NYSBC Microdiffraction (NYX) Beamline 19-ID

IRR Functional Description

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1 INTRODUCTION

Primary Research Capabilities

The primary scientific aim for NYX is focused on the needs of x-ray crystallography users from the nine member institutions of the New York Structural Biology Center (NYSBC) and also those of its DoD sponsor; however, the beamline must also meet needs of NSLS-II General Users. Investigators from the NYSBC community include many pre-eminent structural biologists working on challenging problems at the forefront of the field. Challenges come from the size and complexity of systems under study. Moreover, as technological advances make it possible to deal with new challenges, such as those that come from very small samples, demand grows for the technical capability to meet these challenges. Micron-sized x-ray beams are readily feasible at NSLS-II, and we expect substantial demand for this capability. Micron-sized crystals are commonplace for many problems, and they are intrinsic to the lipidic cubic phase (LCP) methods from crystallization of membrane proteins. To achieve the desired brilliant x-ray microbeams, it is essential to take advantage of the enhancements that come best from undulator sources at a low-emittance synchrotron such as NSLS-II.

A second major emphasis for NYSBC beamlines, and a significant focus of research for NYSBC scientists, concerns the optimization for anomalous diffraction experiments. NYX is an intellectual successor to NSLS beamline X4A, which was designed in part for the testing of multiwavelength anomalous diffraction (MAD). While moving to incorporate microbeams, we also want to preserve the versatility of optimized anomalous scattering experiments across a broad spectrum of atomic resonances from different elements and we propose to capitalize on the intrinsic brightness of NSLS-II to optimize anomalous signals by improving energy resolution. In light of the overwhelming success of single-wavelength anomalous diffraction (SAD) phasing, such optimization has two aspects: one concerns the maximization of on-resonance anomalous scattering at the absorption edges, and the other concerns the off-resonance optimization of anomalous scattering signals in light of complications for diffraction experiments at low energy (long wavelengths).

Recognizing that many problems may not require *de novo* phase evaluation, we would not want to compromise generic performance of NYX to optimize anomalous diffraction experiments. With these considerations in mind, we focus for NYX on the kind of science that will benefit from three attributes that have motivated our proposed beamline design: (1) we aim to provide stable, high brightness beams at the level of cross sections from 50-micron down to 5-micron; (2) we aim to preserve the inherent spectral flux from a monochromator crystal (typically Si 111) within a bandpass down to energy resolution of $\Delta E/E = 5\times10^{-5}$; and (3) we aim to support efficient diffraction experiments in the range of x-ray energies from 5 to 17.5 keV. The NYX energy range covers all conventional phasing elements from the uranium L_{III} edge (17.17 keV) down to the cesium L_{III} edge (5.01 keV) with expected emphasis on the Se K edge (12.66 keV). Because low energy experiments encounter several special technical requirements, we have decided to limit the lower reach of NYX to approximately 5 keV, which includes transition metals through the manganese K edge (Z= 25, 6.54 keV) and accommodates demonstrably effective sulfur SAD experiments, and to develop LAX as a separate beamline dedicated to experiments at energies that can reach the potent uranium M_V edge (3.49 keV) and the biologically prevalent phosphorous and sulfur K edges (2.15 and 2.47 keV).

Beamline Staff

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	Dave Cook	Controls Engineer

2 BEAMLINE DESIGN AND COMPONENTS

2.1 Beamline Performance Goals

Table 1 summarizes the design performance of the NYX beamline. The beam size is based on the needs by the supported experiments. An optical layout of the beamline (described below) and the specifications for the individual components are then determined. The flux performance is estimated using ray-tracing simulations that have incorporated the specifications of the source and the optical components. The actual flux will depend on the configuration of the optical components called for by the experiment.

Table 1. Designed performance of the NYX Beamline

Parameter	Specification/Description
Insertion Device:	1 m long, in a low-β straight section.
Operating Energy Range:	4 – 17.5 keV
Monochromator:	Double crystal monochromator (DCM), Asymmetric-cut Si (111) crystals, tangentially and sagittally bent.
Beam size at sample (FWHM):	Variable from 5 μm to ~50 μm
Photon Flux 500 mA storage ring current:	1.5E14 ph/s/0.1%BW
Detector system	ADSC HF-4M Pixel Array Detector (PAD)

2.2 Beamline Layout

The estimated performance above is based on the conceptual layout of the NYX beamline shown in Fig.1.

