5 XAS: HARD X-RAY ABSORPTION SPECTROSCOPY

5.1 Executive Summary

This document describes the strategic need and preliminary technical design for a damping wiggler-based hard x-ray absorption spectroscopy beamline as an NSLS-II Project Beamline. It represents the synthesis of user and scientific community input, interactions with project and accelerator staff, contracted engineering reports from Accel Corporation, and technical reviews by the NSLS-II EFAC and others.

The technique of x-ray absorption spectroscopy uses each element's characteristic absorption edge(s) to obtain local (within ~10Å) information about that element. It is a non-destructive, element-specific probe of local physical and electronic structure, speciation, and chemical state. XAS measurements may be made by transmission, characteristic x-ray fluorescence, or photoelectron yield. Samples may be crystalline or amorphous, and in nearly any form (solid, liquid, gas, solution, mixture, etc.), and may be measured *in situ* under a variety of conditions.

5.2 Scientific Objectives

Mission and specifications: The mission of this beamline is twofold. One is to provide a versatile and highly productive facility for applications of hard X-ray Absorption Spectroscopy in a wide range of scientific disciplines including material science and catalysis, nanomaterials research, environmental science and geology, and life sciences and biology. This is necessary to address, from the outset of user operations, the significant demand for high-quality XAS at the NSLS. The second, and equally important, mission is to pursue cutting-edge capabilities and techniques in XAS, such as are appropriate to capitalize on the advanced qualities of the NSLS-II source. This beamline therefore is intended to outperform all currently available XAS facilities in the combined metrics of flux, versatility of spatial and energy resolution, and energy range.

Based on these objectives, beamline specifications are summarized as follows:

Source:	Damping Wiggler, length 7 m (or one of two canted 3.5-m segments)
Monochromator crystals:	LN2 cooled, flat crystals; 1st pair Si(111), 2nd pair Si(311) switchable by horizontal translation
Energy range:	~5 – 50 keV, with provisions for reaching 90 keV
Focusing:	macro: toroidal mirror, micro: K-B set
Spot size at sample:	operating values around 1mm macro- and 1 micron micro-focused, but up to 5x40 mm unfocused
Estimated photon flux at sample:	> 10^{13} – 10^{14} ph / s / 0.1% BW (10^{12} for microbeam)
Energy Resolution (standard):	dE/E 3×10^{-4} (Si(111))
High energy resolution mode:	1×10^{-5} or better, with energy-refining monochromator
Scan modes:	continuous-scan (slew) or step-and-count
Endstation 1:	microbeam XAS
Endstation 2:	bulk XAS, three sample positions: classic benchtop, controlled atmosphere, large apparatus

User need: Synchrotron XAS is a cornerstone of material and chemical analyses on the molecular scale. As such, its use has become routine and less "exciting," but not less important. A statistical analysis obtained from NSLS User Administration showed that, in FY06, 653 of 3295 on-site users at the NSLS (22.5%) were at the 8 beamlines devoted to hard XAS (and 2 beamlines performing microbeam XAS). To weigh possible overlap in these beamline-specific numbers, a manual count of unique individuals currently involved in XAS experiments yielded 505, a comparable percentage of the ~2250 current active NSLS users. These users represent a wide range of scientific fields, are interested in nearly every element of the periodic table, and have samples ranging in size from nanometers to centimeters.

In order to benefit the greatest range of users, without compromising the more advanced capabilities and potential future directions, this beamline is equipped with versatile and adaptable endstation components founded on a solid infrastructure and beamline optics designed to deliver the highest quality (and quantity) of beam possible. These details are described more fully in the sections to follow. Efficiency is also an important aspect of productivity, so both the optical configuration and experimental setups are intended to minimize downtime. In addition, an available continuous-scan mode, rather than more typical step-and-count measurement, will significantly enhance throughput.

Scientific opportunities: Current challenges facing application of XAS in more difficult systems are signal strength, fluorescence detection, spatial and energy resolution, time, and energy range. The status of XAS as a more mature technique has left many with the impression that there is nothing new to develop. However, the unparalleled qualities of the NSLS-II open the door to considering new aspects of the technique (e.g. higher energy resolution) and new applications.

The very high flux available will make possible measurements at lower concentrations and using smaller sample mass. This is important in the study of catalysts, environmental contaminants, dispersed nanoparticles, and dilute biological systems. It will also enable “bulk” measurements of very small quantities of material, such as atmospheric particulates and marine colloids, extraterrestrial material, proteins and biological complexes, and sub-monolayer surface adsorbates.

Many problems in XAS analysis are the result of detector-limited fluorescence measurements. For example, an energy-dispersive solid state detector has intrinsic limits of energy resolution and total count rate. Samples with very low target signal and very high background will saturate the detector before enough useful signal is obtained. Interfering fluorescences can similarly overwhelm a weak signal. Increasing flux alone will exacerbate these problems. However, the considerably higher flux available here will enable the use of energy-selective detectors, such as wavelength-dispersive detectors or crystal analyzers. Historically these have not been widely used because they can only accept a very small fraction of the fluorescence emanating from the sample, and therefore required very high sample concentrations. The NSLS-II XAS beamline will provide sufficient flux to use energy-selective fluorescence detectors at trace concentrations. Recent advances in detector technology have made tremendous improvements and are expected to continue to do so. These further developments will be incorporated in the final design to add to the capabilities of this beamline.

Spatial resolution is the primary goal of the nanoprobe beamline. However, XAS experiments cover the whole range of spatial resolution from nm on up. Therefore this beamline is designed with an adaptability of spot size, from unfocused 5x40 mm down to as small as 200x200 microns in the “bulk” endstation, and down to ~1 micron in the microbeam endstation. Many XAS experiments involve processes occurring on a range of spatial scales, but have previously been relegated to separate bulk and micro facilities. Tunability of spot size at a single facility will allow users to better tailor measurements to their needs.

Energy resolution is most important for near-edge XAS structure (referred to as XANES or NEXAFS), where there may be sharp features that are highly sensitive to local electronic structure. These features, such as the pre-edge peak of hexavalent chromium (K edge 5.9 keV), the arsenate peak (As K edge at 12 keV), or pertechnetate (Tc K edge at 21 keV), are narrower than, or exhibit chemically-induced shifts of less than, the best resolution of a Si(311) monochromator. Yet methods of producing higher-resolution monochromatic beams are less efficient and naturally have lower bandpass, and thus have significantly decreased flux as compared with more typical energy resolution monochromators. The high flux and well-collimated beam available at NSLS-II afford the luxury of employing an energy-refining monochromator to accomplish these measurements even at trace concentrations.

Time-resolved experiments are naturally facilitated by high flux. While it will not be possible to move this high-heatload monochromator fast enough for true “quick” XAS (sub-second scan rate), the continuous-scan mode will collect scans on the order of one to three minutes, for time-resolved experiments on the appropriate scale.

Energy range has been a limiting factor in XAS applications. X-ray Absorption edges range from the soft x-ray regime to over 100 keV, while typical Synchrotron hard XAS facilities operate between 4.5 and 30 keV. The 5-90 keV range listed above encompasses the K edges of chromium to bismuth, and L edges of cesium through americium. There has recently been a dramatic increase in applications of "tender" (1-5 keV) XAS as facility development has made that range more accessible. Similar opportunities exist for higher energies (35-90 keV): using K edges as alternatives (or complements) to L edges, probing thick samples or buried regions, penetrating large-volume catalytic or high-pressure cells, avoiding interferences, and reducing radiation damage (e.g. in biological samples containing trace heavy elements such as mercury).

The rare-earth elements are of common interest to both materials research (optoelectronics) and geochemistry, at trace concentrations. Routine XAS work has been limited to the L edges (at 5.5 to 11.3 keV), but these often overlap and interfere (there being 48 L edges in that span). In contrast, the 16 K edges of these elements are nicely resolved over the span of 38 to 65 keV.

One other characteristic quality of NSLS-II that is of important consideration for these applications is its expected world-leading beam stability. While classic XAS experiments involved large uniform samples, most current and expected future samples are heterogeneous on varying spatial scales. These analyses require beam positional stability (e.g. better than 5 microns for a 1 mm spot) over a series of 1000-eV scans. Stable intensity (i.e. top-off injection) is critical to maintain stability of optics under high heat loads. And energy stability, the repeatability of energy calibration over a series of scans, is a prerequisite for useful high-energy-resolution measurements. The design of beamline components must then make a conscious effort to not degrade the inherent source stability.

Research programs: Specific research programs are being developed (or adapted) for this beamline by facility staff and leading user groups. Such programs will be the focus of a beamline workshop scheduled for January 16-17, 2008, following up on the NSLS-II User Workshop breakout session in July, 2007. Several of these will capitalize on the technical opportunities outlined above to address important DOE needs. The following two representative examples highlight research that will do so. In catalysis research, it is important to be able to measure the catalytic reaction *in situ* and/or in time-resolved fashion. However, in order to obtain sufficient signal quality in an appropriate time, this often requires preparation of samples with higher concentrations than are used in actual applications. These may not function the same as their dilute counterparts. The higher flux here will allow measurements at real-world application concentrations. This program will also require the capability of simultaneous XRD to more completely characterize sample systems in one run.

A similar example may be found in environmental stewardship and remediation. Of great concern to DOE is the subsurface contamination present at many of its sites, especially contamination by radionucludes and mercury. Typical concentrations in these environments, however, are below the current EXAFS sensitivity limits of most facilities. Moreover, wet sediment samples are heterogeneous, contain interfering elements, and produce high backgrounds due to fluorescence, scattering and diffraction. Yet detailed knowledge of chemical and physical speciation and processes is essential to determine appropriate courses of action for long-term stewardship and remediation of these DOE sites. The high flux, tunable spot size, energy resolution, and available energy-selective detection are all attributes which make the NSLS-II XAS beamline key in addressing this issue.

Future development and integrated facilities: The expected needs of the user community for XAS facilities at NSLS-II can not be met by a single beamline, however versatile and productive. Therefore, this beamline is part of a larger scope and long-term strategy that includes build-out of this beamline by canting the source, construction of additional non-Project beamlines, and migration of beamlines from NSLS.

The beamline design presented here explicitly includes accommodations for future canting. This is a highly desirable and cost-effective means of doubling capacity, as summarized here:

Sources:	Two canted damping wiggler segments (3.5 m each)
Separation between on-axis beams:	3.5 mrad

Inboard beamline:	microbeam, ~1 micron
	Acceptance up to 0.1 mrad x 0.1 mrad, on axis
	1st experimental station
	additional optical components
Outboard beamline:	Bulk beam size
	Acceptance up to 1 mrad x 0.15 mrad max., on axis
	2nd experimental station
	existing optical components

While the current project scope includes both endstations, it includes only one set of optical components for a single source. It is expected that canting of the source and addition of a second set of optics to make the two endstations independent will be undertaken at the earliest opportunity. There is very high demand, as well, for the lower energy range. This will likely be addressed by installing a complementary 1 to 5 keV bulk/micro XAS beamline at an adjacent dipole source as part of a core cluster. Additional non-Project XAS beamlines are expected to include “quick” XAS (sub-second scan time), perhaps on an undulator source, and specialized catalysis and biological XAS beamlines.

