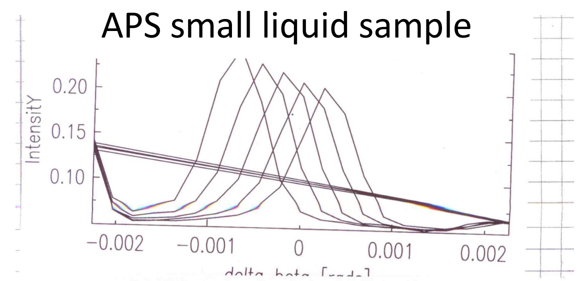
Intensity or positional feedback
Incremental encoder from Renishaw or lookup table

(2) Incident height/slit stage accuracy. Relies on both incident height and incident rotation stages. Limitation is with the rotation stage if there is a long lever arm. Place slit close to center of rotation so that there is only sensitivity to the translation stage. Discussed further below

(3) Sample stage: Walking scans for a small liquid sample 3 µm steps show that the scattered signal moves in a well-define way as expected for a curved sample. Resolution is expected to be < 1 µm.

Will need to validate.

APS small liquid sample



Requirement 2: Reciprocal space range

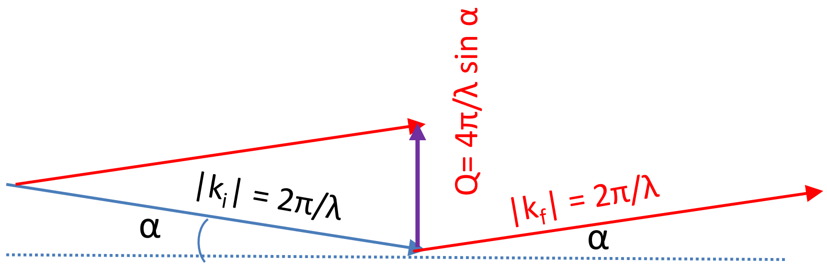
Ge(111) deflector

Maximum range for a liquid surface is for liquid metals, such as Gallium, with high surface tension.

For a fixed Q_z, the angular range depends on the energy.

Typical maximum range in Q_z is 2.5 Å $^{-1.}$ ***(this is in user coordinates). Angle of incidence depends on the energy.

energy (keV)	wavelength (Å)	α (degrees) 1.0 (inv. Å)	β (degrees) 2.5 (inv. Å)
8.000	1.550	7.09	17.96
16.000	0.775	3.54	8.87
24.000	0.517	2.36	5.90



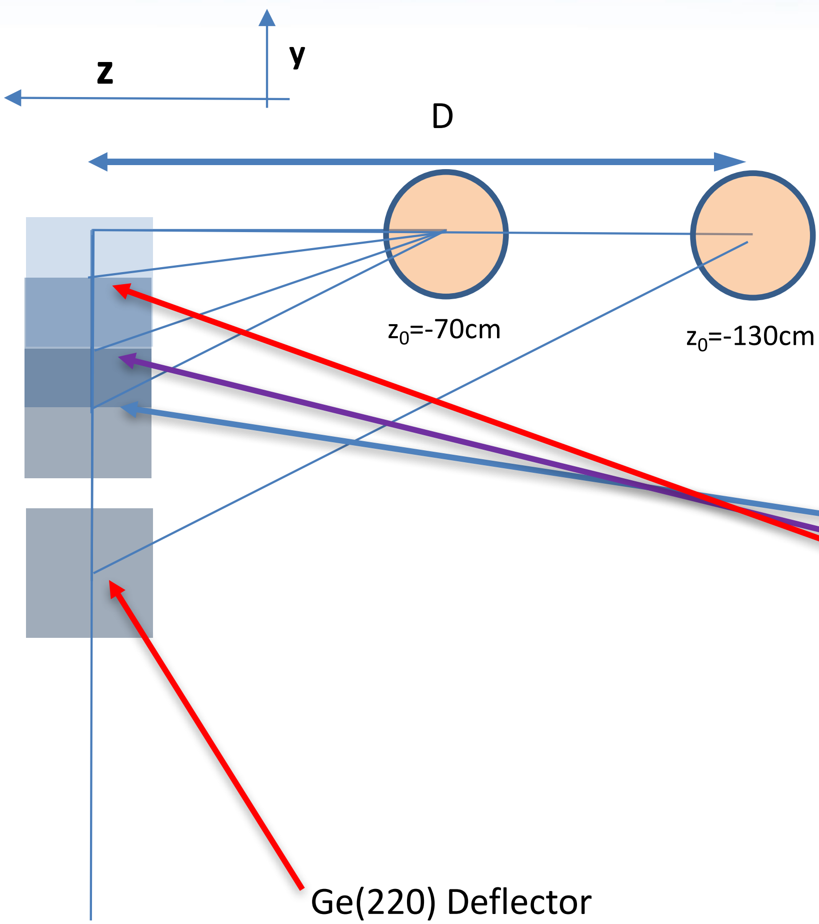
Not likely to require such large Q_z at 8 kev.
For practical purposes, getting to Q_z -2.5 Å $^{-1}$ at 16 keV is adequate.

Would be good to be able to achieve α =15◦, although 10◦ is probably adequate.

*** would be best to get to 3 Å $^{-1}$, achievable at all but the lowest E.

Key finding: desired reciprocal space range is accessible

Requirement 3: Range of Vertical Sample Motion



Ge(111) Deflector

energy keV	wavelength angstroms	D-70cm alpha (2.5A-1) Delta Y (cm)	D=130 alpha (2.5A-1) Delta Y (cm)
8.000	1.550	-24.84	~~-47.48~~
16.000	0.775	-11.16	-20.87
24.000	0.517	-7.30	-13.60

Vertical sample stage has ~30 cm in range.
Sufficient except for largest D and smallest E.

