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NSLS-II Project

PRELIMINARY DESIGN REPORT for the

SUBMICRON RESOLUTION X-RAY SPECTROSCOPY (SRX) BEAMLINE AT NSLS-II

Issued September 2010

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Document Updates

The Preliminary Design Report for the Submicron Resolution X-ray Spectroscopy (SRX) beamline at NSLS-II is a controlled document, revised under change control.

Version No.	Date	Changes made
0	9/26/2010	Initial draft
1	9/30/2010	Uploaded to Sharepoint for comments from BAT and directors
Final		First official release

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Contents

Introduction

Scientific scope

Scientific communities (such as earth and environmental sciences, medical and life sciences, hard and soft condensed matter and materials sciences, chemical and energy sciences) have identified the need to develop analytical resources to advance the understanding of complex natural and engineered systems that are heterogeneous on the micron to nanometer scale. These communities specified a need for high intensity x-ray nanoprobes and made it clear in the Letter of Interest they submitted to the NSLS-II Project for the Sub-Micron Resolution X-Ray (SRX) Spectroscopy Beamline, that NSLS-II would provide one of the best sources in the world for such instruments. The research topics these scientific communities hoped to better address using such an instrument require characterization of elemental abundances and speciation in samples that are heterogeneous at the sub-micrometer scale.

The targeted scientific issues show a wide range of environmental and health issues of high societal impact. Interactions between micro-organisms and minerals control the speciation, migration and toxicity of contaminated materials produced by human activity. Micro-organisms and particulates are likely major players in the cycling of nutrients and metals in the Earth's oceans, processes that can have a significant impact on conditions at the planetary scale, such as global climate change. The properties of airborne particulates can have a profound effect on the toxicity of atmospheric dust introduced to the human body by inhalation. The study of how genetic variations in organisms affect their interactions with contaminant and nutrient metal species in the environment is in its infancy but likely to be greatly advanced with this new technology because of the ability to observe in detail the chemical modifications in organisms caused by genetic modification.

Equally significant will be studies of the varied sources, pathways and functions of metals in organisms, in particular their role in human health. Some metals are required for normal metabolic function, with optimal amounts for maximum benefit. Others are only known to cause toxic effects. Metal ions are also used both as treatments for disease and as image contrast agents within the body. Yet, in order to understand at the molecular level how metal ions function in life, disease, and therapy, a multi-dimensional approach is necessary.

Such an instrument will allow studies of catalysis and chemical processes at the scale of a single particle using coupled µXAS/µXRD of catalytic particles and interfaces to follow processes such as oxidation. In the materials sciences scientists will be able to research the elemental partitioning in microelectronics and elemental diffusion into microcrystalline domains that occur due to aging of plastics and alloys and tracking redox changes of single particle contaminants in batteries and silicon solar cells.

The SRX beamline with its unique combination of high spectral resolution over a very broad energy range and very high beam intensity in a sub-micrometer spot will be a tool very well suited for the study of the scientific issues mentioned above.

The design shows a canted undulator beamline that consists of two branches, each optimized to reach very high spatial resolution for a specific energy range. The first branch is optimized to access higher energy and is included in the initial scope of NSLS-II for the SRX beamline.

It will access an energy range of E = 4.65 keV to E = 25 keV. Mirror optics in Kirkpatrick-Baez (KB) geometry will focus the beam and create a sub-micrometer sized focal spot. The second branch, optimized for lower energies, accessing spectroscopic edges from E = 2 keV to E = 15 keV, will require additional funding to be completed. Zone plates (ZP) will be used as focusing optics for this branch. The high energy cut-off of E = 25 keV of the first branch is determined by the energy at which the brightness of the NSLS-II undulators has dropped by approximately two orders of magnitude from that of the third harmonic at closed gap. Also, the K-absorption edge of rhodium, the best suited coating for mirrors at these high X-ray energies, lies at about E = 23 keV. Consequently, reflectivity drops significant above that energy. The low energy cut-off of E = 4.65 keV is the energy at which the undulator third harmonic begins. This is the energy of the K-absorption edge of titanium, thus giving access to this element. The low energy cut-off of E = 2 keV of the second branch is set by the limits of the Si (111) monochromator. The K-absorption edge of phosphorus, an important element to study in life and environmental sciences, is however still in reach. The high-energy cut-off of E = 15 keV is determined by the need to access the L_3-absorption edge of lead at E = 13 keV.

The wide energy range covered by both branches will allow the scientific community to address a wide range of research topics, as absorption edges of a large number of elements can be reached with the SRX beamline, allowing elemental mapping as well as spectroscopy studies (see Figure 1). The two branches are required to cover this large energy range without compromising the aim of combining X-ray spectroscopy and sub-micron spatial resolution in an optimal way.