5.3 Insertion Device

XAS applications require a stable, broad energy range, non-coherent, high-flux source. The NSLS-II Damping Wiggler is an ideal source for XAS. These IDs are necessary for performance of the accelerator, and will be installed in high-beta straight sections. The standard geometry is a 7 m long device, but this can be effectively divided into two canted 3.5 m segments to provide additional capacity as described above. As shown in Figs. 5.3.1 and 5.3.2, the DW-100 source produces the highest total flux of all NSLS-II sources in the 5-35 keV energy range. Furthermore, it yields the highest brightness of the broad-spectrum sources over the same energy range. However, the spatial distribution of the generated radiation is energy-dependent. As shown in Fig. 5.3.3 and 5.3.4, the higher energy radiation is limited to the central portion of the fan. Since XAS applications require a uniform distribution as energy is scanned, this restricts use to the central on-axis portion of the fan. It also requires narrower acceptance when working at high energies, but that is naturally limited by the angular acceptance of the mirrors and monochromator. These factors make it undesirable to split a single source fan into the two experimental hutches; each application requires on-axis radiation by alternately sharing a single source (and eventually each having its own canted source).

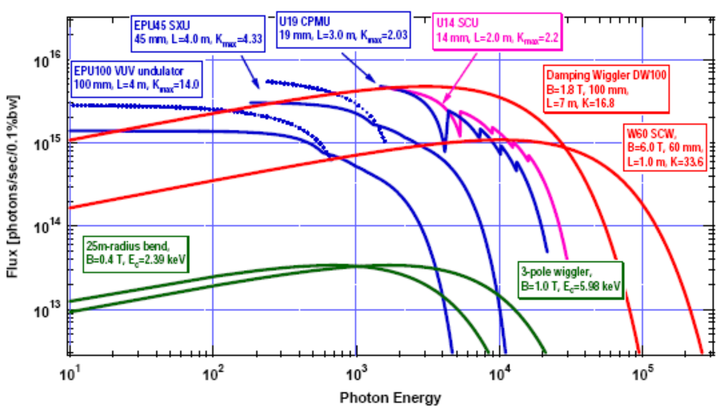


Figure 5.3.1. Flux of various NSLS-II sources.

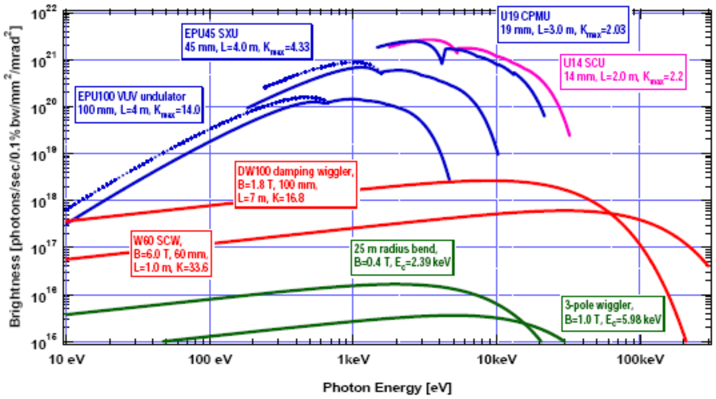


Figure 5.3.2. Brightness of various NSLS-II sources.

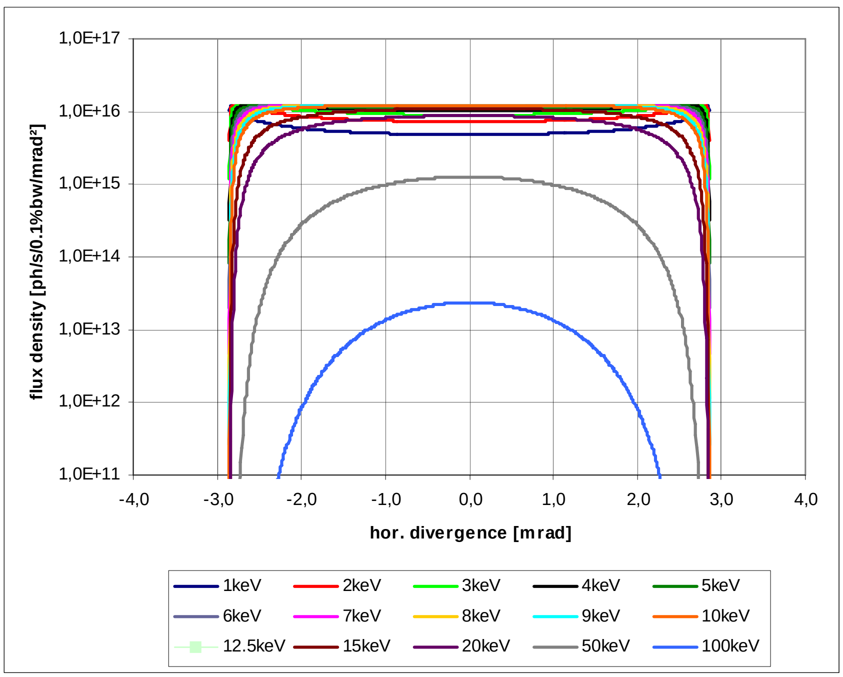


Figure 5.3.3. Horizontal distribution of flux density.

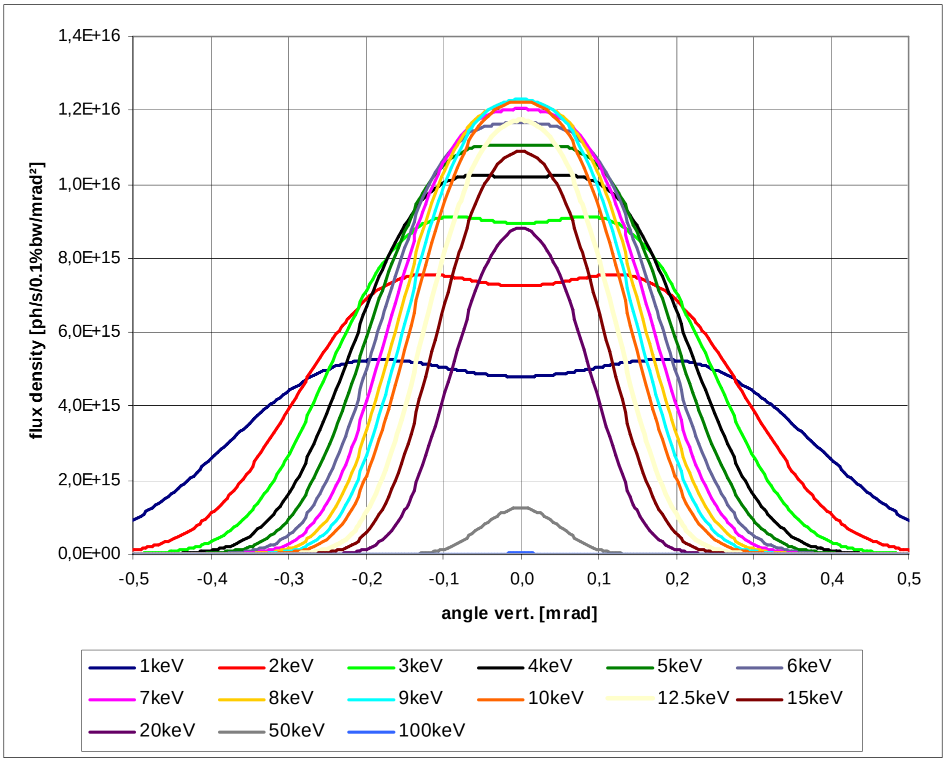


Figure 5.3.4. Vertical distribution of flux density.

The most challenging aspect of using the damping wiggler source is the unprecedented heat load. The full fan of a 7 m ID delivers approximately 65 kW of power, and even restricting acceptance to 1 mrad horizontal and 0.15 mrad vertical still delivers nearly 8 kW to the beamline optics. This maximum angular acceptance (in both dimensions) is defined by the optics (mirrors and monochromator), rather than by heatload limits. Heat load will be discussed further in the description of each component.

Current strategy for canting considers two possible paths. One is to begin operations with 7 m of wiggler in-line and introduce canting at a later stage. This has the advantage of extracting the highest possible flux during early operations when the ring current will not be at the full 500 mA, but has the disadvantage of requiring a repositioning and re-alignment of the ID, front end, and all optical components. The other path is to begin operations with two canted sections but only use one of them. This has the advantage of establishing the positions and alignments that will be used even when canting is fully implemented, and also provides the facility with an early test case for commissioning and refining canted wiggler sources. It has the disadvantage, however, of less flux initially. This second option is favored by beamline design personnel for the added reason that it reduces the heat load issue by an effective factor of two, and improves access to the lowest energy around 5 keV. In either case it is therefore unlikely that the beamline will experience full power from a 7 m wiggler at full ring current. However, component design is based on being able to handle this full heat load, so as to provide a margin of safety and to allow for potential future changes in beamline or project design.

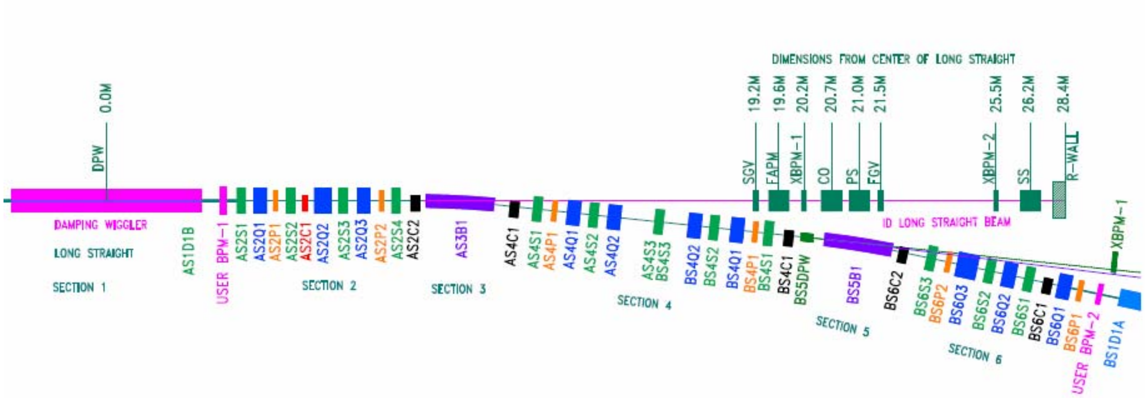
Note that current ray-tracing, heatload and performance calculations are based on a damping wiggler with 100 mm period (DW-100). Current device design now calls for a period of 90 mm (DW-90); this will have a minor effect. Brightness and flux (within the used portion of the radiation fan) will increase slightly, as will heatload and power density. Ongoing thermal and performance modeling will take this into account as component design progresses.