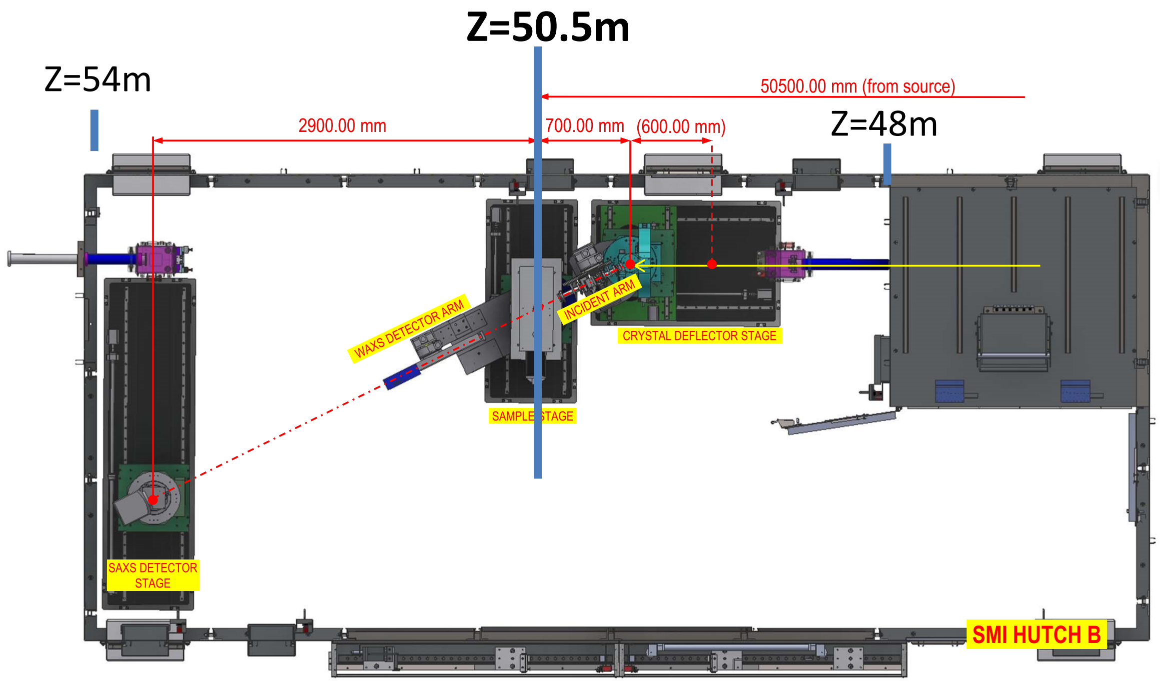
Ge(220) Deflector
Useful for processing measurements than for liquid surfaces since it moves the sample more towards the middle of the hutch.

Key finding: desired reciprocal space range is accessible with D=70cm.

Requirement 4: Position of sample translator *competing factors*

Max angle for SAXS
atan(2.5/4)app=32 deg

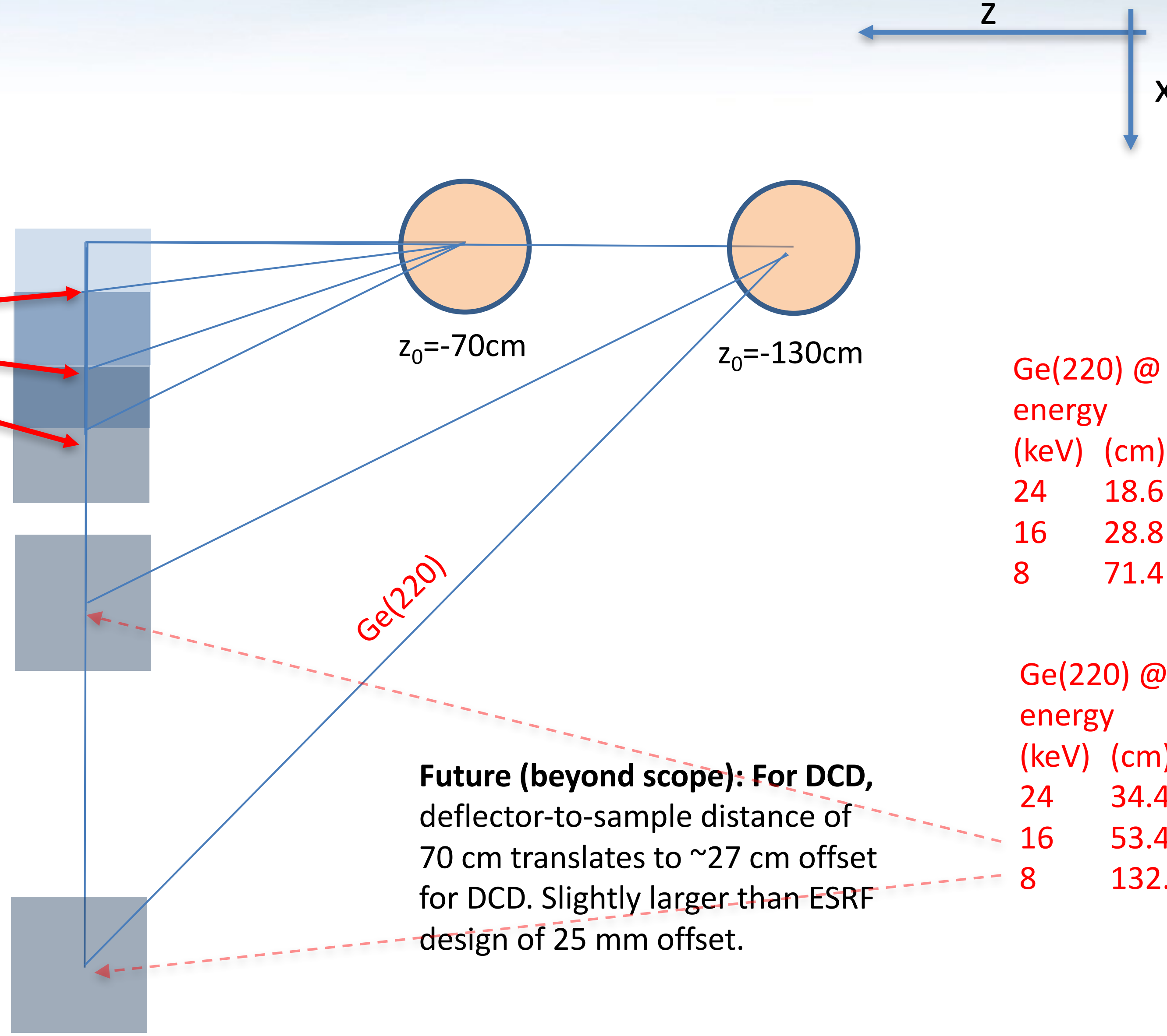
Conclusion: can carry out SAXS studies at 8-24keV