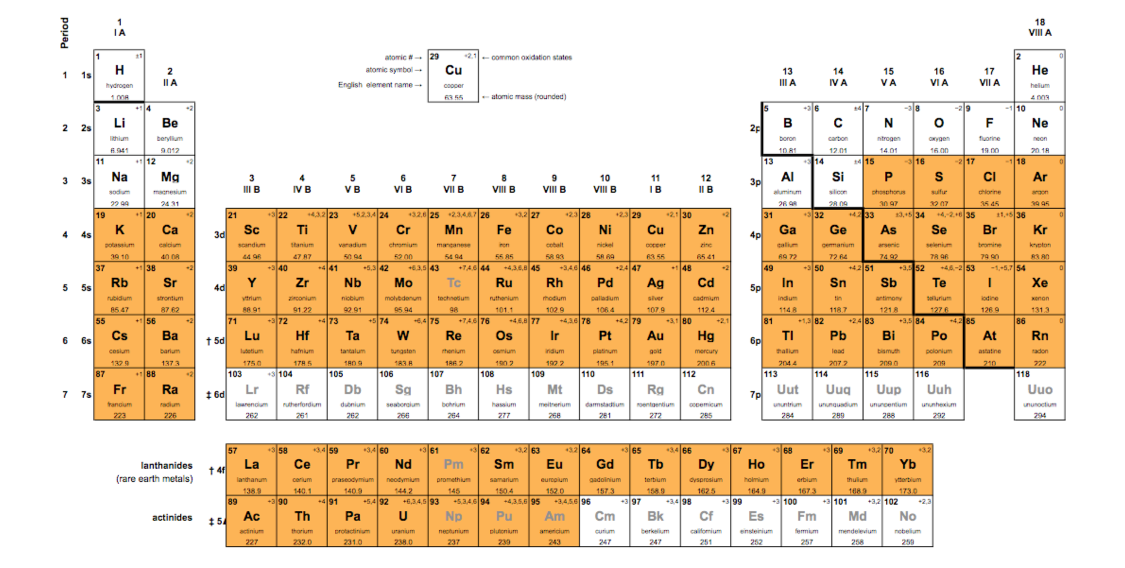

Figure 1. Periodic table of elements showing highlighted the elements with absorption edges at X-ray energies in reach by the SRX beamline.

Team of SRX Beamline

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Insertion Device

Table 1. Source Parameters of NSLS-II.

Electron Energy	E = 3 GeV
Stored Current	I = 500 mA
Electron Beam Emittance:	
Horizontal	$\varepsilon_x = 0.55 \times 10^{-9}$ m rad
Vertical (1.5% Coupling)	$\varepsilon_y = 8.25 \times 10^{-12}$ m rad
Betatron Function:§	
Horizontal	$\beta_x = 1.5$ m
Vertical	$\beta_y = 0.8$ m
Electron Beam Size:§	
Horizontal	$\sigma_x = 28.7$ µm
Vertical	$\sigma_y = 2.57$ µm
Electron Beam Divergence:§	
Horizontal	$\sigma'_{y\,x} = 19.2$ µrad
Vertical	$\sigma'_{y\,y} = 3.21$ µrad
Intrinsic Photon Size*	$\sigma_r = 1.95$ µm
Intrinsic Photon Divergence*	$\sigma'_y = 4.08$ µrad
Total Photon Source Size:§*	
Horizontal	$\Sigma_x = 28.8$ µm
Vertical	$\Sigma_y = 3.2$ µm
Total Photon Source Divergence:§*	
Horizontal	$\Sigma'_{y\,x} = 28.8$ µrad
Vertical	$\Sigma'_y = 5.19$ µrad

§ Low b straight.
*Quantities evaluated for 12.4keV x-rays and a 3m-long undulator.

The two branches of the SRX beamline are designed for different energy ranges, making the undulator design unusual amongst the initial suite of project beamlines at NSLS-II. However, it is expected that in the long run more sectors will be equipped with this design. Two undulators in canted geometry on this short (low beta) straight section will serve the two instruments as light sources. As there is the need to do spectroscopy at the titanium K-absorption edge, the specified lowest energy the undulator for the KB branch should reach is E = 4.65 keV. There are a couple of restrictions applied to the choice of the insertion device as a light source for the SRX beamline. The length of the device and the minimum gap have to be adjusted to the limitations stemming from the beta function of the short straight section (stay-clear condition). A length of 0.5 m minimum has to be removed from the total length of the straight section available for the two undulators to give room for the canting magnets as well as room has to be given to transition pieces and a full canting magnet at the end of the straight, which is also assumed to be 0.5 m long. From the Accelerator Science Division, a stay-clear minimum gap of 5 mm at a length of 1.5 m for the two undulators has been set.

KB Branch

Focusing on the KB-branch, the optimized period of the undulator will depend on the gap (itself related to the length of the device through the beta-function restrictions) and the minimum photon energy (E = 4.65 keV). Oleg Tchoubar from NSLS-II has performed the calculations presented below, which are the basis for the decision on the actual undulator for the KB branch.

