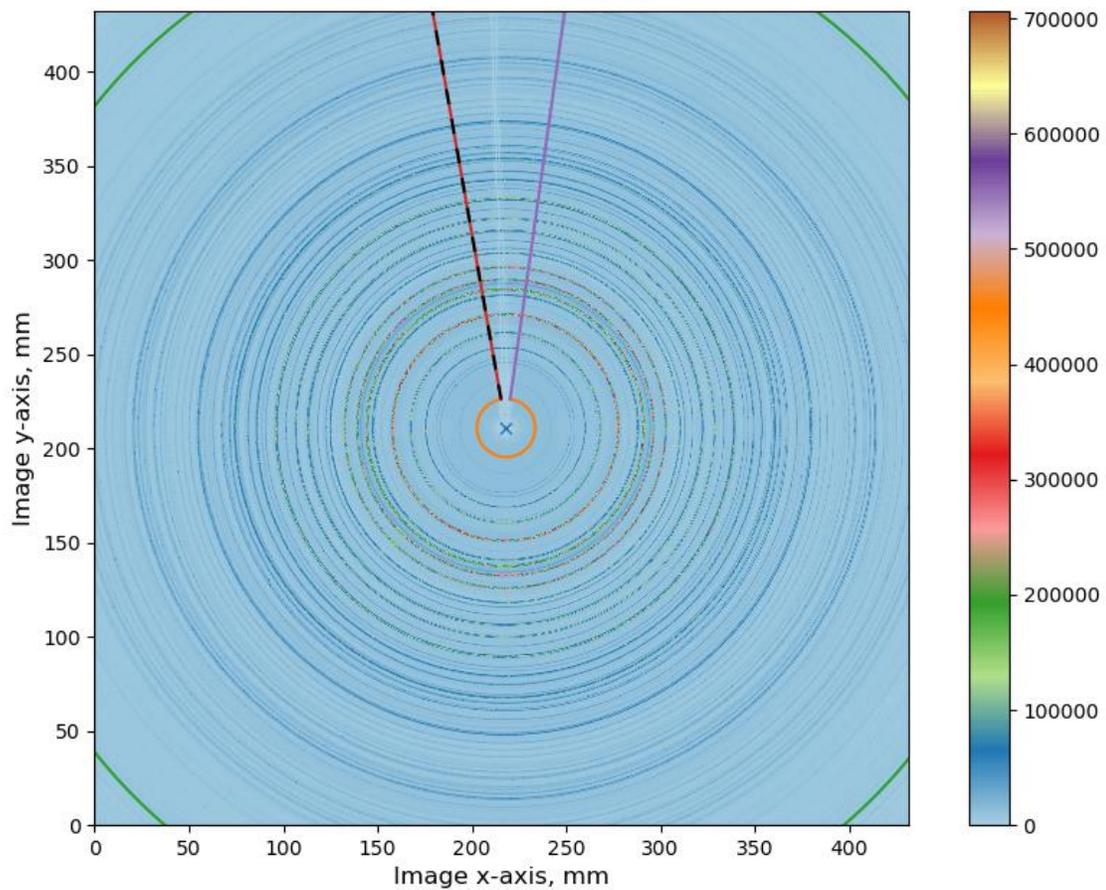# BXDS High Energy Wiggler Beamline User Guide

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BXDS Instrument Scientist

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Revision 3.2

Brockhouse (BXDS) Sector Contact List

** Note: From a CLS phone you must dial "9" to access an external line. For CLS internal numbers you only need to dial the 4 digit number. ***

Serious emergency (Fire/Ambulance):   **911**
U of S Security: 9-306-966-5555

Beamline and CLS staff:

Graham King (High Energy Beamline Lead)	3760	639-998-1886
Al Rahemtulla	3530	519-993-9137
Beatriz Moreno	3868	306-241-1999
Adam Leontowich	3555	306-850-0408
Narayana Appathurai	3648	306-514-1384
Karim Louca	3583	306-504-1169
Garth Steel (controls lead)	3730	
Shawn Carriere (mech. eng. lead)	3541	
Peter Nguyen (elec. eng. lead)	3841	

Call the beamline:

Low Energy Beamline (SOE-1)	657-3821
High Energy Beamline (SOE-2)	657-3830
Undulator Beamline (SOE-3)	657-3832

Other important contacts:

Floor coordinator	657-3639
Control room	657-3570
User services office (USO)	657-3700
Health and safety (HSE)	657-3663
Front desk	657-3500

Contents

1.0 Introduction

The Brockhouse X-ray Diffraction and Scattering (BXDS) Sector at the Canadian Light Source (CLS) synchrotron provides state of the art infrastructure for X-ray diffraction characterization needed by the materials science community in Canada and abroad. The sector, named after the Canadian Nobel laureate Prof. Bertram Brockhouse, has three independently operable beamlines. An undulator sources one beamline (5-22 keV), while a wiggler is the source for two beamlines, the low energy (LE, 7-22 keV) beamline, and the high energy (HE, 25-95 keV) beamline.

1.1   Scope

This document covers a description of the HE beamline, operating procedures, and a guide to experiment planning. A description of the source and front end are provided in the SOE1 manual "BXDS Low Energy (7-22 keV) Beamline Manual" and are not described in detail here.

1.2   History

The HE beamline achieved first light to the endstation location in January 2018, went into the scientific commissioning phase is June 2019, and entered the general user program in July 2020. These events cap a long project history stretching back to the initial funding announcement in 2006. During the in between years, many CLS approved documents describing the HE beamline and its components were written. Three of these contain broad overviews of the project,

- "Beamlines Brockhouse sector at CLS "Preliminary design report" Optics design for the Undulator, High energy and Low energy wiggler beamline", B. Meyer, 2012 (not a CLS approved document)
- 33.2.1.1 - Brockhouse Beamlines Preliminary Design Report
- 33.7.11.1 - Brockhouse High Energy Wiggler Beamline Commissioning Procedure

The beamline is also described in a peer-reviewed journal article:
Rahemtulla, A., King, G., Gomez, A., Appathurai, N., Leontowich, A. F. G., Castle, R., Burns, N., Kim, C.-Y., Moreno, B., Kycia, S. The High Energy diffraction beamline at the Canadian Light Source. *J. Synch. Rad.* (2025) **32,** 750-756.

2.0   POE1 and HE Monochromator

2.1   POE1 Components Shared by the LE and HE Beamlines

The X-rays generated by the wiggler first pass through the front end, where the beam size is defined by a series of fixed masks. The front end is also where photon shutter 2 (PSH2) and the safety shutter (SSH) are located. These shutters are shared by all 3 BXDS beamlines so any user should be aware that closing them will cut off beam to the other 2 beamlines. For this reason these shutters are generally kept open during normal operations. If they are closed, such as happens automatically after a beam trip, and need to be opened the safety shutter must be opened before PSH2 can be opened. After entering the first primary optical enclosure (POE1) the beam passes through a series of graphite filters and a screen and then reaches mirror 1 (M1), which deflects the inboard side of the radiation fan slightly outboard towards the LE monochromator. The two wiggler beams travel together past another pair of slits and screens. The LE monochromator then deflects the LE beam towards secondary optical enclosure 1 (SOE1) to be used for the low energy wiggler beamline. The high energy wiggler beam and undulator beams continue past in a straight line. None of these components are normally adjusted during routine operation. A more detailed description of these components can be found in the SOE1 manual.