5.4 Sector Layout

This sector begins with the insertion device as described above. Front End components are located within the ring tunnel, upstream of the shield (ratchet) wall. On the Experimental Floor, beamline layout consists of a first optic enclosure (FOE) containing all white-beam components and beamline optics through photon shutter, and two experimental endstations in series.

5.4.1 Front-End Layout

A standard front end layout is shown below, with specific items and their positions for this beamline in the table to follow. Note that accommodations are made now for future canting and upgrades.



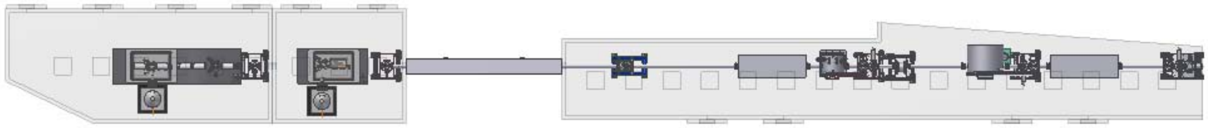
	Position (center) (m)	Acceptance (horiz x vert) (mrad)
Source	0.0	~5.5 x 0.8
Slow gate valve	19.2	
Fixed aperture mask	19.6	(-1.90/+2.35) x (+/- 0.25)
Beam position monitor	20.2	
Bremsstrahlung collimator	20.7	(-2.00/+2.45) x (+/- 0.30)
Photon shutter	21.0	
White beam slits (not shown)	22.5	(+/- 0.6) x (+/- 0.20) centered on outboard cant
room for white beam slits (second canted beam)	23.0	
room for possible future side-deflecting mirror	24.2	
Beam position monitor (removed from design)	25.5	
Safety shutter	26.2	
room for safety shutter (second canted beam)	26.7	
Bremsstrahlung collimator built into shield wall	28.2	(-2.00/+3.40) x (+/- 0.30)
Shield wall face	28.4	

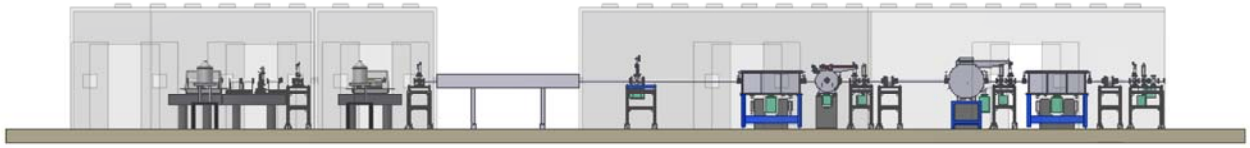
A beamstop and gate valve will be initially installed at the shield wall for commissioning of the ID and front end components.

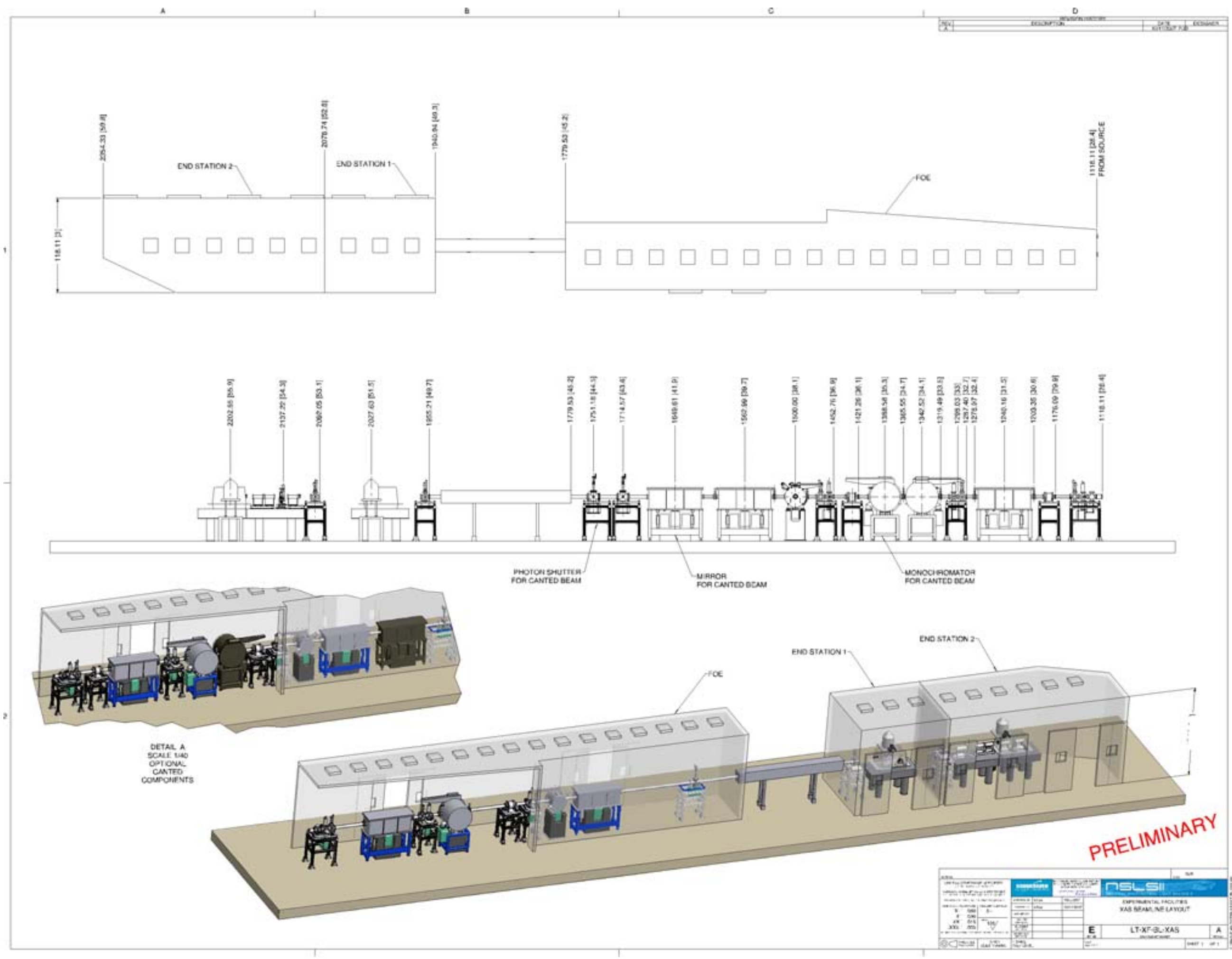
5.4.2 Beamline Layout

The following table provides a detailed layout of beamline components. Schematics and 3-D rendering of the XAS beamline follow the table. More detailed drawings can be found in Appendix 5B.

	Start	Center	Fixed Pos	Length
Shield Wall			28.4	
spool piece, gate valve, bellows	28.4			0.3
Pre-Filter, Attenuators (all water-cooled)	28.7			1.1
Bremsstrahlung collimator	29.8	30.1		0.6
gate valve, bellows	30.4			0.2
Mirror 1	30.6	31.5		1.8
gate valve, bellows	32.4			0.3
Monitoring	32.7			0.3
Steering mask and Be window	33.0			0.5
Double crystal monochromator (high heatload)	33.5	34.1		1.2
gate valve, bellows	34.7			0.2
space for monochromator, canted beam	34.9	35.5		1.2
Bremsstrahlung and white beam stop	36.1			0.5
Beam monitoring and Monochromatic slits	36.6			0.8
gate valve, bellows	37.4			0.2
High energy resolution monochromator	37.6	38.1		1.0
gate valve, bellows	38.6			0.2
Mirror 2	38.8	39.7		1.8
Gate valve, bellows	40.6			0.3
Space for Beam monitoring and Mono slits, canted beam	40.9			0.8
Space for optics, canted beam	41.7			1.6
Photon shutter	43.3	43.55		0.5
Space for Photon shutter, canted beam	43.8			1.0
FOE wall	44.8			0.4
flight tube, shielded	45.2			4.1
Experimental hutch wall	49.3			0.2
Monitoring	49.5			0.5
Exit window and slits	50.0			0.1
Sample location, Endstation 1 (incl. microfocusing optics)		51.5		2.7
Experimental hutch wall	52.8			0.2
Monitoring and slits	53.0			0.5
Sample location 1, Endstation 2		54.3		1.5
Sample location 2, Endstation 2		55.9		1.8
Sample location 3, Endstation 2		58.0		3.0
Hutch wall	59.8			0.2
End	60.0		60.0	







Initial ray-tracing and performance calculations are based on a beamline length of 64.3 m. Subsequent facility design refinements have decreased this to 60.0 m. Adjusting endstation positions required an increase in horizontal focusing to ~2.7:1, resulting in a marginally larger spot size and increase in vertical distortion of partly-focused beam. The ideal value is 2:1, and values of 3:1 or greater result in a decrease of horizontal acceptance of the focusing mirror to less than 1 mrad. Final design will consider this balance between layout and performance in consideration of planned optical elements. For example, it may be advantageous to place the future canted-beam monochromator downstream of this beamline's focusing mirror, thus resulting in more ideal 2.3:1 focusing with the added challenge of having adjacent white beam pass through the high energy resolution monochromator and mirror 2 chambers.

5.4.2.1 Survey and Alignment

All beamline components will be surveyed and aligned in place by the facility. In order to facilitate ease of alignment, all components will be fiducialized to external reference points on their table during assembly. These include horizontal and vertical position and angle, relative to the photon beam. All components are designed with a liberal tolerance allowance greater than 0.5 mm. Provisions will be made for laser pre-alignment of beamline optics.

5.4.2.2 Utility Layouts

This section describes the utility requirements assumed for the damping wiggler beamlines at NSLS-II. The numbers provided are estimations based on the current understanding of the beamline design; significant deviations may be possible depending on possible future evolution of the beamline layouts.

Cooling water. Cooling water is required for all high-heat-load components except the monochromator. Process water, the standard cooling water provided by the facility for this purpose, is clean de-ionized water at a temperature of approximately 20°C. Additional requirements are as follows: temperature stability within 0.1°C, pressure 60 to 100 psi, pressure stability within 5 psi, and free of pump vibrations.

The components and their requirements are listed in the following table.

Component	Number of Circuits	Consumption max.
Pre-Filter and Be-Window	2	4 l/min
Attenuator Units	4	8 l/min
Collimating Mirror and Mirror Protection Mask	1	12 l/min
Steering Mask and White Beam Monitor	2	6 l/min
Thermal Stabilization and Compton shielding of DCM	1	4 l/min
White Beam Stop	1	4 l/min
total:	11	38 l/min

If additional control is necessary, the DCMs temperature stabilization system will be separately supplied with temperature-controlled water by means of a dedicated chiller unit, thus allowing to set the flow to be set and the temperature precisely stabilized, independently from the main water supply in the hutch. This chiller will make it possible to set the water temperature in the range of 25 to 35°C and to keep it constant to ±0.1°C. It can either be equipped with its own electrical cooling system or be connected to the hutch cooling water.

