Australian Synchrotron Development Plan
Project Submission Form

Section A: Summary and Proponent Details

Project Title

hyperSAXS – The Major SAXS/WAXS Beamline Upgrade (MFU)

Spokesperson

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Executive Summary (approx. 100 words)

The SAXS/WAXS beamline is a leading x-ray scattering facility serving a large User base performing challenging experiments across a diverse range of scientific applications. At modest cost this upgrade will facilitate formidable improvements in performance and capability that are readily achievable and increasingly required by Users pursuing the highest quality science. Upgrades needed include sample handling and environments, a double multilayer monochromator, extending the length of the beamline, a basic Bonse-Hart USAXS capability, and detector and hardware upgrades. These investments capitalize on the very high quality of the current setup and allow the facility to remain highly competitive with advanced new storage rings coming online worldwide, for many years to come.

Other proponents (add more rows if necessary)

Name	Institution	Email address

B1: Description of Proposed Beamline/Development Project

The SAXS/WAXS beamline is a high performance beamline which serves advanced x-ray scattering requirements of the Australian, New Zealand and international research community. The beamline aims to extract the most advanced performance from a full length in-vacuum undulator, a premium hard x-ray source at the Australian Synchrotron. Whilst the original beamline design provides solid performance in many aspects of its capability, the experience of running the beamline for 12 months has provided very clear insights into the many strengths and few weakness of the instrument, and reveals opportunities to better suit the needs of Users. This proposal aims to improve outcomes for experiments of the highest quality.

Sample Handling and Environments

It is already obvious that the net scientific output of the beamline is most significantly hampered by a lack of certain sample handling and sample environment equipment. This situation is partly a remnant of the beamline construction budget, but also a part of a strategy to use the early stage of User operations to determine exactly what the Facility should provide to the User community and to better understand the precise details of the equipment required. For example, most commercially available temperature stages turn out to be poorly suited to SAXS analysis, and custom equipment designed on the basis of what we have seen during operations will produce far better results.

This is particularly the case for solution scattering. This requires both automated sample handling and a custom designed sample cell. Whilst the details of this setup are yet to be finalized, it will be based largely on the knowledge gained from prototyping we have done so far. It is clear this requires liquid handling automation and a carefully designed sample cell which allows temperature control, especially cooling, with dry gas blanketing to prevent condensation. The cell itself is most likely to consist of thin quartz capillaries although we may develop a flat sample cell geometry with ultra-thin windows because the signal to noise is critically dependent on low scattering from the sample holder. There are also continuing developments in micro-fluidics. Several existing beamlines have setups for solution handling (ALS, ESRF, Soleil) and these will form part of the basis for this beamline.

One critical capability for efficient operations is having temperature stages that can accommodate multiple samples. With single-sample holders, temperature studies are extremely inefficient because experiment times are often limited by equilibration times rather than measurement times. We have developed some prototype multi-capillary sample holders but the time has come to construct fully developed equipment which a large cross section of the community commonly require.

Sample environments include:

- Temperature and flow controlled cell for protein solution analysis with automated sample handling (described above)
- Stopped flow pumps and multi-fluid mixing cells for time-resolved studies
- temperate controlled multi-capillary holder using Peltier stages (which provide much faster heating/cooling than water baths)
- temperate controlled multi-well holder for gels (again using Peltier stages)

- hot air blower (up to 500 ºC)
- multi-sample humidity controlled stage
- in-vacuum GISAXS
- temperature controlled Langmuir trough with anti-vibration stage for liquid surface scattering. The trough is readily purchased commercially.
- Small tensile stage suitable for polymer and fiber scattering.

Feasibility: Relatively straightforward. Will draw on developments form existing beamlines where needed. Requires engineering input on some custom fabricated equipment.

Optics Upgrades

The most important area to improve the optical performance of current instrument is to bolster performance at very low q, particularly below 0.005 $Å^{-1}$. The instrument is ready to undertake this upgrade because the sources of scattering are precisely identified, the geometry of the problem is now completely understood, and all avoidable sources of scattering have already been eliminated. The need to make improvements is very clear for weak scattering samples where the total scattering is dominated by the instrument background. However there are equally important but less obvious effects such as for time resolved measurements, since the current floor space substantially inhibits the usable flux.