2.2   POE 1 Filters, Slits, and Screens Unique to the HE Beamline

The central portion of the wiggler beam, which contains the greatest flux of high energy X-rays, passes by the M1 mirror and then encounters a second filter assembly. These filters remove most of the X-rays below the beamlines range to reduce the heat load on subsequent components, while only slightly reducing the flux of the useful X-rays. This assembly has 3 filters, 1.8 mm of annealed pyrolytic graphite, 0.41 mm of aluminum, and 1.6 mm aluminum. The filters are clamped in water cooled copper blocks. The first two filters are fixed while the third 1.6 mm aluminum filter can be removed. The graphite removes most of the X-rays with energies below ~5 keV, while aluminum removes most with energies below ~12 keV. A slightly larger flux can be obtained by removing the last Al filter but it is usually left in. Taking it out for more than several minutes can cause an overheating of the beamstop past the HE monochromator, so this temperature should be monitored if it is removed.

Next, the beam passes through two sets of slits, one horizontal and one vertical, which define the shape of the beam which will impinge on the monochromator. Larger slit sizes result in more flux but a greater divergence and energy spread, hence broader diffraction peaks from samples, while smaller slits give less flux but a more uniform beam that gives sharper diffraction data. As the vertical focusing of the beam is greater than the horizontal, the vertical opening of the slits has little noticeable effect on the beam divergence while the horizontal opening has a significant effect. For this reason the horizontal slits are normally kept narrower than the vertical. The vertical slits are normally opened in the range of 2 to 4 mm. The horizontal slits are typically set to a gap between 0.5 and 2 mm, depending on the experimental requirements for flux and resolution. When there is high flux and resolution is important, such as doing diffraction measurements at 30-35 keV, a setting of 0.5 × 2 mm or 1 × 3 mm is usually used. When flux is lower and resolution not as important, such as doing PDF at 55-70 keV, a setting of 2 × 4 mm is usually used. In order to prevent thermal shock to the monochromator crystals the horizontal slits must be closed before the downstream PSH2 shutter can be opened. These slits can overlap with each other so entering a large negative number, such as -20, will move them to the fully closed limits and allow the shutters to be opened. Both of the limit switches for the horizontal slits must be triggered before the shutters can

be opened. There is an indicator light on the bottom of the controls which shows when this is the case. The slits should be opened gradually to avoid thermal shock. Past these slits is a fluorescent screen which can be used to view the beam shape. This is for diagnostic purposes and will normally be taken out for experiments to improve the flux.

2.3　The HE Monochromator

The High Energy Monochromator (HE mono) is a bent Laue design which serves the triple purposes of deflecting the beam inboard away from the undulator beam, selecting a particular wavelength, and focusing the beam. The monochromator carriage contains two Si single crystal wafers oriented at different fixed angles. One crystal is oriented to make use of the (111) reflection while the other is oriented so that the 422 or 533 reflections can be accessed. In general, the 111 crystal is intended to be used for lower energy X-rays (~25-37 keV) while the 422/533 crystal is meant for higher energy X-rays (~39-90 keV). The 422 reflection is generally best for energies between 39-70 keV while the 533 is used for energies from 70-90 keV. The 422 provides higher flux while the 533 provides a narrower energy bandwidth. If PDF is being done using 70 keV then the 422 reflection is preferred for its higher flux. If high resolution diffraction data is needed on thick, highly absorbing samples 70 keV off the 533 will be used since the focal position is far back on the table and a long sample to detector distance can be achieved. These crystals are bent by bars pressing on the top and bottom of the crystal. The monochromator is cooled by liquid N_2 with a cyro-cooler located outside of POE1 on the outboard side. This cryo-cooler also cools the LE mono and has gas tanks that must be periodically changed. These are changed on a schedule but if the shutters cannot be opened an empty tank may be the reason. There is a beamstop past the mono crystals which will absorb any beam that passes through them.

The two crystals can be switched by using the translation motion of the monochromator. It is important that the beam is blocked while the crystals are being switched so that components on the carriage that are not able to handle direct beam do not get damaged as the beam passes over them. Before switching between crystals the horizontal slits behind the mono should be should first be closed. There is an in-position switch that indicates if the monochromator translation is within a certain window around one of the two central positions for the crystals. If the carriage is moved off this in-position switch without the horizontal slits in-position switch being on the front end shutters will close. The 111 crystal is centered at a translation of -34 and the 422/533 crystal is centered at 30. The rotation of the monochromator determines the energy which is selected. As this monochromator contains only one crystal the angle that the beam comes out depends on the energy. The purpose of the translation table in the hutch is to follow the beam as the energy is changed.   For a particular energy the correct combination of mono rotation and translation table angle is needed. Changing the energy is usually done using an app, as explained later. There are also controls to adjust the pitch and roll, which can be used to change the vertical beam position. The Z-stage parameters moves the crystals vertically. It has already been centered at 55.15 mm and should not need to be adjusted when switching energies. The final component is the beamline shutter, which must be closed in order to enter the hutch. This shutter is the mechanism normally used to control if beam enters the hutch or not.

