The yellow mini-hutch for SAXS experiments at MAX IV Laboratory

Ana Labrador¹, Yngye Cerenius¹, Christer Svensson¹, Keld Theodor¹,² and Tomás Plivelic¹

¹MAX IV Laboratory, Lund University, Sweden
²Niels Bohr Institutet, Nanofysik, Copenhagen, Denmark
E-mail: Ana.Labrador@maxlab.lu.se

Abstract. I911-SAXS is the new SAXS (Small-Angle-X-ray-Scattering) beamline at the MAX IV Laboratory in Lund, Sweden. It is one of the 5 stations of the hard X-ray Cassiopeia beamline (I911) at the 1.5 GeV ring MAX II. I911-4 was converted into a multipurpose SAXS station which opened to the scientific community in May 2011. The SAXS users community at this laboratory comes from diverse fields of research with different needs and requirements at the end-station. This results in different set-ups routinely being installed in the easy-accessible experimental mini-hutch. The beam can be focused at sample-to-detector distances between a few hundred millimetres and more than two meters. This versatility permits a selection of q-ranges between 0.006 Å⁻¹ and 2 Å⁻¹. The recent acquisition of a fast readout, low noise pixel detector (PILATUS 1M) and the implementation of a high-throughput solution SAXS are the latest beamline upgrades.

1. Introduction
In 2008 a Swedish-Danish collaborative group took the initiative of converting one of the Cassiopeia side stations (I911-4) to a dedicated SAXS beamline (I911-SAXS) in view of the increasing demand of the MAX-lab SAXS users community at I711 [1]. After some preliminary conceptual feasibility tests, a new experimental hutch was constructed and the final commissioning of the I911-SAXS station started in autumn 2010. In the same year the MAX IV Laboratory was established to include both the operation of the present MAX-lab facilities (MAX I, II, III) and the MAX IV Project, aiming at constructing the new MAX IV facility [2]. The I911-SAXS beamline opened to the scientific community in May 2011.

2. Beamline optics and beam stability
The Cassiopeia I911 beamline (running since 2004) is a unique facility with five independent experimental stations [3]. The I911 source is a superconducting wiggler with 49 poles usually operated at 3T. The central 2 mrad of the 2K/γ = 14 mrad horizontal wiggler fan, are used and split in three parts. The vertical fan is 1/γ = 0.4 mrad. The resulting photon brilliance is 3 · 10¹⁵photons/s/mrad²/mm²/0.1%bw in the energy range from 7 to 18 keV. I911-4, the new SAXS station, uses one side of the central fan, from 0.5 to 1 mrad. The I911-4 monochromator is a Si(111) crystal (asymmetrically cut at 2.5 deg) placed downstream from a, high transmission above 10 keV, diamond(111) crystal. The wavelength of the diffracted beam was tuned by translating the crystal along the synchrotron radiation beam direction. In this case a source-crystal distance of 11350 mm selects the fixed wavelength of 0.91 Å. The crystal is bent meridionally to about 12 m radius to provide horizontal focusing. A curved (400 m
radius) Mo-Si multilayer mirror provides vertical focusing. A top view sketch of the Cassiopeia wiggler fan is shown in Figure 1. Two pairs of slits (JJ X-Ray A/S), two sets of Kapton foil plus scintillation counter (M1,M2), a beam shutter and one removable attenuation foil complete the optical hutch (Figure 2).

Figure 1. Schematics of the fan from the wiggler indicating the part of the beam used at the SAXS experimental side station I911-4. The stations I911-2 and I911-3 are devoted to high-throughput macromolecular crystallography. I911-5 is being refurbished for macromolecular and single crystal diffraction and I911-1 is mainly used for education.

Figure 2. Layout of the I911-4 optical and experimental hutches.

The optical design of the I911 side stations makes them sensitive to any change in the monochromator heat load. This induces changes in the intensity, $I_0$, and position of the focused beam which is critical in a collimated beam. Figure 3a shows some beam snapshots. Figure 3b shows how the $I_0$ intensity decays significantly faster than the ring current. A beam alignment procedure has been developed and has extensively proved its reliability: by scanning the theta and tilt angles and the bending radius of the monochromator, the maximum of the collimated beam intensity is easily recovered. The algorithm is executed in approx. 1.5 minutes due to limitations of the monochromator mechanics and the communication protocol. The alignment procedure has been tested at different time intervals and beam currents. It also works in most critical situation such as after the MAX II injection (Figure 3c) or after eventual instabilities in the stored beam. Figure 3d shows the $I_0$ values after all the alignments done over 60 hours during a real experiment. The users can choose to align the beam before collecting each image; when the intensity has dropped a given percentage or at any other moment when the intensity needs to be optimised (Figure 6c). All choices are easily controlled through the beamline GUI.

The size of the focal spot at the sample position, i.e. at 7 m from the monochromator, is about 0.3x0.2 mm² FWHM (HxV). The flux of the focused and collimated beam was estimated to be 5x10¹⁰ photons/s. More precise measurement will be possible with the recently installed diagnostic set (Figure 6a).

