NSLS-II BDN BEAMLINE 28-ID-1 (PDF) COMMISSIONING PLAN

February 21, 2018

NSLSII-28ID1-PLN-002

REV 1

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February 21, 2018

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2/20/2018

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REVISION HISTORY

REVISION	DESCRIPTION	DATE
1	First Issue	21FEB2018

Acronyms

BDM	Beam Diagnostic Module
BNL	Brookhaven National Laboratory
BPM	Beam Position Monitor
CRS	Comprehensive Radiation Survey
CSS	Control System Studio
DISC	Diffraction and In-Situ Scattering
DLM	Double Laue Monochromator
ECS	Energy Calibration System
FOE	First Optical Enclosure
HV	High Vacuum
GV	Gate Valve
GHS	Gas Handling System
NSLS-II	National Synchrotron Light Source-II
OCM	Optics Conditioning Module
PDF	Pair Distribution Function
PS	Photon Shutter
SAXS	Small Angle X-ray Scattering
SBM	Side Bounce Monochromator
UHV	Ultra-High Vacuum
VFM	Vertically Focusing Mirror
WAXS	Wide Angle X-ray Scattering
XPD	X-ray Powder Diffraction

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1. OVERVIEW

This document presents the technical commissioning plan for the PDF beamline (28-ID-1) at the National Synchrotron Light Source II (NSLS-II) at Brookhaven National Laboratory (BNL).

28-ID Beamline consists of two branch lines, the Pair Distribution Function beamline, PDF (28-ID-1), and the X-ray Powder Diffraction beamline, XPD (28-ID-2). The damping wiggler, DW100 delivers a large energy band-pass white X-ray beam into the First Optical Enclosure (FOE) of the beamline. The first optical component of the PDF beamline, the Side Bounce Monochromator (SBM) receives the transmitted part of the white X-ray beam through the first crystal of the Double Laue Monochromator (DLM) of the XPD beamline. The SBM is used to select the energy and to focus the X-ray beam in the horizontal plane. Water-cooled horizontal white beam slits and a filter array are located between the SBM and the DLM. The second optical component of the PDF beamline, the Vertically Focusing Mirror (VFM) is used to focus the monochromatic X-ray beam in the vertical plane. A Beam Diagnostic Module (BDM) consists of horizontal and vertical slits, and a Beam Position Monitor (BPM) is located downstream of the VFM. The Photon Shutter (PS) is the last component of the PDF beamline located in the FOE. The Optics Conditioning Module (OCM), located in hutch B, consists of 2 Be vacuum windows, a fast photon shutter, a set of attenuators, horizontal and vertical slits, a beam alignment system using a LASER pointer, and a movable 2 circle Diffractometer for energy calibration. A large gantry system that facilitates the translation of 2 area detectors in 3-D space is located downstream of the OCM. A heavy-duty optics table that supports sample environments, and a Small Angle X-ray Scattering (SAXS) flight tube is located underneath the gantry system. Figure 1.1 shows the beamline component layout.