3.0   Translation Table Components

After exiting the HE mono the beam passes through a beryllium window and exits the ultra-high vacuum of the storage ring. It then goes through a spool capped by 50 μm Kapton windows under rough vacuum to reduce beam loss due to air scattering. The presence of the vacuum spool was found to increase the flux at 35 keV by about 12.5%. The improvement will be smaller for higher energies. The spool is attached to the end of the translation table and moves along with it. The spool has two limit switches to prevent crashing into the side of the enclosure. If moving the translation table near its limits these may be triggered and need to be reset. After exiting the spool there are a number of optical components mounted on top of the translation table.
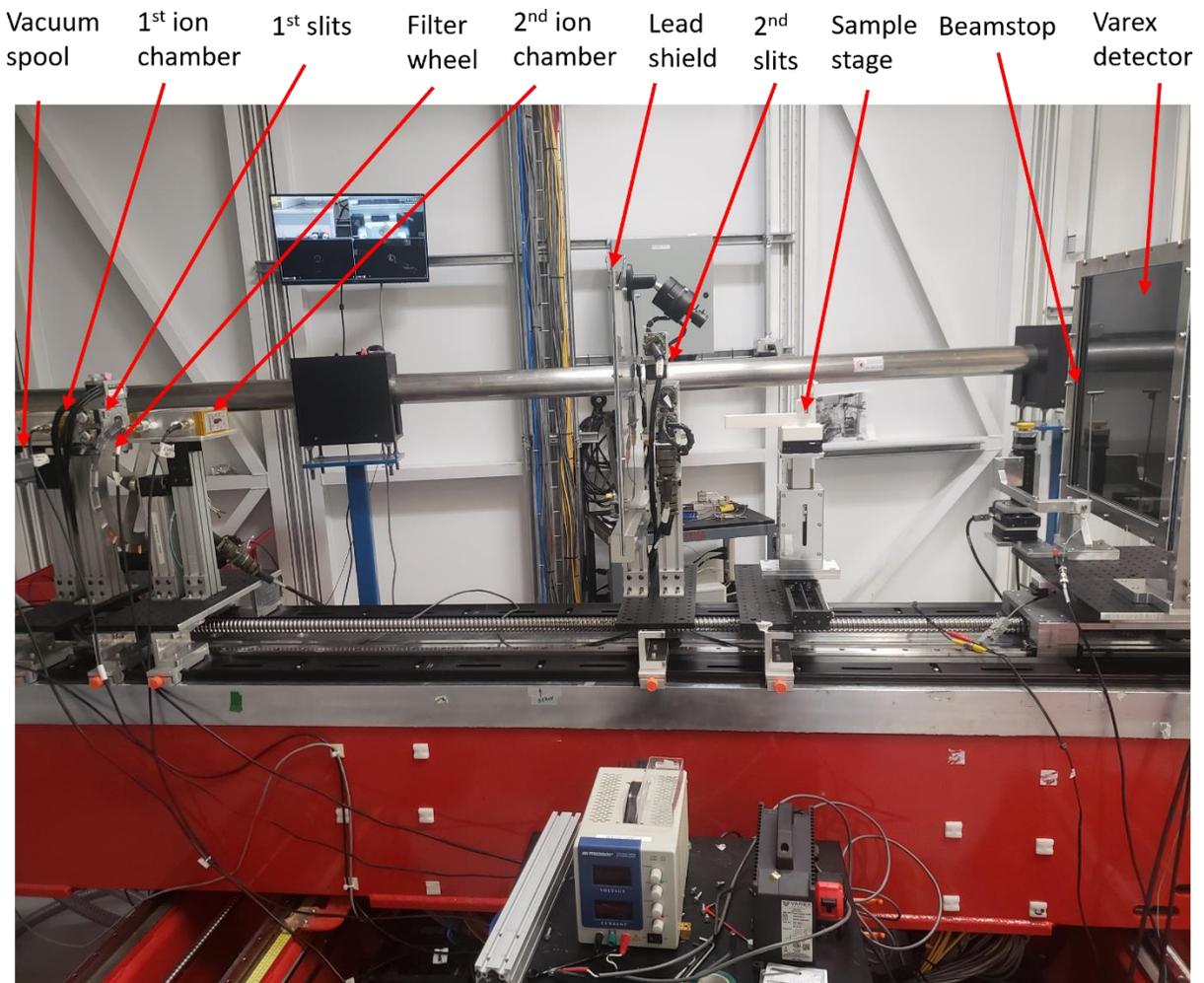
The translation table and its components.

3.1 Ion Chambers

Two ion chambers are present on the translation table to measure flux. The first is positioned at the front of the table and is intended to measure the flux coming out of the HE mono. The second is normally positioned after the first set of slits and the filter wheel but can be moved elsewhere if desired. These chambers are normally filled with air but can be fed Ar gas from a tank outside of SOE2. A spreadsheet is available on the beamline computers to convert the current into flux for a given energy. Both of these are considered the "large" dimensions for the purpose of the spreadsheet. The ion chamber currents can be read in the piccoammeters application. The first ion chamber is connected to port A1604-02-01 and the second ion chamber is connected to A1604-02-02. These are the top two readings on the screen. These parameters are can also be read from the BXDS SOE2 User application screen.

3.2 Slits

Two sets of slits are present on the table and are used to define the shape of the beam. The first set is located near the front of the translation table just after the first ion chamber and the second is located near the sample just after the lead shield. The slits each have four blades, two vertical and two horizontal. The two slit sets are not identical in design. For the first set, the blades going either vertical or horizontal are aligned with their counterpart such that the two blades will touch when the gap is set to 0. For the second set of slits all blades are offset so it is possible to have them completely closed with some overlap by entering negative numbers for the gap. The slits are controlled through SPEC. The spec variables all start with "s" for slit and then have either a "1" or "2" for the first or second slit set, followed by a "u", "l", "i", or "o" for upper, lower, inner, and outer, respectively. For example, to move the upper blade of slit set 1 to 0.3 enter the command "mv s1u 0.3". The exact values to use depend on the angle of the translation table. The sign convention for the blade is that positive opens them and negative closes them.

As the beam has an excellent vertical focus of about 50 µm its size does not need to be reduced in the vertical direction by the slits. The vertical slits are generally opened to be slightly larger than the beam in order to block any stray X-rays but not alter the shape of the beam. In generally, the first set should be opened a little larger than the second set since the beam is further from its focal point at the position of the first slits. The beam size does need to be reduced in the horizontal and the positions of the horizontal slits will have a large effect on the quality of the data obtained. If the beam is much bigger horizontally compared to vertically then the width of the diffraction rings will be wider horizontally and broad, odd shaped peaks will result. The horizontal beam is typically reduce to a width of 200-300 µm but could be made larger or smaller for particular applications. The further the detector is from the sample the larger the horizontal beam can be without large broadening. It is best to define the shape of the beam horizontally using the first set of slits. The second set of slits can then be set slightly larger than the beam. In this way the second set of slits acts as a pinhole to block air scattering but does not alter the shape of the beam. This strategy produces lower backgrounds while avoiding issues of signal from diffraction off the edges of the second slits.

3.3 Filter Wheel

A number of different Al, Cu, or Pb filters of different thicknesses are available to attenuate the beam. This can be controlled through the SOE2 app. The filter wheel can also be controlled through SPEC by the

"fw" command where the user enters the desired angle. The fluxes at these higher energies are not generally large enough to cause detector saturation or sample damage so the wheel is usually set to the empty positon where the beam simply passes through unaffected. A graph of the attenuations using each filter is posted on the inside wall of SOE2. The filter is most often used during alignment when changing an energy to help protect the detector or make the beam more position more precisely seen on filter paper.

3.4 Lead Shield

A sheet of lead covered in aluminum on both sides is used to shield the detector from stray radiation generated anywhere downstream of the sample. It is normally placed just before the second slits to provide as much protection as possible.

3.5 Sample Stage

A general sample stage allows rapid switching of different sample holders such as multi-capillary holder, a capillary spinner, and a high temperature furnace. It has a kinematic magnetic mount to allow the different holders to be pulled off and easily attached. It is controlled in SPEC using motor names of gz or gx for the vertical and horizontal motion. For the capillary spinner, the spinning speed can be controlled by at the power supply or in SPEC using spin # (where # is a value from 1-10 which indicates the voltage) or spin end to stop the spinning.

3.6 Kohzu Stage

The Kohzu stage is an alternative way to position samples in the beam. It can be used instead of the general stage and so only one of these components will be on the translation table at once. It has six degrees of freedom and is controlled through SPEC using the phi, chi, samx, samy, k-th, gonix, and goniz commands. It would general only be used when sample tilting is required. A second Kohzu stage is dedicated to the high pressure seup.

3.7 Beam Stop

The beam stop is normally placed just before the detector in order to stop the primary beam from damaging the detector. It is important that it is properly aligned so that it is blocking all of the beam. It can be attached to the same stage as the detector if it is desired to be close to the detector or on a separate mount if it is desired to be about halfway between the detector and sample. The beamstop can also be moved horizontally and vertically in SPEC using "bsx" or "bsz" commands. The beamstop contains a diode inside so that the flux of the beam hitting it can be measured. This can be used for centering the beamstop, centering samples by looking for their shadow on the beamstop, or aligning the pinhole and collimator for the high pressure setup. The beamstop is usually set as the counter for scans in SPEC. The value of the diode reading is also displayed on the piccoammeters application and BXDS SOE2 User application screen as A1604-02-03.

