XAS Endstation at the X-ray Footprinting & Spectroscopy (XFP) Beamline

IRR Functional Description

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1 INTRODUCTION

1.1 Primary Research Capabilities

The new X-ray Absorption Spectroscopy (XAS) endstation at the X-ray Footprinting & Spectroscopy (XFP, 17-BM) beamline, operated by the Case Western Reserve University Center for Synchrotron Biosciences (CWRU-CSB) in partnership with NSLS-II, will provide XAS capabilities to the NSLS-II structural biology community. It will serve a national and international community of biologists, biochemists, chemists, and environmental scientists with interests in the role of metal centers in biological and environmental processes.

This new endstation will enable bulk XAS investigations of metal sites in dilute (sub-millimolar in absorber) biological and environmentally relevant samples, providing direct insight into metal oxidation state and electronic symmetry through X-ray Absorption Near Edge Spectroscopy (XANES) as well as local metal site structure via Extended X-ray Absorption Fine Structure (EXAFS), for metal centers in metalloenzymes and biomimetic homogenous catalysts. This will enable, for instance, determination of metal site structure in metastable reactive intermediates trapped during enzyme catalytic cycles that are rarely amenable to structural determination via X-ray diffraction techniques, allowing scientific insight into how the changes at the enzyme metal center influence catalysis. These scientific capabilities will be enabled through deployment of endstation equipment optimized for biological XAS experiments, including a low-temperature helium Displex-type cryostat and multi-element germanium fluorescence detector arrays, as well as standard XAS equipment such as ionization chambers, attenuators, slits, and motorized alignment stages.

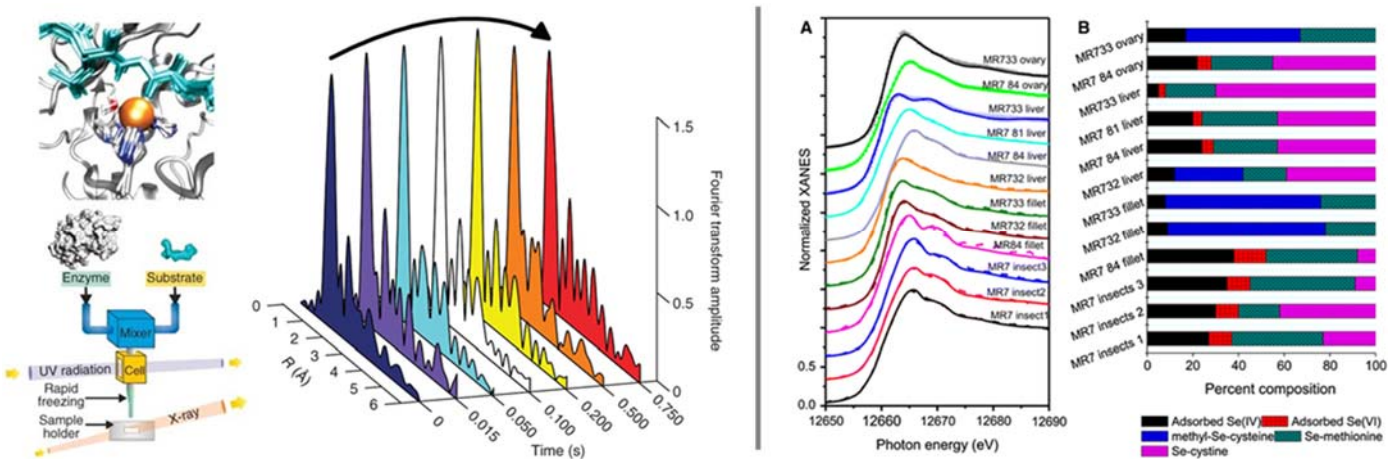

Figure 1. (left) RFQ-XAS of the Zn(II) site in a human matrix metalloproteinase during enzyme turnover. (right) Speciation of the reproductive toxin selenium in insects and fish exposed to coal-mining effluent, determined from bulk selenium K-edge XAS.