Liquid nitrogen. The cryo cooler unit for the DCM requires connection to a liquid nitrogen (LN2) supply. It is recommended to have a LN2 supply tap within 2 to 3 m of the final cryo cooler position. LN2 is also needed within both experimental hutches for sample cooling and for auto-filling of detector dewars.

Compressed air. The following components must be connected to a dry, filtered compressed air supply having a pressure between 70 and 100 psi:

- the monochromatic beam shutter
- all gate valves
- attenuator units, if pneumatically driven

Electrical power. Three types of power are required: standard power for heating, pumps, etc.; low-noise power for measuring equipment; and UPS power for critical systems. Along the beamline a grounding bar is needed, to ground the beamline components. The grounding bar must be connected to the central power distribution ground. A separate low-noise ground is also needed. Standard beamline service of 60 kW is expected to suffice. There are several distinct areas to which electrical power distribution is needed.

1. Beamline Control System Cabinets

A central 3-phase power distribution should be placed near the cabinets. In total, approx. 12 kW are needed.

2. Cryo Cooler Unit

This unit needs a single-phase power distribution of approximately 2.5 kW.

3. *FOE*

The required power in the FOE can be up to 30 kW to accommodate the extreme case where the full beamline might be pumped and baked at the same time.

4. Experimental Hutch

In the hutches, both low-noise and standard power outlets should be distributed along the length of the hutch. Total needs should not exceed 20 kW.

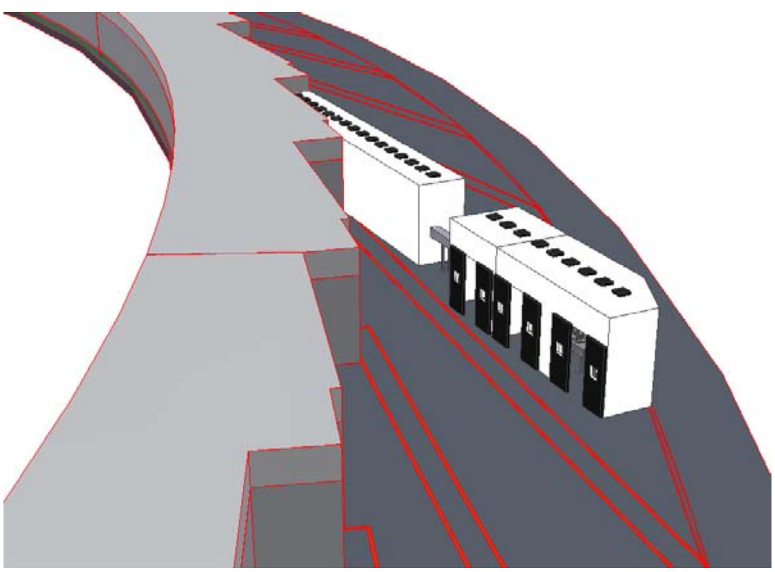
5. *Experimental Control Station*

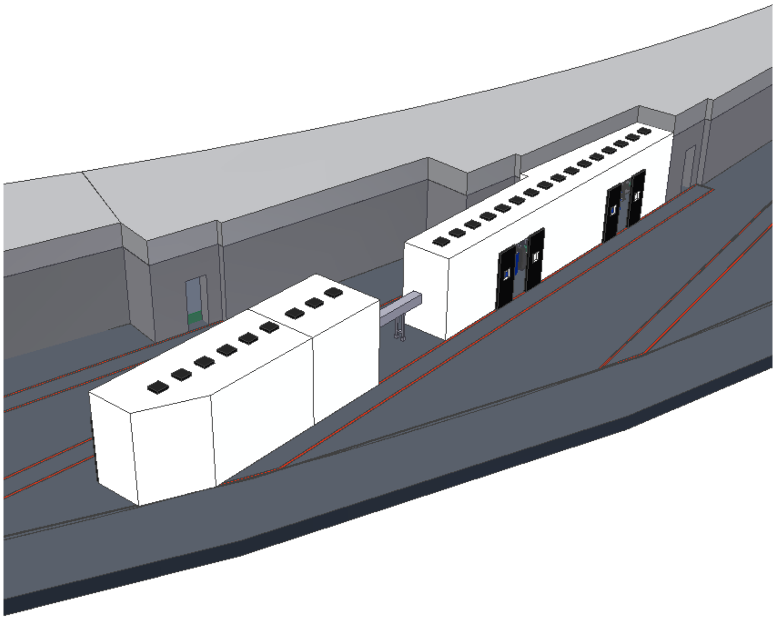
The control station needs adequate power (of all three types) for computer controls, interfaces, and communications, estimated as 5 kW max.

Gases. Dry nitrogen is needed in the FOE, delivered to each vacuum pump-out port as described in 5.4.2.4. In addition, a distribution and control system is needed for local-source gases (helium, Argon, etc.) within the experimental hutches: for enclosures, ion chambers, detectors, sample cells and experimental apparatus.

5.4.2.3 Life Safety Code Compliance

Floor layout and access walkways will be arranged so as to comply with applicable emergency egress requirements. Figures 5.4.2.3a and b show initial access/egress walkways in red. Specific details will be developed in conjunction with layouts of neighboring beamlines, and in conjunction with ES&H personnel and NSLS-II policy. Exit routes will be posted and included in user training.





5.4.2.4 Beamline Vacuum System

There will be several vacuum sections, isolated from each other through gate valves. Each vacuum section will be equipped with an ion pump, full-range vacuum gauge, and pump-out port. Each pump-out port will consist of a rectangular, all-metal valve (CF40) to rough down the vacuum using a pump cart, and a valved connection to dry nitrogen supply to vent the section. The monochromator will also be equipped with a CF63 gate valve where an interlock-protected magnetic bearing (oil-free) turbo molecular pump will be permanently mounted.

The white beam section of the beamline will be bakeable in order to achieve a vacuum pressure below 10^{-9} torr. The monochromatic section is designed to achieve a base pressure in the 10^{-8} torr range or better.

Besides the large flanges of the monochromator doors (that are Viton sealed) and the flanges of the mirror vessels (metal-sealed for M1 and Viton-sealed for M2), all flanges will be bakeable metal-sealed Conflat standard. There will be no mechanical water-vacuum seals anywhere in this design.

The following diagram shows a general schematic overview of the vacuum and its protection system (note that the location of the water-cooled Be window has been changed from this layout):

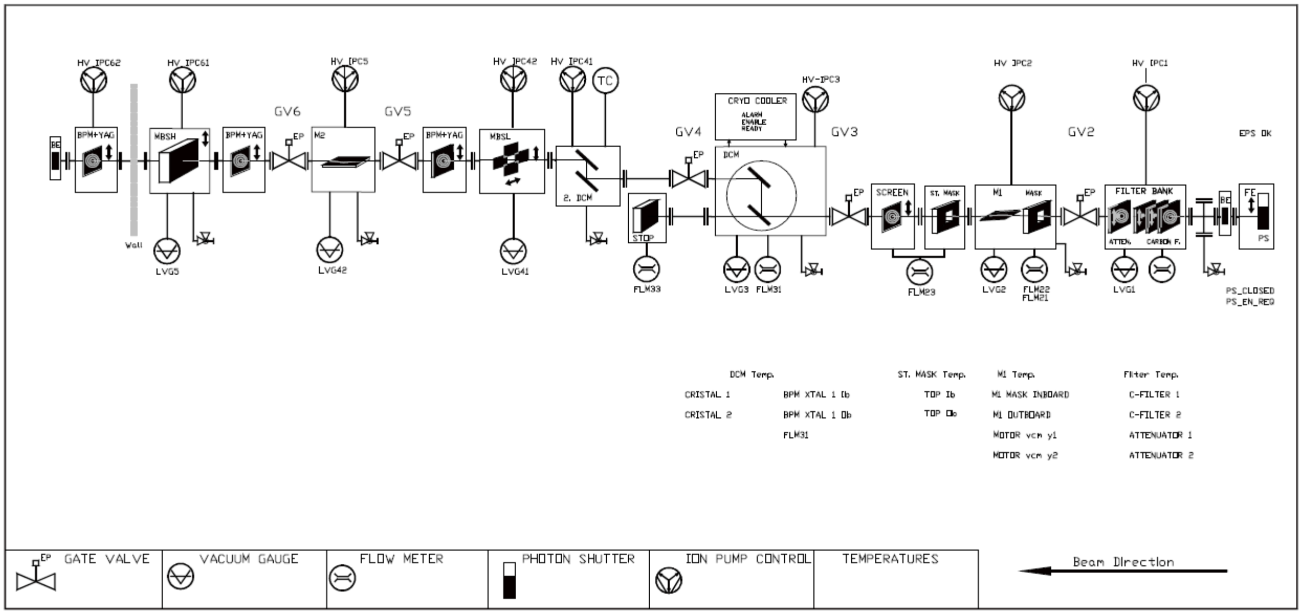


Figure 5.4.2.4

Vacuum control and Equipment Protection System sensors (pressure, temperature, water flow). All components (including valves, shutters and pumps) also have indicators of status or position.

5.4.2.5 Data Acquisition System and Motion Control

See Appendix G for general specifications of an example control system. This document was developed for several Accel components under consideration for this beamline.

5.4.3 Beamline Components

As tabulated in 5.4.2, the following items comprise the major beamline components necessary to deliver controlled monochromatic beam to the experimental hutches.

5.4.3.1 White Beam Slits

While these slits are located in the Front End, they will be under beamline control. Please refer to the appropriate chapter for detailed front-end specifications. In general, these slits will be water-cooled, accept up to 1.0 mrad horizontal by 0.15 mrad vertical, and have resolution and repeatability of better than 10 microns. The 4 independent blades will be tungsten-edged for the high energy range being used, and each capable of closing 2 mm past the centerline. Heat load modeling will need to be conducted in order to ensure that the blades can tolerate the required absorbed power. Accommodations will be made to include future blades to also define the second canted beam.

5.4.3.2 Pre Filter, Be window, Attenuators

Hard x-ray beamlines typically have a beryllium (Be) window to separate the beamline vacuum from the machine vacuum. A Be window also absorbs a significant fraction of the unused low-energy radiation, therefore reducing the overall power delivered to downstream components. However, in a wiggler beamline such as this, calculations of the absorbed power indicate that a Be window in the direct white beam would fail. Due to their high thermal conductivity and mechanical stability, carbon foils are typically used as a protective filter material in front of the Be window.