3.8 Area Detector

The beamline has two area detectors available, a Varex and an Eiger2. These are the final components of the translation table. Each has advantages and disadvantages. See section 5 for detailed descriptions of each and how to choose the most appropriate one for your application.

4.0   Controls for Standard Beamline Operation

There are a number of applications found on the beamline computers that control its various components. The "BXDS User" application displays a visual description of the beamline and can be used to control some aspects of it. It displays the status of the shutters, if the screens are in or out, if the movable filters are in or out, the wiggler gap, and can be used to change these settings. It also displays the vacuum readings, storage ring current, the temperatures of several beamline components such as the monochromator motor temperatures and monochromator beamstop. Generally, only beamline staff should use this application. The "BXDS SOE2 User" application is similar but with functionality limited to only what an external user would need. It is used to open the shutters or reopen the pre-mono horizontal slits. It also displays the wiggler gap and the ion chamber readings. All of the numbers on the diagram should be green or yellow. If there are any red numbers contact a beamline staff immediately.

The "BXDS MaxV motors" application can controls most motors one the beamline, with some overlap with the BXDS user application. For this beamline controls for components in the POE1 are under the VME1604-01 tab while those for the motor on the translation table on in VME1604-02. Within this tab the POE1 tab has controls for the HE slits (also controllable from BXDS User app) and the HE mono. Selecting the SOE2 tab will bring up the translation table controls. Generally it is best to use the pseudo motor control only by imputing the desired angle, as described in section 4.1. There is a "crossslide switch" indicator circle on this screen which should not be lit up green. If it become green this means the front and back of the translation table are misaligned. If this happens contact the controls group for assistance. The Eiger detector motorized stage controls are also located in the SOE2 tab of the MaxV application.

SPEC can also be used to control most of the motors on the translation table. It has some overlap with the MaxV motors and can also be used to execute scans and macros. It can be found inside the SPEC folder on the desktop of the beamline computer. Typing "wa" will show all the variables and their current positions. The most basic commands are "mv" followed by the variable and then a value, which moves that motor to a position of the value entered. The command "mvr" is similar but makes relative moves instead of absolute. SPEC can also be used to trigger the detector and scans can be done using macros to assist in beam alignment. A longer list of commands is posted at the beamline and on the website.

There is also an app called SOE2 App that is used for easier changing energy. It can store pre-calibrated mono and table positions and return to them, automating the energy change process. It can also be used to easily select a filter.

4.1 Selecting a Wavelength

To choose a new wavelength of radiation the translation table and monochromator angles will need to be changed. This is usually done using the SOE2 App. In this app any pre-calibrated position can be selected. It is also possible to select any arbitrary energy and the detector will move there, although if this is done the energy may need to be refined during the initial calibration process. Before moving the translation table be sure that no wires or other objects are in its path. It moves slowly so be patient. The monochromator angle adjusts within a few seconds.

It is also possible to manually input the positions for the table and mono rotation. While energy changes are generally not done this way anymore, this process will still be described so that the reader will understand the processes that the SOE2 app is going through. If the energy ever does need to be changed manually, there is spreadsheet on the beamline computer desktop named "SOE2_TTmonoAngleCalc" that will provide the needed values. Enter the desired energy in keV after the crystal reflection needed and the parameters will update. If you are switching from the 111 crystal to the 422/533 crystal or vise versa the first step will be to change crystals. If you are using the same crystal these next few steps can be skipped. First the HE beamline slits must be closed all the way to prevent damage to components as the carriage is moved. Enter a large negative number such as -20 for the horizontal slits. It will take a few minutes for them to fully close. When the horizontal slits are at their limits it will be possible to change the mono translation parameter. There are in-position switches which are only activated if the translation is within a certain range around the positions for either crystal. Moving the crystals without the slits closed with close the PSH2. After the limit switch for the horizontal slits is activated the mono translation value can be changed. To move to the 111 crystal enter -34 and to move to the 422/533 crystal enter 30. Make sure the mono in-position switch returns to green before trying to open the slits. The slits can then be opened again to their standard positions. While they are moving the monochromator rotation angle can be adjusted. The true position is usually about 1.1 degrees larger than the value output in the spreadsheet. The vertical positon may need to be adjusted using the tilt parameter. What seems to work best is putting a piece of phosphor paper over the first ion chamber and getting it centered on that. It is currently a good idea to shield the detector using lead and then use phosphor paper to confirm the beam is centered on the beam stop and going through all the optics. Cameras are set up in the hutch and can be moved around to view the phosphor paper while aligning the beam.

The focal position of the beam depends on the X-ray energy and monochromator reflection used. The sample is generally placed at the vertical focal position. It is acceptable for the sample to be slightly away from the focus, sometimes even desirable for samples with large grains which require a larger beam for better powder averaging. The bent Si monochromator crystal provides a good vertical focal size of about 50 μm. The beam is only slightly focused in the horizontal direction and is usually roughly 2 mm in size. Its size is typically reduced using slits to 200-300 μm. The following table provides guidance for commonly used energies. A useful reference is that the beginning of the translation table is 4.4 m from the mono and the translation table is exactly 3 m long, so the end is at 7.4 m. The focal position is proportional to the energy, so a focal position for any energy can be calculated by using the ratio of the desired energy to one in the table below. For example, the focus at 33 keV can be calculated from the 35 keV position as (33/35)*6.65 = 6.27 m.

Reflection	Energy (keV)	Vertical Focal Position (m)	Focal Position from Downstream end of Table (m)
111	30	5.70	1.70
111	35	6.65	0.75
422	55	5.91	1.49
422	60	6.45	0.95
422	65	6.99	0.41
422	70	7.52	-0.12
533	70	6.07	1.33
533	80	6.93	0.47
533	90	7.80	-0.40

4.2 Operating the Varex Detector with QXRD

The Varex detector is controlled through the QXRD program. This is a complex program but only a few parameters need to be considered for standard data collection. If more complicated issues arise the QXRD user manual can be consulted. When opened the program should automatically detect the Varex detector if it is on. However, if the detector is turned off while the QXRD program is running QXRD will loose its connection and will not automatically re-establish it. Therefore, the detector should be turned on before QXRD is opened and, if the PE is turned off, QXRD should be closed and reopened.

The "acquire" tab at the bottom brings up the key controls for data acquisition. The exposure time is one key parameter that needs to be carefully chosen. Exposure times can range anywhere from a minimum of 0.1 seconds to a maximum of 8 seconds. It is important to choose an exposure time that does not saturate a significant number of pixels. The Varex detector is 16 bit, which means that the values of each pixel can range from 0 to 65,535 (2^{16}). The values displayed are after subtraction of the dark current, so the actual saturation point is typically between 56,00 and 61,000, depending on the exposure time, the gain setting, and the individual pixel behavior. It is possible in QXRD to have any pixel above a certain value display in green so that it is easy to see if pixels are saturating. The value of a pixel can be read by putting the cursor over the pixel. This will be the V value at the bottom of the screen. If the pixel is near the saturation range it is possible to tell if it is saturated by seeing if there are small random variations in the value. A saturated pixel will not change values, while a non-saturated one will show small random variation. The amount of dark current that will need to be subtracted decreases with longer exposure times up to about 2 seconds then increases. As such, exposure times longer than 2 seconds are usually not used. An exposure time of 0.5 to 1.0 seconds is usually best. If the signal is very weak 2 seconds can be used and if the signal is strong shorter times can be used to avoid saturation. It is generally better to err on the side of shorter exposure times and sum up a greater number of these to compensate in order to avoid needing to change the exposure time due to saturation.