1.2 Beamline Staff

Lead Beamline Scientist	Erik Farquhar	
Authorized Beamline Staff	Jennifer Bohon	Beamline Scientist (footprinting)
	Michael Sullivan	Technical Support Staff
	Donald Abel	Technical Support Staff

2 XAS ENDSTATION DESIGN AND COMPONENTS

2.1 XAS Endstation Performance Goals

Table 1 summarizes the projected design performance for the XAS endstation at 17-BM. This endstation builds on existing infrastructure installed at the 17-BM beamline as part of the initial beamline construction project, summarized in the XFP Beamline IRR Functional Description (PS-C-XFD-XFP-PLN-001). Notably, the beamline is equipped with a single optic consisting of a Rh-coated toroidal mirror at a fixed 4.2 mrad angle in the 17-BM front end, which provides user-adjustable collimation and focusing of the beam from the three-pole wiggler (3PW) source at multiple sample locations within the 17-BM-A experimental hutch.

One goal of the XAS endstation is to provide a monochromator that will, in combination with optimization of the 17-BM front-end mirror configuration and horizontal acceptance from the 3PW source, provide an optimal combination of monochromatic photon flux and resolution over an energy range of 4.5-16 keV, encompassing the K-edges of all first row transition metals and the L-edges of the lanthanides and third-row elements. Monochromatic photons are delivered to an experimental table housing cryogenic sample environments and detectors optimized for biological XAS.

Table 1. Designed Performance of the XAS Endstation at 17-BM

Parameter	Specification/Description
Energy Range	4500 eV to 16000 eV (defined by Rh-coated FE mirror at 4.2 mrad)
Monochromator	Channel-cut Si(111) water-cooled double crystal monochromator
Expected Source Acceptance	1.0 mrad x 0.33 mrad (VxH)
Beam size at sample (FWHM)	4 mm (V) x 6 mm (H) (collimated, unfocused)
Flux at sample at 500 mA storage ring current	7×10^{11} ph./sec at 10 keV (1x5mm (VxH) aperture).
Sample enviroment	He Displex-type vacuum cryostat (6.5 – 300 K), mounted on three-axis motion stage
Detector system	Ionization chambers, multi-element Ge fluorescence detector

2.2 XAS Endstation Layout

Figure 2 depicts the layout of the new XAS endstation at 17-BM. Briefly, the new endstation consists of a UHV chamber repurposed from NSLS beamline X6A comprising water-cooled pre-mono slits (4 independent blades, with X and Y pairs), a water-cooled channel cut Si(111)XAS monochromator (3.0 mm crystal offset, Ga-In eutectic for heat transfer), a water-cooled pink-beam absorber coated with phosphor to act as a diagnostic, and a water-cooled beryllium exit window. The UHV chamber is surveyed into place and aligned such that the center of rotation of the XAS monochromator first crystal is +32.45 m from the 3PW source and +155 mm above the 1400 mm nominal beam height. This device delivers monochromatic X-rays to a motorized lift table containing independent x-y-z motion stages for the sample and x-y stages for the detector. The sample stage houses a He Displex-type cryostat (6.5-300K with temperature control) as standard beamline equipment, which can be removed for other user-supplied sample environments. The detector stage houses a permanently installed multi-element germanium fluorescence detector, which is kept cold using a liquid nitrogen supply line in the experimental hutch. Finally, ancillary supporting instrumentation such as ionization chambers, monochromatic slits, and attenuators are installed on optical rails attached to the lift table.

This endstation builds on existing infrastructure documented in the XFP Beamline IRR Functional Description (PS-C-XFD-XFP-PLN-001), including two experimental endstations in the 17-BM-A enclosure designated as the High-Dose Footprinting Endstation (ES:1), located 28.0 m from the 3PW source, and the High-Throughput

Footprinting Endstation (ES:2), located 30.0 m from the 3PW source. For experiments at ES:1 and ES:2 the beamline UHV environment terminates in a diamond exit window at the appropriate sample position, while the XAS monochromator chamber remains isolated and under vacuum. For experiments at the XAS endstation, UHV transport pipe is installed to the XAS monochromator, with the XAS monochromator gate valve on the upstream end of the monochromator chamber separating chamber vacuum from upstream beamline components, including the front-end mirror.