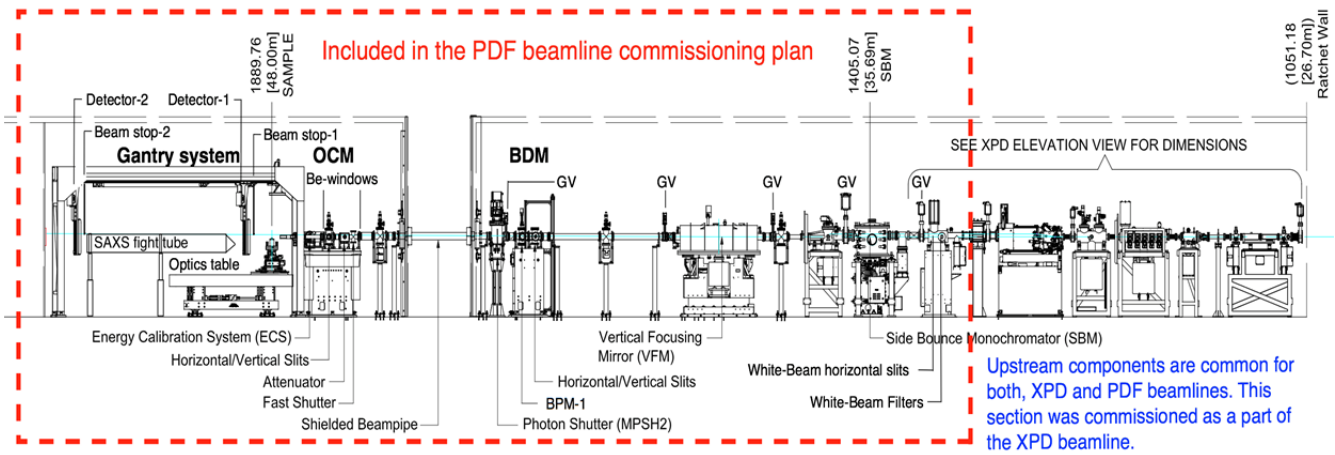

Figure 1.1: PDF Beamline Component Layout

The PDF beamline components are listed below (upstream to downstream):

<u>White beam components</u>

1. Horizontal slits
2. Filters
3. SBM

<u>Monochromatic beam components.</u>

4. VFM
5. BDM
   - 5.1 Horizontal and vertical slits
   - 5.2 BPM
6. PS
7. OCM
   - 7.1. Fast shutter
   - 7.2. Pneumatically actuated filters
   - 7.3. Horizontal and vertical slits
   - 7.4. Beam intensity monitor equipped with a filter wheel
   - 7.5. LASER alignment system
   - 7.6. Energy calibration system equipped with a movable 2 circle Diffractometer

7.7. Support table

7.8. Telescopic beam guide

8. End station gantry system that facilitates the 3-D translation of 2 area detectors and a sample support.
9. 2 Beamstops equipped with photodiodes.
10. Optics table
11. (0-5) T superconducting magnet coupled to (0-500) K liquid He cryostat
12. (80-500) K liquid nitrogen cryostream
13. Automatic sample alignment system (based on 2 optical cameras)
14. Portable gas enclosure
15. SAXS setup (flight tube + detector)
16. Gas Handling System (GHS) (excluded from beamline IRR)

All components located upstream of the PDF white-beam horizontal slits serve both, PDF and XPD beamlines. These components are fully commissioned and currently operational. The first DLM crystal and the SBM crystal always receive a low-energy filtered white X-ray beam of ~300 W (Figure 1.2). The upstream Diamond windows and filters remove the low-energy part of the spectrum. By design, the water-cooled SBM can stand the power of the filtered beam. Should there be a need to further reduce the beam power, additional filtering is available upstream of the SBM (Figure 1.1). Therefore, we do not see a need to commission the new components in the FOE of the beamline at a lower beam current. Since the B hutch and the shielded beam transport across the egress isle have never been exposed to X-rays before, initially they will be commissioned at 120 mA.

We identify 3 different phases of technical commissioning. During phase 1, the Si (311) SBM crystal that corresponds to 74.8 keV will be mounted, and the VFM will be moved out of the beam. Temperature of the SBM assembly and the vacuum pressure of the beamline up to the photon shutter will be closely monitored with the X-ray beam on the SBM crystal to ensure the function of the equipment cooling and vacuum systems. A radiation survey will be performed to ensure there are no detectable radiation leaks through the FOE under surveyed nominal and horizontally mis-steered beam conditions. Next, the beam direction, size, and flux will be optimized by tuning the parameters of the SBM using the BPM output. Then the VFM will be moved into the beam, and the radiation survey will be continued to ensure there are no radiation leaks under nominal and vertically mis-steered beam conditions. During the following studies/maintenance period, a 120 mA beam will be delivered to the B hutch. A fluorescent screen mounted on the optics table at the sample position will be used to observe the beam. A radiation survey will be conducted around the B hutch and the shielded beam transport across the egress aisle to ensure there are no radiation leaks under all beam conditions. If there are no leaks found, the beam current will be increased to 360 mA, and the radiation survey will be continued.

Following a successful radiation survey, the first area detector data set of the beamline, a diffraction pattern from a standard sample will be acquired.

If at any point during the Comprehensive Radiation Survey (CRS), a radiation dose rate of 5 mrem/hr or higher on contact is identified, the radiation survey will be terminated and the cause investigated, and any hazards will be mitigated before continuing.