The other parameter that needs to be chosen is the summed. This is the number of frames that will be acquired and saved as a single file. The total data collection time is therefore the exposure time multiplied by the sums. Increasing the sums will improve the counting statistics and lead to lower random noise in the data. With data collected using an area detector, even a single frame generally produces low noise data as the entire diffraction rings are integrated. If very fast data collection is needed to monitor a rapid change a single frame can be used and data collected in less than a second. When speed is not critical a good rule of thumb is 16 to 64 frames for powder diffraction data and 128 to 512 for PDF. This assumes

that the chosen exposure time is producing peaks with intensities of at least 20,000. A greater number of sums is needed if the signal is weaker or most of the signal is quite weak as the exposure time has been reduced to avoid saturation of a few bright spots. When collecting PDF data on samples containing only weakly scattering elements an even greater number of exposures could be beneficial. To start the data collection press the "acquire" button at the bottom of the screen. Note that if live view is on the view pauses while the data collection is in progress.

Subtraction of dark current is also essential for good quality data. Having an accurate dark current is always important, but more so for PDF experiments than for high resolution powder diffraction. The "dark" button is used to collect a background. The number of darks frames collected does not need to be the same as the sums used for the data collection. Generally, the number of dark frames should be at least half the number of sums used and never less than 16. It is essential that the shutter is closed for the dark measurement. It is also important if the detector was recently exposed to X-rays that any residual image has had time to fade away. The dark current depends heavily on the gain setting and the exposure time. If these are changed a new dark is always needed. When the exposure time is changed it will take a while for the detector to adjust to the new setting, as the internal temperature of the detector changes. Therefore, the dark current will drift slowly for several minutes after the exposure time is changed. How long it takes to stabilize depends on how much the exposure time has changed by. It is therefore important to wait a few minutes before collecting the new dark. For this reason it is best to choose setting and plan experiments so that frequent changes to the exposure time are not needed.

4.3 Operating the Eiger Detector

The Eiger detector is controlled through an EPICs interface and can also be controlled through SPEC. There is a separate manual at the beamline with more details. In the EPICs controls screen it is important to set the beamline energy so the correct flat-field correction is applied. This should also automatically set the lower threshold to half of this energy. The Albula program can be used to automatically see an image after it is collected for a live view. Once this program is opened go to the Detectors and click on Eiger. The lower and upper limits for counts need to be manually set. Once this program has been running for a while it may become slow and need to be closed and reopened. The following SPEC commands can be used for data collection:

eigname 'imagename' -- sets image name

eiginc 1/0 -- toggles whether to add index number to images

eigindex NUM -- sets image index number

eigexposure CT_TIME -- sets acquisition time for eiger images

eigct -- triggers an acquisition

eiglive 1/0 -- toggles a 'live mode'

eigmasterct -- creates a filewriter HDF5 image that contains all extra metadata not typically included

eigpath '/beamlinedata/BXDS/projects/prjfolder/' – sets save directory for Eiger images (subfolders must be present)

eighelp – displays eiger macros.

eigmasterct – Takes an acquisition preserving all the inherent metadata from the eiger detector (flatfields, etc.)

eigh5 1/0 – Toggles between saving images as HDF5 and TIFF – need to also toggle liveview for it to kick in

4.4 Data Processing Using GSAS-II

GSAS-II can be used to calibrate the detector, integrate 2D images into 1D diffraction patterns, and also create PDFs from these patterns. The first setup is to import the 2D data by going to the "Import" tab, select "Image", and then select "GSAS-II known TIF Image" or "HDF5". You can then select the image or images desired and import them.

The first step is usually to do a calibration using a standard sample. Ni is most commonly used but we sometimes also use LaB_6. All the calibrations can be done by going to the image controls of an image and using the parameters on the left side and the bottom. The wavelength and approximate sample to detector distance should be entered. The cursor can be put in the center of the beamstop shadow to get good starting values for the beam X and Y. The tilt parameter and penetration can start at 0. At the bottom choose the calibrant. The pixel search range should generally start at 10 and then be lowered once the program has found the rings. The larger the value the wider the range it looks for peaks so the easier time it has finding them. However, the smaller the range the more accurately it locks onto the position of the peaks. When there are closely spaced rings a large value can sometimes lead to the fit drifting between closely spaced peaks. The Min ring I/Ib is the minimum ratio of the intensity of the ring to the intensity of the background. If this value is too large only the strongest peak or two will be used, but if it is too small the program cannot tell the peaks from the background and the ring positions will just drift around. This always needs to be reduced, a value between 1.6 and 3.0 is usually good.

The program has a method to find the peaks if the sample to detector distance is not known well. This is the calibrate command under the calibration tab. With this method the first peak can found by marking several points on it. As the wavelength and distance are usually already known to a good approximation it is usually possible just to skip to using the recalibrate command. Below are some steps to follow for calibrating data collected in the two types of configuration typically used.

The following procedure has been found to generally work well for data collected in a high resolution configuration. For the first step just refine the two beam centers and the distance, leave the tilts and penetration fixed as they are only small corrections. Change the Min I/Ib to 3 and leave it as this value for the whole refinement. Change the min d-spacing to 0.75. The pixel search range can be left at 10 for the first iteration. Go to the calibration tab and select recalibrate. Repeat this until the calculated and actual peaks show good overlap. Manually adjust parameters if the starting configuration is too far off for the program to find the peaks. The distance affects how close in or far out the peaks are and the centering affects where the rings are centered. Once a fairly good fit is obtained reduce the pixel search range to 2, add in the two tilt parameters, and then do recalibrate once. Then add in the penetration and continue recalibrating until the parameters no longer change.

Calibrating data collected in PDF configuration is trickier as peaks of significant intensity do not extend to the edge of the detector. Start by making the minimum d-spacing 0.55, the pixel search range 5, and the

I/Ib 2. Do recalibrate to find the approximate beam centers and distance. Once this looks reasonably good, reduce the pixel search range to 2, change the min d-spacing to 0.45, add in the tilts, and then do recalibrate once. Then add in the penetration and continue to recalibrate until the parameters do not change anymore. The "show ring picks?" box can be used to see what rings the program is finding. If it is not finding much of the outer rings the I/Ib can be reduced to 1.8 or even 1.6.

Once the calibration is complete the next step will be to integrate the data. The controls for this are on the upper right of the image controls. First, un-check the "do full integration" box. It is also helpful to click the "show integration limit" box. An inner 2-theta value should be chosen which excludes the shadow of the beamstop but does not exclude any features in the data. The choice for the outer 2-theta depends on the type of data collected. For high resolution diffraction data it is generally fine to use most of the partial rings that are observed in the corners of the detector. If there is a high level of graininess or texture then peaks generated from the corners of the detector may not have accurate intensities. If the rings or smooth it should be fine to use this data. It is still best to exclude the data from the very corners as these data have low statistics, poorer powder averaging, and a large portion of the pixels which make them up are at the edge of the detector. For PDF data the artifacts caused by the pixels at the edge of the detector are amplified as the data multiplied by Q prior to Fourier transform. Therefore, for PDF data it is generally best to integrate to only just before the edge of the detector.