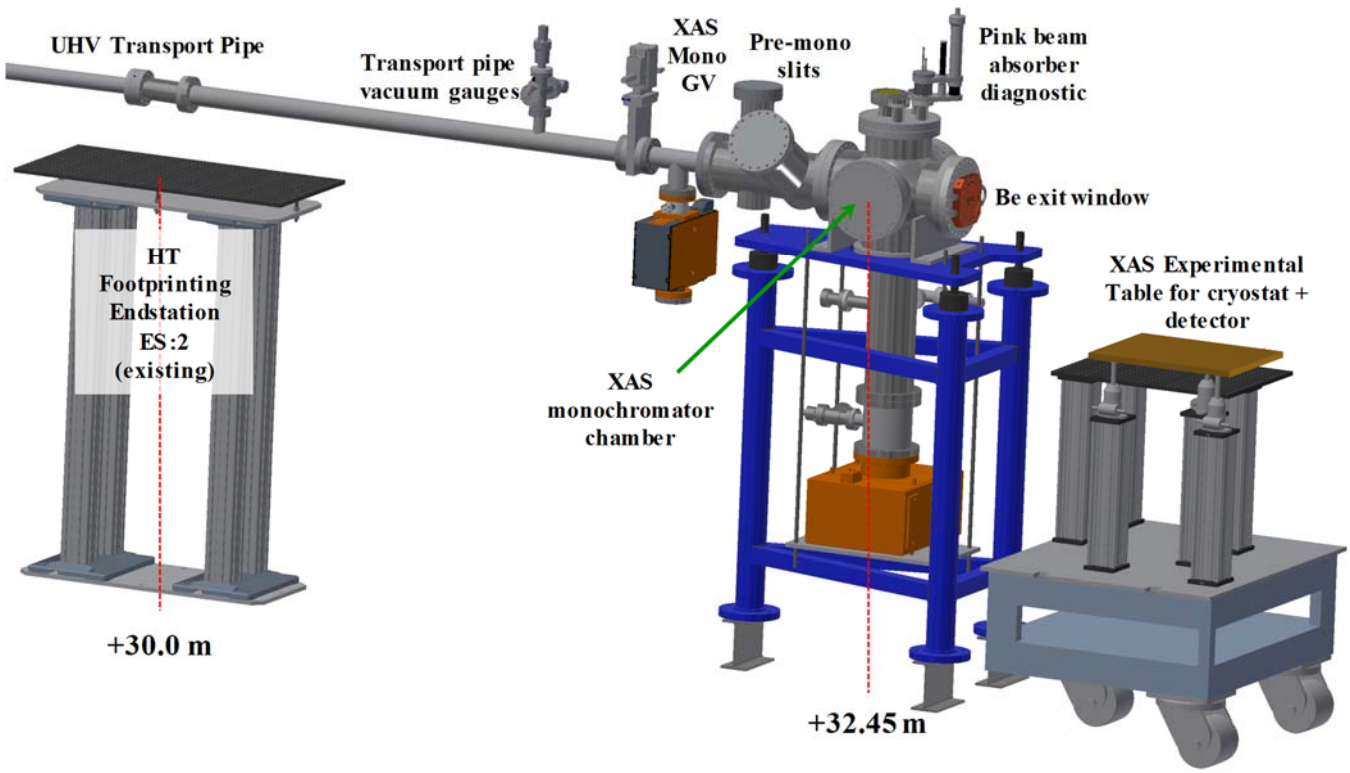

Figure 2. The layout of the XAS endstation at 17-BM.

3 BEAMLINE SAFETY

3.1 Radiation Shielding

Radiation shielding is as specified in the XFP Beamline IRR Functional Description (PS-C-XFD-XFP-PLN-001) and follows released shielding guidelines.[1] The 17-BM-A first optical enclosure continues to also function as the beamline experimental hutch.