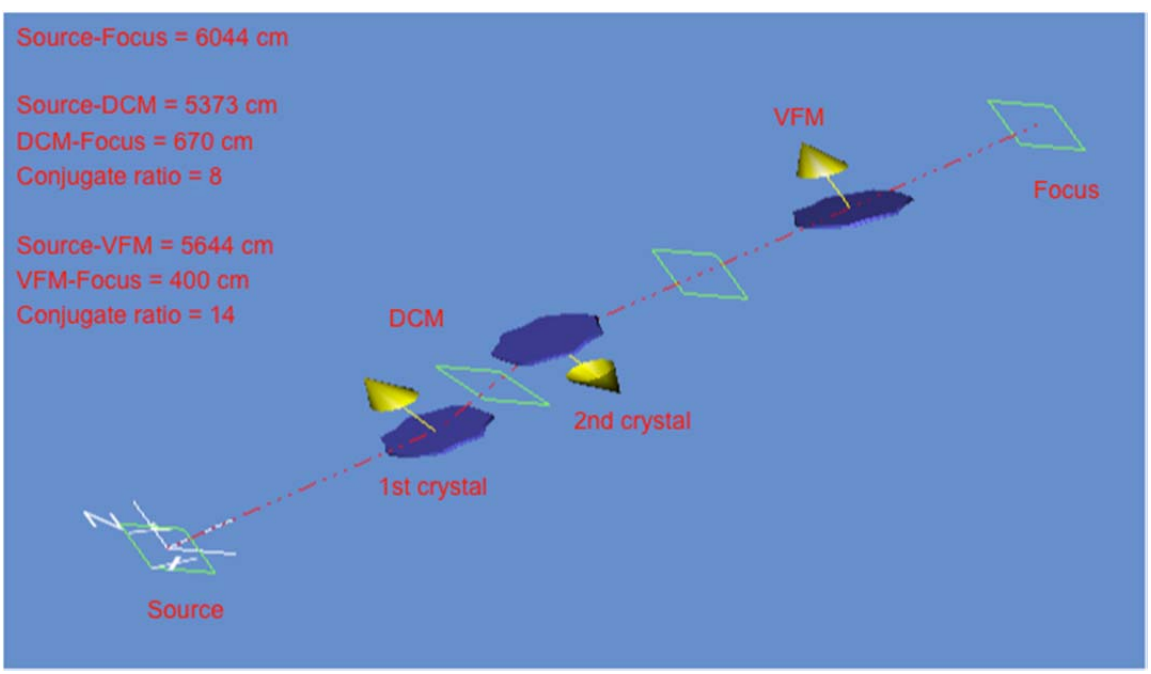


Figure 1 Optical configuration of 19-ID-NYX photon delivery.

The source for the 19-ID-NYX beamline is an in-vacuum undulator (IVU) installed asymmetrically in a canted low-β long straight section. The IVU is a 1m long with a magnet period of 18 mm. The asymmetric configuration of the straight section is to accommodate an upgrade of the 1m source to a 2m source as well as install a second insertion device in the straight section. The undulator parameters shown in Table 2.

Table 2. Main characteristics of the 19-ID undulator source for NYX.

Device Name	**X25**
Device Type	In-vacuum undulator
Period [mm]	18
Length [m]	1m
Minimum Gap @ Low-β Straight [mm]	5.6
Maximum K	1.55

The monochromator is the heart of the NYX beamline and its novel design will define the energy resolution, pre-focus the beam vertically, and focus the beam horizontally. A downstream vertical mirror will complete the vertical focus and also provide harmonic rejection. The overall beamline view is shown in Figure 1 where a double crystal monochromator (DCM) is positioned before a vertical focusing mirror (VFM). We have designed x-ray optics that will capture the full spectral brilliance passed by a Si(111) monochromator but focused into a beam of 10 micron cross-section and while achieving an energy resolution under 6×10^{-5} in $\Delta E/E$ (0.8 eV at 12 KeV). Beams can readily be defocused to 50 micron cross section, and they can be vertically focused to 5 micron cross section with approximately 50% loss in flux in the horizontal plane. Technical details of the layout of beamline components are included in the appendix.

3 BEAMLINE SAFETY

3.1 Radiation Shielding

The design of all radiation shielding (hutches and radiation safety components) follows guidelines to reduce radiation levels external to the beamline enclosures during normal operation to < 0.05 mrem/hr and as low as reasonably achievable. The shielding wall thicknesses follow released shielding guidelines:[1]

Hutch 19-ID-A (FOE, white beam):

- Lateral wall: 18 mm lead
- Downstream wall: 50 mm lead
- Roof: 10 mm lead

White Beam Transport (Beam path, white beam)

- Path length: 17m
- Pipe, bellows and ion pump shielding: 7mm lead

Hutch 19-ID-C (Optics, white and mono beam):

- Lateral wall: 18 mm lead
- Downstream wall: 50 mm lead
- Roof: 10 mm lead

Hutch 19-ID-D (Endstation: monochromatic beam):

- Lateral wall: 6 mm steel
- Downstream wall: 6 mm steel
- Roof: 3 mm steel

A monochromatic beamline shutter of the standard NSLS-II design is installed at the downstream end of the 19-ID-C hutch. The C and D hutches share a common wall downstream of the beamline shutter.

3.2 Radiation Safety Components

These are the components that help contain the synchrotron radiation from the undulator source and the gas Bremsstrahlung radiation from the storage ring. The major components are shown in Figure 2 and described in details below. The location and dimensions of these components are determined based on the ray-tracing analysis and documented in the ray-tracing drawing. Radiation safety components also include devices such as labyrinths and guillotines. A full list of components are identified in the 19-ID radiation safety components checklist (PS-R-XFD-CHK-007).

[1] W.-K Lee et.al., *Guidelines for the NSLS-II Beamline Shielding Design*, (LT-C-ESH-STD-001).