The "Start/End azimuth" can be used to exclude the shadow of the beamstop holder. As this appears at the top of the screen, to wrap around the end value must be higher than 360 ˚. Good starting values are usually 100˚ and 440˚. These can then be tweaked so that none of the inner most rings of the diffraction pattern which are being shadowed are included. The number of bins can be set at 3600 if data is being integrated into the corners and 2600 if it is being integrated only to just before the edge of the detector.

It is also possible to create pair distribution functions (PDFs) using GSAS-II. To do this 1-D patterns for the sample and for the empty Kapton capillary are needed. Go to "Calculate" and select "Setup PDFs". Then select the data file for just the sample (but not the Kapton yet). Under PDF controls enter the composition, select the Kapton file for "Container File", and enter the capillary thickness for "Sample diameter". Optimize PDF will generate I(Q), S(Q), F(Q), and G(r) files. You can look at the S(Q) and F(Q) to decide what a reasonable value of Q_{max} is. If things look strange a first thing to try is setting the flat background to 0 and then running optimize PDF again.

5.0 Detector Descriptions and Comparisons

5.1 The Varex Detector

The Varex XRD 4343CT is a large area detector with a total active area of 43.2 × 43.2 cm^2. There are 2880 × 2880 pixels, giving a total of about 8.3 million pixels. Each pixel is 150 × 150 μm. It works by using a CsI scintillator to absorb X-rays which then emit visible light which is absorbed by amorphous Si to create an electrical signal. It is meant to operate in the energy range of 20 keV to 15 MeV. Its quantum efficiency is 80% or above for energies up to 60 keV, and drops to a little under 50% at 90 keV. It has its own power supply and is turned on and off from a button on this supply. A data signal cord and a ground are also

attached to it. The detector has several gain settings. For gain higher numbers mean less amplification of the signal. To help avoid issues with saturation the gain is usually set at the lowest value of 8 pf, although sometimes the second lowest setting of 4 pf is used when the signal is weak. The highest gain setting should not be used as up to 40% of the dynamic range can be used up by dark current at this setting. More details can be found in the owner's manual, which is on the beamline computer.

The electronics surrounding the detector area do not come with built in shielding and can be damaged by exposure to X-rays. To protect them and ensure a long life-time for the detector a lead shield sandwiched between aluminium plates has been constructed and mounted on the front of the detector. The detector is only meant to be operated between 15 and 35 ˚C and the temperature has a large effect on the magnitude of the dark current. The hutch has air conditioning and the rapid flow of cool air within the hutch is able to keep the detector at a reasonable temperature. The dark current changes rapidly when the detector is first turned on due to temperature changes inside. It is recommend to have the detector on for at least 20 minutes prior to collecting any data that will be used for analysis.

The detector is mounted on a motorized rail which can be controlled through the SPEC command "dety". A higher Q-range can be achieved, at the expense of sampling statistics, by raising and tilting the detector 45˚. It is recommended that any movement of the detector be done by two people.

5.2 The Eiger Detector

The Dectris Eiger2 4M is a fast solid state area detector. It has 2,000 × 2,000 pixels for a total pixel count of 4 million. Each pixel is 75 × 75 μm. The active area is 155.1 mm × 162.15 mm. The detector contains gaps between the panels. These gaps are larger in one direction than the other, which is why the active area is not square despite there being the same number of pixels in each direction. When X-rays are absorbed by the CdTe material this directly creates an electrical signal. As such, there are no gain settings and there is no dark current. The counts obtained should correspond to the actual number of photons registered. It operates with a continuous readout.

The Eiger is mounted on three motorized stages which allow it to be moved up, inboard, and along the beam direction. The detector is connected to a water chiller which should be on when the detector is operating. The chillers purpose is just to keep the detector near room temperature. As it does not cool the detector below ambient temperature it is fine to collect data right after it has been turned on. It also has a port for dry air on an inert gas. It is important that the humidity inside stays below 40%, so a slow flow of dry gas over the pixels keeps the humidity down. However, the humidity is usually well below this level in the hutch so the detector is often run without the gas flow.

5.3 Choosing a Detector

The main advantages of the Varex are its large size and the large number of total pixels it has. The large number of pixels means that better resolution can be obtained at a given distance, while the large overall size allows the detector to be placed far back and still cover a large angular range. This combination makes

it possible to obtain diffraction data which has both good resolution and covers a large range of d-spacing. This detector is also advantageous for PDF measurements as it can cover over 50˚ of 2Θ when brought close to the sample, allowing a large Qmax. The larger pixel size and overall large size can also be advantageous when measuring thick samples as the detector can be placed far away, which minimizes peak broadening due to sample thickness. The QXRD interface is also well developed and easy to use.

The Varex also has several large drawbacks. The detector has memory, which means a small amount of signal from the last image will still be read after the signal is gone. It is necessary to wait between images for this ghost signal to fade, which can slow down data collection. The detector also has a large dark current which must be measured and subtracted. This dark current fluctuates slightly over time so it is necessary to occasionally collect new dark images. The detector is also slow, being able to read out no faster than 1/16 of a second. As such it is possible to saturate and loose counts for some pixels.

The Eiger has several advantages. One is the lack of any image memory is the largest one. There is also no dark current; when the X-rays are off there should be no signal at all, except for the occasional cosmic ray. It is also a fast detector which reads out continuously, making it nearly impossible to saturate. It also has no pixel spread function, meaning that signal hitting a pixel should never cause any reading in a neighboring pixel. It's smaller pixel size can be an advantage, as it allows the detector to be closer to the sample. This can be beneficial if the XRD data is desired at an energy which has a focal position near the end of the translation table.

The main drawback of the Eiger is its small size, which limits the angular range that can be collected. While the detector can be moved closer to the sample due to its smaller pixel size, as it has less total pixels compared to the Varex the d-spacing range will be less for a given resolution. This is partly alleviated by the fact that it is on a motorized stage. The detector can be moved so that the beam is near an edge or corner to expand the d-spacing range collected. This comes at the cost of powder averaging as only parts of the rings will be collected, but if the rings are fairly smooth this is not a significant cost. The detector also has gaps between panels. This means some sections of the rings are not observed. For most purposes this is not a significant issue, but if texture is being analyzed it could be.

6.0 Experimental Planning and Setup Considerations

The high energy beamline of the Brockhouse sector is well suited to rapid collection of powder diffraction data, collecting powder diffraction data by penetrating through absorbing samples, and collecting total scattering data to generate pair distribution functions (PDFs).

The beamline uses area detectors, which has several advantages. First, the entire diffraction pattern is collected simultaneously. Second, most of the powder diffraction ring is collected and can be integrated together to give very high counting statistics and average out small amounts of preferred orientation or graininess in the sample. These first two advantages mean that a diffraction pattern can be collected very quickly but still have very high counting statistics. It is often possible to collect data in a few tenths of a second or a few seconds and still have very flat backgrounds. The setup can be optimized for either resolution or d-spacing coverage by altering the sample to detector distance. High resolution can be

achieved but it is not possible to get very narrow peaks using an area detector. If very high resolution is required, it is better to use the Huber end station at the low energy beamline.

