BL45XU
Structural Biology III

1. Introduction

One of the important requirements of macromolecular crystallography (MX) beamlines is to provide high-throughput diffraction data collection. BL45XU started operation in 2019 to perform high-throughput diffraction experiments of protein crystals with automated measurements using the ZOO system [1]. The optics layout consists of a double-crystal monochromator of SPring-8 standards, a horizontal focusing mirror, a virtual source slit, and a Kirkpatrick–Baez mirror [2]. The available energy range is 6.5 to 16 keV. The beam size at the sample position can be changed from 5 (H) × 5 (V) to 50 (H) × 50 (V) μm^2 with a photon flux of 5.70×10^{12}–1.75×10^{13} photons/s at 12.4 keV.

2. Optimization of automatic data processing system after automatic measurement

The automated high-throughput data collection produces a large number of diffraction images. The conversion of the images to the structure factor is also automated using the data processing software KAMO [3]. This process is performed on-the-fly and then the users can check the processed data immediately. This process consists of a series of several subprocesses, and all these can be monitored and progressed by the beamline staff. To improve work related to the subprocess efficiency, we are developing shell/python scripts to perform each process in succession. Briefly, we investigated the checking of the result of each process, error handling, and parameter setting and launching for the next process, and then incorporated these schemes into the scripts. Furthermore, the process of separating a whole data set into several parts to correct diffraction data depending on the oscillation angle and the error processing in merging the separated parts of the data were implemented and are now in operation.

3. Development of UniPuck exchange system for continuous automatic measurement

We are now commissioning a UniPuck exchange system (puck stocker) in order to automatically install UniPuck in the sample changer SPACE [4] (Fig. 1). SPACE can load eight UniPucks, a universal cryogenic crystal sample container, and then a total of 128 (= 16 × 8) samples can be handled at once. The developing puck stocker will enable the installation of up to 42 UniPucks to perform longer automated measurements within 2 or 3 days. Although there were several technical difficulties in achieving this, the servo system for the stocker

Fig. 1. Sample puck stocker under development.

operating axis and improvements to the hand part were considered, and continuous testing has made stable operation possible.

4. Automatic liquid nitrogen supply system installed in SPACE and puck stocker

To further improve the efficiency of this beamline, the control and recording of beamline devices are automated through the Internet of Things (IoT). As part of this effort, an LN2 supply system using the self-pressurized tank has been installed for the sample changer SPACE and the puck stocker. To manage the remaining amount of LN2 in the tank, a load cell is used (Fig. 2). The SPACE and puck exchange system can be operated continuously for 4.5 days and 3 days, respectively, during the test operation using a 210-L self-pressurized tank.

Fig. 2. Self-pressurized tank installed on a load cell.

5. *In situ* X-ray diffraction measurement using a crystallization plate

X-ray diffraction data measurement for crystals in a crystallization plate at room temperature is suitable for evaluating crystal quality without additional sample treatments, such as picking-up fragile crystals, cryoprotectant soaking, and cryocooling. To adapt to this need, we have been developing an *in situ* diffraction measurement instrument for crystallization plates at BL45XU. To speed up switching operation from measurement in a cryogenic environment to measurement using a crystallization plate, the plate diffractometer was modified, including the addition of automatic axes for the height adjustment of the stage. Further development of the automation of the switching operation is underway to provide users with the plate diffraction measurement system.

6. Improvements for efficient and smooth operation of automatic measurements

Automated measurements at BL45XU are performed for multiple users in a single day. Multiple samples of each user are stored in the beamline. The following efforts are being made to prevent mistaken identifications of user specimens handled in large volumes and to ensure efficient operation. (1) Each UniPuck ID is photographed and recorded even in a frozen state (Fig. 3). (2) The user's dry shipper and data storage media (HDD

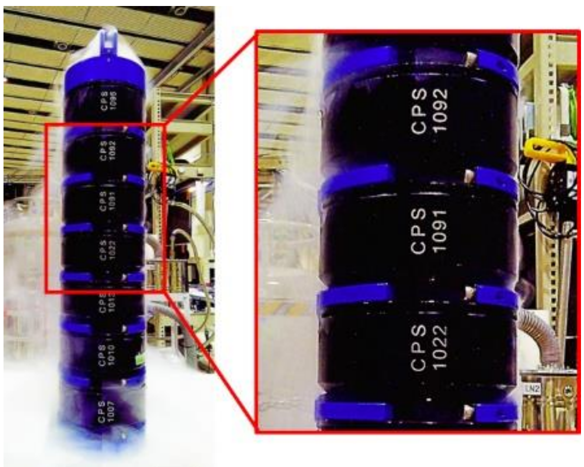

Fig. 3. User's UniPuck recorded in a frozen state.

etc.) are managed by attaching a sticker with user name and other information printed on it upon arrival (Fig. 4). (3) To ensure that overseas users receive/ship their samples securely and promptly, a dedicated Google spreadsheet was used to facilitate information between the beamline staff and user offices.

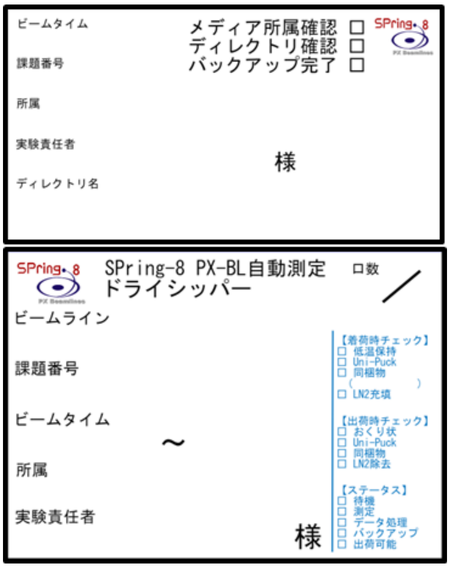

Fig. 4. Information sticker for user's samples.

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