There are three solutions to improving the instrument at low q:

- install the multilayer monochromator. This optic simply provides a large increase in total flux (a factor of approximately 20-fold increase) so that with or without lengthening the beamline, more *useful* flux can be extracted from the *total* available flux.
- Lengthen the beamline by 3m. This significantly improves the collimation geometry of the optics, whilst maintaining acceptable focused beam size at the sample position. This benefits all camera lengths.
- Add Bonse-Hart USAXS optics, which can be inserted into the incident and scattered beams during an experiment. This is a new capability and offers the potential for rapid change to low q limits for isotropic scattering samples without the delay of a conventional camera length change.

Beamline Extension and Double Multilayer Monochromator

Intense scrutiny and experimentation on the beamline optics and slit system during 2009 led to a breakthrough in understanding SAXS optics, a clear method to manage the instrument background at all camera lengths and produce very low instrument backgrounds at very low q. A simple geometric model precisely describes all observed properties of the optics and slits and represents a substantial advance in the understanding of SAXS optics in the beamline community. This approach can readily optimize the optical setup of during beamline operations, and makes adjustments to existing setups and design of new beamlines very straightforward and reliable.

The rapid advance in optical performance in 2009 came largely out of relentless work to increase data quality for protein solution scattering. Weak scatters, such as dilute solutions, interfacial wetting studies of super-repellant surfaces, and weakly scattering biological tissues require a very low instrument background intensity to extract reliable data, particularly if other techniques are unavailable or unable to provide corroborating data. The beamline has reached the limits of its current setup to advance this frontier – we have eliminated the need to measure incident flux (and consequently have lack any scatter from incident intensity monitor), we have reached the limits of ultra-thin vacuum windows, and are at the limits of the optical collimation system possible in the current floor space.

The limitations of the current beamline stem directly from it being too short, preventing the collimation system from extracting full value from the source. Fortunately the added length required to accommodate the correct length of collimation space is not great, only 3m is sufficient. The new slit positions would be at 23650, 29000 and 32700 mm from the source, with the sample position moved downstream from 30000 to 33000 mm. The most important effect of additional length is to make it much easier to contain all parasitic scattering from beamline optics within the beamstop. Currently this can only be done by setting the beam defining slit small, allowing at the longest camera only 5 to 10% flux, and with very small tolerances (< 20 μm) on anti-scattering and guard slits to produce a useful beam. These tolerances require significant skill whilst being done by beamline staff, and prevent User camera length changes after hours. They also place significant pressure on the stability of the instrument and consequently the stability of the background intensity, which has been a concern for data quality. An additional benefit of this change is to bring the real-space distribution of the instrument background and the direct beam at the sample closer together. This is because the geometry allows a considerably smaller guard slit to be used. This is one of the interesting paradoxes of decreasing the optical demagnification of this beamline – whilst it slightly widens the real space distribution of the direct beam it makes the real-space distribution of the background smaller which benefits experiments where spill of background intensity outside small samples must be avoided. The additional length more than doubles the useful flux, which whilst certainly useful is not the main reason for the modification.

One carefully considered option for increasing the collimation length was to remove the space reserved in the white beam hutch reserved for the DMM upgrade and move the focusing mirrors upstream to free additional space for the slit system. However this option has been abandoned because the benefits of installing the DMM itself are too large. For each camera length there is a finite angular acceptance that provides useful flux, i.e. with manageable parasitic background intensity near the beamstop. As the camera lengths increase the angular acceptance of the collimation system becomes progressively lower. The main purpose of adding the DMM is to greatly increase the *total* flux, so that proportionally more *useful* flux can be maintained at the sample position. This is particularly the case at 3m and longer camera lengths, and particularly for weak scattering samples where the collimation system must maintain essentially zero parasitic scattering right down to the beamstop for reliable results, and of course for time resolved analysis. We are finding that weaker scattering samples are currently flux limited, particularly for time-resolved work, and that the counting detectors are able to cope with much higher flux for many of the current range of experiments.

