BL02B1
Single Crystal Structure Analysis

1. Introduction

BL02B1 is designed for single-crystal structure analyses and is equipped with a two-dimensional (2D) hybrid pixel detector, PILATUS3 X CdTe 1M (Dectris). The silicon double-crystal monochromator with an inclined geometry can select monochromatic X-rays between 8 keV and 115 keV from synchrotron X-ray radiation of the bending magnet source. Most recent experiments use monochromatic X-rays with energies between 18 keV and 60 keV. BL02B1 is currently promoted for charge density studies and *in situ* experiments for functional materials using the PILATUS3 X CdTe 1M. The 2D detector with CdTe sensors is very useful for detecting high-energy X-rays because of their high quantum efficiency. By using high-energy X-rays, crystal structure analysis can be performed for inorganic materials with heavy atoms. Because the PILATUS3 X CdTe 1M has a wide dynamic range, it is used for precise structure analyses, especially in charge density studies.

2. Development of centering system for single-crystal structure analysis of small molecule

In FY2020, we continued a project to develop a fully automated measurement system for the single-crystal structure analysis of small molecules. This project aims to load the mounted sample into the sample magazine, start the measurement, and obtain the data needed for single-crystal structure analysis. One of the most challenging technical developments in this project is the centering of samples that are difficult to recognize in camera images. In FY2019, we developed an automatic centering program that recognizes a microloop from the camera image and then moves the microloop center to the center of the goniometer. On the basis of this achievement, in FY2020, we developed a program for the precise adjustment of the sample position using Python3. The program recognizes the diffraction pattern and automatically adjusts the sample position using a motorized goniometer in the ω direction with YZ translation stages to maximize diffraction intensities, as shown in Fig. 1. For the calculation of diffraction intensity, the intensities of all detected diffraction spots were integrated. A combination of sample-shape recognition and diffraction-pattern recognition enables quick and precise centering. It also enables the automatic centering of small crystals or crystals

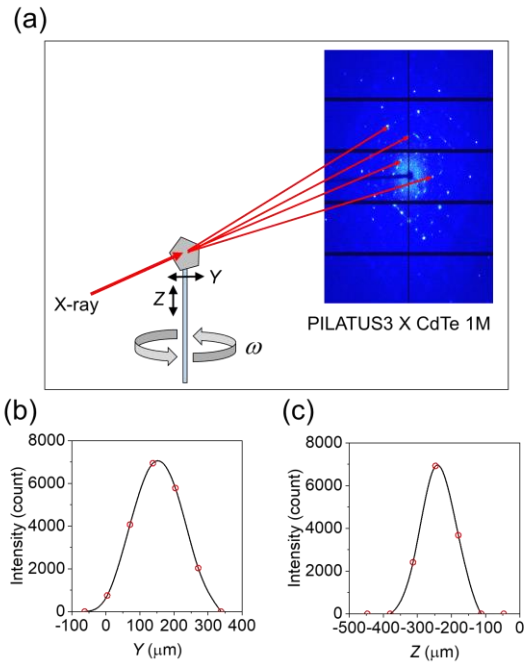

Fig. 1. (a) Schematic image of automatic precise adjustment. (b) Y-scan and (c) Z-scan.

with indistinct shapes that are difficult to approximate by image recognition. This program will contribute to the development of a measurement system independent of the user's operational skills and is essential for the complete automation of measurements.

3. Updating the data storage server for experiments using the 2D detector

In this beamline, we are conducting single-crystal structure analysis experiments using the 2D detector PILATUS3 X CdTe with 1M pixels and have been using a DELL storage server because each sample occupies several to several tens of GB of data. However, owing to the deterioration of the server over time and the unavailability of hard disks for replacement in case of failure, we decided to update the server. The specifications of the newly installed storage server made by Fanatics, shown in Fig. 2, are as follows.

- CPU: Intel Xeon Gold 6248R (24 core 48 threads, 3.0 GHz, 35.75M cache) ×2
- Chipset: Intel C622 Chipset
- Memory: 384 GB (32 GB×12) DDR4 2933 MHz ECC RDIMM
- SSD: 480 GB 2.5 inch SATA3 SSD ×2 （for OS／Software RAID1）
- HDD: 16 TB 3.5 inch SAS3 7200 rpm HDD ×36 （for data）
- RAID: RAID60 （16 TB×34＋Hot spare×2）（for data）
- LAN: RJ45 10GbE ×2 (Intel X722 + PHY Intel X557)
- IPMI: IPMI2.0 (dedicated RJ45 LAN port)
- Video: VGA×1 (ASPEED AST2500 BMC)
- USB: USB3.0×2、USB2.0×2
- Case: 4U Rack mount type (W: 437 mm, H:178 mm, D: 699 mm)
- Power supply: 1000/1200 W 80PLUS titatinum certified redundant (1+1) power supply
- Drive bay: 3.5 inch SAS3/SATA3 Supported hot-swap bays×36
- OS: CentOS 7

Fig. 2. New (top) and old (bottom) data storage servers in server rack.

The old storage server had a capacity of 60 TB, but the new one can store 480 TB of data, which will enable us to cope with the expected explosive increase in the amount of data produced at the beamline in the future. We hope that the experiments to observe nonequilibrium phenomena, which are expected to require high data capacity in the future, will be promoted.

In summary, we developed an auto-centering system and updated the data storage server. We are planning to fully automate all single-crystal X-ray diffraction measurements by combining the auto-centering system with an automatic exchange robot for single-crystal samples.

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