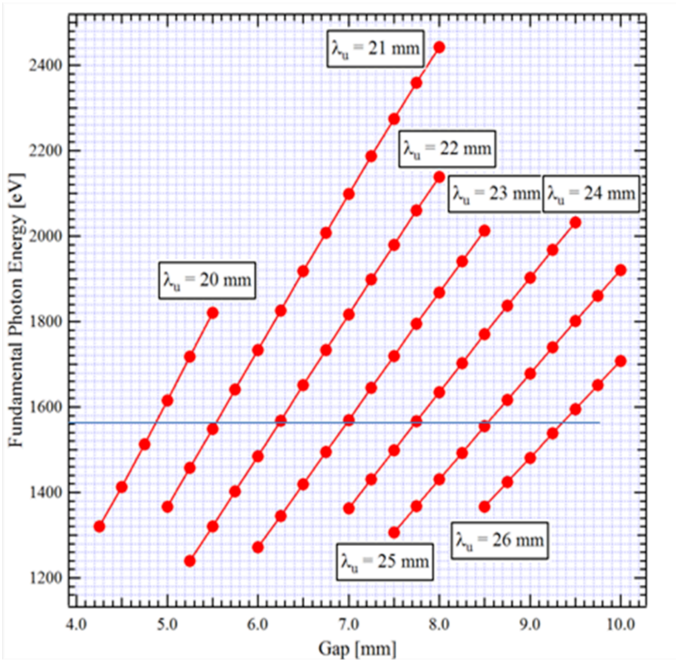

Figure 2. Fundamental photon energy as a function of the gap for IVU periods from 20 – 26 mm. The blue line indicates a fundamental energy of E = 1560 eV.

In Figure 2 the fundamental photon energy as a function of the gap for in-vacuum undulator (IVU) periods from 20 – 26 mm are plotted. The blue line in figure 2 indicates the energy E = 1560 eV, of the first undulator harmonic. With this, the 3rd harmonic would start around the desired X-ray energy of E = 4.65 keV. From the graphs in Figure 2 it becomes visible that for a minimum gap of 4.9 mm a period of 20 mm would be allowable, a minimum gap of 5.5 mm would relate to a period of 21 mm and for a gap of 6.2 mm the period would be 22 mm.

The maximum device length for a given minimum gap is determined by the beta function for the straight. Toshi Tanabe from NSLS-II calculated the possible lengths, assuming a canted device placed into a half-straight, as listed in Table 2.

Table 2. Maximum canted device lengths for given gaps in a canted device setup.

Gap	Maximum canted device length.
5.0mm	1.25m
5.5mm	1.5m
6.0mm	1.75m
6.26mm	1.85m
6.6mm	2.0m
7.2mm	2.25m

As a result of these consideration three options have been calculated, an undulator with 20 mm period and 1.25 m length at a gap of 4.9 mm, an undulator with 21 mm period and 5.5 mm gap and 1.5 m length, and an undulator with 22 mm period and 6.2 mm gap and 1.8 m length. For these calculations the center of the device is located approximately 1 m from the center of the low-beta straight, resulting from the design described above.

Figure 3 shows the spectral flux in phot/sec/0.1%BW for these three configurations at odd harmonics as a function of photon energy. Figure 4 shows for the same configurations the calculated spectral flux through an aperture of 0.1 x 0.1 mrad^2 at minimal gap. As a result and taking the stay-clear minimum gap of 5.5 mm into account, an IVU21 is the undulator of choice. Table 3 summarizes the parameters. Figure 5 shows the flux coming from this undulator in Watts/eV/mm^2 as a function of X-ray energy for odd and even harmonics, calculated using the SRW program package of Oleg Tchoubar.

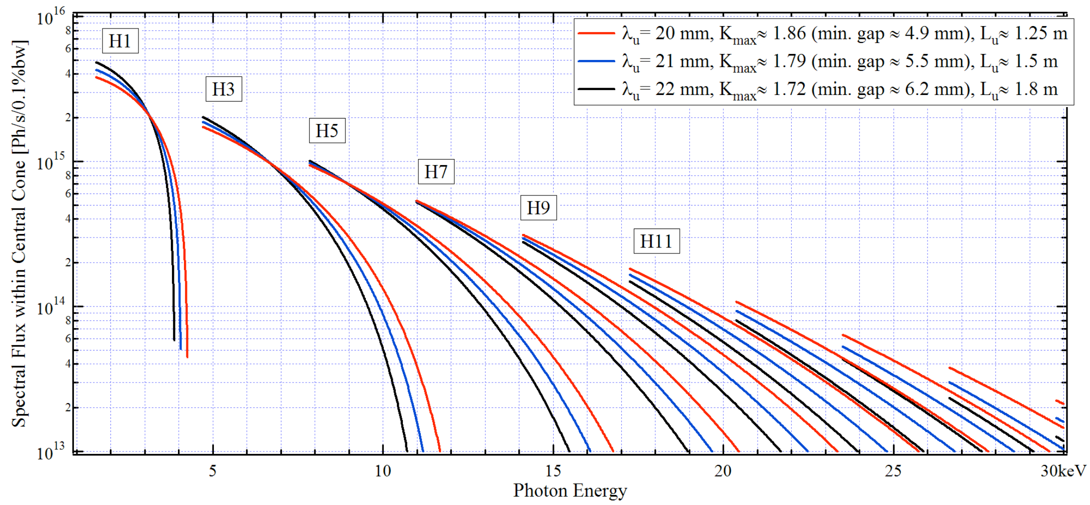

Figure 3. Spectral flux at odd harmonics for undulator setups as seen in the legend.