The length extension and DMM options are both part of improving to the collimation properties of the beamline. This proposal recommends implementing both. Length extension benefits the performance of the beamline with both monochromators, and assists all camera lengths not just the 7m camera length.

Feasibility: Straightforward. DMM is an off the shelf component designed to fit into the existing optics. Beamline extension is straightforward as the beamline fits well inside exterior walls of building. Hutch extension simply requires more side and ceiling panels of existing design. Building modifications limited – minor extension to external walls needed only for access to other sectors and extension to isolated concrete experiment floor is required. All works can be performed in advance and in stages for minimal interruption to experimental program.

USAXS

The third approach for additional low-q performance is to implement a basic Bonse-Hart type USAXS setup. This involves adding multi-bounce collimator crystals before the sample, and a multibounce analyser crystal after the sample inside the SAXS camera. The crystal optics are intended to move readily into and out of the analysis position on precision translation stages. This would provide a new and unusual capability similar to BL4-2 at SSRL, that is a rapid changeover to low q-minima at most camera lengths, without the delay of a full camera length change of the 2D SAXS camera. It should be noted this technique applies only to isotropic scatterers, but this is quite often the case. Relatively simple crystal optics would provide a slightly lower q-minimum than possible by the existing pinhole camera.

The detailed design of the USAXS setup has not yet been done. It is not currently intended to be a full wide angular range USAXS setup nor do 2-dimensional crystal collimation such as at ID02 at the ESRF. What is currently intended is simpler 1-dimensional setup aiming at a modest q-min of approximately 0.0008 $Å^{-1}$. Without 2-D crystal collimation, the actual q-minimum achieved would depend on the resolution-blurring in the pinhole collimated direction and the quality of the crystals used. The upper q-limit of the crystal optic setup would deliberately be set relatively low, approximately 0.1 $Å^{-1}$, in order to achieve short scan times of approximately 10 seconds, with pinhole data used at higher q.

The setup requires precision rotation and translation stages both before and after the sample position, several crystals and several point detectors for alignment and for measurement. The first extension tube of the SAXS camera would be modified to include the additional in-vacuum stages and hardware required.

Feasibility: uses readily available commercial parts, development and testing can be done offline. Design to be based on existing installations at other facilities. Will require engineering resources for design and fabrication work.

<u>Detector upgrades</u>

For its current capabilities, the SAXS/WAXS beamline has amongst the most advanced detector setup worldwide. We are finding that advanced detectors are crucial for the quality of science that Users are able to achieve as well as increasing the quantity. Some of these capabilities could be shared with other beamlines to maximize the benefits for the facility. To maximize the outcomes of the beamline it is important to stay at the forefront of detectors.

- Upgrade the 200k Pilatus detector to 300k by simply adding the third module to the empty position in the existing housing. The current detector was limited by the original construction budget rather than the needs of Users. We are already finding that Users are looking to increase the number of pixels in the detector to improve the q-range/resolution capacity for WAXS. This will also reduce the need to use the MARCCD for WAXS, which is disruptive to install, requires mechanical shuttering which causes problems when used in combination with a counting detector, and has poor time resolution.

- add additional readout electronics to all Pilatus detectors for full-speed readout. This is needed for very fast time resolved experiments, and will be particularly when combined with the DMM where much higher flux will be available. The highest quality science done on the beamline is often the time resolved work.

- Add a custom 2D detector for 2D WAXS able to run simultaneously with the SAXS detector. The beamline construction budget could not accommodate this detector. This aspect is particularly useful for polymer science requiring in-situ anisotropic structure analysis at multiple length scales. The beamline has already conducted such work in a crude way by putting the Pilatus 1M at the WAXS position, however the goniometer is not rated for such loads and the geometry of the 1M SAXS detector is poorly suited and cannot provide adequate azimuthal angular range. This application requires a customized time-resolving detector with large azimuthal coverage (> 180º), similar to DND-CAT at the APS. We would intend to construct a custom detector, very likely using Pilatus hardware.

- Other minor purchases such as point detectors and electronics.