This beamline design incorporates three components, a set of graphite foil pre-filters to protect the Be window, a Be-window for vacuum isolation, and an additional attenuator package to further manage the power load on the optical components. The C pre-filter and Be window combine to absorb the lowest-energy radiation, and effectively establish the low-energy limit of the beamline's range.

The approach described here is to design a filter assembly that can be safely used whenever required in order to reduce the power levels on the optical components. Reduced power will improve the performance and stability of the white beam optical components such as collimating mirror and monochromator. Considering the high heat load which is produced by the wiggler the carbon filter unit is an essential component to maintain sustainable power levels down the beamline.

When considering the heat load absorbed by the most upstream filter, a standard water-cooled graphite filter is not able to cope with the high heat load. From experience at other facilities using similar powerful sources, there are two possible solutions:

A) Thin C foils which are only radiation cooled. In that case the foils become extremely hot (up to 1500°C or higher). This system is in place at NSLS X25, formerly a wiggler but now an undulator beamline. A stack of radiative cooled foils successfully withstand the power load (3.4 W/mm^2). They use seven foils from 5 µm up to 51 µm in thickness. After a few years operation, the foils do not show any visible damage. In addition it has been verified that the temperature of the foils is somewhat lower than calculated. This system has proven to work reliably up to temperatures over 1000°C.

B) High thermal conductivity Highly Oriented Pyrolytic Graphite (HOPG) foils, mounted in a water-cooled filter frame. This system is employed at ALS Beamline 5.0. HOPG is clamped between two water cooled copper frames and absorbs 10 W/mm^2 (250 µm). The contact pressure is finely adjusted by springs. Temperatures up to 1000 K are possible. After more than one year operation, there is no visible damage on the HOPG foil.

We have carefully evaluated the behaviour of specifically the first filter unit for the power load of the damping wiggler source; please refer to Appendix D. Both options, radiation cooled filter elements as well as contact cooled filter, are possible solutions for this beamline. A final careful FEA has to be done using the filter and source parameters for the configurations needed.

Be window and pre-filter

This component is very similar to the set-up used at different beamlines at BNL. It consists of a water cooled frame which holds the different filters. The radiation-cooled foil is hold in a Tantalum frame, since it must withstand the extremely high contact temperature, and will turn hot itself. The power then is dissipated into the surrounding environment and is absorbed by a water cooled copper surface positioned around the Tantalum frame. The Tantalum frames, together with the carbon foils, are placed in a water-cooled cartridge made of OFHC copper. The cartridge is held and mounted on a DN40CF flange. The assembly is mounted to a DN100CF base flange and fits in a DN100CF standard cross. PT100 temperature sensors (e.g. two sensors) will be installed in the copper cartridge and monitored by the control system. If any PT100 sensor exceeds a temperature limit the beam shutter will be closed. The PT100 sensors will be connected to an electrical feedthrough, the water pipe (SF-copper, Ø8x1) will be brazed on the cartridge and on a DN40CF flange avoiding water-to-vacuum joints.

A possible filter combination could be as shown:

Table 5.4.3.2 Possible pre-filter combination.

Filter no.	Filter thickness [µm]	Absorbed power [W]	Absorbed power density [W/mm²]
1	5	222	1.6
2	5	96	0.7
3	5	72	0.5
4	25	240	1.8
5	25	162	1.2
6	50	237	1.8
7	50	180	1.4
8	100	277	2.1
9	135	283	2.2
10	300	457	3.6
11	300	337	2.6

Attenuator Unit: This unit consists of a number of different filter setups, that allow tailoring the power load on the optical components to the right level of operation for each particular operational mode of the beamline. The design foreseen for your beamline is based on the one we have realized recently for other high heat load beamlines and is described in the following paragraphs. Currently, we assume that for the Damping wiggler beamlines it might be reasonable to work with two pneumatically driven attenuator units and two or even three motorized filter banks with different filters mounted.

The latest ACCEL design is based on a series of three water-cooled filters mounted on three pneumatic drives. These three carbon foils are of Annealed Pyrolytic Graphite (APG). These APG foils have a similar thermal behaviour than HOPG and are available down to thicknesses of 50µm. The foils are clamped to copper frames where the thermal contact is improved by (i) polishing the surface of the copper within the contact area and (ii) using springs that apply a well defined and well distributed contact pressure. The thermal conductivity of APG is similar to diamond. Due to its excellent mechanical properties and together with the advanced cooling each foil is designed to remove about 500 W. The power density on the first foil presents the most challenge and therefore limits the maximum thickness of the foil, while heat conduction as well as physical integrity under thermal stress define the minimum thickness of the first foil. The APG foil used in the existing system is 125 µm thick. This thickness presents a good balance between the transmission at lower energies and the still very good stability of the foil. The second foil can only be inserted in the beam when the first foil is already in.

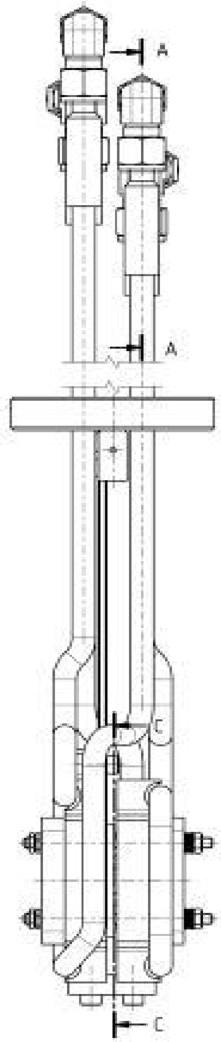


Figure 5.4.3.2a Directly cooled high power filter set-up, which can be pneumatically driven.

The fourth filter drive is motorized and consist of a cooled frame with five positions that can be used for graphite foils (Pyrolytic graphite) of different thickness to be used to further attenuate the beam. This filter design avoids water-to-vacuum joints. Water pipes are brazed to the each cooled copper frame and to the vacuum feedthrough, where edge-welded bellows permit the translation of the frames with the filters.

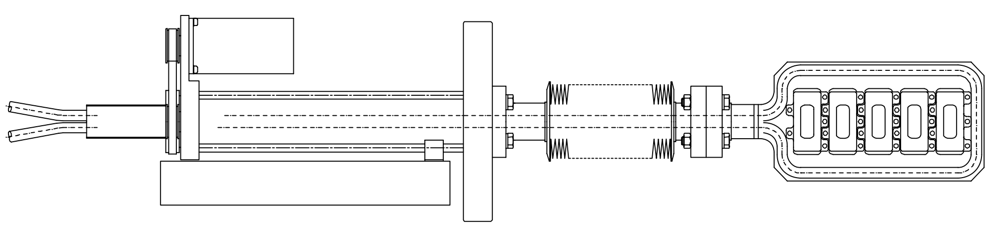


Figure 5.4.3.2b Example motorized water-cooled attenuator assembly

Please note that the filter bank must be equipped with a special protection, as the 2nd filter foil can only be introduced into the beam, when the 1st foil is already in the beam. Similarly the standard motorized filters units can only be put into the beam when both pneumatic filters are in the beam.

As an option the two first PG foils could be replaced by a 250 µm HOPG foil based on the above mentioned ALS design. Using such a design, the maximum temperature is reduced significantly and therefore we do not expect any influence by infrared radiation on the downstream optical components. On the other hand the use of HOPG will significantly reduce the flux at the sample at low energy.

The vessel of the filter assembly is equipped with view ports which permit visual inspection.

General*:*

UHV rated

Two pneumatic driven actuators:

- APG foils clamped on a double sided cooled copper frame
- 1000 W cooling capacity for each actuator
- position monitored by limit switches

Two motorized actuators:

- PG foils and metal foils clamped on a cooled copper frame
- 500 W cooling capacity
- position monitored by limit switches

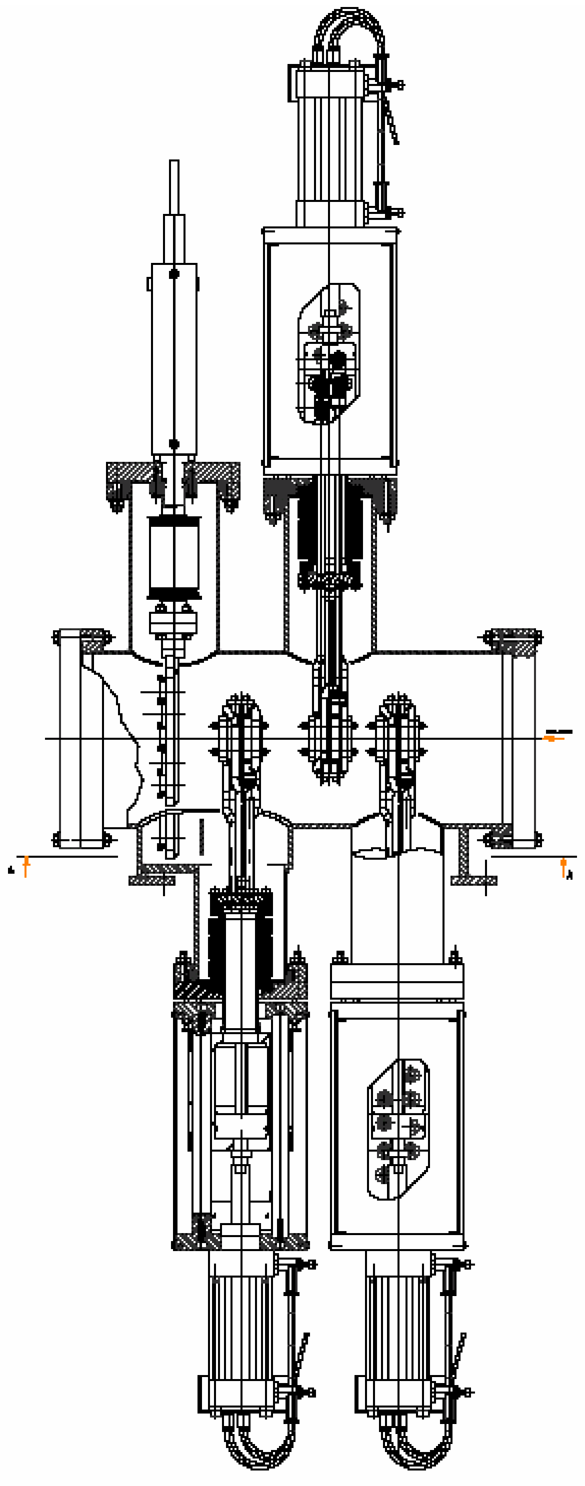


Figure 5.4.3.2c Example high heat load filter system as installed at the XAS beamline at ASP.