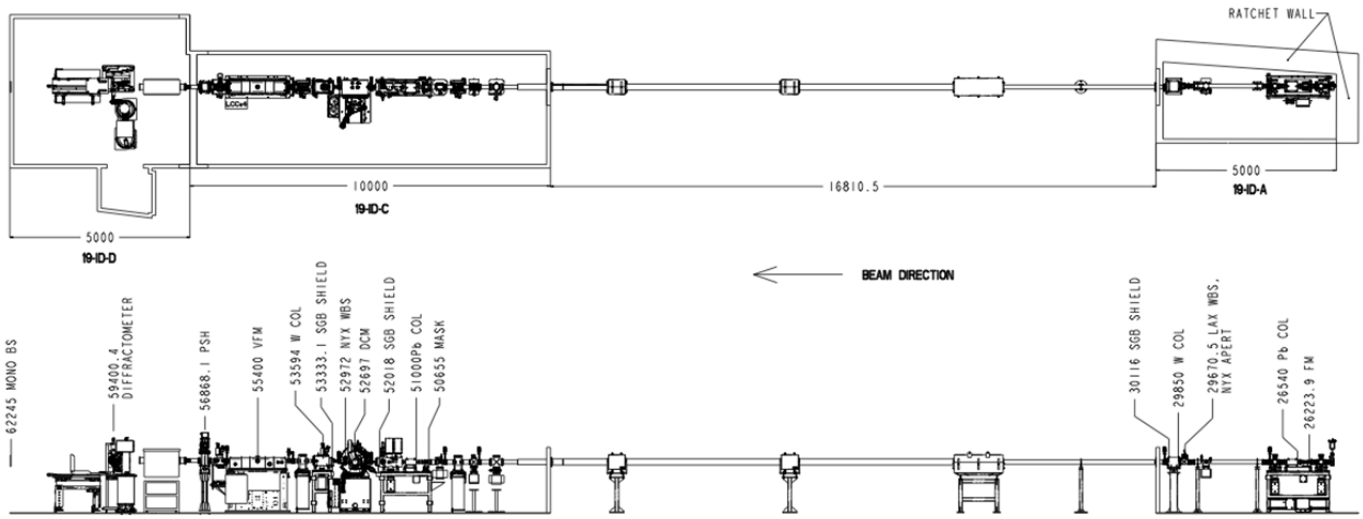


Figure 2. Radiation safety components with location. Details are given in the text.

3.2.1 Heat Load Management (white and pink beams)

All radiation containment and shielding components as well as all photon delivery component have been designed to handle the combine power of the IDs listed in PA-C-XFD-RSI-NYX-001. This combined power includes upgrading NYX with the 7600 watt SAGU and installing a 2500 watt ID in the LAX straight resulting in 10,100 watts maximum combined power from the undulators. The table below lists the expected power upstream and downstream of each water-cooled mask based on the actual acceptance angle of each mask.

19-ID Component Map

Position	Beamline Component	Acceptance Angle	Combined Power Upstream	Combined Power Downstream	Power Absorbed
mm		*mrad²*	*kW*	*kW*	*kW*
-545	Center of Source IVU				
0	Center of Straight				
17893	Fixed Mask-Front End (water cooled) NYX	0.142			
17893	Fixed Mask-Front End (water cooled) LAX	0.122	10.100	9.590	0.510
----------	Ratchet Wall				
26224	Fixed Mask (water cooled) NYX	0.013			
26224	Fixed Mask (water cooled) LAX	0.013	9.590	0.908	8.682
26540	Lead Collimator				
29670	NYX Fixed Mask & LAX Beamstop (water cooled)	0.016	0.908	0.700	0.208
29850	Tungsten Collimator				
30116	Lead Shield (SGBS-1)				
----------	Downstream Hutch Wall 19-ID-A				
	White Beam Transport				
----------	Upstream Hutch Wall 19-ID-C				
49840	XBPM #1 DN160				
50655	Movable Mask (water cooled) on ± 1mm Center	0.012	0.700	0.659	0.042
51000	BS Collimator (Lead)				
51465	Vertical Slit-upper (water cooled)				
51535	Vertical Slit-lower (water cooled)				
51711	Filters # 1-4 (water cooled)		0.659	0.350	0.309
51782	Filters # 5-8 (water cooled)		0.350	0.350	0.000
51919	Screen #1 Diamond (water cooled) DN63				
52018	Lead Shield (SGBS-2)				
52697	DCM (cryo-cooled)	0.012	0.350	3.498E-05	0.350
52972	White Beam Stop (water cooled)				
53333	Lead Shield (SGBS-3)				
53550	Screen #2 (YAG) DN160				
53594	Tungsten Collimator				
54152	XBPM #2 DN160				
54283	4-Jaw Slits				
55400	VFM				
56414	XBPM #3 DN160				
56461	Screen #3 (YAG) DN160				
56868	Photon Shutter				
----------	Downstream Hutch Wall 19-ID-C				
57413	Beryllium Window #1				
57818	Energy Analyzer				
58068	Ion Chamber				
58318	Screen #4 (Phosphor)				
58838	Beryllium Window #2				
59400	Microdiffractometer (goniometer)				
60400	Pixel Array Detector				
62245	Beam Stop - Downstream Wall				
----------	Downstream Hutch Wall 19-ID-D				

The front-end water-cooled mask limits the combined power downstream of the ratchet wall to 9590 watts combined. The first water-cooled mask in hutch 19-ID-A with the aperture size of 4.3 mm (H) × 2.2 mm (V), limits the maximum combined power downstream to ~908 watts. This mask is expected to absorb a total combined power 8,682 watts and water flow rates were verified with FEA for a total power of 9820 watts to accommodate the full upstream power for worst case of misalignment. The total beam power transported downstream to the first optical element is further limited by two additional water-cooled fixed masks and a 500um pyrolytic graphite that serves as a high-pass filter reducing the incident power on the first Si crystal in the monochromator to 354 watts.

The first fixed mask in 19-ID-A hutch is fabricated with Glidcop (body) and the other two downstream masks and white beam stop are fabricated with oxygen-free copper. The masks and white beam stop are cooled by the DI water circuit and interlocked in the personnel protection system (PPS).