Feasibility: All but 2D WAXS detector are straightforward commercial off the shelf equipment purchases. 2D WAXS detector is custom layout/design using proven commercial components – will do development primarily with vendor where possible, with local collaborations where needed.

Liquid Surface Scattering Upgrade

The beamline optics are designed to be capable of in-plane scattering on liquid surfaces, and we are getting interest from Users looking for this capability. This technique allows determination of in-plane ordering and structure of surfaces and thin films on liquids, such as packing and self-assembly of monomers, surfactants and macromolecules, absorption of nanoparticles etc. There have long been some thoughts within the community of constructing a dedicated liquid surface diffractometer at the Australian Synchrotron capable of diffraction and reflectivity, such as in the Decadal Plan. For very little expense this beamline can readily implement the diffraction capability. All that is required is a horizontal goniometer and receiving collimators, plus the associated sample cell and anti-vibration stage. This can readily be installed on existing equipment already designed for the purpose. We can readily use the existing WAXS detector which is ideal for this technique.

Feasibility: Straightforward off the shelf equipment purchase. Existing beamline equipment is designed for the purpose.

B2: Applications and Potential Outcomes to Australian Scientific Community

How does the project advance synchrotron-based research in Australia/NZ? What are the likely outcomes? Include specific examples where possible.

The SAXS/WAXS beamline is a facility in heavy demand by a diverse range of synchrotron Users, and these upgrades will benefit Users in all areas. Many of the experiments are challenging and involve a very wide range of samples, high throughput, time- and temperature resolved studies, analysis of very weak scatterers, and very large ranges in length scales. The beamline is already a world leader in many aspects of its capabilities. Within one year of first light the absolute optical limits of the current installation have been achieved and our User community is already pushing the boundaries of the current facility. The following are some highlight and general examples of where this very advanced capability will be directly used by our research community.

It is clear that many Users are ready to take advantage of improved performance at low-q. Amongst the most scientifically important and technically demanding applications is large proteins and complexes in solution. These samples require extreme optical performance for low-q data quality and typically also have challenging sample handling requirements as they typically aggregate easily. These experiments are scientifically important, are an active focus of current research and demand for very advanced instruments that can perform both static and dynamic experiments in this area will rapidly grow.

There are many experiments running at the fringes of the current facility which will quickly benefit from improved optical performance. In the biological field these include analysis weakly diffracting structures in biological tissues such as disease diagnosis in hair and biopsy samples, fundamental studies of membrane proteins, and studies of large hierarchical structures such as insect scales and membranes.

A key application of the upgrade will be in-situ muscle diffraction studies for fundamental, disease and medical purposes, where extreme flux, optical performance and advanced detector capabilities must all come together on one facility. The first part of these studies by local groups has commenced with in-vivo proposals already submitted.

Other examples include in-situ colloid formation studies, in-situ studies of drug-delivery systems, dynamics of liquid crystals and surfactant mesophases, surface wetting studies of super-resistant coatings, in-situ polymer membrane processing such as for gas separation and solar energy.

The 2D simultaneous WAXS detector is targeted directly at the polymer sector of the User community, ready to undertake in-situ processing studies involving anisotropic structures over very large length scales of textured crystalline and super-crystalline structures. There is an acute shortage of instruments able to perform this research worldwide, and we expect strong interest in this capability from the national and regional research community. The extremely flexible setup and existing capability puts the SAXS/WAXS beamline in an excellent position to serve this need, since only an additional detector is required.

The beamline and its User community is well placed to make use of targeted upgrades to extract truly extraordinary performance from its in-vacuum undulator source, alongside the most important sample environment equipment required to make the most use of it.