3. The I911-SAXS experimental hutch

The four I911 side stations are quite similar and the main difference that converted I911-4 to allow SAXS experiments is the longer sample to detector distance. The experimental hutch, which resembles a conventional home-lab SAXS system, is a stainless steel construction (900x800x4500 mm³, HWL). The hutch encloses the downstream parts of the beamline: the sample experimental area, the evacuated and modular SAXS chamber and the 2D detector to acquire the X-rays scattered by the sample. The cabinet has two sliding doors to access the sample environment area and to modify the SAXS chamber lengths and another door to access the 2D detector. There are several chicanes to allow the control, from the outside, of equipment inside. The sample to detector distance can be changed from a few hundred millimetres to more than 2 meters. The accessible q-range of a typical I911-SAXS setup is 0.01 Å⁻¹ - 0.3 Å⁻¹ ($q = (4\pi sin\theta)/\lambda$). Lower values (0.006 Å⁻¹) or higher (2 Å⁻¹) can be reached by changing the setup.
Figure 3. (a) Beam snapshots (b) $I_0$ decay of a collimated beam without alignment. (c) Three alignment routine iterations done after a MAX II injection. The beam snapshots before and after the alignment are shown in (a). (d) Up: $I_0$ values during all alignments performed over a 60 hours long, experimental run. Down: the linear fit of $I_0$ respect to the ring current shows that the intensity decay of the collimated beam is proportional to the ring current. Note that alignments after the MAX II injection are also included here. (e) $I_0/I_t$ collected, without any sample, over one injection cycle. Alignments were done every 30 minutes.

The easy accessible experimental “mini-hutch” (Figure 4) has been equipped with (Figure 2): two pairs of JJ-X-ray slits (collimating and guard slits) a scintillator for $I_0$ measurements; a fast ms shutter (Uniblitz), a modular evacuated SAXS camera, a X-Y motorised Xenocs 4x4 mm$^2$ beamstop with integrated pin diode (active area: 1.8x1.8 mm$^2$) for transmission intensity measurements (Figure 6b), a X-Y sample stage and the data acquisition 2D detector (a MarCCD 165mm from Rayonix, L.L.C.). Recently a low noise, fast read-out pixel detector (PILATUS 1M) has been acquired and will soon be commissioned. The whole set-up was conceived to work under pressure better than $10^{-2}$ mbar.

Figure 4. View of the experimental hutch I911-4 for SAXS experiments at the MAX IV Laboratory. Left: hutch interlocked. Right: hutch open.

A very useful and practical feature of this set-up is the design of the SAXS chamber which allows to offset the exit window without changing the beamstop position. In this way the q-range can be easily changed without modifying the chamber length (Figure 5). Also available is a diagnostic set-up, that can be easily used, composed of an X-ray camera to look at the beam and an aperture and pin diode set for measuring the beam profile (Figure 6a).

3.1. Sample environments: Present and Foreseen

The currently available sample environments are: An in vacuum flow-through capillary, for solution scattering; Several multiple positions sample-holders, in air, for solids (films and powder), gels or liquid samples. Both systems can be used with a water-bath temperature control. A multiple position sample-holder is being modified to work in an evacuated chamber. Complementary techniques, such as Raman and UV-VIS spectroscopy, are currently under development to provide simultaneous information to the structural scattering results. The users are encouraged to suggest new set-ups and to come with their own ones. So far, since the station was opened there have been successful measurements of diverse systems: proteins and
membranes in solution, natural and synthetic polymers, colloids and nano-particles in general, therefore proving the multi-purpose character of the current set-up.

4. Beamline Control, Data Acquisition and Data Reduction
Like many other facilities, beamlines provide a unique service to scientific communities. Therefore, in addition to the characteristics and quality of the source and equipment, the success of a beamline also depends on the control system that users interact with, to provide a reliable system for data collection and processing. To complete the recipe for success a competent and friendly users support should not be forgotten.

The low level beamline control system is implemented using Tango middleware (device servers). The experimental logic is coded with SPEC macros and most of the functionality needed for the user to run the experiment is driven with an in-house developed Graphical Interface, which includes the beam intensity optimisation routines after injection and during data collection. The **Beamline I911-4 GUI** also provides SAXS data reduction and multiple frame processing. On-line data reduction of isotropic and anisotropic SAXS data is under development.

5. Conclusions
The new SAXS beamline at the MAX IV Laboratory fully replaces, improves and expands the SAXS activities, previously shared, at I711. The optimized experimental conditions at I911-4 result in more than 10 times higher photon flux on the sample when measured under similar conditions. This has decreased the required exposure time to about 2 minutes for standard protein solution experiments. It has also increased the quality of the acquired data. An important achievement for the beamline performance is the beam alignment automation routine, to easily maximize the beam intensity during data collection.

**Acknowledgements**
K. N. Toft and Y. Gapanov for their early and preliminary work on the beamline. D. Haase for help and support in some SAXS activities. T. Ursby help in the overall I911 station and K. Noren as the Hard X-ray coordinator. D. Spruce and the IT group for the implementation of some specific software developments. C. Dicko and S. Haas for his current work with the Raman technique. L. Arleth, J. Nygaard and N. Skar-Gislinge for the input to validate and improve the biological measurements and S. Nielsen for his work on the high-throughput solution SAXS.

**References**
[1] Knaapila M et al 2009 *J. Synchrotron Rad.* **16** 498-504
[2] https://www.maxlab.lu.se/
[3] Mammen C B 2005 Design and construction of the Cassiopeia beamline for protein crystallography at MAX-lab, Lund. *Ph.D. thesis*. Also in *Acta Physica Polonica A* **101** 595-602 (2002) and *AIP Conf. Proc.* **705** 808-11 (2004).