3.2 Radiation Safety Components & Radiation Management

Beamline radiation safety components and radiation management are as described in the XFP Beamline IRR Functional Description (PS-C-XFD-XFP-PLN-001) and documented in the NSLS-II Beamline 17-BM Radiation Safety Component Checklist (PS-R-XFD-CHK-012).

The impact of added XAS endstation components in the form of new scatter sources (slits, monochromator, pink beam absorber diagnostic) was assessed through FLUKA simulations, as documented in 17BM Beamline Shielding Analysis Revisited with the Addition of the XAS Experimental End Station (NSLSII-ESH-TN-281). These simulations show that radiation dose rates continue to meet NSLS-II radiation dose and shielding design criteria, and thus no changes to as-built beamline shielding or radiation safety components were required.

3.3 Oxygen Sensor

The 17-BM-A hutch is outfitted with an oxygen sensor inside the hutch, which has been certified in accordance with the Beamline Enclosures and LOB Receiving Area Cryogen Fill Stations ODH Monitoring and Alarm System Certification and Inspection Procedure (NSLSII-ESH-PRC-028). This system will mitigate against hazards arising from a liquid nitrogen fill line in the hutch enclosure that will supply the integral 15L dewar of a cryo-cooled germanium detector, in addition to the hazards previously identified in the XFP Beamline IRR Functional Description (PS-C-XFD-XFP-PLN-001).

3.4 Personnel Protection System (PPS)

The PPS system functions as specified in the XFP Beamline IRR Functional Description (PS-C-XFD-XFP-PLN-001). The search and secure path was reviewed as part of the XAS endstation installation process, and was adjusted in response to changes in accessibility from the new eqipment, as documented in the 17-BM Beamline Search Sequence Diagram.

3.5 Hazard Identification and Mitigation

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

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

4 INSTRUMENT READINESS

4.1 Survey and Alignment

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

4.2 Utilities

Utilities services/capabilities are as documented in the XFP Beamline IRR Functional Description (PS-C-XFD-XFP-PLN-001). The following components were added / altered for the XAS endstation installation:

- Distribution of process chilled water for water-cooling of a compressor for the XAS cryostat.
- Liquid nitrogen distribution in 17-BM-A (note that the supply source is the FMX/AMX LN2 distribution system).

4.3 Vacuum System and Pressure Safety

The original XFP beamline vacuum system installation and specification is as described in the XFP Beamline IRR Functional Description (PS-C-XFD-XFP-PLN-001).

For the XAS endstation monochromator chamber, vacuum pressure is expected to be in the 10^{-9}-10^{-7} torr range. The Be window terminating the XAS endstation is of a standard NSLS-II design used for direct white beam in NSLS-II 3PW beamline front ends. As the monochromator chamber components (slits, absorber, and monochromator) are internally water cooled, the monochromator chamber is equipped with a certified burst disk to prevent overpressure. Venting of this vacuum section to atmospheric pressure with dry nitrogen gas will employ a pressure relief valve with a very low relief pressure (1/3 psi) to prevent any internal overpressure condition. A beampipe proximity sensor switch is used to determine when the beamline is in the XAS configuration and thereby appropriately control vacuum interlock signals.

4.4 Controls

All motorized components have been tested and documented on the endstation installation traveler. Control System Studio (CSS) screens have been prepared to access the motors on the components.

4.5 Equipment Protection System (EPS)

The Equipment Protection System (EPS) capabilities for the original XFP beamline installation were documented in the XFP Beamline IRR Functional Description (PS-C-XFD-XFP-PLN-001).

Scope additions for the XAS endstation include:

1. Vacuum pressure monitoring and interlock for the XAS monochromator and transport pipe.
2. DI water flow monitoring and interlock for cooling of the XAS components including the pink beam slits, monochromator first crystal, pink beam diagnostic absorber, and Be exit window.
3. A beampipe proximity sensor switch interlock to control which vacuum and water flow components interlock the beamline shutter, depending on which of the XAS or footprinting endstations are in operation.
4. PCHW water flow monitoring for the XAS cryostat compressor.
5. Liquid nitrogen pneumatic supply valve control.
6. EPICS interface for components that require I/Os installed on the EPS PLC, including an XIA attenuator box assembly.