Status and Developments of BL13-XALOC, the macromolecular crystallography beamline at Alba

J Benach, G Cuní, C Colldelram, J Nicolás, J Lidón¹, F Gil-Ortiz, J Juanhuix
Alba Synchrotron, ctra. BP1413 km 3.3, 08290 Cerdanyola, Catalonia, Spain
¹Current address: MAX IV Laboratory, Ole Römers väg 1, 223 63, Lund, Sweden
E-mail: juanhuix@cells.es

Abstract. BL13-XALOC is a Macromolecular Crystallography beamline at the newly built 3-GeV synchrotron ALBA (Barcelona). The optics design is based on an in-vacuum undulator, a Si(111) channel-cut crystal monochromator, and a pair of KB mirrors. The optical design allows three main operation modes: an unfocused configuration, where one or both mirrors are removed from the photon beam path; a focused configuration, where both mirrors can focus the beam to 49×9 µm² FWHM (H×V), and a defocused configuration that can match the size of the beam to the dimensions of the crystals or to focus it at the detector. To achieve a uniform defocused beam, the slope errors were reduced by using novel method together with our high-accuracy metrology laboratory. Thorough commissioning has also demonstrated that the X-ray beam has excellent energy and spatial stability. The End Station includes a high accuracy single axis diffractometer, a removable mini-kappa goniometer, an automated sample mounting robot and a Pilatus6M, photon-counting detector. This equipment, together with the operation flexibility, allows the beamline to tackle a large variety of crystals, from medium sized crystals with large unit cell parameters to microcrystals. Several examples of high-quality data collections measured during beamline commissioning are described. The beamline started user operation on 18th July 2012.

1. Source and optics
Alba is a third generation 3-GeV, 4.3-nm-rad storage ring built near Barcelona and the Universitat Autònoma de Barcelona [1]. Out of the seven first-phase beamlines, BL13-XALOC is the only one dedicated to Macromolecular Crystallography (MX), delivering an X-ray beam between 5 and 24 keV (optimally in range 6-16 keV). The photon source is a 2-m long in-vacuum undulator, a Si(111) channel-cut crystal monochromator, and a pair of KB mirrors. The optical design allows three main operation modes: an unfocused configuration, where one or both mirrors are removed from the photon beam path; a focused configuration, where both mirrors can focus the beam to 49x9 µm² FWHM (H×V), and a defocused configuration that can match the size of the beam to the dimensions of the crystals or to focus it at the detector. To achieve a uniform defocused beam, the slope errors were reduced by using novel method together with our high-accuracy metrology laboratory. Thorough commissioning has also demonstrated that the X-ray beam has excellent energy and spatial stability. The End Station includes a high accuracy single axis diffractometer, a removable mini-kappa goniometer, an automated sample mounting robot and a Pilatus6M, photon-counting detector. This equipment, together with the operation flexibility, allows the beamline to tackle a large variety of crystals, from medium sized crystals with large unit cell parameters to microcrystals. Several examples of high-quality data collections measured during beamline commissioning are described. The beamline started user operation on 18th July 2012.
in excess of the full central cone of the undulator, show that the meridional RMS slope error of the first crystal surface induced by heat load is reduced 3-fold when the LN\textsubscript{2} temperature (T\textsubscript{LN2}) is increased from 78K to 90K (figure 2). Temperatures at the first crystal surface are 97K and 111K, respectively. The reduction of the RMS slope error when increasing T\textsubscript{LN2} may be explained by the reduction of the absolute value of the Silicon thermal expansion coefficient $\alpha$ which is zero at ~124K.