6.1 Choosing Energy and Sample to Detector Distance

The two main parameters to choose when planning an experiment are the energy of the X-ray beam and the distance between your sample and the detector. The higher the energy the smaller the d-spacing that can be accessed. In other words, high energy leads to greater d-spacing coverage (higher Q range). However, as more d-spacing is covered the more the peaks are squished together and the lower the resolution of the peaks. Lower energies have less d-spacing coverage but provide better separation of peaks. Another consideration is that the higher the energy the lower the flux of photons. The flux drops by a factor of roughly 3 for every 10 keV increase in beam energy. However, higher energy X-rays are more penetrating and are needed for thick or highly absorbing samples.

The sample to detector distance can also be used to adjust the resolution and d-spacing coverage of the diffraction pattern. A long sample to detector distance will lead to fewer but sharper peaks (smaller d-spacing coverage but higher resolution) while a short distance will lead to more peaks but greater peak overlap (large d-spacing coverage but lower resolution).

When choosing an energy it is also important to avoid absorption edges of the elements in the sample. Being at or just above an absorption edge will increase the absorption of the sample and also increase the background due to fluorescence from the sample. The energy should be either at least 0.5 keV below the absorption edges or at least 50% above the edge. In some cases it might not be possible to meet this criteria for all elements. For the energies used at this beamline only samples with elements that are heavier than Mo need to have their absorption edges checked. The absorption edge energies can be found online in several places, including this website:

http://skuld.bmsc.washington.edu/scatter/AS_periodic.html

6.2 Typical Configurations

Standard Powder Diffraction: One common setup is measuring high resolution diffraction data on powder samples contained in Kapton capillaries. For these measurements an energy of 30 or 35 keV is typically used. There is very high flux at these energies and these lower energies are well suited for high resolution data. For 30 keV a sample to detector distance between 400 and 650 mm is typically used, depending on if resolution or d-spacing coverage is being prioritized. If a good balance between the two is desired a distance around 500 mm should work well. At 35 keV the range is 450 to 700 mm, use around 600 for standard measurements. For these lower energies the slits behind the monochromator can be kept small to decrease the divergence of the beam, as there is plenty of flux even with small slits. Typical slit settings are either 0.5 × 2 mm or 1 mm × 3 mm. For samples with significant concentrations of very heavily elements (typically 3rd row transition metals or heavier) or with absorption edge just below these

energies it might be necessary to use 55 keV with a sample to detector distance around 950 mm to reduce absorption.

Penetrating powder diffraction: One great advantage of using high energy X-rays is their ability to penetrate through thick samples, giving bulk structural information or allowing changes in devices to be monitored *in-operando*. Such measurements are typically made using 35, 55, or 70 keV X-rays. These energies are useful as they are high enough to be very penetrating and the focal positions for the X-ray beams also fall at positions far enough back on the translation table to allow long enough sample to detector distances for collecting high resolution powder diffraction data. 35 keV off the 111 mono reflection can be used for many less absorbing samples and has the advantage of having higher flux. 55 keV off the 422 mono reflection is probably the most used energy for this purpose. This energy is very penetrating for most samples and has been found to work well for battery experiments. For highly absorbing samples 70 keV off the 533 reflection can be used with a sample to detector distance around 1.1 meters. This will penetrate through almost any sample. The disadvantage of using this energy is that the lower flux and very long distance makes the measurements take longer, often several seconds to several minutes. This is still not a very long time, so as long as the sample is not rapidly changing it works well.

For thick samples absorption is not the only concern. The thickness of the sample will also cause a broadening of the diffraction peaks, as scattering from different parts of the sample along the beam direction occur at slightly different angles. If the sample is greater than ~2mm thick it is advisable to use higher energies (55 or 70 keV) as they allow for longer sample to detector distances while maintaining a reasonable d-spacing coverage. The smaller the thickness of the sample relative to the distance to the detector the smaller this broadening will be.

High Pressure Diffaction:

A special setup is available for high pressure experiments using a diamond anvil cell (DAC). This setup uses a collimator and pinhole to reduce the beam size. The collimator/pinhole setup is mounted a breadboard that can be moved onto the translation table as a single piece, thereby speeding up the setup. The pinhole has several holes of different sizes. There is a second dedicated Kozhu stack that is used for holding the DAC.

LPSD Cleanup Aperture Assembly, February 2018
Material: Re, 250 µm thick, unpolished

Aperture diameters: 20, 30, 50, 75, 100, and 150 µm

Alignment slot, 50 µm wide, about 1.0 mm long
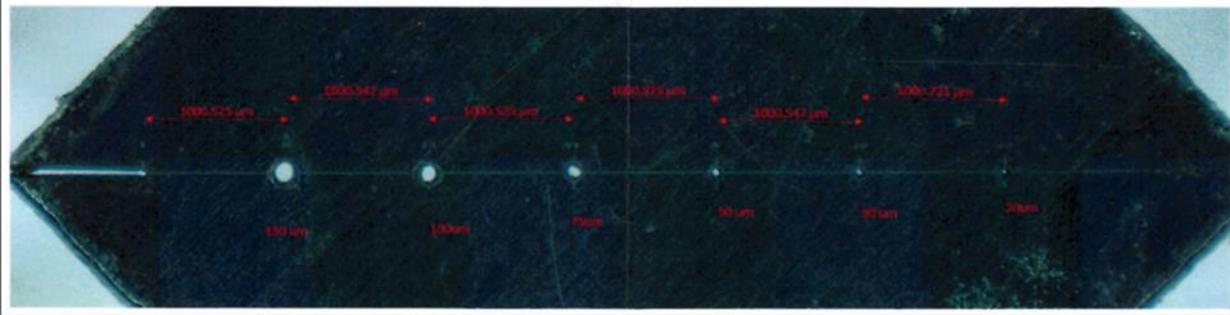
A special software called BXDS Bluesky has been developed to facilitate high pressure measurements. It can be used for alignment scans as well as centering scans for the DAC. Data collection can also be done while rotating the DAC to increase powder averaging. It made to interface with the Eiger detector. A separate manual is available at the beamline with details on how to operate this program.

PDF configurations:

The resolution of a PDF is determine by the maximum Q value used to generate it, where Q is $2\pi/d$. As such, PDF data is typically collected using high energies and short sample to detector distances. It is possible to obtain distances around 130 mm, although we usually use around 160 mm. The most common energy used is 60 keV off the 422 mono reflection. It is also possible to use 55, 65, or 70 keV off the 422 mono reflection. We can use 80 keV off the 533 mono reflection to obtain very high Qmax values, although the flux is lower in this case so this is only advisable for more strongly scattering samples and when the very high resolution is needed to answer the scientific question.

When choosing an energy there are several factors to keep in mind. The higher the energy the higher the Qmax and therefore the sharper the peaks will be in the PDF. However, higher energies also lead to lower resolution of the diffraction pattern, which leads to a more rapid dampening of the PDF. If the sample is amorphous this is not a concern as the PDF will fall off quickly anyway. If the nearest neighbor distances are the primary interest then this is also not a concern. However, if medium range order out to many 10's of Å is desired then too high an energy should not be used. The flux also decreases as the energy is increased, so for more weakly scattering samples a very high energy should not be used.

