Current Status of TPS 07A

TPS 07A, a micro-focus protein crystallography (PX) beamline, is currently under commissioning and expected to be operational in December 2021. Different from **TPS 05A**, which is optimized for crystals with a large unit cell, **TPS 07A** is particularly optimized for micro-crystals (small ratio of signal to noise) and large inhomogeneous crystals (difficult to measure). In building a reliable micro-focus PX beamline, three main design guidelines for the optical system have been pursued: a small beam size, high-flux density and a highly stable beam. Based on these demands, beamline specifications and a simulation of beam performance appear in detail in **Tables 1 and 2**, respectively.

Table 1: Beamline specifications of **TPS 07A**.

Energy range	5.7–20 keV
Beam size	1 x 1 to 30 x 30 μm^2 @ 12.4 keV (H x V, FWHM)
Monochromatic flux	≥ 10^{12} photons/s @ 12.4 keV @ 1 x 1 μm^2
Beam divergence	< 1 mrad (in both directions)
Energy resolution	≤ 1.7 x 10^{-4} with Si(111) crystal
Energy stability	< 0.25 eV h^{-1}

Table 2: Simulation of beam performance of **TPS 07A**. Simulation energy at 12.39 keV, 3 m-IU22, 500 mA, min gap 5 mm, with heat load on first crystal.

Second source H-opening /μm	MD3 position /mm	Beam size /μm^2, V x H	Beam flux /photons s^{-1}	Flux density /photons s^{-1} μm^{-2}	Interval to Henderson limit /s
2	2	0.7 x 1.5	1.4 x 10^{12}	1.3 x 10^{12}	0.031
4	11	3.3 x 3.2	2.8 x 10^{12}	2.7 x 10^{11}	0.15
6	15	5.0 x 5.3	4.1 x 10^{12}	1.5 x 10^{11}	0.27
12	25	10 x 11	7.9 x 10^{12}	7.0 x 10^{10}	0.57
25	46	21 x 24	1.4 x 10^{13}	2.9 x 10^{10}	1.38
>100	62	29 x 34	1.7 x 10^{13}	1.7 x 10^{10}	2.35
>100	82	40 x 45	1.7 x 10^{13}	9.5 x 10^{9}	4.21
>100	102	50 x 56	1.7 x 10^{13}	6.1 x 10^{9}	6.55

Because of the ongoing commissioning, some vital beam characteristics for PX experiments, including beam size, flux and stability, have been measured at the sample position. The focused beam size at the sample position was measured on scanning a tungsten knife edge through the beam along the vertical and horizontal directions. Based on the Gaussian profile, it clearly shows that the focused beam size at the sample position is 1.7 × 3.7 μm^2 (V x H) (**Figs. 1(a) and 1(b)**). In addition, notably, **TPS 07A** can provide continuously adjustable beam sizes from 1.7 to 97 μm (**Fig. 1(c)**). The flux at the sample position can be determined with a Si-PIN photodiode and was found to be 6.6 x 10^{11} photons s^{-1} at 12.7 keV and magnet gap 7.575 mm when normalized to current 400 mA in the storage ring. As mentioned above, it is reasonable that the measured flux is less than the expected flux (**Table 2**) about one order.

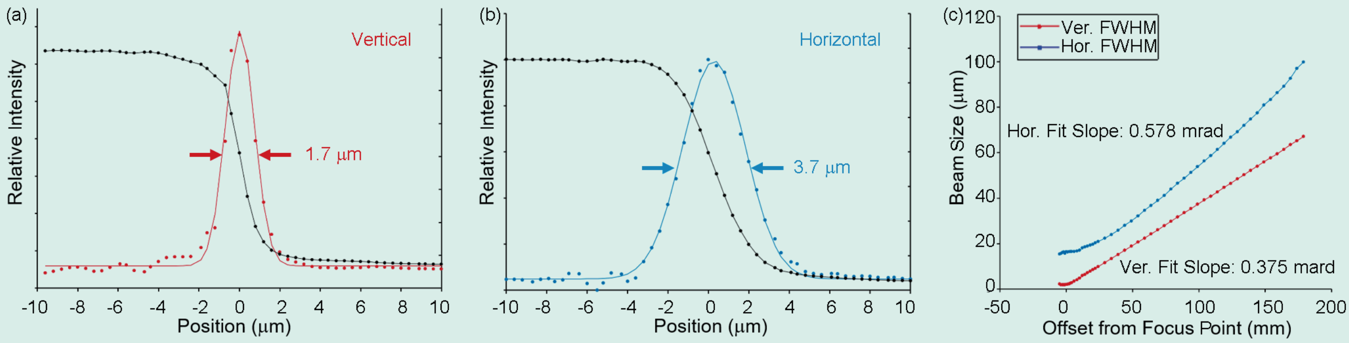

Fig. 1: (a, b) Focused beam size at the sample position in vertical and horizontal directions. Black and red solid dots show the raw data and the derivative defining the beam profile, respectively. The red solid line shows the Gaussian fitting. (c) Range of beam size. The red and blue lines stand for vertical and horizontal positions, respectively. The red and blue lines show vertical and horizontal beam sizes at varied offsets from the focus point. The measurements were conducted under fully open horizontal secondary source slits.

