9th Edit. of the Mech. Eng. Des. of Synchrotron Radiat. Equip. and Instrum. Conf. MEDSI2016, Barcelona, Spain JACoW Publishing
ISBN: 978-3-95450-188-5 doi:10.18429/JACoW-MEDSI2016-WEPE36

DESIGN, CONSTRUCTION AND COMMISSIONING OF TWO HIGHLY INTEGRATED EXPERIMENTAL STATIONS FOR MICRO-FOCUSING MACROMOLECULAR CRYSTALLOGRAPHY BEAMLINES AT NSLS-II

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Abstract

We present the final engineering design and first commissioning results of two highly integrated experimental stations for macromolecular crystallography (MX) at the National Synchrotron Light Source II (NSLS-II). One of the beamlines will provide micro-focusing (FMX) and the other high automation (AMX). These beamlines will support a broad range of biological and biomedical structure determination methods from serial crystallography on micron-sized crystals, to structure determination of complexes in large unit cells. The experimental stations were designed and fabricated largely in-house to obtain highly flexible and modular units that meet the challenging stability and integration requirements required by the small beam sizes and the limited work envelopes. Key components to meet these criteria are a highly compact beam conditioning unit and exchangeable main and secondary goniometers (sample rotation axes) integrated in a granite support structure.

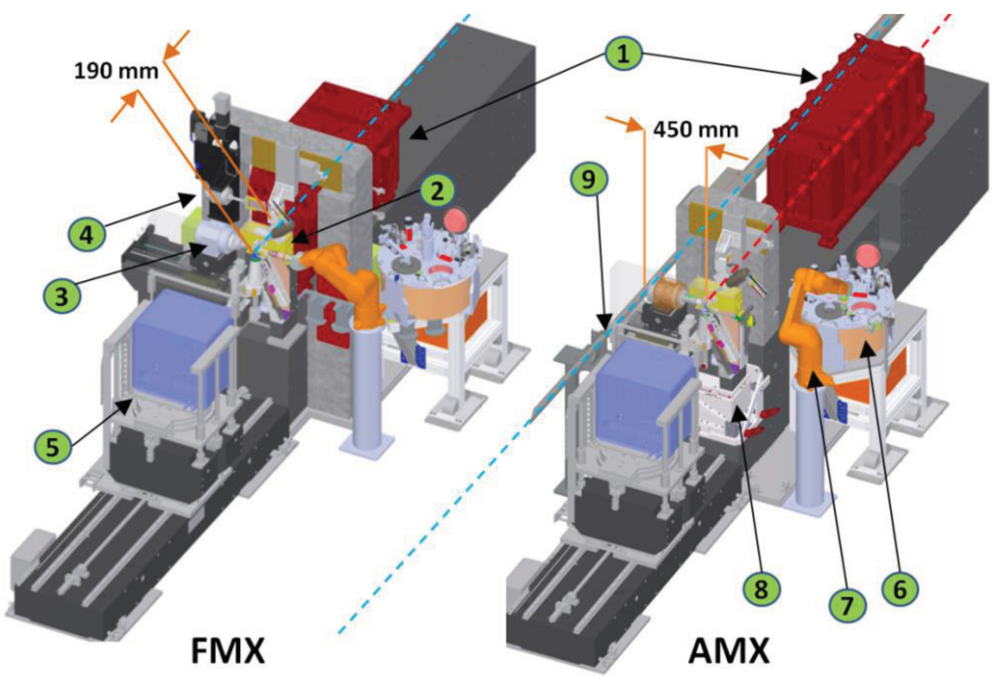

Figure 1: The FMX (left) and AMX (right) experimental stations. The beams come from the top right (FMX cyan dashed line, AMX red dashed line). Devices (identical at both endstation if not indicated otherwise): (1) KB focusing mirror tanks, (2) beam conditioning unit, (3) main goniometer, (4) FMX secondary goniometer, (5) detector support, (6) LN2 sample Dewar (7) Stäubli 6-axis sample mounter (8) AMX XY lift stage (9) FMX beam pipe passing the AMX endstation.