Proposal to put the sample rail at 50.5 m from the source (SMI proposal 50.7m)
JPLS >3m sample-to-detector, after accounting for 0.5 m long detector.
CMS 5m sample-to-detector
SMI 8m sample-to-detector

Key Finding: sufficient room in the hutch for up to a 3m sample-to-detector spacing

Requirement 5: Sample lateral motion



Ge(111) @ 70 cm

energy (keV)	x (cm)
24	11.2
16	16.8
8	36.4

Ge(111) @ 130 cm

energy (keV)	x (cm)
24	20.8
16	31.5
8	67.5

Ge(220) @ 70 cm

energy (keV)	x (cm)
24	18.6
16	28.8
8	71.4

Ge(220) @ 130 cm

energy (keV)	x (cm)
24	34.4
16	53.4
8	132.7

Future (beyond scope): For DCD, deflector-to-sample distance of 70 cm translates to ~27 cm offset for DCD. Slightly larger than ESRF design of 25 mm offset.

Key Finding: sufficient room in the hutch for the required sample's horizontal motion.

Requirement 6: Detectors configurations/resolution

soller slits, are they require to get the desired resolution?

How to get high in-plane resolution for GID?

2nd generation sources: GID, large beam on the sample

*Large parallex error: **solution** use Soller slits, ~0.1 degree resolution*

3rd generation sources: GID; small beam on the sample

In order to get the same angular resolution as Soller slits a small beam height is required so that the beam does not spread out too much.

$\boldsymbol{\alpha=2/3\ \alpha_c \simeq 0.1}$ ∘(water surface) @ q_r= 1.5 Å^{-1} (chain packing peaks)

Result: 0.05 mm vertical beam required @ 0.5 m sample-detector distance

Key finding: Simplest and best to use 2D detector without Soller slits.

Requirement 6: Detectors configurations/resolution

D/d legend, highlight what is the base configuration
d(pixel size), D(sample-to-detector), pixels (no. of pixels)

What combination of detectors seems most suitable?

		S-D dist.	pixel size				16 kev	16 kev	8 kev	8kev	
.	type	D(mm)	d(mm)2	δ2θ(°)	<pixels>	Δ2θ	δq (inv Å)	Δq (inv Å)	δq (inv Å)2	Δq (inv Å)3	pixels
REFLECTIVITY*	Pilatus 100k	1000	0.172	0.010	308	3.0	0.00144	0.44	0.0007	0.22	487x195
GID	Pilatus 1M	300	0.172	0.033	1010	33.0	0.00480	4.78	0.0024	2.39	981x1083
GID	Pilatus 1M	500	0.172	0.020	1010	19.8	0.00288	2.90	0.0014	1.45	981x1083
GID	Eiger 1M	500	0.075	0.009	1000	8.6	0.00126	1.26	0.0006	0.63	1030x1065
SAXS	Princeton/Roper	3000	0.06	0.001	2082	2.4	0.00017	0.35	0.0001	0.17	2082x2082
SAXS	Eiger 1M	3000	0.075	0.001	1000	1.4	0.00021	0.21	0.0001	0.10	1030x1065
*REFLECTIVITY	Pilatus 100k	1000	0.172	0.010	487	4.8	0.00144	0.70	0.0007	0.35	487x195
*REFLECTIVITY	Pilatus 100k	1000	0.172	0.010	195	1.9	0.00144	0.28	0.0007	0.14	487x195
SAXS	Princeton/Roper	1500	0.06	0.002	2082	4.7	0.00034	0.70	0.0002	0.35	2082x2082
SAXS	Eiger 1M	1500	0.075	0.003	1000	2.9	0.00042	0.42	0.0002	0.21	2082x2082

Baseline detectors

100K and Princeton/Roper are existing

Distances D still have to be refined
For reflectivity we may want D to be closer to 750 mm.

Other factors

detector speed?

energy range: CdTe or thicker silicon?

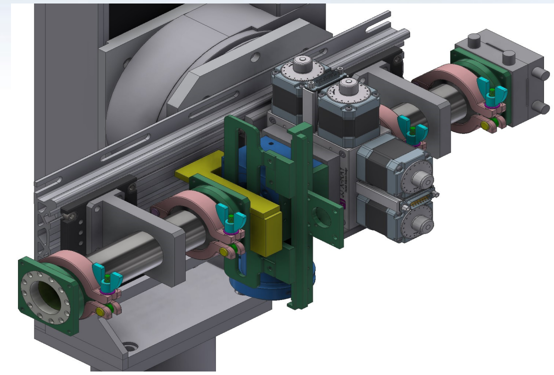
Fluorescence detector to be mounted above the sample

Beam stop (since the detector moves is a moveable beam stop required)?

Key finding: 2D detectors provide reasonable resolution and range, SAXS Princeton is slow.