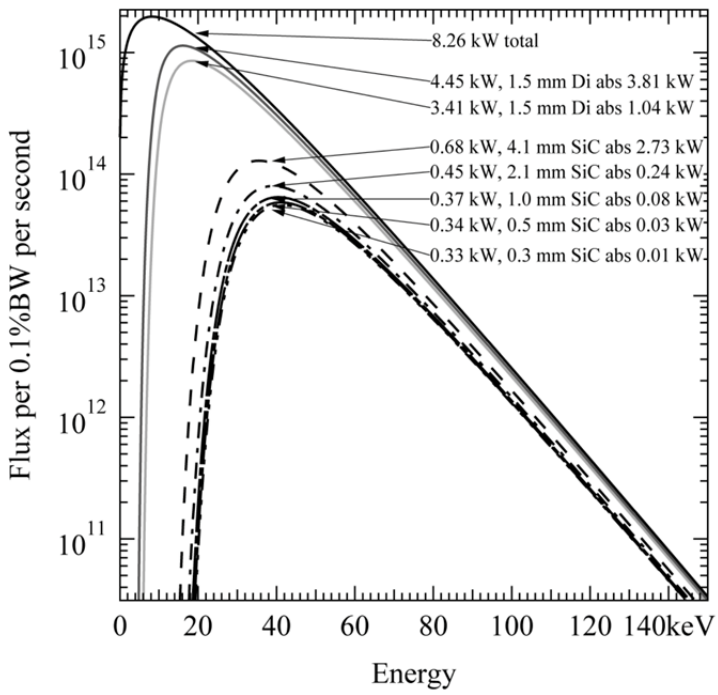

Figure 1.2: Power spectrum of the 28-ID beamline under different amount of filtering. The 330 W spectrum will be delivered to the SBM.

During phase 2, function of all process variables, CS-studio, BlueSky, and Data Broker, and XPD/PDF software will be verified. Alignment of all beamline components will be also verified. The SBM and VFM parameters will be optimized simultaneously with the other downstream components to obtain the best beamline performance. Data will be acquired on several standard samples, and the beamline energy will be precisely calibrated using the ECS. Then the stability of the monochromatic beam over a time period of one day will be investigated using standard sample measurements. At this stage, the beamline performance at 75 keV will be baselined against calculations and the performance of well-developed beamlines at other facilities.

During phase 3, beamline equipment parameters will be calibrated for the other three discrete energies, 117 keV, 39 keV, and 64 keV. This step requires opening of the SBM chamber several times to mount

three different monochromator crystals, and should be coordinated with the XPD user operations and the NSLS-II machine schedule. The beamline SAXS system will also be commissioned during this phase. Finally, all PerkinElmer area detectors that are currently being used at the XPD and PDF beamlines will be calibrated to determine the optimal operating parameters and protocols.

Scientific commissioning will then follow, with the participation of the user groups, to verify that the beamline meets the design performance goals for routine User Operations. Technical and Scientific Commissioning will follow a phased approach, wherein initial capabilities are commissioned for User Operations, while later capabilities are still in the Technical or Scientific Commissioning stage, provided this is done in a safe manner.

The 28-ID beamline GHS delivers a variety of gases to both XPD and PDF branch lines. Technical and scientific commissioning of the GHS will take place in the second half of 2018. A commissioning plan for the GHS will be presented at a later date.

2. Different Phases of Technical Commissioning

2.1 Phase 1

The goal of phase 1 is to ensure the safe operation of equipment in the FOE, and safe delivery of the X-ray beam into hutch B. The CRS defined in the BSA document, NSLSII-28ID-PRC-001, *Beamlines XPD/PDF (28-ID) Radiation Survey Procedure* will also be completed during phase 1. Ideally, the positions and orientations of the equipment would be surveyed to their nominal positions, but we expect to make adjustments away from the surveyed positions that will reflect the actual beam path as opposed to the nominal designed beam path. The steps below will be completed during phase 1:

2.1.1 Without Beam

a. Obtain a safety system work permit to open the SBM chamber and the PDF white beam filter vacuum enclosure (this step should be coordinated with the vendor, the XPD user operations and NSLS-II machine schedules).

b. Close the relevant upstream and downstream gate valves, bleed the SBM chamber, and open the top lid.

c. Mount the Si(311) crystal that corresponds to 75 keV, fill Galistan, and mount the strain gauges.

d. Replace the flange and 15-pin D connector at the bottom of the SBM chamber.

e. Verify all motions of the crystal assembly and the function of thermocouples and strain gauges.

f. Close the top lid, and pump-out the SBM chamber.