3.2.2 Radiation Management of Primary Gas Bremsstrahlung (GB)

There are four components for primary Bremsstrahlung shielding. Four Bremsstrahlung collimators are located immediately downstream of the fixed mask; with the last one located just downstream of the DCM also acting as a Bremsstrahlung stop. An aperture in this Bremsstrahlung stop permits the vertically offset monochromatic beam to pass through.

Ray tracing and computer modeling (FLUKA) indicate that 19-ID is shielded for the maximum planned stored electron beam current of 500 mA. The primary Gas Bremsstrahlung ray traces have been completed in accordance with *Synchrotron and Bremsstrahlung Ray Trace Procedure* (PS-C-XFD-PRC-008) and *Insertion Device Front End Ray Tracing Procedure* (PS-C-ASD-PRC-147) and top-off safety analyzed. In accordance with NSLS-II shielding guidelines the following radiation shielding components are implemented to contain and attenuate the primary gas bremsstrahlung to allowable levels. Two collimators (one Pb and one W) are located in hutch 19-ID-A and two collimators (one Pb and one W) are located in hutch 19-ID-C. The last tungsten collimator in hutch 19-ID-C additionally acts as the primary bremsstrahlung stop.

3.2.3 Radiation Management of Secondary (Scattered) Gas Bremsstrahlung (SGB)

The following radiation containment components are implemented to occlude the Secondary Gas Bremsstrahlung within an 8 deg. cone in accordance with NSLS-II shielding guidelines: one lead shield is located in hutch 19-ID-A and two lead shields are located in hutch 19-ID-C. Additionally two polyethylene shields were located in hutch 19-ID-A and 19-ID-C to attenuate neutrons generated in the primary shields. The scattering of synchrotron and bremsstrahlung radiation has been simulated using FLUKA, which verified that the 19-ID-NYX shielding geometry is adequate at 500 mA beam current and verify that the dose rate outside the hutches is below the limit of 0.05 mrem/hr.

3.2.4 Configuration Control

All radiation safety components are under configuration control, following the NSLS-II Radiation Safety Component Configuration Management procedure (PS-C-ASD-PRC-055). Examples are given in Figure 3.

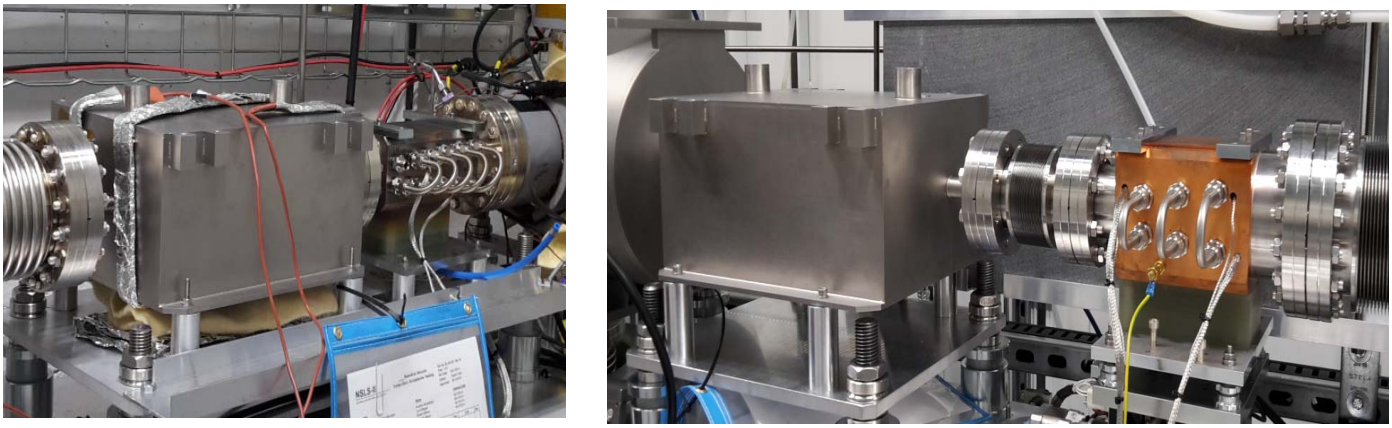


Figure 3. Examples of radiation safety components under configuration control. (Left) Pb collimator & water cooled-mask in the 19-ID-A hutch. (Right) Pb collimator & water-cooled mask in the 19-ID-C hutch.

3.3 Area Radiation Monitor (ARM)

Radiation level in the area is actively monitored through an area radiation monitor (ARM) installed on the outboard wall at the downstream end of the 19-ID-A (Figure 4). The monitor has been certified in accordance with the procedure PS-C-ASD-PRC-008, *NSLS-II Area Radiation Monitor PPS Test.*

Figure 4. Area Radiation Monitor (ARM) installed on the beamline.

Figure 5. Oxygen sensor installed in the FOE to monitor potential oxygen deficiency hazard from LN2 used for monochromator cooling.

3.4 Oxygen Sensor

The Si(111) monochromator uses liquid nitrogen (LN2) for cooling the crystals. The use of LN2 creates potential oxygen deficiency hazard (ODH). This is mitigated by the installation of an oxygen sensor inside the 19-ID-C and 19-ID-D (Figure 5). The system has been certified in accordance with procedure PS-C-XFD-PRC-005, *Beamline Enclosures ODH Monitoring and Alarm System Certification and Inspection.*

3.5 Personnel Protection System (PPS)

The Personnel Protection System (PPS) controls access to the hutches through interlock system and search and secure procedure to ensure personnel safety during normal operation of the beamline. Each hutch is equipped with a PPS-interlocked user labyrinth to facilitate temporary equipment access during user experiments. The C hutch is also equipped with a PPS-interlocked sample access door, to allow for rapid loading of sample cassettes during automated measurements.