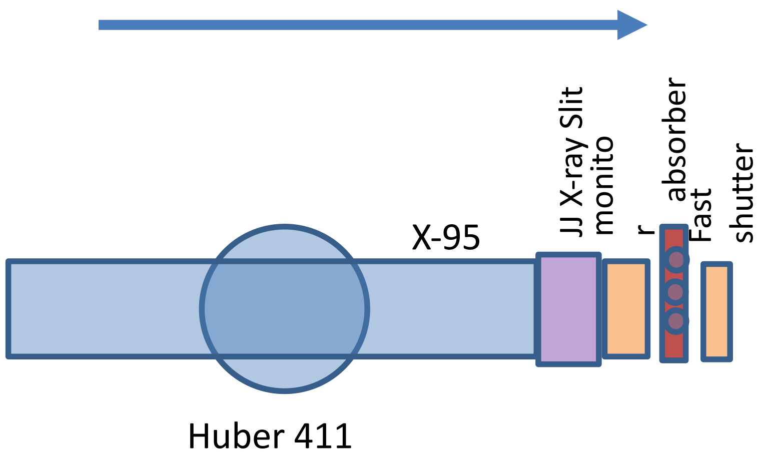
Requirement 7: Flight Paths Concept

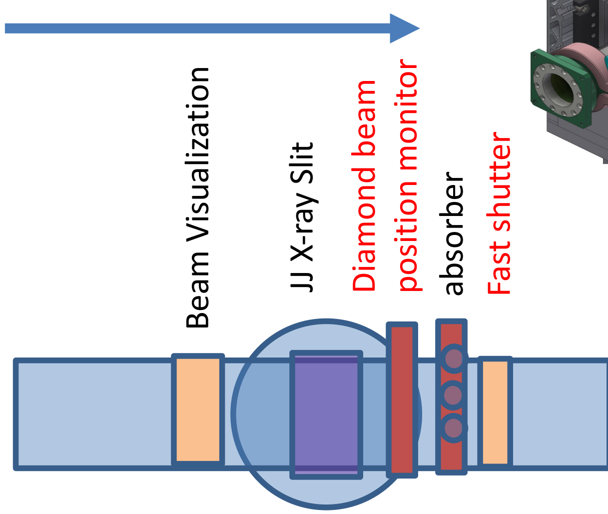
Incident flight paths located between deflector & sample



New design:
components mount on a rail

Old Design:
Flight path is structural





Advantage: Places slit closer to the sample
Disadvantages: Slit height sensitive to position and rotation angle. May affect tracking.

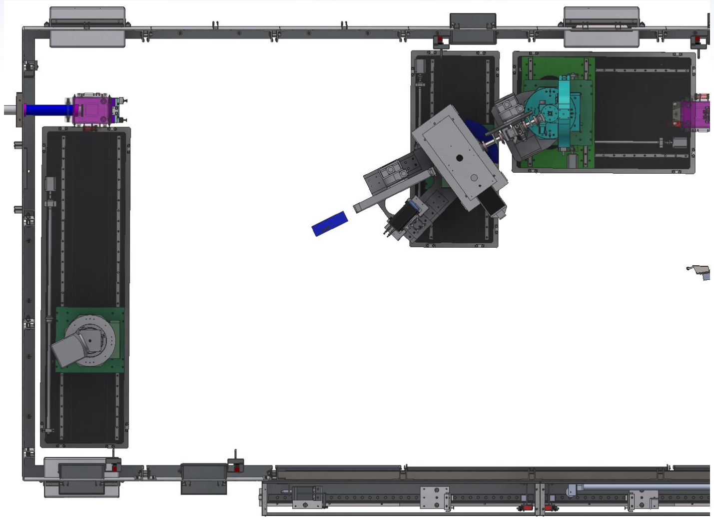
Advantage: (1) height less sensitive to the rotation angle, (2) easier to change the configuration
Disadvantages: (1) Slit is a bit farther from the sample, *ok due to the low divergence. (2)* May require two extra windows, *at higher energies flight paths are less important.*

Should an additional intensity monitor be included or will the diamond beam position monitor suffice?
Should the rail be horizontal or vertical?
Key finding: revised layout reduces the sensitivity to angular error in the rotation stage.

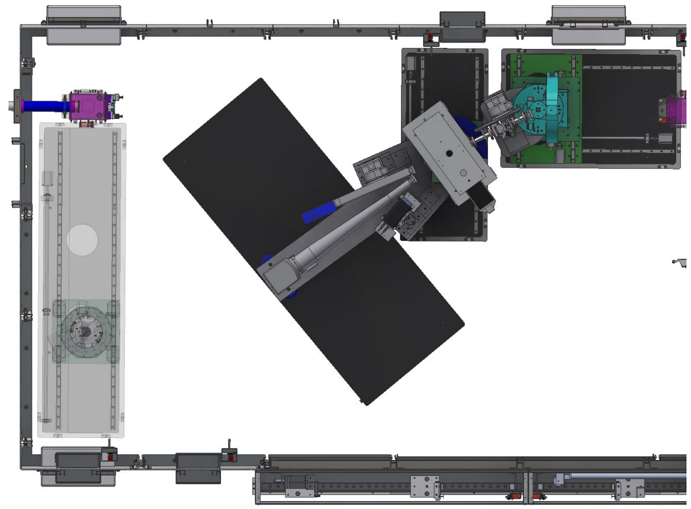
Requirement 7: Flight Paths Concept

Outgoing "detector" arm

Plan A: (JPLS proposal)
Concept: Reflectivity and GID share the same 2-theta arm, fixed offset angle. SAXS is decoupled from the detector arm.
Advantage: Maximum SAXS sample-detector distance (3 m from sample)
Disadvantage: No easy way to attach SAXS flight path, difficult to change configuration.



Plan B: (new approach)
Concept: Reflectivity, GID, and SAXS share the same 2-theta arm, fixed offset angles
A granite slab has to be added with air pad support for long 2-theta arm
Advantage: SAXS flight path moves with the 2-theta arm
Disadvantage: SAXS sample-detector distance (~1.5m), loss of a factor of 2 in resolution



Consideration: A factor of two in resolution versus an improved better design

Requirement 8: Vibrations

Vibrations excite long wavelength "sloshing modes"

important when there is viscous damping and for longer samples

Best if no active vibration isolation required

if active vibration is used mechanical stiffness is critical

Long Island is a big liquid surface, vibrations travel far (following slides)