g. Turn on the water-cooling circuit and verify the correct water pressure level. Observe the vacuum pressure in the chamber for 15 minutes.

h. Close the relevant upstream and downstream gate valves, bleed, and open the white beam slit vacuum enclosure. Mount the filter plates.

i. Close and pump out the white beam slit vacuum enclosure.

j. When the vacuum pressures of the SBM chamber and the white beam slit/filter enclosure reach 1 x 10 E^{-8} mbar, open the gate valves.

k. Move the VFM out of the beam

l. Wide open the BDM beam defining slits.

m. Set the PDF white beam horizontal slits to allow a 1 mm wide X-ray beam to pass-through.

n. Search and secure the FOE and the B hutch.

2.1.2 With Beam

a. Deliver a 360 mA X-ray beam into the FOE of the beamline

b. Monitor the temperature of the SBM crystal and the vacuum pressure of the beamline up to the photon shutter for 1 hour. The vacuum pressure should comply with LT-C-XFD-SPC-COM-001, *Standard Technical Specifications for NSLS-II Beamline Components*.

c. If the temperature of the crystal exceeds 100 °C, apply additional filters.

d. If the vacuum pressure does not comply with the standard specifications, investigate the cause, and fix the underlying problem before continuing.

e. Keep opening the slits in steps of 1 mm until they allow the full beam. Monitor the temperature of the mono at each step. Apply additional filters if necessary.

f. Perform a radiation survey to ensure there are no detectable radiation leaks through the FOE under nominal and horizontally mis-steered beam conditions.

g. Tune the SBM 2-Theta and observe the beam on the BPM. Track the beam position over a period of 1 hour to verify the stability of the monochromator over time.

h. Tune all SBM parameters and qualitatively optimize the flux on the BPM at the surveyed nominal angle (5.8 degrees outboard with respect to the white beam).

i. Move the VFM into the beam and measure the vertical shift of the beam on the BPM at the surveyed nominal mirror angle for the specific energy.

j. Continue the radiation survey to ensure there are no detectable radiation leaks from the FOE under surveyed nominal and vertically mis-steered beam conditions.

Below steps should be performed during the following maintenance/studies period.

k. Adjust the height and the angle of the OCM to the surveyed nominal values for the specific energy.

l. Move the VFM out of the beam.

m. Wide open the cleanup slits and the fast shutter, and take all pneumatic filters out.

n. Move the (telescopic beam guide + ECS) and both area detectors out of the beam.

o. Obtain a safety system work permit to open the photon shutter.

p. Open the photon shutter, and deliver a 120 mA beam into the B hutch.

q. Observe the beam on a fluorescent screen mounted at the surveyed nominal sample position on the optics table.

r. Mount a scattering source/s in B hutch according to the guidelines of the BSA document, NSLSII-28ID-PRC-001, *Beamlines XPD/PDF (28-ID) Radiation Survey Procedure*, and perform a radiation survey around the B hutch and the shielded beam transport, across the egress aisle to ensure there are no detectable radiation leaks.

s. If there are no detectable radiation leaks found, increase the beam current to 360 mA and continue the radiation survey.

Below steps should be performed during the following operations period.

t. Adjust the beam size to (0.5 x 0.5) mm using BDM slits.

u. Align the detector 1 beamstop using the signal from the embedded I_0 monitor.

v. Then move detector 1 to its surveyed nominal position in the X-Y plane (when the detector is at its nominal position, vertically undeflected beam should hit the center of the detector).

w. Apply OCM pneumatic filters, and precisely align the beam stopper using the detector.

x. Quantitatively maximize the flux using I_o from the beamstop.

y. Mount a standard sample in the beam and acquire the first diffraction data of the PDF beamline.

z. Calibrate the energy and analyze the data.