INTRODUCTION

The FMX and AMX beamlines were designed and constructed as part of a suite of Advanced Beamlines for Biological Investigations with X-rays – the ABBIX project [1–3]. The beamlines' layout and scientific mission greatly influence the endstation design: beam sizes of down to 1 and 4 µm respectively require high vibrational stability. The two beamlines share sector 17 of the storage ring in a canted undulator arrangement. Combined with the Kirkpatrick-Baez (KB) focusing mirrors' short focal distances, this layout poses great spatial constraints for the experimental stations and beam conditioning units. To support different measurement

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modes such as serial micro-crystallography or crystallization plate screening, and to open the possibility to accommodate novel experiments that have not been specified at the time of design, the design needs to be modular and flexible.

The experimental stations comprise a beam conditioning unit, an on-axis microscope, a specimen goniometer, an array of auxiliary devices, and a sample changing robot (Figure **1**). Each of these major components can be aligned independently into the beam. The specimen goniometers consist of a high precision air bearing main goniometer with horizontal spindle axis, interchangeable with a secondary goniometer at FMX for specialized experiments. The on-axis microscopes are reflective optics telescopes (Questar QM100 [4], with customized housing) complete with front and back illumination. The auxiliary apparatus includes collimation and diagnostic units, a beamstop, and a nitrogen gas cryo cooler. The endstations are highly integrated in a compact space on a granite support structure with high modularity for future upgrades and extensions.

This article highlights the construction of the self-contained beam conditioning unit and the collimating and diagnostic system, as well as the main goniometer.

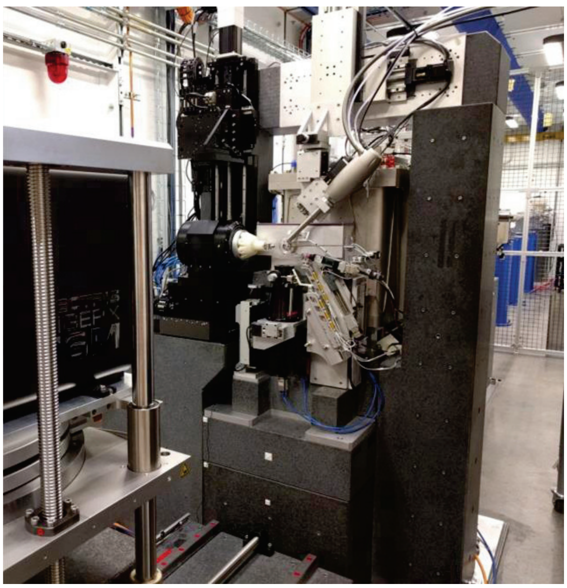
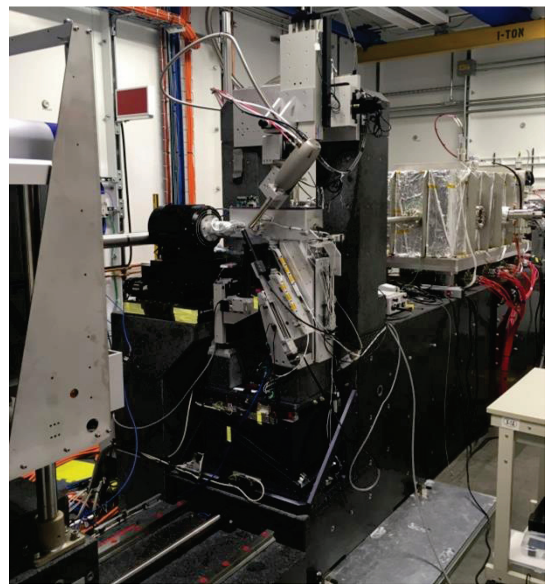

Figure 2: Current status of the experimental stations of FMX (left) and AMX (right). Both endstation core modules are mounted on the main granite block supporting the focusing mirrors. The CryoStream sample coolers and the FMX secondary goniometer are mounted on granite arches.

END STATION DESIGN, INTEGRATION AND ENGINEERING

In the design of the experimental stations (Figure 2), two main spatial restrictions had to be overcome which result from the beamlines' optical layouts. The so-called experimental station core module aligns these components along the beam path from upstream to downstream: the compact beam conditioning unit, the on-axis microscope, an x-ray fluorescence detector, the beam collimator, a diode and scintillator unit, the beamstop, and the backlight. At FMX, due to the KB focusing mirrors' short focal length, the distance from the mirror tank's downstream face to the sample is only 190 mm along the beam (Figure 1). This extreme restriction led us to design and develop a highly compact beam conditioning unit that contains, within 140 mm, a **B**eam position monitor, **A**ttenuator, **S**lits, **I**ntensity monitor, sub-millisecond **S**hutter, and secondary **S**lits – the BASISS unit (Figure 3). The remaining 50 mm to the sample position fit the on-axis microscope and the beam collimation unit. At AMX, there is ample space along the beam, but here the lateral distance between the adjacent FMX beam pipe and AMX focal spot is just ~450 mm (Figure **1**), which greatly restricts the space for the main goniometer. In each case, the more restrictive environment sets the boundary conditions to keep the design identical for the two endstations wherever possible.