PPS also monitors critical DI water flow to the fixed mask, the white beam stop and the pink beam stop (on a single circuit) to ensure the safe operation of the radiation safety components. In the event that water flow is lost, the PPS system will close the frontend photon shutter to shut off the beam.

3.6 Hazard Identification and Mitigation

Overall, the NYX beamline is similar to other beamlines that are already in operation at NSLS-II. A USI evaluation has been conducted and it was determined that the anticipated activities at the beamline do not violate the existing SAD and ASE. All relevant NSLS-II procedures and safety practices are followed during the design and construction of the beamline to mitigate the hazards identified in these document.

4 INSTRUMENT READINESS

4.1 Survey and Alignment

The beamline components are installed according to the specifications and the respective final designs. Installation of the components are verified and documented by the NSLS-II Survey group working closely with the beamline staff.

4.2 Utilities

The following services/capabilities are deployed at the beamline:

- Electrical power distribution: to all electrical power outlets, light fixtures, fans, etc. in the enclosures and along the beamline
- Distribution of deionized water: A hutch only, for high heat-load components
- Distribution of process chilled water: A and C hutches, water-cooled racks
- LN2 distribution and phase separator.
- Compressed air: for pneumatic valves
- Dry nitrogen gas
- Network connectivity
- Cabling and piping support structures, for all utilities including EPS and PPS.

4.3 Vacuum System and Pressure Safety

The vacuum pressure for 19-ID-NYX components is expected to be 10^{-8} mbar or better. There is no physical barrier between the beamline vacuum and front end vacuum. Instead, two ion pumps are installed upstream and downstream of the fixed mask to provide differential pumping. The effectiveness of these pumps have been verified by testing. A fast gate valve sensor is installed just downstream of the fixed mask in 19-ID-A close the fast gate valve in the Front End to protect the storage ring in case of vacuum failure in the beamline vacuum system (e.g. window failure in the experimental station). Ion pumps maintain the vacuum for all components in the A, C and D hutches, to a pressure better than ~10^{-8} mbar. Two Berylium windows (127 micron thick) are installed on 19-ID-NYX. The beamline vacuum is separated from the endstation vacuum by a beryllium window installed downstream of the beamline shutter. This window will normally have vacuum on both sides; however, it is designed to withstand full atmospheric pressure against the vacuum load. The second beryllium is installed before the microdiffractometer. The direction of the pressure difference across both windows will maintained whenever the vacuum vessel is vented to avoid any flexing and work hardening of the of the beryllium.

All vacuum vessels are installed with 1/3 psi pressure relief valves to avoid over pressure when the vessel is vented using dry nitrogen. Three burst discs are used along the beamline for cooling loops internal to vacuum vessels. It is worth noted that the two beryllium vacuum windows were original installed at NSLS and moved for reuse at NSLS-II. The original design and test document has been reviewed by BNL and was used along with the history of performance to determine the acceptance for reuse at 19-ID-NYX.

4.4 Controls

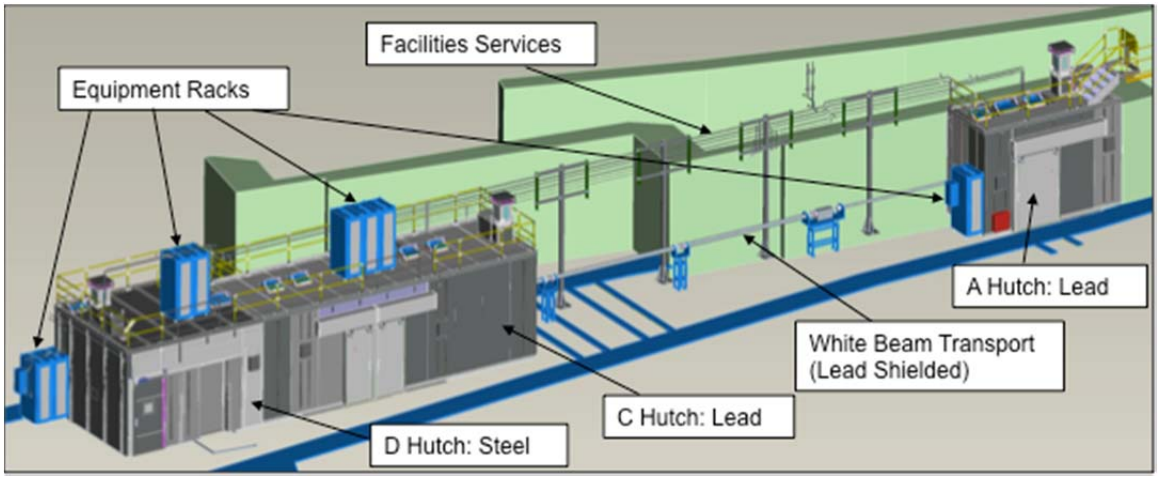
All motorized components have been tested by the control engineer and documented in a checklist. Controls System Studio (CSS) screens have been prepared to monitor all vacuum pumps and gauges as well as temperature sensors.