One of the most common issues that causes beam instability is the X-ray heat load on the double-crystal monochromator (DCM) that results in a loss of parallelism of its two crystals. At **TPS 07A**, the heat-load test showed that the period required to attain thermal equilibrium of the DCM upon X-ray exposure is about 22 min (**Fig. 2(a)**). The period of thermal equilibrium was subsequently decreased to about 2 min using a feedback system to correct the beam position continuously. Thermal drift and vibrational problems that also lead to beam instability will be analyzed in future commissioning.

Regarding the energy resolution and energy stability, the results are as follows. The energy resolution ($\Delta E/E$) varies from 1.85×10^{-4} to 1.95×10^{-4} as the energy alters from 5.7 to 20 keV (**Fig. 2(b)**); the energy drift is about 1 eV within 10 h (0.1 eV/h) (**Fig. 2(c)**). Taken together, **TPS 07A** has effective energy resolution and stability for SAD/MAD experiments.

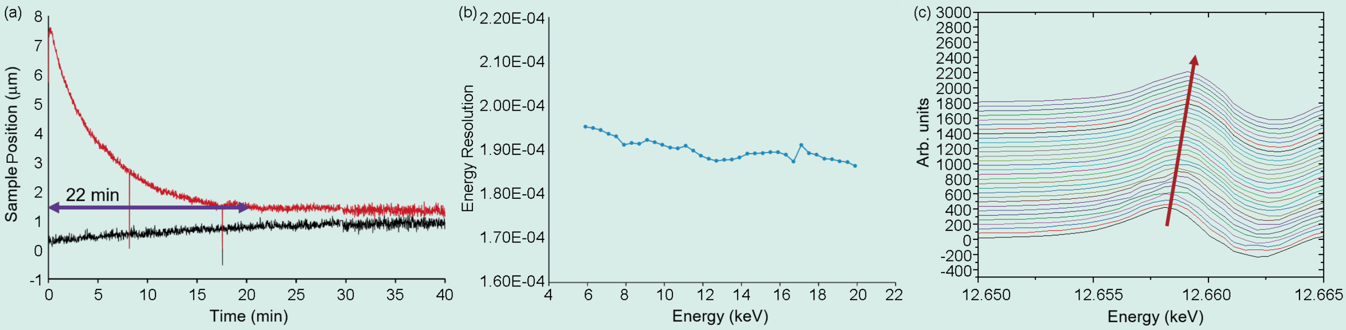

Fig. 2: (a) Thermal equilibrium period of DCM. Red and black lines represent vertical and horizontal beam positional variations, respectively. (b) Energy resolution. (c) Difference of energy drift.

To achieve a rapid and effective data collection from many micro-crystals or large inhomogeneous crystals with a raster/grid or helical scan, the endstation of **TPS 07A** is equipped with two state-of-the-art devices -- area detector Eiger2 X 16M and vertical axis micro-diffractometer MD3. Compared with MX300HS (at **TPS 05A**), the major benefits of Eiger2 X 16M are the increased frame rate, a superior point-spread function and no readout noise. This detector hence becomes more suitable for efficiently obtaining diffraction data from micro-crystals.

Many PX synchrotron beamlines worldwide, including **TPS 05A**, utilize a horizontal-axis diffractometer to rotate the crystal for data collection. In this geometry, polarization effects caused by the horizontal polarization of synchrotron radiation could cancel the Lorentz factor, but a diffractometer with a vertical spindle has superior mechanical stability (smaller sphere of confusion) for micro-crystals. After thorough consideration, our group chose a vertical-axis MD3 diffractometer to install at **TPS 07A**. In so doing, the necessary marginal cost is that the average intensity of symmetry-equivalent reflections decreases about 10% in a 2 Å bin using 1 Å radiation.[1]

In addition, an ISARA high-capacity sample changer (29 UniPucks/464 samples) will be installed; it changes samples within tens of seconds to increase the efficiency of data collection for many micro-crystals. Finally, **TPS 07A** will be embedded with powerful software, *MeshBest*, that automatically analyses the results of the raster scan from a loop with many micro-crystals. The strategy of whether and how to collect those crystals will be given. The ultimate goal is to provide an automatic work-flow to make multi-crystal data collection even more efficient.

In conclusion, **TPS 07A** with a high-flux mode and special features will be useful for the toughest crystallographic projects, such as those with large amounts of micro-crystals and those with large inhomogeneous crystals (Reported by Chung-Kuang Chou, Chia-Liang Lin and Chun-Hsiang Huang).

Reference

1. J. M. Holton, K. A. Frankel, Acta Cryst. **D66**, 393 (2010).