B3: Match to Selection Criteria

- All upgrades target well understood properties of the beamline to aid identifiable and active research groups, many of whom have already used the beamline. This proposal represents a convergence of highly detailed understanding of the beamline with the needs identified and expressed within its User community.
- The proposal is complemented by the related CD/SAXS beamline proposal. The two proposals produce highly effective and efficient outcomes for a rapidly growing SAXS community, without requiring two straight sections. This proposal aims to extract the full remaining capability of the 3m in-vacuum undulator source to support experiments of the highest quality.
- SAXS/WAXS is able to deliver on proposals it makes. The beamline has demonstrated innovative and highly successful use of the most advanced insertion device currently installed at the facility. It has demonstrated the most rapid progression from first light to full User operations, very high rates of usage, an intensive experimental program and a high level of reliability. Within one year, every possible performance impediment within the beamline optics has been identified and removed, and upgrades are required to make further progress. SAXS/WAXS has a demonstrated capacity within the beamline for efficient controls implementation, and for rapid development of highly effective data acquisition and processing software to allow Users to make the most of capabilities.
- The established beamline provides an ideal base for further development. Its design and function are sound and readily upgraded. The beamline serves a wide range of Users across the Australasian research community, many of whom perform ambitious and challenging experiments. Many of our previous Users are rapidly expanding their plans knowing what is already possible. We have a highly advanced endstation which is readily able to take advantage of the optical upgrades and immediately accommodate the additional equipment required.
- All of the upgrades are readily achieved, most within 18 months. All upgrades are compatible with the existing setup and represent increases in capabilities rather than replacements: all existing equipment remains an integral part of the beamline. Most expenditure uses off the shelf components or are complete systems. We will require some engineering resources to assist in designing some new equipment, and to implement hutch and building modifications: the intention is to second a single engineer to see the entire project through. Most of these upgrades were actually foreseen and allowed for years ago in the original planning and design, which was explicitly made flexible enough to accommodate modification and upgrades.

B4: Potential Users

The following table is a short summary of some of the current Users who will rapidly exploit the proposed upgrades:

Name	Institution	Application
James Pearson	Monash University.	Fast time resolved in-situ muscle diffraction
Leanne Dyksterhuis, John Ramshaw	CSIRO	Very difficult large proteins such as membrane proteins in solution. Membrane proteins structures in tissues.
Matthew Wilce, Nathan Cowieson, Marcel Hijnen	Monash University, AS, Burnet Institute	Proteins solutions, including recombinases, in-situ dynamics of in-situ proteolysis and membrane interactions.
Terry Mulhern, Ren Dobson et al.	U. Melbourne and Bio21	Wide range of biological and medical research. Protein solution research, drug development, antibiotics, pathogenesis, self assembling systems.
Ben Boyd et al.	Monash University	Drug delivery. Performing fast time resolved studies relating to drug delivery systems.
Graham Edward, Robert Knott	Monash University, ANSTO	In-situ polymer formation, particularly time-resolved simultaneous 2D WAXS with 2D SAXS.
Calum Drummond, Liliana De-Campo, Charlotte Conn, Tamar Greaves, Kate Nairn, Xavier Mulet et. al	CSIRO, ANU	Properties and dynamics of liquid crystal systems, proteins in ionic liquid systems
Philip Heraud, Karen Sui, Rob Lewis et. al	Monash University	Disease studies in tissue, diagnostics development
Andrew Whitten, Jennifer Martin	University of Queensland	Protein solution studies
Kevin Jack	University of Queensland	Colloid chemistry, protein solutions.
Mary Ryan, Bridget Ingham	Imperial College (UK), Industrial Research Ltd. (NZ)	Functionalized nanoparticles
Roger Newman, Bridget Ingham	Scion Ltd. (NZ), Industrial Research Ltd.	Fundamental and industrial studies of cellulose and wood materials

	(NZ)	
Christopher Squire, Edward Baker	University of Auckland	Protein solution scattering
Ainsley Seago, Sarah Weisman	CSIRO	biology of large structures in insect tissues
Paulo Falcaro, Anita Hill et al.	CSIRO	Polymer membranes for separation technologies, surface scattering, mesophase materials
Irving Liaw, Chao Yan et al.	University of Melbourne	Super non-wetting coatings, polymer blend materials
Patrick Hartley, Durga Acharya, Hsin-Hui Shen, Jacek Jasieniak et al.	CSIRO	Liquid crystal systems, protein solutions, self assembled proteins, polymer coatings
Nicola Scarlett, Ian Madsen, Helen Brand	CSIRO	In-situ studies of mineral processing
Chanh Tran et al.	LaTrobe University	Development of novel x-ray imaging techniques