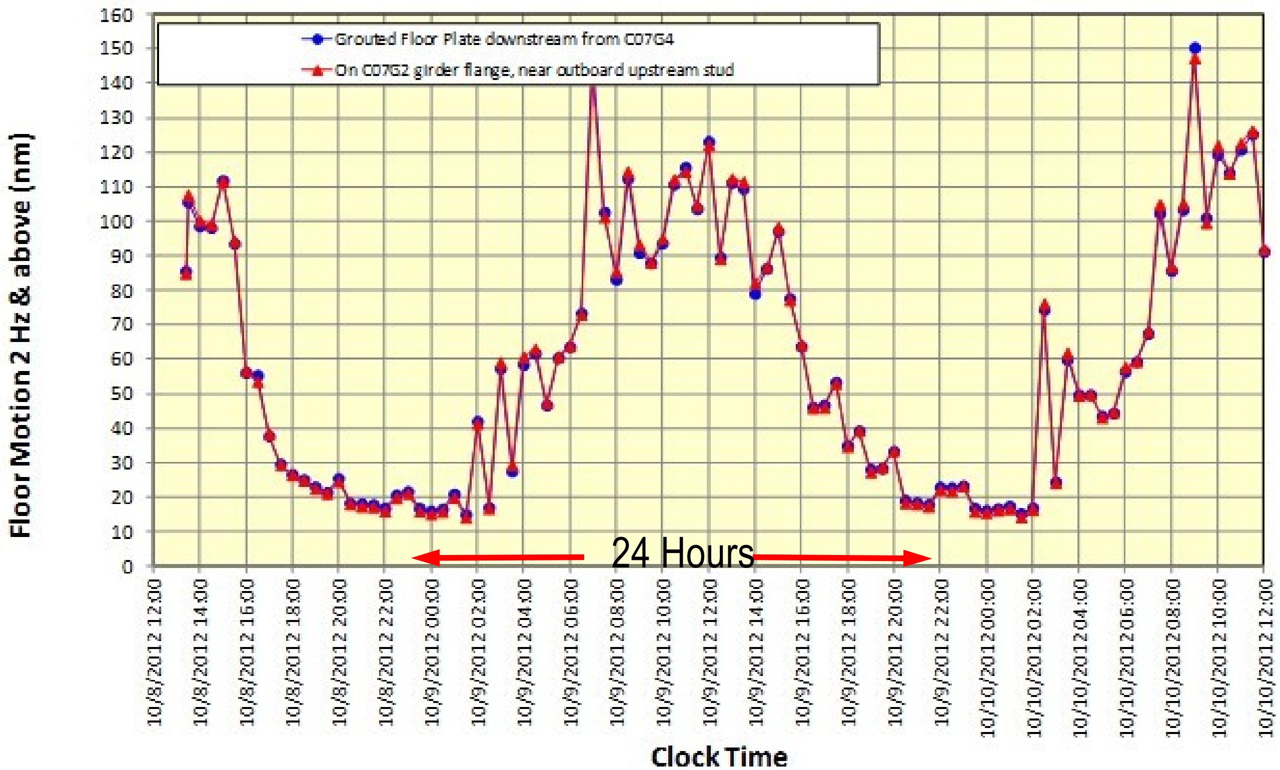
Studies at the beamline planned using existing stages

with & without active vibration isolation.

Remediation

Discussions with Charlie Spataro, Mechanical Engineering Group, NSLS-II

Ground Motion Measurements–NSLSII SR Floor and Girder



The maximum day-time amplitude is ~ 5 times the minimum night-time amplitude.

U.S. DEPARTMENT OF ENERGY | Office of Science

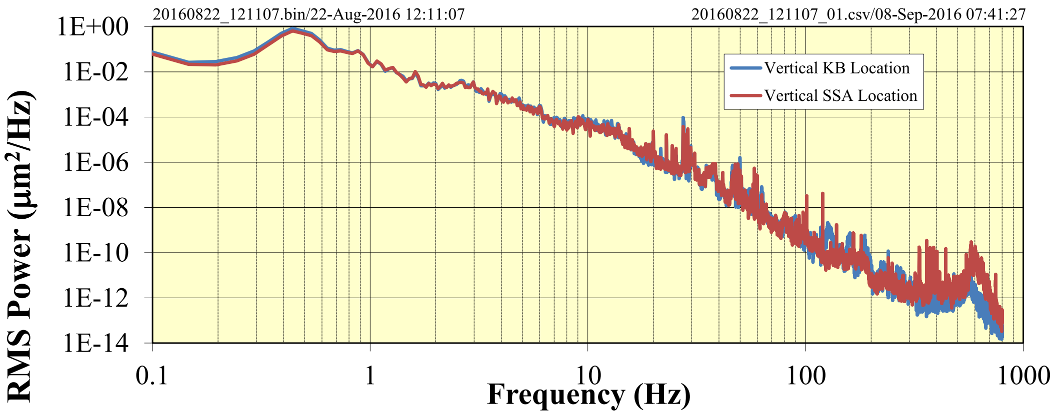
BROOKHAVEN NATIONAL LABORATORY | National Synchrotron Light Source II