5.4.3.3 Bremsstrahlung Collimator

The collimator will absorb high-energy Bremsstrahlung radiation originating inside the ring and defines the extent of the remaining Bremsstrahlung extremal rays on which the dimensions of the downstream white-beam stop must be defined. The outer dimensions and dimensions of the aperture will be determined through ray tracing. Appendix B shows the initial Bremsstrahlung ray-tracing, using two different types of collimators and stops. A lead Bremsstrahlung collimator basically consists of a rectangular vacuum pipe mounted inside a block of lead. Because of the vacuum pipe, some tolerances and the necessary clearance, the openings in such lead collimators are rather large. An in-vacuum tungsten collimator allows a much smaller beam opening and therefore much better collimation. This is the recommended option. In addition, the tungsten collimator is more compact; the absorption length for lead is determined to be 300 mm along the beam direction; for tungsten, 200 mm. The collimator assembly will sit on an adjustable support that will permit translation and tilting for precise alignment, then will subsequently be locked in place as part of the safety system.

5.4.3.4 White beam Mirror

A directly cooled Si mirror offers several advantages. This technology is in use at beamlines at NSLS and the ALS, and mirrors with the required quality characteristics can be procured. As long as the bond of such a mirror is not directly exposed to the x-ray beam, the stability of the frit-bonding seems not to be a problem, even after several years of use. This being the case, and to protect the end of the mirror from inadvertent exposure to direct beam, a water-cooled protection mask will be employed.

An additional feature required for such a high-intensity beam will be a shielding shroud over the sides and above the face of the mirror, to keep scattered radiation from heating the enclosure and positioning/bending mechanism components.

5.4.3.5 Steering Mask

A protective water-cooled steering mask just upstream of the monoichromator will serve to protect the DCM interior and any uncooled downstream surface from being hit by a miss-steered direct beam. Furthermore, a steering mask would serve as a conductance-limiting aperture for the vacuum performance between these two sections. To accommodate all operation modes of the beamline, this mask must sit on a vertical stage and will be equipped with edge-welded bellows to allow for the necessary translations.

The mask will have water channels, I.D. 8 mm, with copper tubes brazed to it (no water-to- vacuum joints are used in this design). The material will be Glidcop and the impinging area will have a suitable slope so it can withstand the high thermal stress that will be produced under worst-case conditions (i.e. full beam).

Specifications

Material	Glidcop
Total power capacity	≤ 6 kW
Maximum power density on surface	12 W/mm^2
Angle of cooled surface	Approx. 10°
Length of water cooled body	Approx 150 mm
Water flow maximum	6 l/min.
max. pressure	120 psi
Aperture size	1 mrad (h) x 0.15 mrad (v)

5.4.3.6 High-Heatload Monochromator

This component is the critical item of the wiggler-based XAS beamline. It must be able to handle the large heat load (perhaps as high as 2.5 kW) while still maintaining the requisite stability and optical quality.

Crystal design. As discussed in Appendix D, there are two ways to cool the crystals, either by direct or by indirect cooling. The monochromator designed by Accel is typically equipped with indirectly cooled crystals. This cooling method is certainly more robust and more reliable in terms of leak tightness, but is less efficient for high heat loads. In such a case, the crystal set-up must be redesigned to accommodate direct-cooled crystals.

In-house monochromator development and design is based on the “hockey-puck” crystal geometry, and this will be pursued within the NSLS-II Project R&D program. This is the preferred option, as it is proven technology (on a smaller scale) and appears to be able to effect more efficient cooling.

Cryocooler. For cooling the crystals to LN_2-temperatures, a closed loop LN_2 cryo-system will be used. Originally developed for the ESRF, the Accel Cryotherm model is most highly regarded. Its control system is based on a PLC with an interface to a standard PC to manage the temperature controls system via EPICS.

5.4.3.7 High-Energy-Resolution Monitor

This device will be based on a high-precision version of a standard DCM. Choice of crystals, since cooling is not an issue, will be made on the basis of optimal energy range, bandpass, available crystal quality, and testing work to be performed at the NSLS.

5.4.3.8 Monochromatic Focusing Mirror

For the VFM, a configuration of two sagittal cylinders with different sagittal radii in the substrate is technically feasible; the polishing of such a mirror will be a very challenging process, but a vendor has accomplished several similar mirrors already.

The following table summarizes possible specifications for the two mirror substrates and benders, based on similar existing beamlines.

	M1	M2
Mirror Substrate	Monocrystalline Silicon	Fused Silica or ULE or Zerodur
Direction of Reflection	Downwards	Upwards
Shape	FLAT cylindrically bent to tangential cylinder	Double sagittal cylinder cylindrically bent to torus + central flat
Tangential Operational Bending Radius Range	5.0 km - flat (> 40 km)	3.5 km - flat (> 40 km)
Sagittal Bending Radius	flat (> 1 km)	(i) $R_{sag}1$= tbd mm (Pt coated) (ii) $R_{sag}2$= tbd mm (uncoated)
Substrate Length	approx. 1400 mm	
Substrate Width	~ 120 mm	~ 135 mm
Substrate Thickness	60 to 70 mm	60 to 70 mm
Optical Active Surface: Length	1200 mm	
Width	2 x 35 mm (Si & Pt)	2 x 35 mm (cylinders) polished width ~ 45 mm
Slope Error: Sagittal	< 15 µrad rms for M1 < 25 µrad rms for M2 (best effort < 15 µrad rms)	
Tangential	< 2.5 µrad rms on 1200 mm (Best effort: < 2 µrad rms)	
Micro Roughness	< 3 Å rms; best effort < 2 Å rms	
Coating	Pt> 600 Å; Cr underlayer	Pt> 600 Å; Cr underlayer Bare central flat

Cooling	YES, directly cooled	No

5.4.3.9 Beam Monitoring Elements

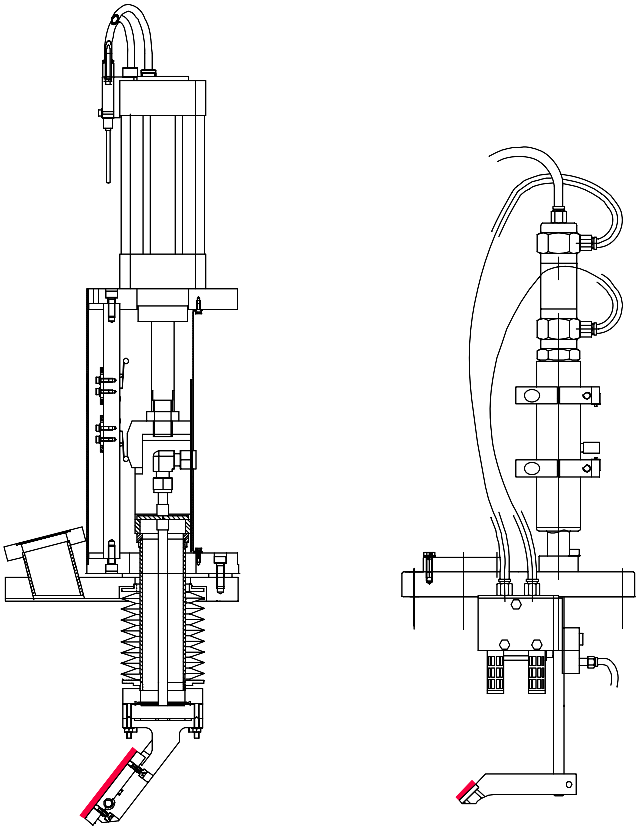
a) Water-cooled white beam CVD fluorescence screen: The device consists of a retractable water-cooled CVD diamond foil, acting as x-ray screen, mounted to a pneumatic drive; the fluorescent effect is based on the residual doping with nitrogen atoms. The diamond screen is transparent; i.e., beam detecting further downstream is possible. The assembly is mounted to a DN100 CF cross with one view port permitting a side view onto the screen. The pneumatic drive is equipped with limit switches. The vacuum feedthrough is made of edge-welded bellows. The water lines are brazed to the screen support to avoid vacuum-to-water joints. The foil is clamped to the cooled support.

The projection of the beam onto the 45° inclined foil will be monitored with a CCD camera. This system is capable of staying in the beam. However, because of the resulting absorption at photon energies below 10 keV, the screen should be withdrawn when not in use. Moreover, to increase the lifetime of the foil and prevent overexposure of the camera, this screen should only be used at reduced power levels—i.e., in combination with some of the carbon filters.

This screen has been installed at the high power wiggler beamline at the Australian Synchrotron Project.

General	
Screen material	CVD Diamond foil less than 0.125 mm thick
Screen slope	45°
Field of view	40 mm (h) x 20 mm (v)

These fluorescence screen monitors typically are mounted to a pneumatic drive via a vacuum feedthrough on a conflat flange. The water-cooled monitor is inserted in the beam by a stepper motor. Modeling of heat load and power absorption will be required. The flange is also equipped with a view port for the camera that provides side view of the screen. Examples can be seen below, for a water-cooled device and an uncooled device.



General:	Screen 1	Screen 2
Beam Size (hor. x vert.)	20mm x 15mm	≤ 40mm x ≤ 20mm
Screen Size (fluorescent region; hor. x vert.)	20mm x 20mm	50mm x 60mm
Water Cooling	yes	no

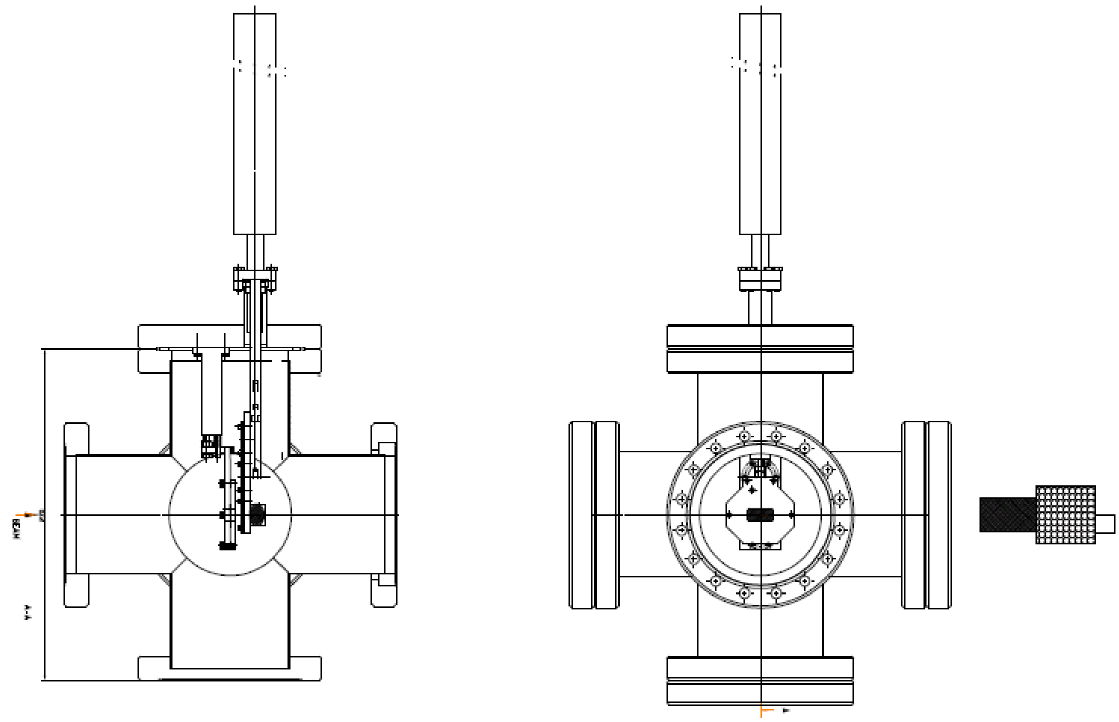
b) Quadrant diode beam position monitor (4-diode BPM): A standard monitoring device for monochromatic beams of large size is a quadrant type detector that monitors the fluorescence yield of a target foil. Beam position information is derived from the intensity ratio of one pair of diodes. The device is rated for UHV and consists of a diode holder (holding four diodes) and a fluorescence foil holder.