2.2 Phase 2

The goal of phase 2 is to determine the optimal beamline equipment parameters for 75 keV X-rays. All data acquisition, management, processing and analysis software will be tested and optimized at this stage. Beamline automatic sample alignment system will be commissioned and its operating software will be tested. Finally, the beamline performance will be compared against calculations and the performance of well-developed PDF beamlines at other facilities. The steps below will be completed during phase 2:

a. Verify the range, accuracy, repeatability, and limits of all beamline motions against acceptance test results using properly tuned, dedicated Delta Tau channels. If necessary, Delta Tau motor controllers will be further tuned at this time.

b. Install filters in both filter wheels of the OCM.

c. Test and verify the function of process variables, CSS-studio, BlueSky, Data broker, xpdAcq and XPDsuite software.

d. Test and verify the function of detector control software.

e. Move the VFM into the beam at the surveyed nominal angle.

f. Quantitatively and iteratively optimize the SBM and VFM parameters simultaneously with the parameters of other downstream equipment.

g. Determine the VFM parameters for collimated and focused beams.

h. Measure focal length and focus size vs. bending of the VFM and SBM.

i. Test the effect of asymmetric bending.

j. Perform slit scans to determine Horizontal/Vertical beam intensity profiles.

k. Align the LASER with respect to the X-ray beam. Then align the telescopic beam guide with respect to the LASER.

l. Align the detector 2 beamstop.

m. Commission the ECS:

    i. Make a sample holder and mount the analyzer crystal.

    ii. Make EPICS conditions to apply a default filter when the sample 2-Theta reaches a pre-defined minimum angle.

    iii. Move the ECS into the beam.

    iv. Mount a standard sample and align it on the Theta/2-Theta rotation axis.

    v. Adjust the height of the OCM support table to bring the capillary sample to beam height (if necessary adjust the vertical slits).

vi. Move the Analyzer 2-Theta to 0, and translate the analyze crystal out of the beam to ensure the scintillator detector is directly looking at the sample.

vii. Do a sample 2-Theta scan to find a strong peak.

viii. Lock the sample 2-Theta at the center of the strong peak.

ix. Move the analyzer crystal into the beam and analyze 2-Theta to the nominal angle for the particular energy.

x. Then fine-tune analyzer Theta, 2-Theta, and roll to optimize the scintillation detector counts.

xi. Do a sample 2-Thera scan, and acquire a diffraction pattern on a standard sample (Ex: LaB_6).

xii. Refine the X-Ray wavelength.

n. Acquire diffraction/PDF data on a standard sample under ambient conditions every 5 minutes over a time period of one day, and verify the beam stability using structural model based refinements.

o. Measure the photon flux, energy bandwidth, and beam size at the sample position (relative numbers would be enough for this purpose) as a function of the opening of the horizontal white beam slits, and as a function of SBM crystal bending to determine the opening width of the horizontal white beam slits for an acceptable energy resolution.

p. Acquire data on 3-5 standard samples to base line the optimal flux/energy spread at 75 keV.

q. Verify the alignment of the detector 1 beamstop along the full Z travel range. If necessary, fine-tune the alignment of the bridge with respect to the beam direction.

r. Align the detector 2 beamstop.

s. Commission the automatic sample alignment system:

i. Complete the assembly of the system.

ii. Verify the function and limits of all motions.

iii. Determine an alignment strategy.

iv. Write python functions to perform the alignment.

v. Optimize the parameters of the python functions iteratively.

2.3 Phase 3

The goal of phase 3 is to commission, calibrate and baseline the beamline performance at the remaining three discrete energies, 117 keV, 39 keV, and 64 keV respectively. Since it requires access to the beamline FOE, phase 3 activities should be coordinated with the XPD user operations and the NSLS-II machine schedule. The XPD and PDF beamlines use several area detectors for data acquisition. During phase 3, time will also be allocated to calibrate these detectors to find the optimal operating protocols and parameters, and to baseline their performance against the manufacturer specifications, and each other.

2.3.1 Without Beam

a. Obtain a safety system work permit to open the SBM chamber.

b. Close the relevant upstream and downstream gate valves, bleed the SBM chamber, and open the top lid.

c. Remove the SBM crystal.

d. Mount the next crystal and top off Galistan.

e. Verify all motions of the crystal assembly, function of thermocouples, and strain gauges.

f. Close and pump-out the chamber.

g. Turn on the water-cooling circuit and verify the correct water pressure level. Then observe the vacuum pressure in the chamber for 15 minutes.

h. When the vacuum pressure of the SBM chamber and the white beam slit/filter vacuum enclosure reach 1 x $10E^{-8}$ mbar, open the gate valves.

i. Wide open the BDM vertical slits.

j. Move the VFM out of the beam.

k. Search and secure the FOE and hutch B.