In view of this result, the cryocooling system may be optimized not to increase heat exchange, but rather to reduce vibrations. Several actions were taken in this direction, essentially trying to bring the LN\textsubscript{2} flux closer to a laminar regime. First, the circuit in the heat exchanger in contact with the Si crystal was modified with the aid of computational fluid dynamics (CFD) simulations to reduce the Reynolds number and to make the flow speed more uniform in the microchannels of the heat exchanger. Second, the average speed at the microchannels was reduced to 0.4m/s or lower. Third, the pipe diameter upstream and downstream of the heat exchanger was increased from 8 to 10mm. Finally, cavitation conditions were also modelled and avoided in all critical points. To validate these modifications, the vibrations of the monochromator with circulating LN\textsubscript{2} flow were measured using a laser interferometer at frequencies of the LN\textsubscript{2} flow cryopump of 20-70Hz, in 1 Hz steps. The vibrational modes of the monochromator crystal appeared to be decoupled from the cryopump, indicating that monochromator is vibrationally stable. Other metrology tests show that first resonance of the monochromator mechanics is well above 100 Hz, which are not likely to affect the quality of diffraction data.

Energy stability was also tested with an X-ray beam. A Ni K-edge (8.333 keV) scan was performed every half hour for 10h, with a beam injection in between. The error at the inflection peak energy was

---

**Figure 1.** Lay-out of the BL13-XALOC beamline.

**Figure 2.** Meridional slope error of the first crystal surface of the monochromator at two different temperatures of the LN\textsubscript{2} flow.

**Figure 3.** Normalized fluorescence signal measured on a diode from a Ni foil over 11 h. Monochromator energy was set at the Ni K-edge inflection point, the most sensitivity to monochromator energy changes. Overall flux variation is 2% PV, corresponding to <0.1eV.
0.1 eV peak-to-valley, which is much better than the Si(111) crystal bandwidth at the same energy (~1.6eV). Static energy stability was also checked by measuring the normalized fluorescence of a Ni foil from the X-ray beam set at the inflection point of the Ni K absorption edge, showing very little variation for hours (figure 3).

The focusing system consists of a vertical focusing mirror (VFM) and a horizontal focusing mirror (HFM), both manufactured by IRELEC. This system is critical to successfully defocus the beam while preserving beam uniformity as the quality of the diffraction can be reduced by beam striations that appear during defocusing [4]. To minimize these striations, we have developed a new method to correct large sized mirrors by using mechanical spring actuators [5]. The method, based upon Elastic Beam Theory, minimization algorithms, and the in-house profile metrology (Alba-NOM) [6], lead to slope errors of only 55 nrad and 83 nrad RMS for the VFM and the HFM, respectively (figures 4 and 5). The VFM slope error has also been measured in the final working position with the X-ray beam by the pencil-beam technique, fully matching the metrology performed at the Alba-NOM two years earlier.

2. End station

The End Station is based on two high-precision positioning tables that move the diffractometer and the detector independently. Both have been designed in-house with excellent mechanical and vibrational behaviour [7] (figure 6). The high accuracy single axis diffractometer (Maatel-Bruker MD2M) is complemented by a removable mini-kappa goniometer and an automated sample mounting robot [8] (Irelec CATS) that works with both cryogenic samples and crystallization plates. The photon-counting detector [9] (Dectris Pilatus 6M), that can be placed from 90 mm to 1356 mm from sample, offers a large sensitive area (431×448 mm²), a fast framing rate (12.5 frames/second), a large dynamic range (20 bits), and a negligible dark current noise. The X-ray beam at sample shows Gaussian profiles. The beam can be easily defocused while preserving the beam centre position (figure 7). Beam profile striations are not severe, accounting for less than 20% of the maximum.

Figure 4. The slope error of the VFM after correction, before (black) and after (red) bending to the nominal ellipse. The pencil-beam results (blue) fully agree the metrology.

Figure 5. The profile error of the HFM before (red) and after (black) applying surface error correction through 4 pairs of spring actuators. The HFM slope error is reduced from 201 nrad to 83 nrad RMS.