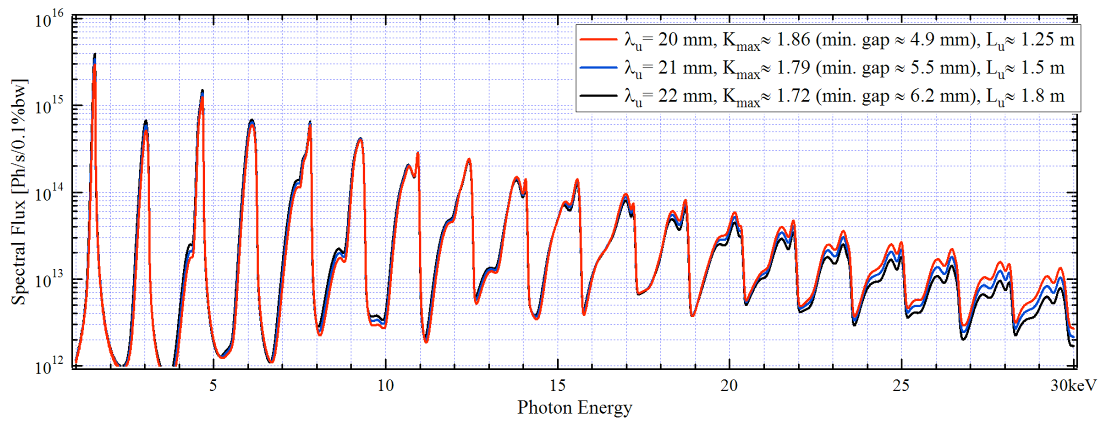

Figure 4. Spectral flux through an aperture with 0.1 x 0.1 mrad^2 size at minimal gap for

undulator setups as seen in the legend.

Table 3: Parameters in-vacuum undulator U21.

Undulator length	1.5 m
Period length	21 mm
Minimum gap	5.5 mm
Lowest energy in 3rd harmonic (E=3GeV)	4.65 keV
Maximum energy	28 keV
K-Value	1.79

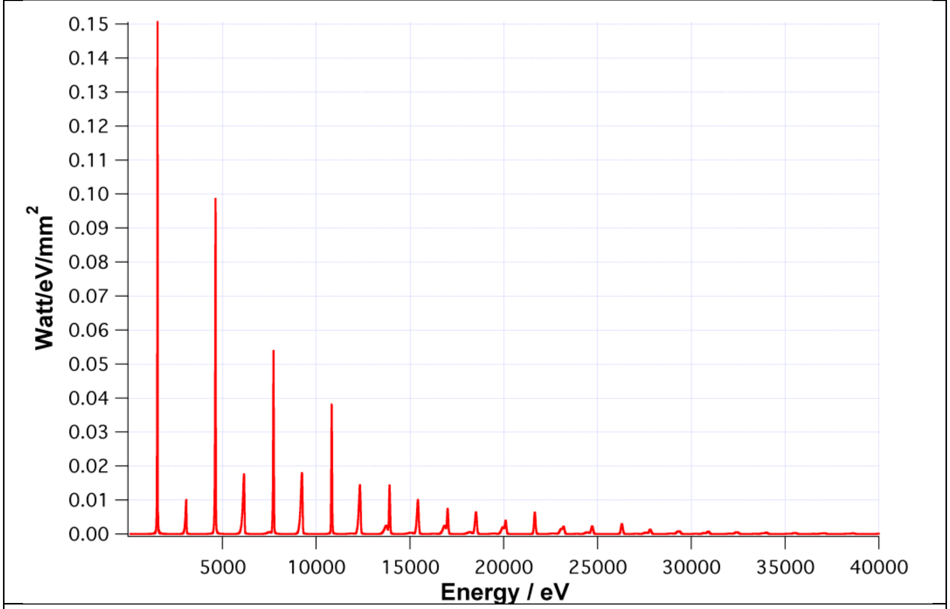

Figure 5. Spectrum of the undulator IVU21

ZP Branch

The ZP branch is dedicated to an energy range from E = 2 keV up to E = 15 keV. From figure 3 it becomes clear for undulator types IVU 20, IVU 21, or IVU 22, again considering the allowable minimum gap, that due to these limitations there will be an energy range between E = 4 keV and E = 5 keV, where no radiation can be used for experiments. From the viewpoint of scientific applications this cannot be accepted, therefore a solution has to be found to close this energy gap. Several ways to solve this problem are possible. One solution would be to shorten the length of an undulator IVU 22 to 1.2 m. This would allow the minimum gap to be reduced without deteriorating the electron beam. Due to the resulting higher magnetic field strength the full desired energy range would be covered. However, due to the shorter length, the overall performance in terms of photon flux will be poorer. Another solution would be to use a cryo-cooled undulator. Due to cryo-cooling a higher K-value would be achievable resulting in a full coverage of the desired energy range without hampering the electron beam. A super-conducting undulator could be another solution for filling the energy gap. A decision has not been made yet, as the ZP branch is not in the baseline scope of the NSLS-II project. Figure 6 shows the spectral brightness as a function of X-ray energy for bend magnet, wiggler and undulators as sources of X-radiation. This plot will be the starting point for discussions about the right choice of insertion device. As the CPMU17 shown in figure 6 still has a gap at medium X-ray energies, probably a higher K-value above 2 and a longer period length will be a solution.