9ID: Axis PSD Plot
noisy periods

0.13 Hz resonance

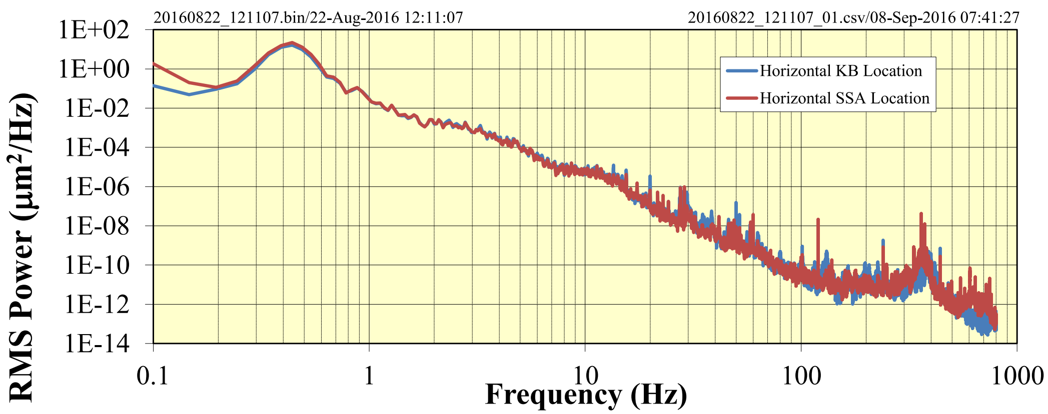
Noisy period

Vertical



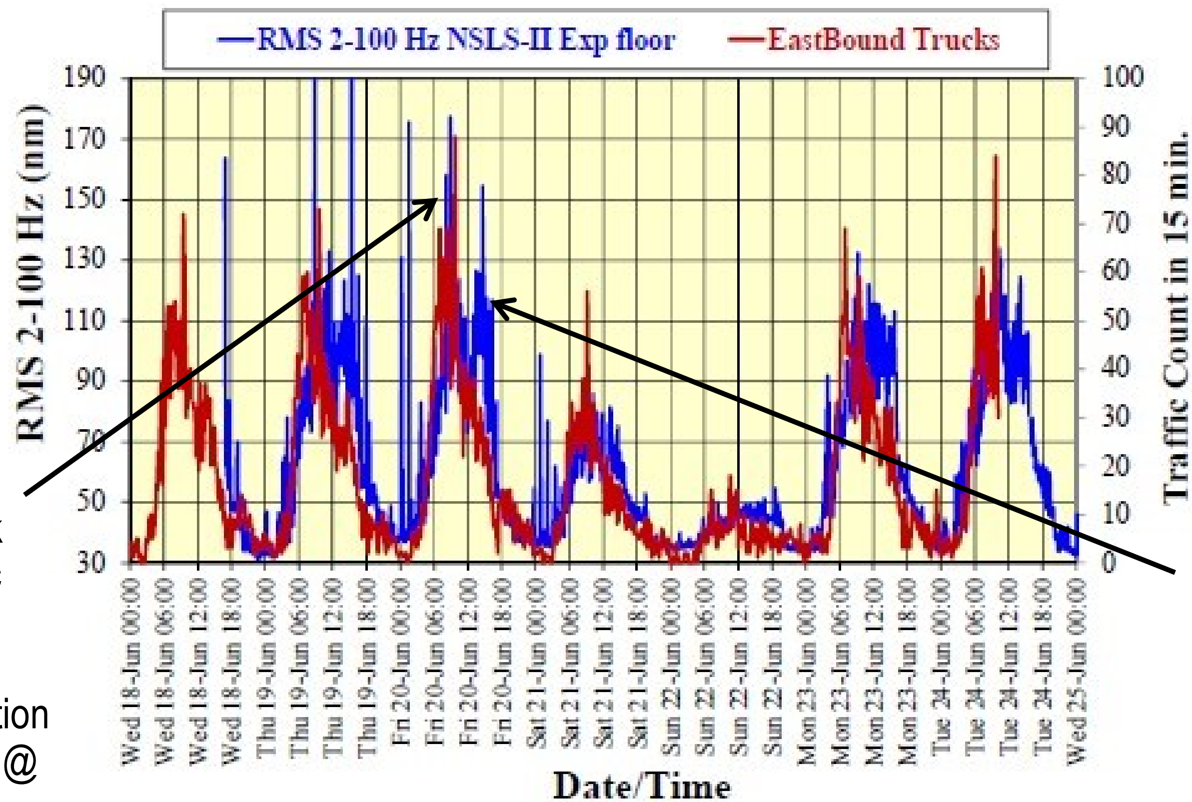
According to C. Montag (BNL) & J. Rossbach (DESY) , this '7 second hum' appears in all spectra at various labs around the world and is a 'Microseismic peak around 1/7 Hz; caused by ocean waves hitting coasts.'- *Handbook of Accelerator Physics and Engineering, Chao,et al.*

horizontal



Horizontal noise is about 5 times worse than vertical. Vibrations are a factor of 5 worse than at Argonne.

Exper. Floor Vibration Levels vs LIE Eastbound Truck Count



Truck traffic peak and vibration peak @ 9AM

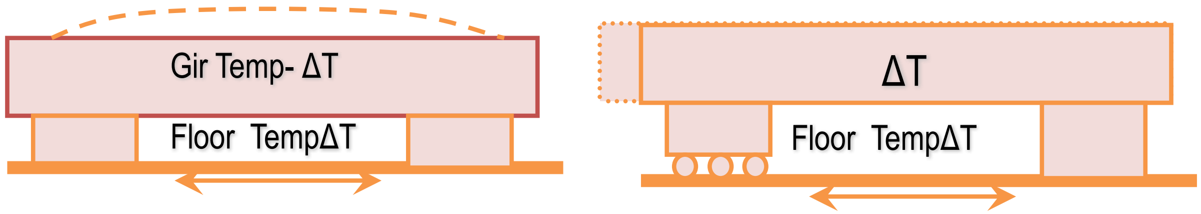
Second vibration peak @ 3 pm

The eastbound truck traffic shows a peak in the morning consistent with the vibration pattern but does not explain the second vibration peak in the afternoon.

The maximum day-time amplitude is ~ 5 times the minimum night-time amplitude.

Key finding: vibrations may be an issue and remediation efforts might be required.

Remediation: Viscoelastic Pads



The viscoelastic film allows top plate to move relative to the bottom plate freely at slow cycles (< 0.1 Hz). Used for ring beam supports and also under some mirrors

Will further explore the implications of using such pads at JPLS

Herzan AVI Specifications, with/without LFS

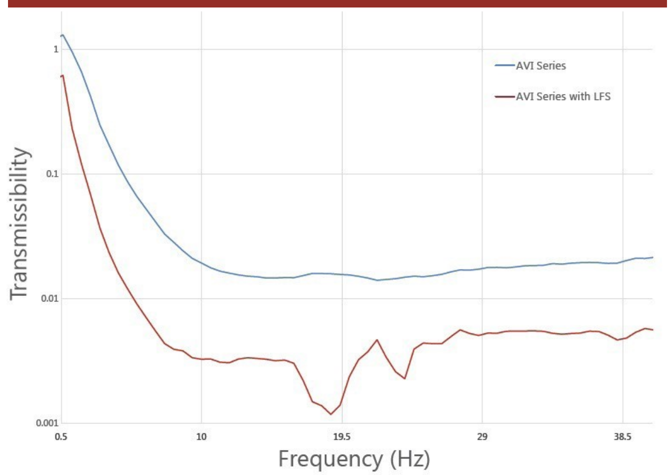
Modular & Scalable Active Vibration Isolation Platform

The AVI Series is a modular, low profile, active vibration isolation platform capable of sub-hertz isolation performance across all six degrees of freedom. The AVI Series is available in three standard sizes (AVI-200, AVI-400, and AVI-600) to accommodate instruments of all shapes and sizes (from 0 – 9,000 Kg), from compact AFMs to room scale TEMs.

Vibration units previously used at APS & NSLS

Improvements at low frequency

TRANSMISSIBILITY



PERFORMANCE

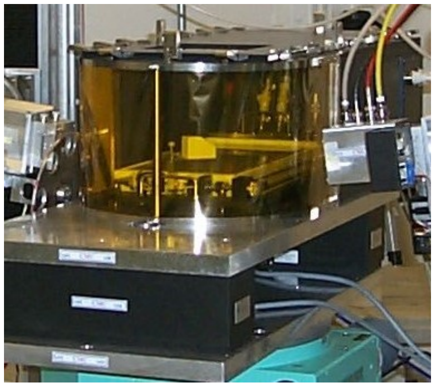
The LFS System enhances the low-frequency vibration isolation performance of the AVI Series, enabling the line of platforms to begin isolation at 0.5 Hz. To the left is a performance comparison graph depicting the standard performance of the AVI Series platforms and the AVI Series platforms with the LFS System.