2.3.2 With Beam

a. Locate the beam on BPM and tune the SBM parameters using the BPM.

b. Move VFM into the beam and measure the vertical shift of the beam on the BPM at the corresponding pitch angle of the VFM for the particular X-ray energy.

c. Wide open the cleanup slits and the fast shutter. Take all pneumatic filters out, and move the telescopic beam guide + ECS, and both area detectors out of the beam.

d. Adjust the height and tilt of the OCM table to the surveyed nominal values for the corresponding mirror angle.

e. Observe the beam on a fluorescent screen mounted at the sample position on the optics table.

f. Adjust the beam size to (0.5 x 0.5) mm, and then align the detector 1 beamstop using the signal from the embedded I_0 monitor. Then move detector 1 to its surveyed nominal position.

g. Apply OCM pneumatic filters, and precisely align the beam stop using detector 1.

h. Align the detector 2 beamstop using the embedded IO Monitor and detector 2.

i. Align the LASER with respect to the beam.

j. Mount a standard sample in the beam and acquire diffraction data.

k. Calibrate the energy using the ECS, process, and model the data.

l. Fine-tune and calibrate all beamline and instrument parameters for the optimized flux using diffraction data. Note down all optimal beamline parameters for the particular energy.

m. Acquire data on 3-5 standard samples to base line the optimal flux at the particular energy.

n. Repeat 2.3.1 and 2.3.2 for the remaining 2 energies.

o. Commission all PerkinElmer detectors that are currently being used at the PDF and XPD beamlines. The following steps will be taken for each detector:

   i. Measure the flat field.

   ii. Determine the best strategy to trigger the detectors.

   iii. Acquire data on standard samples and baseline the performance of each detector with respect to each other.

3. DELIVERABLES FROM TECHNICAL COMMISSIONING

3.1 Generate a matrix/spreadsheet of optimal beamline parameters for each energy.

3.2 At each energy, acquire data on 3-5 standard samples with optimized flux, process, analyze, and model the data and record the results for future comparison.

3.3 Generate a matrix/spreadsheet of optimal detector parameters.

3.4 Generate a comparison of beamline performance with calculations and the performance of other PDF beamlines.

4. SCIENTIFIC COMMISSIONING

The PDF beamline is designed for PDF, complementary Wide, and Small angle X-ray Scattering (WAXS and SAXS) measurements on structurally challenging novel materials in a variety of sample environments. The goal of the scientific commissioning is to calibrate the equipment and to define the optimal beamline parameters and configurations needed for the anticipated scientific program, and to bring the complete beamline into routine user operations. During the initial phase of scientific commissioning, the following equipment will be commissioned.

4.1 Commission the (80-500) K liquid nitrogen cryostream:

a. Implement and verify the function of all control PVs of the cryostream on CSS-Studio screen.

b. Perform T-dependent measurements on calibration standard samples that show well-known phase transitions.

c. Analyze the data and produce temperature calibration plots.

4.2 Commission (5-500) K liquid helium cryostat”

a. Implement and verify the function of all control PVs of the cryostat on the CSS-Studio screen.

b. Implement and verify the function of all control PVs of the translation stages on the CSS-Studio screen.

c. Verify all translations of the mounting stages.

d. Perform T-dependent measurements on calibration standard samples that show well-known phase transitions.

e. Analyze the data and produce temperature calibration plots.

4.3 Commission (0-5) T superconducting magnet coupled to the liquid helium cryostat:

a. Assign a work control coordinator and obtain the BNL safety groups approval to commission the magnet.

b. Implement the function of all control PVs on the CSS-Studio screen.

c. Verify all translations of the mounting stages.

d. Measure the stray magnetic field, and determine safe working conditions.

e. Perform B and T dependent measurements on calibration samples that show well-known phase transitions.

f. Analyze the data and produce magnetic field calibration plots.

4.4 Commission the SAXS system.

a. Move the flight tube into the beam.

b. Fill the flight tube with He.

c. Measure a SAXS calibration standard sample.

d. Measure a nanoparticle standard sample.

e. Process and analyze the data and baseline the performance.

5. DELIVERABLES FROM SCIENTIFIC COMMISSIONING

5.1 Generate a spreadsheet of data, and temperature calibration plots for the (80-500) K liquid nitrogen cryostream.

5.2 Generate a spreadsheet of data, and temperature calibration plots for the (5-500) K liquid helium cryostat.

5.3 Generate a spreadsheet of data, and magnetic field calibration plots for the (0-5) T superconducting magnet.

5.4 Generate a calibration procedure for the SAXS system.