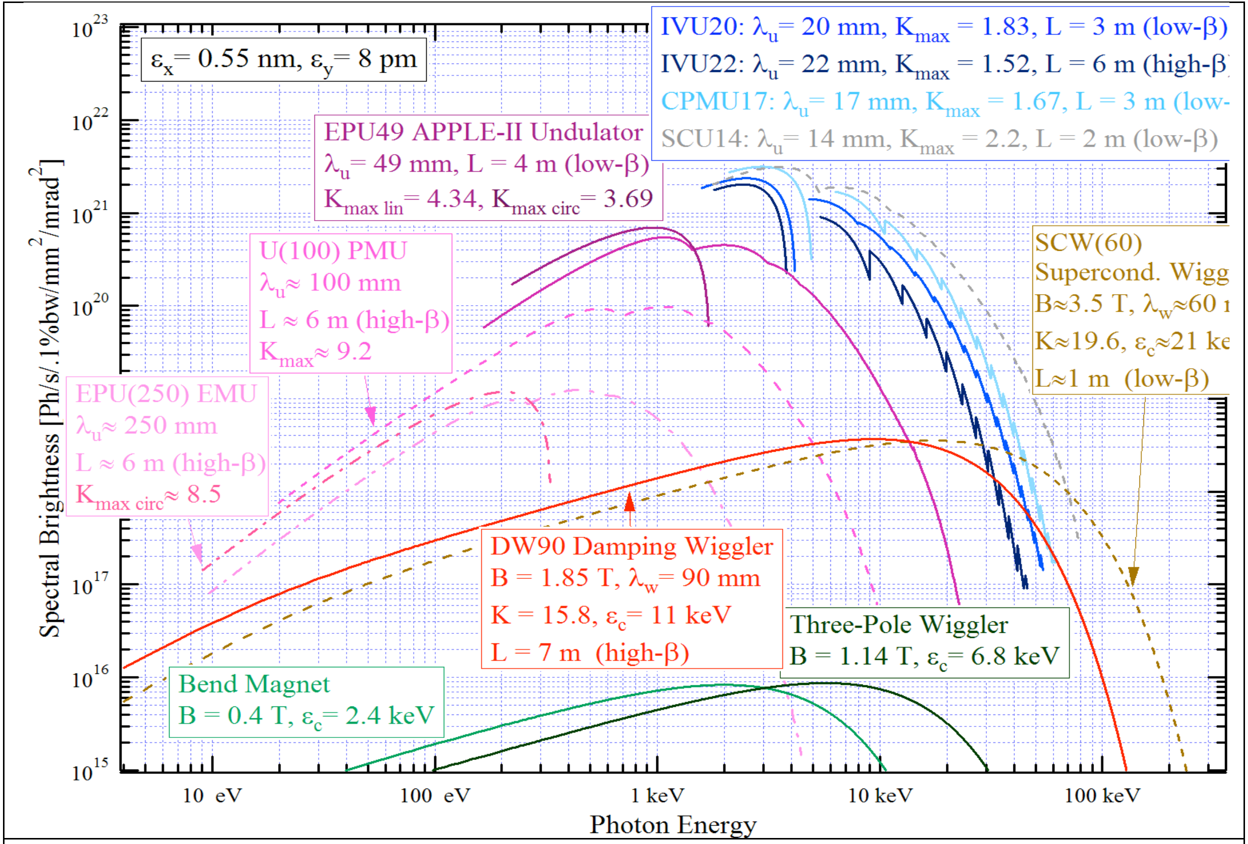

Figure 6: Spectral Brightness of different NSLS-II sources for X-radiation.

IVU 21 Scanning Requirements for the KB Branch

The SRX beamline is dedicated to X-ray spectroscopy with high spatial but as well high spectral resolution. To achieve the task of measuring spectra with stable high beam intensity, with very precise energy tuning and with reliable reproducibility, the undulator control has to be very accurate. The undulator gap needs to be changed during the experiment to obtain a usable spectrum of a sample. The following calculations have been made to determine, what requirements are needed to perform reasonable XANES- and EXAFS-spectroscopy. Significant contributions to these calculations came from Roger Dejus, Matt Newville and Mark Rivers, all from the Advanced Photon Source at Argonne National Laboratory, and Antonio Lanzirotti, Univ. of Chicago/NSLS.

Figure 2 shows the fundamental photon energy as a function of the gap G for different IVU periods. Looking at the curve for l = 21 mm, the two end points of the line are:

$$G = 5.0 \text{ mm} \quad \Rightarrow \quad E = 1370 \text{ eV}$$
$$G = 8.0 \text{ mm} \quad \Rightarrow \quad E = 2440 \text{ eV}$$

For the following calculation it is just to determine the slope, assuming a straight line, which results in

$$\Delta E/\Delta G_{1st\ order} = 0.357\ keV/mm$$

The slope of the so-defined line for the n-th order is the now calculated using the slope of the 1st order multiplied with n.

The full width half maximum (FWHM) of single harmonics of IVU 21 have been determined using the result of the SRW calculation, which is shown graphically in figure 5. With this and to cover the extremes, the FWHM of the 3rd and the 17th harmonic have been determined to

3rd harmonic at E = 4635 eV $\Rightarrow$ FWHM = 35 eV corresponds to 0.75%
17th harmonic at E = 26300 eV $\Rightarrow$ FWHM = 130 eV corresponds to 0.50%

To cover these FWHM's or the σ-values the following gap changes are necessary:

3rd harmonic: ΔG_{FWHM} = 32.45 µm, $\Delta G_{Sigma} = \Delta G_{FWHM}$ / 2.35 = 13.81 µm
17th harmonic: ΔG_{FWHM} = 21.65 µm, $\Delta G_{Sigma} = \Delta G_{FWHM}$ / 2.35 = 9.21 µm