4.5 Equipment Protection System (EPS)

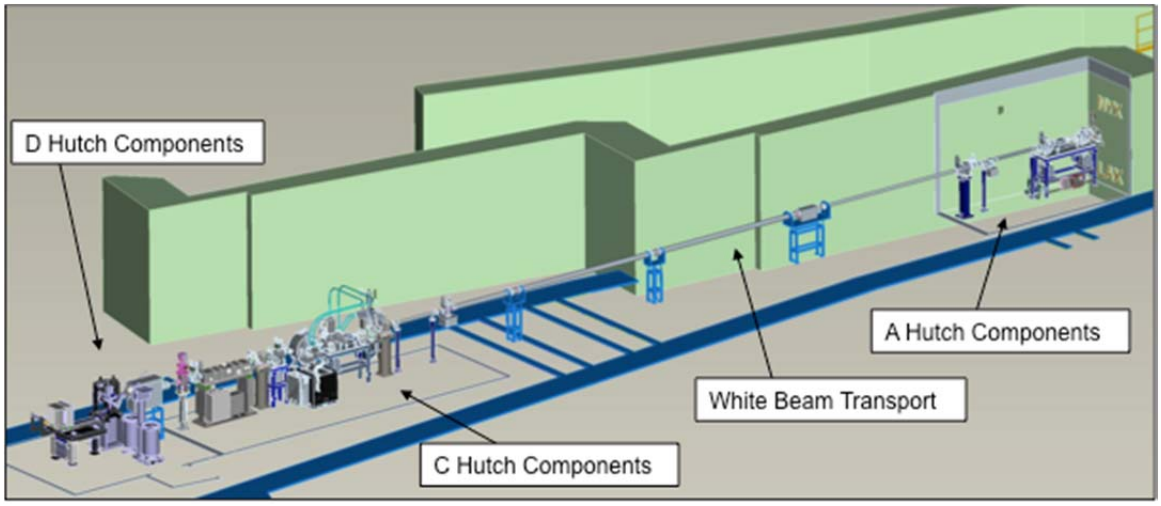
The Equipment Protection System at the NYX beamline performs the following functions:

1. Vacuum pressure monitoring and interlock for all the vacuum sections of the beamline.
2. Temperature monitoring and interlock for all non-safety related components, including components exposed to heat load in the monochromator and white beam mirror system, and in-vacuum motors.
3. Water flow monitoring and interlock for the cooling of the white beam mirror.
4. EPICS interface for components that require I/Os installed on the EPS PLC, including the readout of LVDTs and control of venting and evacuation valves in the experimental station.

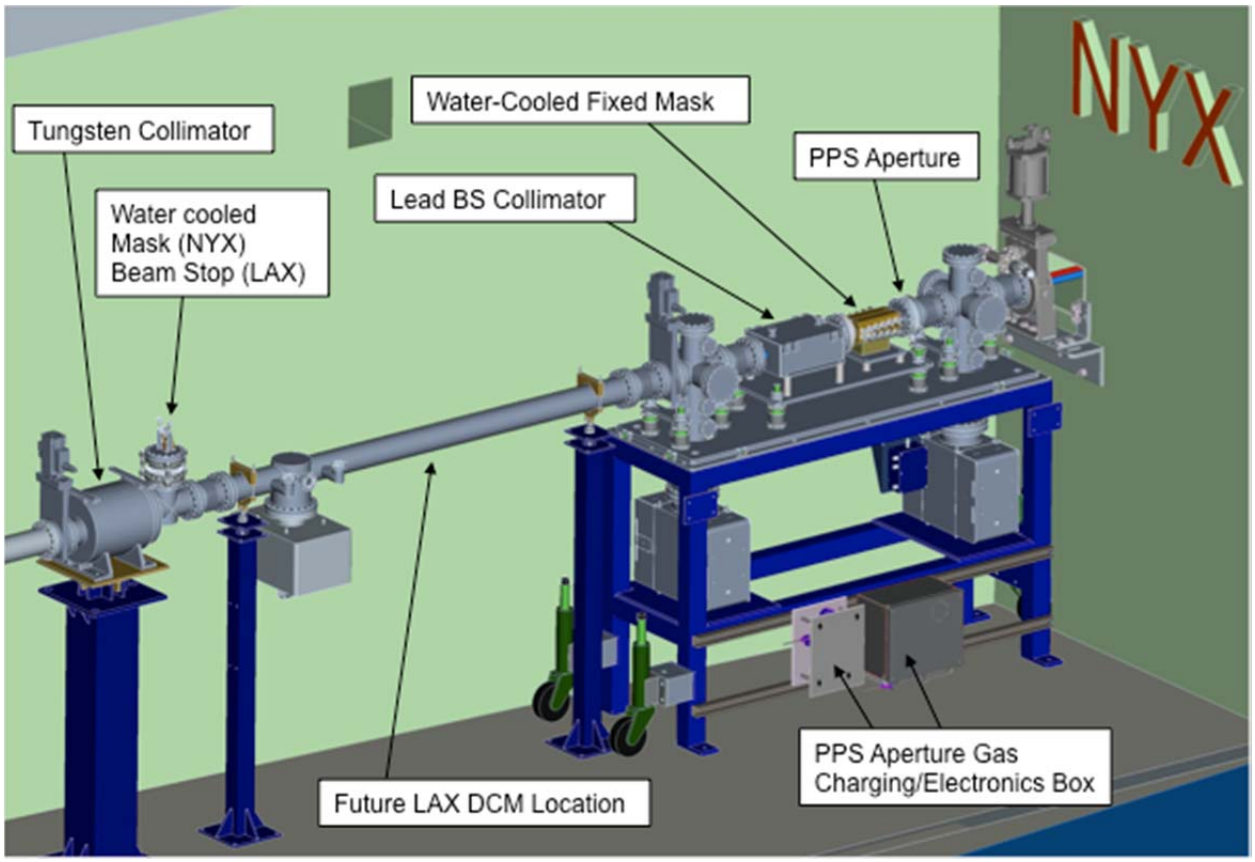
APPENDIX: LAYOUT OF THE 19-ID-NYX BEAMLINE COMPONENTS