Figure 6. BL13-XALOC End station. Detector is supported by red table, diffractometer by blue table, automatic sample changer in independent support.
3. First Results

The beamline has been tested using a variety of protein crystals, as summarized in Table 1. Results are excellent and yield very good statistics. Therefore, BL13-XALOC is shown to be able to deal with a large variety of data collection scenarios: automated x-ray diffraction experiments, non-standard and difficult ones, as well as experiments involving a range of crystal sizes and unit cell parameters. The beamline started to receive its first users on July 18th 2012.

The authors wish to acknowledge all XALOC team for hard and good work, and A Camara-Artigas, L Campos, M Coll, G Montoya, I Usón and N Verdaguer for test crystals.

Table 1. Some first data collections taken during beamline commissioning. \(t\) stands for exposure time and \(\Delta \omega\) for oscillation angle per image. Storage ring current was between 60 and 90 mA.

| Sample | Space group | Cell parameters | Total resolution | Overall \(R_{\text{sym}}\) | Lowest resol. shell (resolution) | Reference | Comment |
|--------|-------------|----------------|------------------|----------------------|-------------------------------|-----------|---------|
| Lysozyme | P4\(_2\)_2\(_2\) | \(a=b=79\) Å, \(c=37\) Å | 90° | 1.26 Å | 3.8% | 2.0% (3.76 Å) | [10] | High quality data, \(\lambda=0.979\) Å, \(t=1\) s, \(\Delta \omega=1\)° |
| SH3 mut of c-Src tyr kinase | P3\(_2\)_1 | \(a=b=31.5\) Å, \(c=106.8\) Å | 90° | 0.93 Å | 3.9% | 3.6% (2.34 Å) | [11] | Atomic resolution data, \(\lambda=0.827\) Å, \(t=2.6\) s, \(\Delta \omega=0.5\)° |
| meganuclease | | \(a=106.6\) Å, \(b=70.3\) Å, \(c=107.1\) Å, \(\beta=119.9°\) | | 2.40 Å | 6.9% | 3.8% (6.0 Å) | [12] | First Se-SAD dataset, \(\lambda=0.979\) Å, \(t=1\) s, \(\Delta \omega=1\)° |
| I-Dmol D. Mobilis | P2\(_1\) | \(a=464\) Å, \(b=374\) Å, \(c=461\) Å, \(\beta=98.8°\) | 187° | 4 Å | 14.4% | 5.5% (11.7 Å) | | Large unit cell Sample-det. dist. 752mm \(\lambda=0.979\) Å, \(t=1\) s, \(\Delta \omega=0.5\)° |
| Human Rhinovirus 2 with inhibitor | C2 | \(A, c=461\) Å, \(\beta=98.8°\) | 187° | 4 Å | 14.4% | 5.5% (11.7 Å) | [12] | | |

References

[1] Einfeld D 2012 Proc. of IPAC2011, San Sebastián, Spain 1
[2] Doelling D, Vogel H P, Fischer B, Hobl A, Komorowski P, Krischel D and Meyer-Reumers D 2008 Proceedings of EPAC 2008, Genoa, Italy 2261
[3] Juanhuix J and Ferrer S 2007 AIP Conference Proceedings 879 824-829
[4] Moreno M, Belkhou R, Cauchon G and Idir M 2005 Proc. SPIE 5921 59210F
[5] Nicolas J, Ruget C, Juanhuix J, Ferrer S 2012 These proceedings
[6] Nicolas J and Martínez J C 2012 NIM A In preparation
[7] Collidelram C, Ruget C, Nikitina L 2010 Diamond Light Source Proceedings (MEDSI-6), 1 e44
[8] Hülsen G, Broennimann c, Eikenberry EF and Wagner A 2006 J. Appl. Cryst. 39 550
[9] Jacquamet L et al. 2009 J. Synch. Rad. 16 14
[10] Camara-Artigas A and Bacarizo J 2012 Acta Cryst. D In preparation
[11] Marcaida M J et al. 2008 Proc Natl. Acad. Sci. USA. 105(44) 16888-93
[12] Verdaguer N, Blaas D and Fita I 2000 J. Mol. Biol. 300 1179