To achieve useful spectroscopy data, it would be necessary, to allow fluctuations in the intensity of the maximum of an undulator harmonic of only 1%. Assuming a Gaussian shape of the harmonic, this would demand a positioning accuracy of 14% of the σ-value,

3rd harmonic: $\Delta G_{1\%}$ = 1.9 µm
17th harmonic: $\Delta G_{1\%}$ = 1.29 µm

Therefore, to achieve reasonable spectroscopy data across the covered energy range, a minimum stepsize of 1 µm in driving the IVU21 undulator will be necessary.
The minimum gap of IVU21 allowed by the stay-clear constraints of the Accelerator Science Division is G = 5.5 mm, corresponding to an energy in the 1st order of E = 1560 eV, see figure 2. As the X-ray energy of a higher harmonic is obtained by multiplying this fundamental energy with the order n of the harmonic, this gap corresponds to an energy of E = 4680 eV in the 3rd harmonic. The K-absorption edge of rhodium lies at E = 23.2 keV, which is probably the highest X-ray energy useful for experiments, as the reflectivity of the mirrors will drop drastically above that edge. This would be reached at the same gap in 17th harmonic, reducing however significantly the usable beam intensity. At a gap of G = 8.0 mm, the fundamental energy lies at E = 2440 eV, corresponding to the 9th harmonic for energies above E = 21 keV. This assessment clarifies, that practically all spectroscopy experiments will need gap changes in the gap range of G = 5.5 mm up to G = 8.0 mm.

The following list summarizes the above mentioned and further parameters necessary for the undulator IVU21. A homonymic list can be found in the RSI (Requirements, Specifications, and List of Interfaces) document for the SRX beamline.

Gap Adjustment for Energy Positioning

- Range of gap: 5.5 mm to maximum gap.
- Minimum reproducible step: less than 1 micron.
- "Stepping time," time for accomplishment and confirmation of position and parallelism at minimum step: less than 1 sec.
- Position readback precision: smaller than 1 micron.
- Speed: greater than 1mm/min

- Parallelism at each position as required for less than 1% variation in intensity .

Gap Adjustment for Energy Scanning
- Range of gap for energy-scanning: 5.5 to 10.5 mm
- Minimum reproducible step: less than 1 micron
- Position readback precision: less than 1 micron
- Position readback rate: greater than 1 khz
- Continuous smooth scanning speed (min., max.) range: 10 to 1,000 microns/minute
- Parallelism maintained during continuous scanning motion: as required for less than 1% variation in intensity

Motion Control
- Gap under beamline control
- Parallelism maintained by machine and/or feedback loop, but can be checked by beamline
- Elevation under machine control

Duty Cycle
- Periodic stepping at 0.1 sec to ~4 hour frequency
- Repeated continuous-motion energy scans (0.02 to 2.0 mm in length) for >24 hours

Front End

The design of the front end for the SRX beamline is not finished yet, therefore only interim design considerations can be presented here. The preliminary layout of a front end designed by NSLS-II for two undulators in a canted geometry can be seen in Figure 7. All elements shown there will accommodate both beams from the two undulators. It is taken into account, that the control of the two beams for the two branches needs to be independent, meaning that a change in parameters of components for the KB branch should not cross-talk to the ZP branch and vice versa. The layout incorporating these constraints and special demands is work in progress. A list of the components can be found in the next chapter, a first ray tracing is attached in Appendix A.