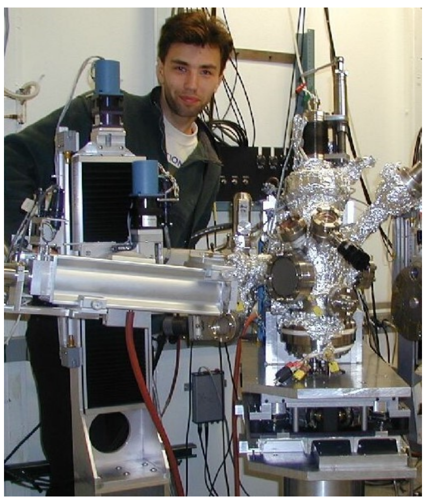
Performance Details

- Enhances vibration isolation from 0.5 - 100 Hz
- 90% vibration attenuation at 2 Hz
- 99% vibration attenuation at 5 Hz and beyond
- Up to 50 dB of vibration reduction

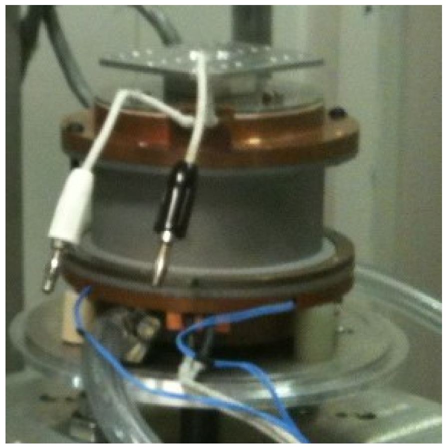
Requirement 9: Sample cells, and weight loading

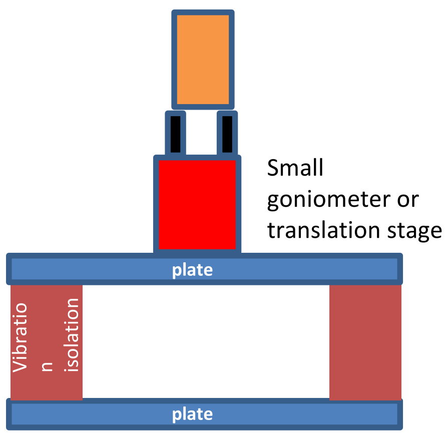
Large liquids chambers with vibration isolation