Both beamlines' focusing KB mirrors can be retracted from the beam to provide a more parallel beam for large unit cell crystals – at the expense of dose rate. At FMX, this leads to a beam movement of 5 mm vertically and 2.5 mm horizontally, accommodated within the BASISS unit. At AMX, the corresponding beam movements are 19 mm and 14 mm respectively. To accommodate this large beam shift, a wedge-based XY lift and translation stage carries the endstation core module.

BASISS Beam Conditioning Unit

Several constructional details are the key to fitting all six components of the beam conditioning unit into a 140 mm long space. Instead of the commonly used vacuum

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tank, a helium-filled aluminum enclosure was built with lower requirements on wall thickness. Compact stick-slip piezo positioners with nm resolution (SmarAct SLC-17XX) and integrated encoders were used not only to achieve high positioning repeatability and speeds (up to 20 mm/s) but also to minimize component dimensions. Additionally, the thickness along the beam of each device was carefully minimized: The *X-ray beam position monitor* (Sydor Instruments SI-DBPM-M405, [5]) is driven by two SmarAct SLC1740-D positioners that have a repeatability of 25 nm and provide holding power against its 5 coaxial signal cables. The *attenuator* was constructed as a slim stack of 4 sliders with 8 attenuator slots each. In order to provide attenuation with sufficient dynamic range over FMX's energy range of 5-30 keV, an octal system of layered thin Al and Sn foils was implemented [3]. Both *four-blade slits* and the *intensity monitor* inherit their compactness from the careful arrangement of SmarAct SLC1720 and SLC1740-D positioners, respectively. The design of the fast *shutter* is derived from a highly compact unit developed at the APS [6] which uses a galvanometer motor (Cambridge Technologies 6220H) with ±20° opening angle to rotate two tungsten blades bracketing a channel to pass the beam. Depending on the local beam size, this shutter provides opening times of a few 100 µs, which is required to limit the sample exposure before the actual data collection in high-flux experiments.

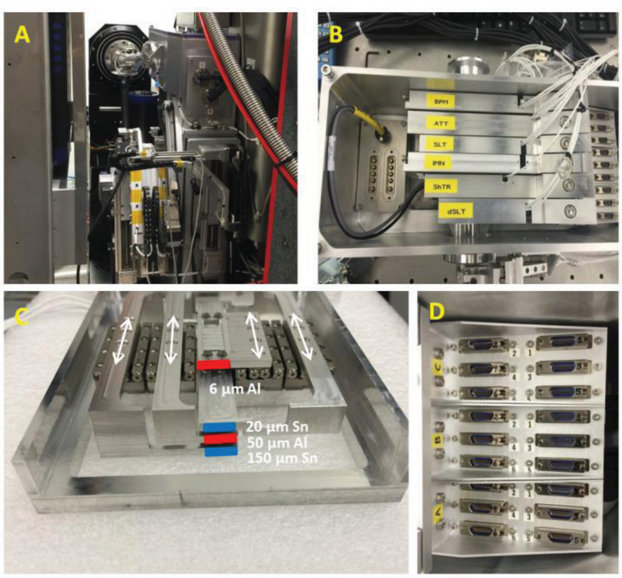

Figure 3: (A) Closeup view of the BASISS beam conditioning unit integrated in the endstation. (B) All devices aligned inside the unit, (C) attenuator module, (D) Patch panel: Omnetics micro-D latch connectors for 17 nano-positioners (1 spare).

An important design goal for this unit was for it to be highly modular and enable rapid exchange of modules to reduce service times. All devices are mounted on a specially designed rail with 2D kinematic mounting brackets for easy and reproducible access to each device. A central patch panel and short cable loops allow for quick exchange of components.