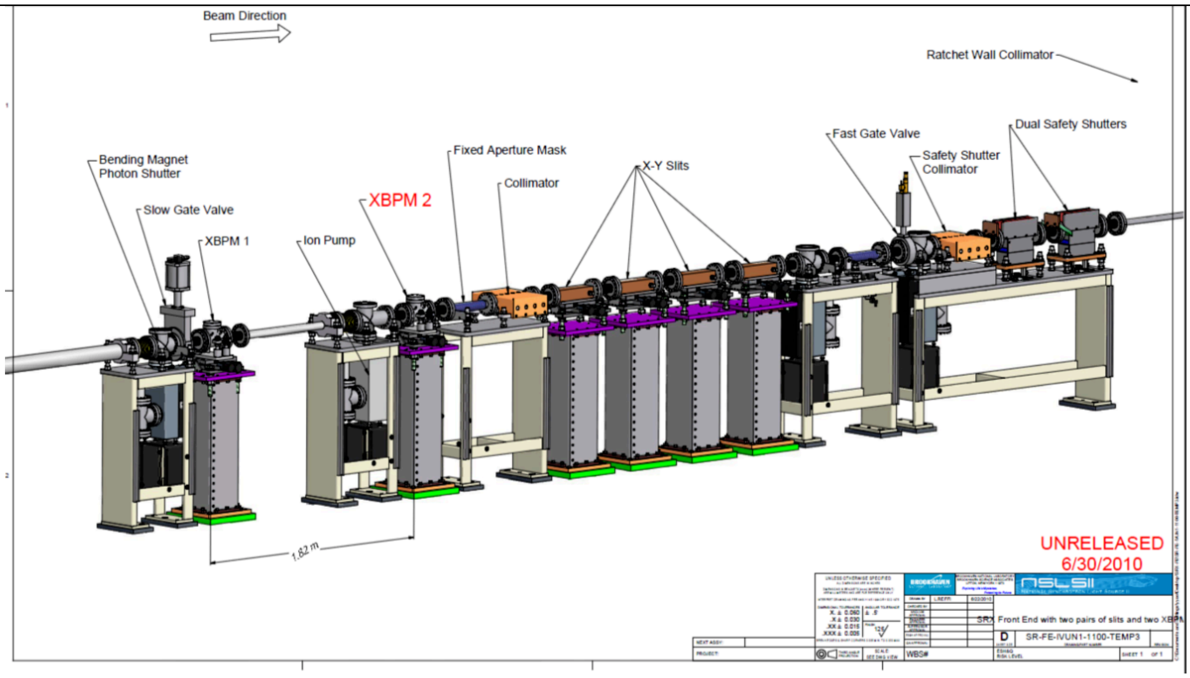

Figure 7: Model of components of front end for the SRX beamline, having two undulators in canted geometry as independent X-radiation sources.

Description of components

Bending Magnet Photon Shutter (BMPS)
The BMPS is designed to protect the slow gate valve (SGV) from bending magnet radiation before the upstream straight is fitted with an insertion device and a complete front end.

Slow Gate Valve (SGV)
The SGV is included to isolate the machine and front end, but will not withstand white beam from insertion device or bending magnet radiation. The SGV is controlled and monitored by storage ring vacuum PLC using a voting scheme with inputs from vacuum sensors at both sides of the valves and position of BMPS.

Beam Position Monitor 1 (XBPM1)
The XBPMs shall be designed to work with the insertion devices. It will consist of tungsten blades and water cooled mountings and it will be motorized to allow centering of the device around the beam. The XBPMs shall be mounted on X/Y stages with the very high position stability in the 1 µm-range and a position resolution well below 1 µm.

The X/Y stage for the XBPM and the X/Y slits are expected to be the same design, including the stand, where possible.

Beam Position Monitor 2 (XBPM2)
This device and X/Y stages shall be identical to XBPM1, however the blades shall be relocated to avoid masking effects.

Fixed Aperture Mask (FAPM)
The fixed aperture mask shall provide radiation fans to the first optical enclosure (FOE). No tolerance shall be added to the mask for mis-positioning, however a manufacturing tolerance

of +/-0.2mm for the aperture (at the downstream end of the mask) shall be included in the downstream fan definition. For IVU beamlines, it is permissible for the mask aperture to have corner radii equivalent to half the aperture height.

Bremsstrahlung Collimator (BC1)
BC1 restricts the bremsstrahlung radiation fan exiting the shield wall. This should be as tight to the beam as is reasonable without undue mechanical tolerances or alignment difficulty.

X-Y Slits
The X-Y slits shall be of the SPring8 dual "L" type design, connected with bellows to allow full adjustment of all four "blades" via two X-Y stages. General specifications are: The material will be water-cooled Glidcop with Tungsten blocks. A pre-mask may be included for power protection. The maximum opening angle shall be sufficient to allow full FAPM fan to continue to the FOE without clipping. The slits will be motorized to allow selection of any part of the FAPM fan. As mentioned, the same X/Y stage shall be used for the XBPMs. The aperture stability shall be in the 1 µm range. This aperture stability specification is governed by the differential movement between the two X/Y slit units. For the high stability stages some form of additional coupling between stands may be required to constrain any differential movement.

Two sets of slits will be needed to operate independently both, the KB-branch and the ZP-branch. It has to be ensured that manipulating the beam of one branch does not influence the other branch.

Fast Gate Valve (FV)
The fast gate valve is to shut within a few milliseconds once triggered by FV sensors located in the front end and beamline whenever there is a sudden increase of pressure of a few decades. The stored beam has to be dumped prior to FV closing, and the cause then investigated and mitigated.

Safety Shutter
The safety shutter is actually a pair of shutters, required for redundancy, air actuated with independent redundant and diverse position sensing.

Preliminary ray-tracing

The design of the front end is not yet finalized, therefore a concluding ray-tracing cannot be presented at this time. However, in Appendix B the results of a ray-tracing calculation using a standard arrangement of the front end components is presented.

Beamline Design

Optical layout

The general concept of the SRX beamline is to offer two end stations where x-ray spectromicroscopy experiments can be performed with a spatial resolution in the sub-micrometer range. The energy range from E = 2 keV up to E = 25 keV will be covered. This concept will be realized by dividing the beamline into two branches, each served by an optimized, in-vacuum undulator with the two insertion devices arranged in a canted geometry. One branch will be equipped with focusing mirrors in a Kirkpatrick-Baez (KB) setup. As described later in this document, two sets of KB's will offer sub-µm focal spot sizes, where one set gives high flux at moderate resolution and the second set aims for a spatial resolution in the range of 100 x 100 nm^2. Using a slit system as a secondary horizontal source, the user of the SRX beamline will be able to choose between high flux or very high spatial resolution, depending on the application. By changing the slit settings, it will be possible to a certain extend to tune the focal spot size, as to better accommodate certain samples. The KB branch will operate with X-ray energies ranging from E = 4.65 keV up to E = 28 keV. The maximum of flux that can be delivered to the focal spot will be around 1.5 x 10^{13} phot/sec, and even with the highest possible spatial resolution 3.6 x 10^{11} phot/sec will be possible, according to the calculations. The second branch will use a Fresnel zone plate to reach a spatial resolution in the range of 30 nm or smaller in the energy range of E = 2 keV up to E = 15 keV, where the expected flux will be up to 10^{10} phot/sec. Photon flux and spatial resolution in that energy range will be unprecedented when comparing these key parameters to similar beamlines worldwide, which is due to the excellent performance of NSLS-II. The KB branch is a project beamline of the NSLS-II project; the ZP branch is not in the original scope. To ensure that a reasonable design and layout for the latter will be available when an approving decision will be due as well as to avoid mistakes when placing critical optical components of the KB branch, the design of the KB branch has to take basic design results for the ZP branch into account, as will be described in this report. A schematic layout of both branches starting with the two canted undulators and showing major optical components of the two branch lines is seen in figure 8. Note, that the angle between the two branches is exaggerated for illustration purposes.