The four detecting silicon diodes are mounted to a vertical stage that permits a vertical positioning of the diodes which is needed to operate at different beam heights.

There are mounts for two foils, which can be of different kind, or be the same. Typically, thin chromium and copper foils are used, but silver foils might be used as well. Chromium and copper have been working up to photon energies of around 20 keV. There is an option to mount a YAG crystal underneath this foil holder. Than this YAG can be used to visualize the beam by means of a camera looking from the side onto a prism which is positioned behind the YAG.

The assembly is designed such that it mounts to a CF100 flange and fits a standard size DN100 CF cross.

The detection electronics for the diodes will consist of an integrated four-channel picoammeter.



UHV rated BPM with diodes and fluorescent foil and screen

General:

Energy range	5 KeV to 25 KeV
Foils	0.5 micron Cr, Cu, or Ag
Photo current	~2 µA @ 10^{13} ph/s

Translation stages:

Stepper motor drives	linear actuator with 2-phase stepper motor and limits
Maximum stroke diodes	50 mm
Maximum stroke foils	100 mm

c) Endstation X-ray beam monitor and camera: This type of x-ray beam monitor is a commercially available visualization system for x-rays, or can be contrived with minimal effort. Such a system provides a field of view large enough to study the beam size, beam profile and the beam position stability of a focused beam in the endstation (at atmosphere), but must be removed from the beam path during data collection.

Feedback systems: electronic beam position monitors (type b above) will be strategically incorporated into at least two feedback systems.

The first of these will monitor beam position at the entrance slits for the high energy resolution monochromator. When this monochromator is in use, the BPM will be incorporated into a synchronization control loop that maintains tune between the two monochromators. While it is conservatively expected that the high-resolution mode of operation will preclude use of continuous-scan data collection (so as to allow for stabilization at each data point), this would be a desirable feature and is worth pursuing as part of final design.

The second feedback system will employ BPMs at the upstream end of each experimental hutch. These will be utilized (being highly sensitive due to the long lever arm) to control fine monochromator (and possibly mirror) adjustments to maintain beam position at the sample. This type of feedback is in place at a number of existing beamlines. While the NSLS-II source will have exemplary stability, feedback systems are likely to still be required to eliminate instabilities created by the beamline optics and their mechanisms.

5.4.3.10 Monochromatic slits and photon shutter

A standard design for in-vacuum monochromatic slits and for photon shutter will be developed for the NSLS-II experimental facilities. These designs will be used for all applicable beamlines.

5.4.4 Instruments

Instrumentation for the XAS beamline will consist of two endstations providing advanced capabilities to address cutting-edge challenges in local-scale physical, chemical, and electronic structures. The guiding philosophy for design of these endstations is to provide the widest range of high-quality XAS tools to fit current scientific needs. It is also intended to ensure a solid infrastructure that remains versatile and adaptable to both expected and unforeseen experimental needs of 2013 and forward.

A central aspect of this is detector selection. Preliminary design calls for a suite of detectors to answer the basic needs of various methods of measurement. It is expected that more detailed specifications for these detectors will be defined after further interactions with the community and through the Beamline Advisory Team process. Further technical developments are likely in the next few years; incorporation of these into final beamline design will enhance capabilities. Also, beamline design will explicitly include provisions to accommodate the subsequent addition of detectors beyond current project scope.

The following sections describe Endstation 2, the main experimental hutch for bulk XAS, and Endstation 1, the smaller upstream hutch for microbeam XAS. At initial operations, these endstations will operate from a single control station and share a single source and set of beamline optics. For efficient experimental setup, users will be able to access Endstation 2 while Endstation 1 is in use, but not vice versa. Eventually (but beyond Project scope), each endstation will operate independently, thus doubling capacity. In that arrangement, Endstation 2 will be served by the outboard of the two canted sources; Endstation 1 the inboard source.

This combination of endstations will provide a range of spot size from 5 x 40 mm to 1 x 1 micron, to cover the range of scale needed for the planned research programs. It will also complement efforts at the nanoprobe Project beamline which, combined with the planned soft x-ray spectromicroscopy facility, will pursue finer spatial resolutions.

Instrument control and data collection will include all motor drive channels, ion chamber and detector outputs, sample image capture, temperature, sample-cell and illumination control. Data collection software will include standard XAS scanning parameters, sample mapping functions (micro and macro beam), programmed sample locations and experimental control, and will also incorporate XRD data and image capture from sample cameras.

5.4.4.1 Endstation 2

This endstation will serve bulk applications at three sample positions. Each will have its own set of components to minimize downtime and effort for setting up or reconfiguring each experiment.

Sample position 1: The scientific mission of this classic benchtop style setup will be for *in-situ* analyses, grazing-incidence surface measurements, high concentration and fast-scanning applications, and use of high energy resolution fluorescence detection or simultaneous XRD.

Flow-through cells are important for *in-situ* measurement of catalysts at controlled temperature and gas flow, and for chemical (e.g. ion exchange), environmental (e.g. contaminant adsorption), and geochemical (mineral-water reactions) solution flow experiments. Flow-through cells are also important for some biological materials where a static solution may suffer radiation damage. Other *in-situ* sample cells include those for electrochemical and fuel-cell research, laser or small magnet units, or high pressure measurements.

Surface XAS is an important measurement technique that is ideally suited for the highly-collimated high-brightness beam provided by this source. Measurements are typically made in total reflection, using either the reflected beam (analogous to transmission through a thin sample) or fluorescence. The high flux at this beamline will greatly improve the ability to measure very dilute (sub-monolayer) surface species, and the higher energy range will create additional opportunities to do so at buried interfaces or under solutions.

An important mode of data collection will be continuous-scan (slew) mode, where the monochromator is kept in continuous motion while measurements are made. Scans in this mode will take only one to three minutes each, resulting in very high throughput for more routine measurements. Time-resolved studies on this scale are thus possible.

Fluorescence detector-limited applications involve samples with low target signal but high background, or those with strong interference by overlapping fluorescence peaks (in a solid-state detector spectrum). These benefit from use of a high energy resolution or energy-selective detector, but such detectors typically accept only a very small solid angle of fluorescence from the sample. The very high flux and excellent macro focus of this beamline will make energy-selective detectors much more attractive. The wavelength-dispersive spectrometer included in this preliminary design will be useful on samples where conventional detection is unsuccessful.

Simultaneous XRD is of great interest to many of the catalysis and in-situ research programs, as well as for bulk characterization of samples in general.

Components at sample position 1 include the following (all within project scope):

- Optical table, fixed height
- Separate table for detector(s) at 90° to beam
- Beam-defining x-y slits
- Ionization chambers for I-zero, transmitted and reference measurements
- Optical rail
- Sample stage with x, y, z, rotation, and horizontal and vertical tilts
- LN2-cooled sample mount
- Stage adapter for flow-through cells
- sample temperature control
- Camera on sample position (45° to beam), with illuminator
- Compressed-helium cryostat
- Fluorescence detectors: Lytle, PIPS, and solid-state Si (e.g. the Vortex type)
- High energy resolution fluorescence detector, such as a wavelength-dispersive spectrometer
- Area detector for simultaneous XRD, to be placed downstream of sample stage
- Light-weight helium-filled flight tube to deliver beam to sample position 2 when needed

It is expected that each experiment will provide its own sample cells, but the support infrastructure (e.g. gas supply connections, flow control, temperature control and monitoring, etc.) is part of the beamline. The sample stage must have sufficient capacity to support the cryostat or other sample cell. Tilt geometries are needed for both horizontal and vertical polarization-dependent grazing-incidence measurements. Detectors will be positioned with appropriate stages. The area detector will be moved back, out of the beam path, when not in use, and may also be used in Endstation 1.

Sample position 2: The scientific mission of this station will be for low-concentration samples, hazardous radioactive or nanomaterials, and samples requiring clean environment or a controlled atmosphere. The “multi-use enclosure” employed here is designed to serve the needs of such samples. Based on designs in the planning stage at two NSLS beamlines, this enclosure would serve as a glove box, open up to operate as a fume hood, or simply be open to atmosphere. Sealed glove-box mode would be necessary for air-sensitive samples or those requiring a specific atmosphere. Examples include a variety of catalysts, redox-sensitive environmental samples, anaerobic biological samples, and atmospheric science applications. Radioactive samples, nanomaterials, and other hazardous materials require containment and often also need ventilation as in a fume hood. The enclosure would operate in that mode with the glove-bearing face opened for access. Policies and requirements as to HEPA filtration and monitoring for these materials will be more clearly defined in conjunction with ES&H personnel as the Project develops. The bottom of the enclosure will be lined with trays made of Teflon or similar materials for ease of cleaning and decontamination. Samples requiring no special care would simply be measured in air.

An important consideration for low-concentration measurements will be the requirement for a clean sample environment. Experience at NSLS X15B with ultra-low (sub-ppm) concentrations demonstrate the need to keep the sample free of dust during analysis, and to eliminate stray scatter from other parts of the hutch. This enclosure will be kept clean. Dust will not be a problem in glove-box mode; for open and fume-hood modes, an air-filtering curtain will be placed across the opening during analysis to reduce dust. As for contamination of the signal by scatter, that will be addressed by applying a collimating cone to the detector snout, and a scatter shield behind the sample.

Components at sample position 2 include the following:

- Support table, fixed height
- Separate table for detector at 90° to beam
- Multi-use enclosure (described above)
- Beam-defining x-y slits
- Ionization chambers for I-zero and transmitted beam measurements
- Sample stage with x, y, z, and rotation motion
- LN2-cooled sample mount
- Camera on sample position (45° to beam), with illuminator
- Multi-element Ge fluorescence detector, with a sealed feed-through into the enclosure
- Nose cone collimator and filter holder for detector
- Set of filters (shared)
- Light-weight helium-filled flight tube to deliver beam to sample position 3 when needed

The detector will be retractable and have height adjustment to center on beam. Experiments at this sample position can take advantage of components at position 1 for additional beam monitoring, analysis of reference samples, etc., or may employ a helium-filled flight tube to efficiently traverse position 1.