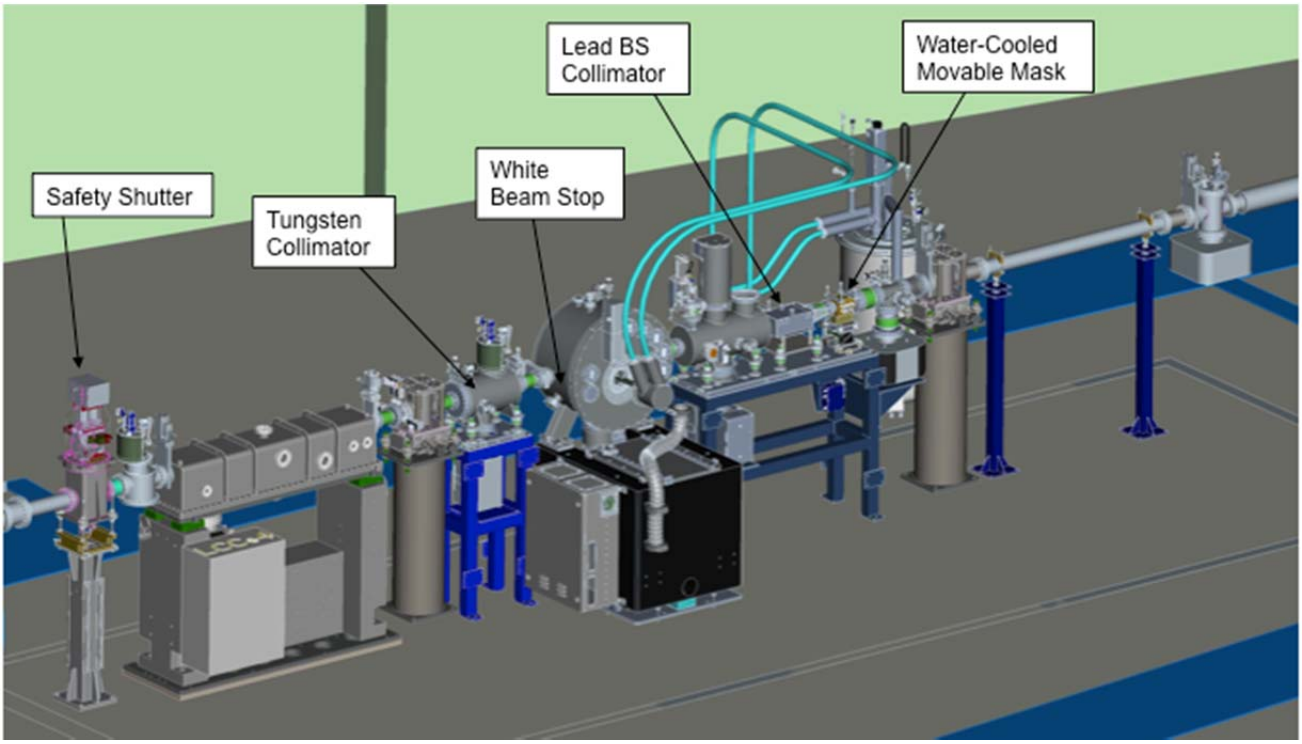
19-ID Hutch Layout



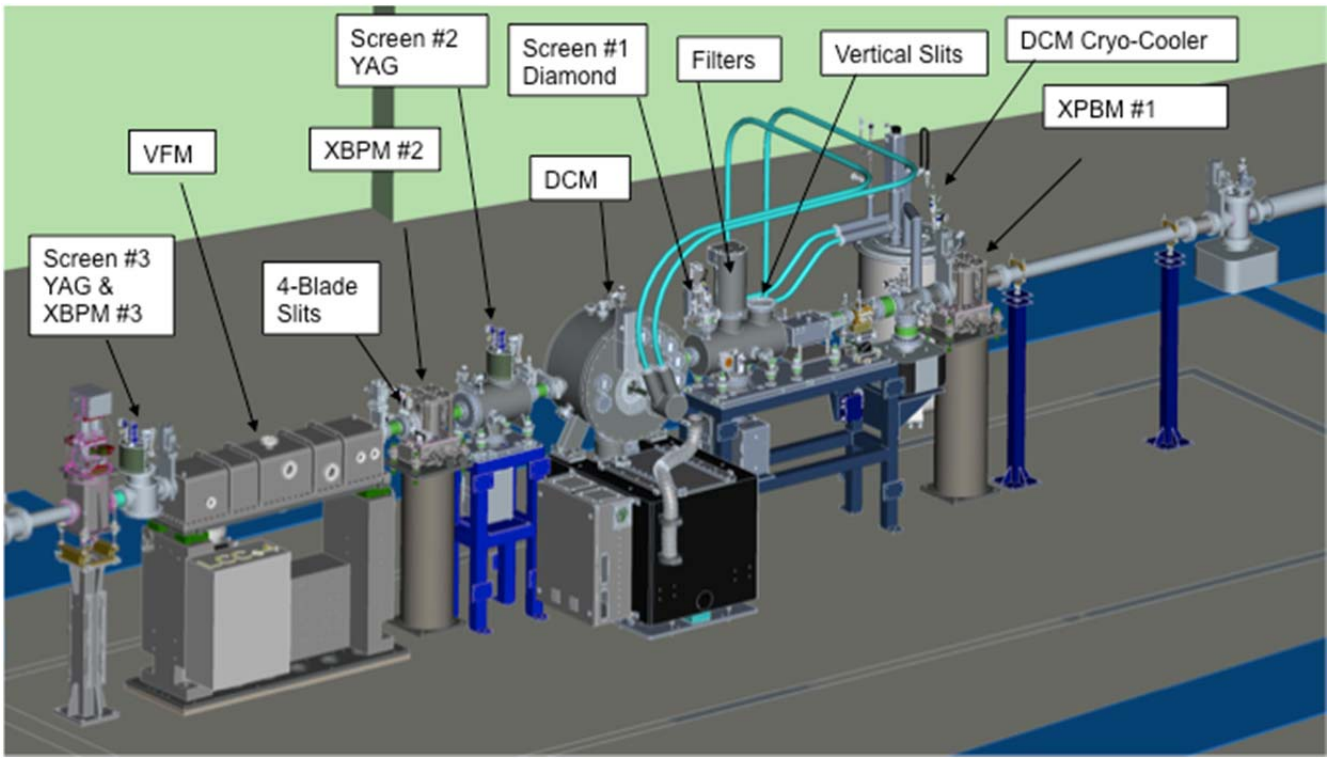
19-ID Component Layout



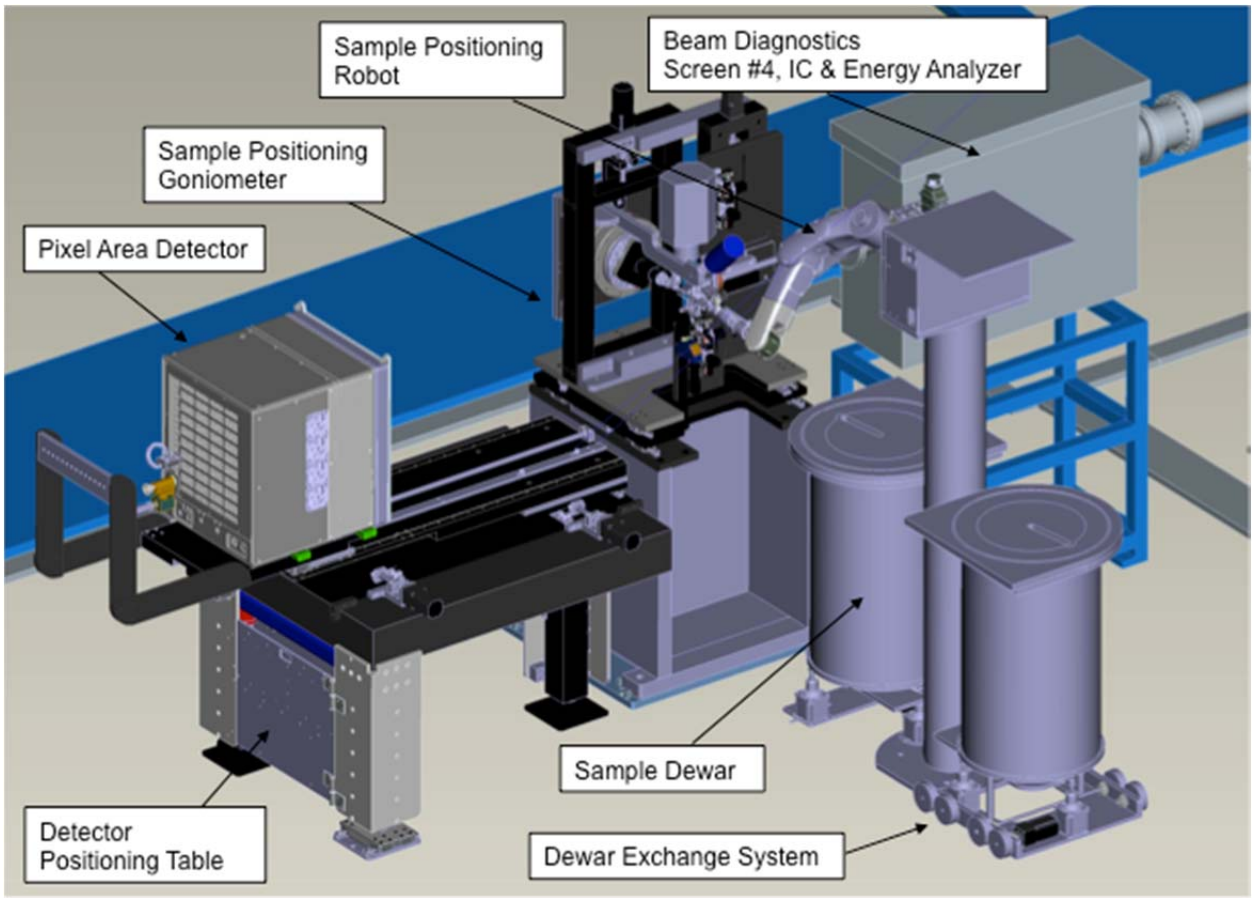
Hutch 19-ID-A Components



Hutch 19-ID-C Radiation Containment Components



Hutch 19-ID-D Photon Delivery Components



Hutch 19-ID-D Endstation Components (Microdiffractometer, Robotic Sample Mounting, Pixel Area Detector, Beam Diagnostic)