For stability and performance reasons the double crystal monochromator for the KB branch will be horizontally deflecting. A positive side effect is an increase in distance between the two branches. The horizontal focusing mirror creates at the position of the secondary horizontal source aperture an image of the source. The image of the HFM is the source point for the horizontally deflecting KB mirror located downstream, whereas the vertically deflecting KB mirror is still looking at the original source. The KB mirrors will create a demagnified image of these both source points as a focal spot through which the sample will be scanned. A variety of detectors will be available for fluorescence, transmission and diffraction measurements. In the ZP branch two horizontally outward deflecting mirrors will create a secondary source spot as well, which is then again the source point for a zone plate creating a very small focus at the position of the sample. The two deflecting mirrors advantageously increase the distance between the branches even more, creating a manageable distance of about 57 cm to the KB branch at the downstream end of the ZP branch.

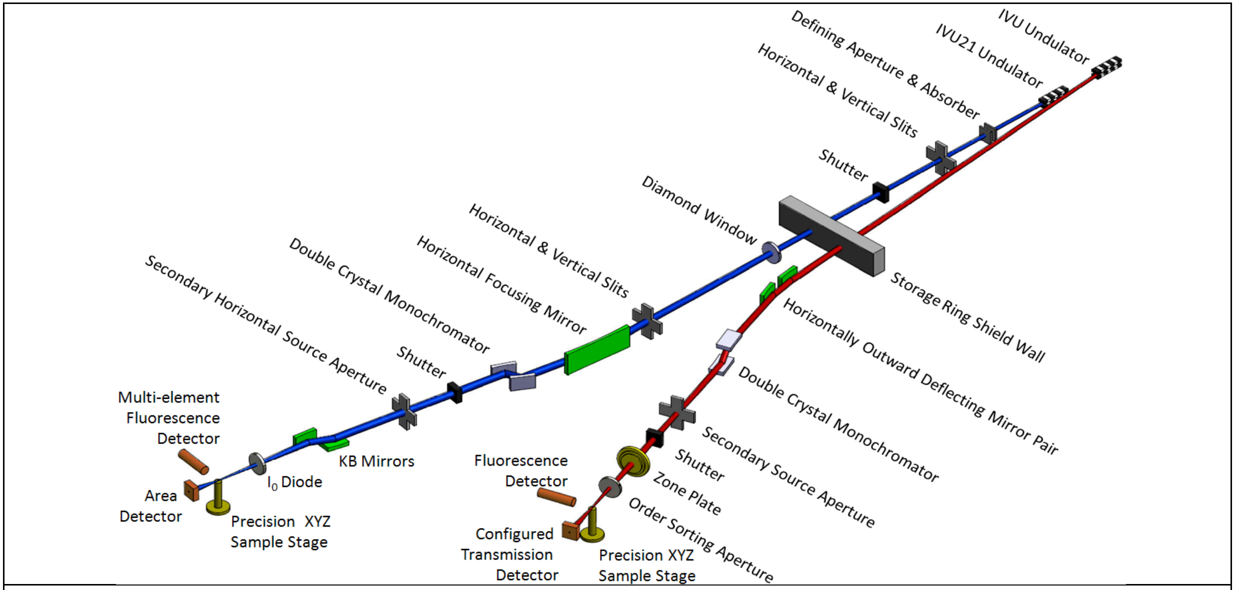

Figure 8: Sketch of optical layout of both branches of the SRX beamline. The branch hosting the ZP ist not in the baseline scope of the NSLS-II project.

Figures 9, 10 and 11 show the layout of the optical components of the KB and the ZP branch of the SRX beamline itself including the surrounding hutches and the beam pipes. The first hutch, starting at the ratchet wall, will be the First Optical Enclosure (FOE). Here, the horizontally focusing mirror and the double crystal monochromator of the KB-branch, and the mirror pair and the double-crystal monochromator of the ZP-branch will be installed. Due to the canted design of the undulators the x-ray beams for the two branches are very close. Therefore, the optical components for the KB branch have to be constructed in a way that room is available for the x-ray beam feeding the ZP-branch, which may be implemented at a later time. The hutch in the middle will host the end station of the ZP branch, the hutch farthermost downstream will be the place for the end station of the KB branch, including the KB mirror, the sample holder and the detectors. The secondary horizontal source aperture of the KB branch will be at the upstream end of the ZP branch hutch. Positions and dimensions are given in figure 9 and will be explained in later chapters of this document.