It is also important to make sure the Q_{min} does not become too large. If a strong high d-spacing peak is lost behind the shadow of the beamstop this will create a long wavelength artifact in the intensity of the G(r). If the sample has a larger unit cell it might be necessary to give up a little Q_{max} by putting the detector a little further away to make sure all the peaks are captured. If a high Q_{max} is still need when very low Q peaks are present the detector can be vertically offset to still collect high Q data even at a slightly longer distance.

When using 60 keV data is typically available out to about 27 Å^{-1}. For many samples the data needs to be cut down as the noise at high Q is too high or the normalization is imperfect and the F(Q) does not go flat. A typical usable range is around 21-25 Å^{-1}, although sometimes the full range can be used.

6.3 Choosing a Capillary Size

In general, the larger the capillary the more sample is in the beam, the stronger the signal, and the better the powder averaging is. Larger capillaries are also easier to load. However, the thicker the capillary the higher the absorption. A thicker capillary will also lead to more peak broadening, although this effect is quite small. On the high energy beamline capillaries with inner diameters ranging between 0.5 and 1.8 mm are typically used. 0.81 mm capillaries are the most commonly used size and work well for most samples. If the sample has a higher absorption or getting the very sharpest peaks is a priority 0.63 mm capillaries can be used. 0.5 mm can be used if the sample is very absorbing or there is limited sample available. For samples with only lighter elements 1.0-1.8 mm capillaries allow more signal, especially when higher energies are being used such as for PDF experiments.

The absorption of the sample at the energies to be measured should be checked prior to choosing a capillary size. The APS has a nice online calculator for doing this:

https://11bm.xray.aps.anl.gov/absorb/absorb.php

The APS calculator is quick to use but guesses at the sample density and can sometimes overestimate it by a factor of as much as 2, giving absorptions that are higher than they really are. The MAC program available on the data analysis computer can also give more accurate values if the density of the sample is known.

A μr less than 0.5 is ideal and considered negligible. Values between 0.5 and 1.5 are low and will also work fine. Values between 1.5 and 3 are acceptable but somewhat undesirable and should be avoided if this is easily achievable by using a thinner sample or different energy. If the value is 3 or higher a different energy or thinner sample is strongly recommended.

6.4 Higher Harmonics

A particular X-ray energy is obtained by rotating the Si single crystal monochromator to a specific angle that satisfies Braggs law for that energy. However, any multiple of that energy will also satisfy the Bragg condition as well. This means that we are not truly selecting a single wavelength but a set of harmonics. These higher energy harmonics will also diffract and produce additional peaks. In practice, peaks from these higher harmonics are always much weaker than those produced from the primary energy. This is for several reasons. The first reason is that higher harmonics are less efficient at diffracting from the mono crystal than the primary harmonic. The second reason is that the flux of the higher energy X-rays are much lower than that of the primary harmonic. The third reason is that the higher energy X-rays are more penetrating and less likely to scatter off the sample, so they produce a lower signal for a given flux.

Using filters or having highly absorbing samples can increase the relative intensities of higher harmonic peaks. If a filter is used to reduce the beam intensity, the filter material will absorb a greater proportion of the primary harmonic relative to the higher harmonic simply due to the fact that the higher energy X-rays are more penetrating. This means that the flux of the beam reaching your sample will contain a greater relative flux of the higher harmonic energy. If the sample itself is highly absorbing then this can also increase the relative intensity of higher harmonic peaks for similar reasons. If the sample absorbs most of the primary harmonic but little of the higher harmonic, than the transmitted signal of the higher harmonic will be relatively higher.

When using the 111 mono reflection (typically for 30-37 keV energies) there is no second harmonic. This is because for Si the 222 reflection is systemically absent. However, very weak third harmonic peaks can usually be seen for moderate or strongly absorbing samples. This is possible as the 333 reflection is allowed and there is still a significant flux of 90-105 keV photons. These third harmonic peaks are typically very weak as the relative flux of photons at triple the energy is very small and these photons are much more penetrating. For Ni powder in a 0.63 mm capillary the third harmonic peaks are roughly 0.02% of the intensity of the primary peaks when using 35 keV. While these peaks are very tiny, the background of the data are usually so flat that they can still be seen for the most intense peaks in the pattern. As they are third harmonics, they appear at d-spacing which are three times those of the regular peaks. If energies below 30 keV are selected the third harmonics will be more pronounced. This is because below about 30 keV the flux of the beam actually drops significantly due to the filters in POE1 while the flux of X-rays with triple that energy increases. This is one reason why the 25-29 keV range is not often used.

The 422 mono reflection is typically used for energies of 55-70 keV and the strongest harmonics are seen in this situation. As the 844 reflection is allowed second harmonics are permitted. Peaks coming from X-rays with energies between 110 and 140 keV can typically be seen. For Ni powder in a 0.63 mm capillary the second harmonic peaks are roughly 0.05% of the intensity of the primary peaks when using 60 keV. While stronger than the third harmonics seen for the 111 reflection, these peaks are still very small and generally not problematic. It is still important to be aware of their existence so they are not falsely attributed to an additional phase. They will appear at twice the d-spacing of the strongest primary peaks. Third harmonics are not usually seen when using the 422 reflection as there are essentially no photons at 3 times these energies.

The 533 reflection is used to access energies from 70-90 keV and no higher harmonics have ever been observed when using this setup. As the 10,6,6 reflection is systematically absent no second harmonics are produced. As there are essentially no photons at 210 keV or higher no third harmonics are present.

7. Macros and Sample Environments

7.1 Macros

Motor movements and data collection can be automated using SPEC macros. If macros are desired please inform beamline staff at least two week prior the experiment so we have time to write them.

7.2 High Temperature Furnace

The high temperature capillary furnace can achieve temperatures up to 1200 ˚C. It works by heating a capillary of sample inside a heating coil. The beam only passes through the capillary and sample so there is no additional background. Pictures and a more detailed description are available on the beamline website.

To use the furnace it is necessary to load the sample into a quartz capillary. We normally use 2.0 mm diameter capillaries with two open ends. Powders can be loaded or solid metal pieces cut so their diagonal is smaller than the capillary diameter. If the user will run more than 4 samples they should purchase such capillaries prior to their beamtime.

It is also possible to flow gas through capillaries. As we do not have a hazardous gas exhaust system, currently only non-hazardous gases are permitted. The beamline has a regular supply of N_2 and Ar that users can use. If other gases are desired please check with beamline staff well in advance of the experiment to see if we have the gas available. If not, the user may need to purchase it themselves and have it shipped to CLS.

The furnace is controlled by the SPEC command "mv m_euro #" where # is the temperature set point. When the power to the furnace is turned on or off it is necessary to type "reconfig" in SPEC. The heating rate can be set by going typing "euro2400menu".

7.3 Cryostream

The cryostream is mainly for cooling to low temperature but can also heat to a little above room temperatures. It can access temperatures between 80 and 500 K. It has some trouble stabilizing at very low temperature so it is recommend not to go below 100 K unless it is scientifically necessary. The cryostream is very stable in temperature, although not that quick to change temperatures. It is controlled by its own app that came with the machine, called CryoConnect. We have also made macros for automated temperature control. There is a manual at the beamline specifically for using the cryostream. Since the nozzle needs to approach from the top unobstructed it is usually used for samples in capillaries mounted on the capillary spinner.