Beam Collimator and Diagnostics

A molybdenum collimator tube (OD 0.51 mm, ID 0.35 mm) acts as a guard aperture for radiation scattered from upstream BASISS components and suppresses air scatter from the beam path between the drilled microscope deflection mirror and the cold stream used to cool the sample (Figure 4, left). The collimator is aligned along the beam by a compact retractable mechanism with a motorized XYZ stack and manual flex pivot-based pitch and yaw adjustments. A wire-eroded 0.5 mm thin holder minimizes the shadowing of the on-axis microscope. On a second retractable positioner, a diode and scintillator provide beam shape, position and intensity at the focal spot (Figure 4, right).

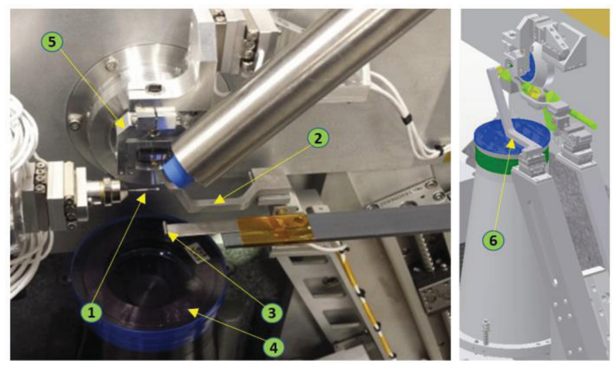

Figure 4: *Above*: FMX sample environment. (1) sample pin, (2) collimator tube holder, (3) beam stop, (4) microscope objective, (5) microscope on-axis deflection mirror. *Right*: Same region viewed from right, showing collimator positioner and (6) scintillator / diode positioner.

Both collimator and scintillator fingers are supported by magnetic kinematic mounts for rapid exchange and protection against accidental touching. Due to the mounts' high reproducibility, after an initial collimator alignment the collimator's pitch and yaw does not require re-adjustment even after repeated dismount/mount operations, only a minimal translational realignment in X and Y direction was required; this can be automated.

Main Goniometer

The main sample goniometers employ a Nelson Air SP150 rotary air bearing for the horizontal rotation axis. Its total spindle runout 50 mm above the rotation table has been determined to ±20 nm. The spindle is carried by an XYZ translation stack with a linear air bearing stage in the X-direction for sample scanning and positioning (Aerotech ABL15050) and Y and Z stages to align the rotation axis into the beam (Aerotech ANT180 and custom AVS lift stages). For sample centering, a stick-slip piezo XY centering stage (SmarAct SLC-1720) is mounted on a light weight extension arm, with a

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customized base plate containing the positioner's sensor modules. The final mounting arm (Figure 5) is just coming out of fabrication.

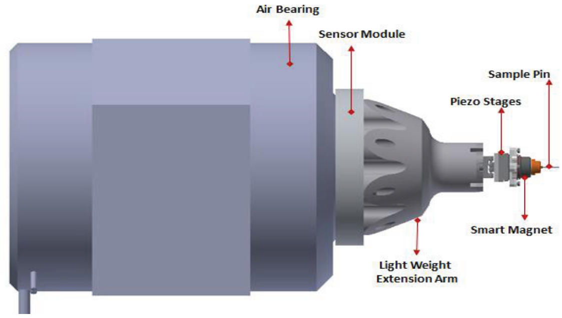

Figure 5: Main goniometer arm assembly on air bearing spindle.

In first metrology tests with a prototype arm, a sphere of confusion of 860 nm peak-peak at the sample position was obtained. The main contribution to this error is reproducible, so these first tests and previous measurements on an identical SmarAct centering stage setup [7] suggest that using active correction and the final centering arm, a target sphere of confusion on 100 nm can be achieved. At FMX, the main goniometer can be retracted laterally and replaced by a secondary goniometer for specialized experiments such as crystallization plate screening. This goniometer would be lowered from above and is currently being commissioned.

CONCLUSION

Two highly compact experimental stations for macromolecular micro-crystallography have been developed at the FMX and AMX beamlines. After the beamlines' first light on March 8, 2016, the first diffraction images were recorded in June, and the first user groups collected their first datasets in August.

ACKNOWLEDGEMENT

This work is supported by US Department of Energy BER contract DE-SC0012704 and by an US National Institutes of Health NIGMS grant P41GM111244. NSLS-II is operated under DOE BES contract DE-AC02-98CH10886.

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