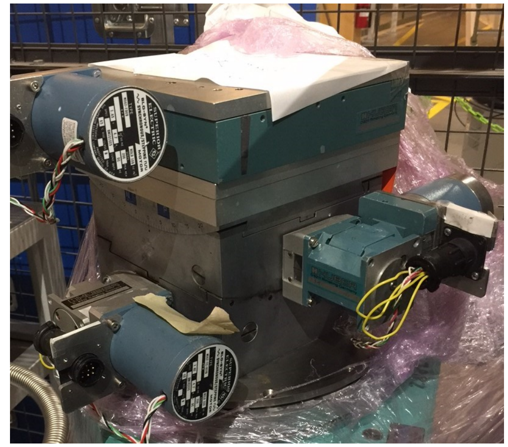


Small liquids chambers with vibration isolation





Large Huber goniometer, no vibration isolation

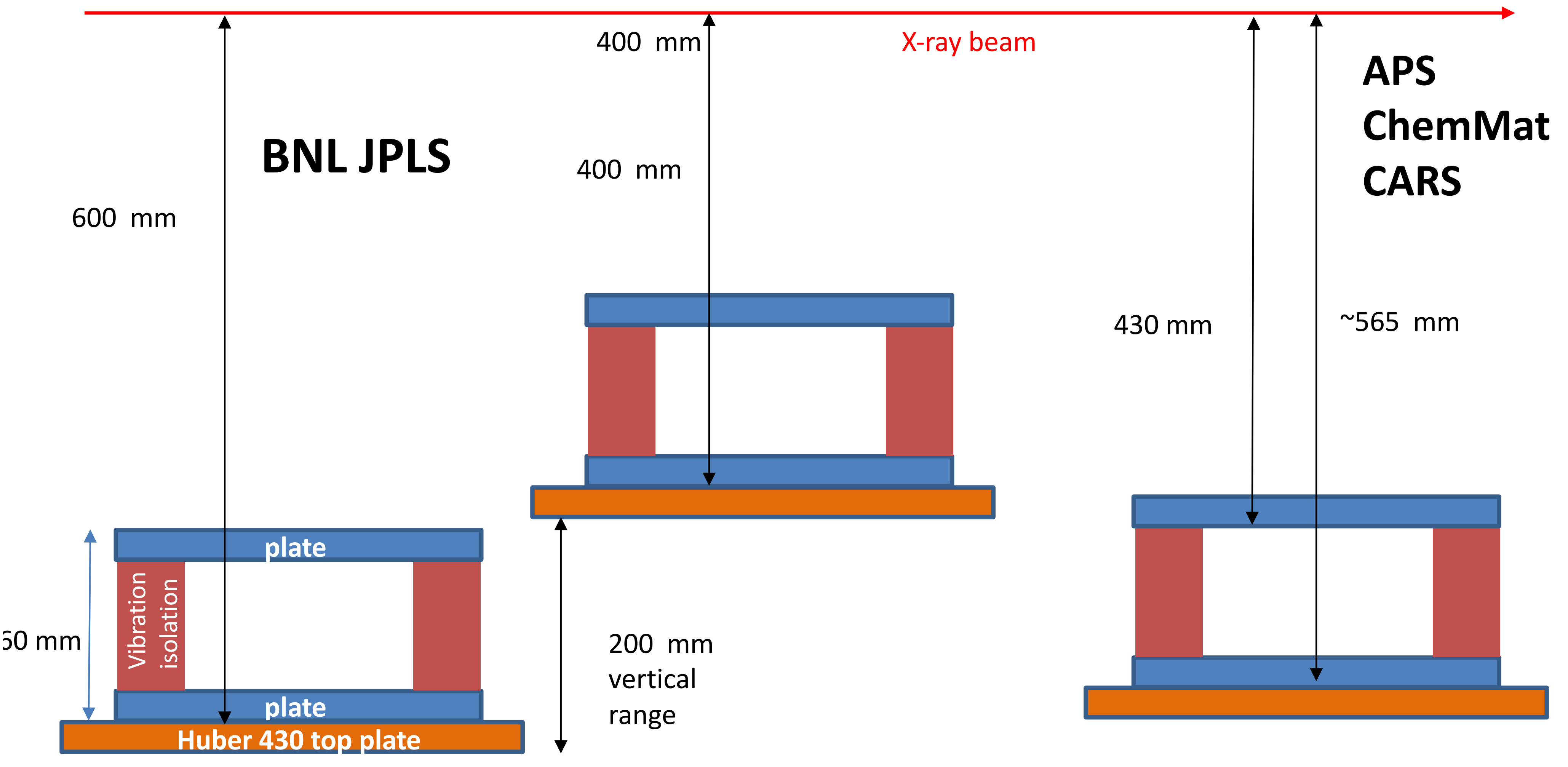


Large Processing chamber, no vibration isolation, no goniometer (this would be too large)

Would like to achieve 200 kg weight loading and chambers 1 meter^3.

Key finding: sufficient space above vibration stage to accommodate a wide variety of sample chambers

Requirement 10: Height above vibration unit to beam (proposed)



Safety Considerations

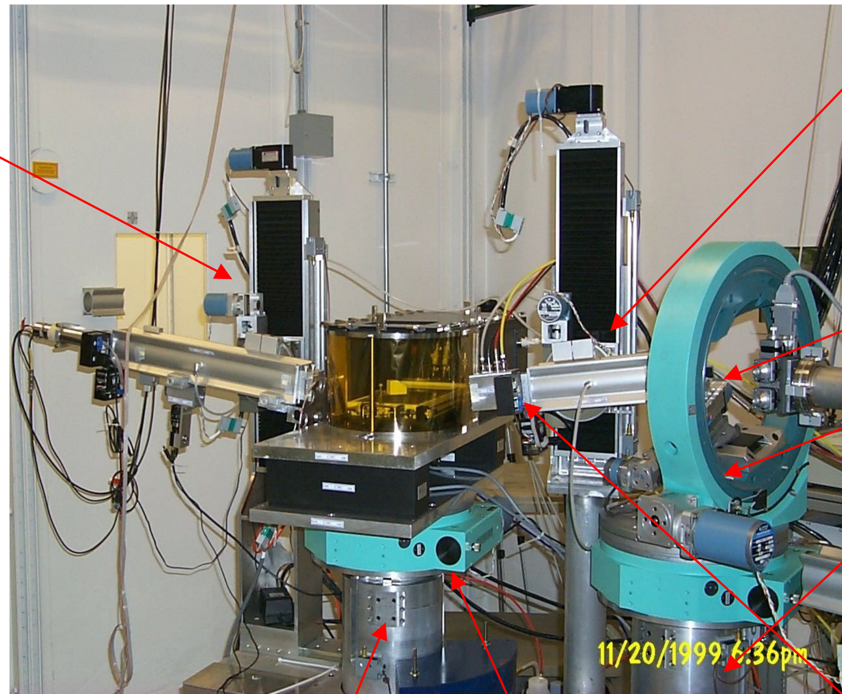
- Chemical exhaust, will eventually require ventilation
  flange already exists in the hutch
- Working in the JPLS hutch with SMI beam
  PPS system for the beam pipe exists
  Requires reevaluation with motion in the hutch
- EPS system for complex motor operations
  the flight path to the SAXS chamber will require safety evaluation
- Reevaluation of the search procedure.

Controls and DAMA

- Controls will build on existing infrastructure implemented through NEXT Project (or already in hand):
  - 24 existing Delta Tau channels, requires additional Delta Tau & new EzAxis motor controllers
  - Motor cables (24) already dropped into hutch
  - Equipment racks exist atop hutch and on the floor outside hutch
  - Will share timing IOC, archiver server, and storage server with SMI
- Need to acquire:
  - Network switch and cable
  - Linux IOC server
  - Workstations and displays
  - Some camera hardware
  - New motors, gearboxes, and connectors to replace existing ones
- Labor to modify existing and implement new controls has been estimated
- Experimental control will adopt BlueSky framework; some DAMA effort to customize for JPLS instrument is needed and has been estimated
- Effort is also needed to update the controls environment to allow facile sharing of the upstream beamline optical system controls between the JPLS and SMI stations

Recycled components from APS Liquid Spectrometer
will be shown in Daniel's talk

Vertical translation rotation (Huber 411)



Vertical translation rotation (Huber 411)

Crystal mount & translation

Eulerian Cradle

Crystal deflector stage

Detector rotation rotation (Huber 430)

Sample rotation (Huber 430)

JJ x-ray slit

Vertical sample stage

Present State of the Design

Verification and Validation of open issues prior to PDR

Vibration isolation
Choice of Detectors
Design of the flight paths
Angular precision of the chi stage
Huber 440 versus 430 for sample 2-theta arm

How the conceptual design supports the JPLS scientific objectives?

JPLS will provide a versatile and flexible platform for studies of liquid surfaces and polymer processing during real time conditions using the existing, high quality undulator beam at SMI. Well suited to existing and expected developments.

The proposed instrument will provide the resolution, intensity, beam size, energy range, stability, accuracy, weight loading for studies of liquid and polymer processing.

The capabilities provided at JPLS complement existing instruments, NSLS II SAXS/WAXS facilities at SMI and CMS, with new capabilities. The proposed instrument will allow the beam to be tipped downward to study liquid surfaces and for processing with liquids.

Spectrometer with SMI undulator beam will provide a world-class instrument.

Cost effective and rapid deployment: design will recycle many costly parts from NSLS, ~$1.5M budget, construction completed in 18 months.

Thanks for your attention
and to the entire JPLS Team

Daniel Bacescu
Lonny Berman
Chris Stebbins
Ron Pindak
Mary Carlucci-Dayton
Mark Schlossman