Sample position 3: The function of this position will be for any large apparatus that needs more room than is available at the benchtop position. Large magnets, catalytic cells, and large-volume high-pressure assemblies can be wheeled into the hutch in this position.

Components at sample position 2 include x-y slits, available channels for motor control, sample monitoring, and detectors, and approximately 2 x 2 m floor space.

Experiments at this sample position can take advantage of components at positions 1 and 2 for additional beam monitoring, analysis of reference samples, etc., or may employ a helium-filled flight tube as described above.

5.4.4.2 Endstation 1

As a complement to the bulk endstation (having a minimum spot size of about 0.2 x 0.2 mm), Endstation 1 will address microbeam applications for XAS, providing a very high flux (estimated 10^{12} ph/sec at 8 keV) in an approximately 1 x 1 micron spot size, and having the important ability to maintain focus and positional stability over a 1000-eV EXAFS scan. Its scientific mission will center on a) relating micron-scale elemental distribution to physical structure and chemical speciation in heterogeneous materials, and b) XAS measurements of small samples. Obtaining local information in heterogeneous samples is important for such examples as measuring micron-scale variations in catalyst systems, identifying reactive particles and local chemical transformations in environmental samples, exploring biogeochemical processes involving microbes, and relating structure and chemical processes in biological samples. This tool will also be useful for studying reaction and transport processes of nanoparticles in industrial, environmental, and biological systems.

The microbeam endstation also meets a critical need in the study of small samples, such as single crystals, small samples for grazing-incidence surface XAS, small-mass samples of atmospheric or marine particulates, and in nuclear forensics. Recent work at NSLS X27A employs orientation-dependent single-crystal XAS to examine site-dependent substitution chemistry. Atmospheric particulates, critical in cloud formation and global climate research, are naturally difficult to obtain in quantity. The field of nuclear forensics is a new application for microbeam XAS, as it becomes more important to be able to identify, on the basis of trace particles, evidence of nuclear materials processing or weapons testing. And in high pressure research, microbeam XAS can be applied to diamond anvil cell experiments.

Simultaneous microbeam XRD and XAS is also an important aspect of this endstation, which will share an area detector with Endstation 1. Planned applications include identification of crystalline phases in heterogeneous samples to further characterize structure-function relationships, and to measure crystalline structural variations during local-scale reactions in, for example, catalytic or ion-exchange materials.

The primary mission of this microprobe will be XAS, as it is expected that other beamlines (at undulator sources) will specialize in x-ray fluorescence and XRD imaging at micron or submicron resolution. The optics and source described here are optimized for the stable energy scans required for XAS.

Components at Endstation 1 include the following:

- Support table, fixed height
- Multi-use enclosure (described above)
- Separate table for detector at 90° to beam
- Beam-defining x-y slits
- Miniature ionization chambers for unfocused and focused I-zero, and transmitted beam measurements
- Kirkpatrick-Baez microfocusing mirror set, within enclosure
- Sample stage with x, y, z, and rotation motions
- LN2-cooled sample mount
- Low- and high-resolution microscope cameras on sample position (45° to beam), with illumination
- UV illumination for sample imaging
- Light-weight helium-filled flight tube to deliver beam to Endstation 2 when needed

The sample stage will have sufficient precision for microbeam applications. Conventional K-B mirrors will be used to focus up to 0.1 x 0.1 mrad of on-axis wiggler beam to a ~1 micron spot. The detector will be retractable and have a positioning stage to center it on beam. The area detector for XRD listed in Endstation 2 may also be used in Endstation 1; in order to accommodate it outside the enclosure a large sealed x-ray transparent window will be installed on the downstream end of the enclosure.

This endstation is separated from the three sample positions in the “bulk” endstation for several practical reasons. First, it is a sufficiently different application of XAS that it will benefit from the distinction and the future opportunity to operate in an independent and optimized manner. Second, it improves efficiency to be able to take beam in the upstream hutch while setting up more elaborate experiments in the other. And third, its requirements for beamline optics (angular acceptance, heat load, and the like) differ from the bulk techniques.

5.5 Preliminary Safety Analysis

This section is concerned with Synchrotron and bremsstrahlung radiation protection.

5.5.1 Beamline Radiation Analysis

Geometrical Synchrotron and Bremsstrahlung Ray-Tracing

Please find the relevant synchrotron as well as bremsstrahlung ray tracing schemes in Appendix A. The results of these drawings were derived based on the following documents:

1. Technical Bulletin 20 of the Advanced Photon Source
2. NSLS II Technical Note no 020, Guidelines for NSLS II beamlines..
3. Document SR_lattice_frontend_longstraight_8_22_07.pdf
4. Comments in e-mail of Sushil Sharma about first aperture within the front end

For the creation of the initial ray-tracing files we used the following parameters:

Source point	center of long straight section
Fixed Aperture Mask (FAPM)	19.8 m
First Pb collimator	20.7 m
Safety shutter position	26.2 m
End of Front End	28.6 m
Bremsstrahlung source	4 m downstream from center of straight section
Bremsstrahlung lateral source dimensions	+35 mm outboard Inboard unknown Vertical ± 12.5 mm

In addition we defined the following parameters and used those in the evaluation:

Synchrotron radiation miss-steer	±2.0 mm
Size of the First Aperture Mask:	2.5 mrad (h) x 0.5 mrad (v)

The horizontal size of the first front end aperture was assumed to be 2.5 mrad to take a practical start. We assume that the size of this aperture will be part of future discussions, since this challenging aperture has to be designed together with both ID beams and the goal is that the Front-End delivers only a fan of 1mrad (h) x 0.15 mrad (v). For our ray-tracings we have assumed that there is a further mask just outside the Shield Wall defining the beam to 1 mrad x 0.15 mrad,

Please see Appendix A for the ray-tracing files which were realized using the above input parameters. Those file can be used for detailed discussions and have to be updated when the detailed parameters of the Front-Ends are available.

Further thoughts are needed on the first collimator. Most likely concerning two ID lines (3.5 mrad apart) this collimator will be a combination of an in-vacuum tungsten together with an outside lead collimator.

As it can be seen in the horizontal schemes the size of the beamline collimator inside the FOE is still reasonable. Most interesting is the result on the vertical ray tracing. As it is typically (and practically) done at other beamlines we have placed the beam defining second aperture at the end of the front end assembly right after the ratchet wall. This allows a rather small vertical opening of the collimator in the FOE and therefore an efficient collimation of the Bremsstrahlung at the beginning of the FOE.

Our target is the smallest possible collimator aperture resulting in the smallest possible beam offset of the monochromator. The conflict arises from the requirement to provide enough absorbing material inside the Bremsstrahlung stop between the Bremsstrahlung extremal and the aperture for the monochromatic beam. In this evaluation we follow the assumption that 11 mm are necessary.

In the ray-tracings we have assumed a vertical opening of the first lead collimator in the FOE of 15mm. Under this assumption one needs at least a DCM offset of 30mm to fulfill the requirements at the beam stop. This should be possible and acceptable. By using an in-vacuum tungsten collimator and optimizing the positions one could probably reduce the vertical offset at the DCM to 25mm. But this needs further detailed evaluations based on the more detailed design of the Front-End.

Enclosures:

There will be three beamline radiation enclosures at the XAS beamline, one first optics enclosure (FOE) and two contiguous experimental hutches. The FOE contains all beamline optics, and will be shielded for white beam and Bremsstrahlung scatter. The FOE with its lead-shielded sides interfaces to the ratchet wall of the storage ring. Following the standard regulations for white beam hutches, the labyrinths to run electricity as well as media and power connections will be located on the roof. The hutch will be long enough (approx 17 m) to accommodate the optics for both the original beamline and the canted beamline to be built later. One double door with sliding panels, large enough to accommodate larger components being moved via fork lift, will provide access to the hutch.

The beam transport between the FOE and the first EH will be a tunnel type (coffin style) transport. Such an enclosure design will provide enough flexibility to accommodate vacuum pipes for both canted beamlines. Shielding interfaces such as guillotines are included.

The FOE will be equipped with an overhead crane (1 metric ton). A summary of the specifications is as follows:

First Optics Enclosure, white beam hutch (FOE)

	Sides 17 m x 3.3 m	Downstream wall 3 m x 3.3 m	Roof 17 m x 2.5 m
Shielding requirements: Lead (mm)	23 mm	50 mm	14 mm

- one extra panel of size 1 m x 1 m x 50 mm at the downstream wall centred at 1400 mm above the floor
- one sliding double door (white beam hutch)
- 10 lockable, hinged chicanes on the roof
- one hutch crane (1 metric ton), on trolley above trace of beam
- one set of guillotine, adjustable shielding around beam pipe on downstream wall
- painted with primer

Shielded Beam Transport (coffin style, base with lid)

- lead shielding of 7 mm thickness
- dimensions: 8 m long, 0.4 m x 0.4 m cross section
- support stands every 2 m with gussets
- painted with primer

First Experimental Enclosure, monochromatic hutch (EH-1)

	Upstream wall 3 m x 3.3 m	Sides 3.5 m x 3.3 m	Downstream wall 3 m x 3.3 m	Roof 3.5 m x 3 m
Shielding requirements: Lead (mm)	6 mm	6 mm	6 mm	5 mm

- one sliding door
- three lockable, hinged chicanes on the roof, three on the sides
- one set of guillotine, adjustable shielding around beam pipe on upstream wall
- painted with primer

Second Experimental Enclosure, monochromatic hutch (EH-2)

	Upstream wall 3 m x 3.3 m	Sides 7 m x 3.3 m	Downstream wall 3 m x 3.3 m	Roof 7 m x 3 m

Shielding requirements: Lead (mm)	6 mm	6 mm	6 mm	5 mm

- one sliding double door
- one sliding single door
- seven lockable, hinged chicanes on the roof, four on the sides
- one set of guillotine, adjustable shielding around beam pipe on upstream wall
- painted with primer

Provision will be made to run cable trays and utilities inside and outside the hutches.

5.5.2 Personnel Safety System

This will be a beamline-specific application of the NSLS-II standard PSS, and will encompass hutch door interlocks, beam-stops, photon and safety beam shutters. Specific components are described elsewhere, and this beamline will utilize one touch-screen panel, one shutter control, emergency-stop and personnel-check provision for all three enclosures, and door interlocks for 1 door on FOE, two on EH1 and two on EH2.

5.5.3 Equipment Protection System

This will be a beamline-specific application of the NSLS-II standard EPS, and will include vacuum, temperature, water flow, beam status, and cryo-cooler status. Specific components are described elsewhere.

5.6 Additional Requirements Imposed on the Conventional Facilities

Endstation multi-use enclosures will need to interface with common exhaust system planned as part of facility.