Structural Biology I (BL41XU)

1. Introduction

The bio-crystallography beamline at the station BL41XU has been open for the public use from October 1997. This beamline has the following two goals:

- * To realize routine analyses of macromolecular crystallography by the MIR-OAS method, which is the heavy atom multiple isomorphous replacement (MIR) method combined with the optimized anomalous scattering effect (OAS) for heavy atom derivatives.
- * To expand the applicable range of macromolecular crystallography in molecular weight and crystal size by taking advantage of the high brilliance characteristics of the beamline.

2. Beamline

The light source of the bio-crystallography beamline is an in-vacuum type undulator with a magnetic periodicity of 3.2 cm and a total length of 4.5 m. The undulator gap can be changed from 8 mm to 50 mm. The energy range of fundamental emission is from 6 keV to 18 keV, and the corresponding energy range of third harmonics is from 18 keV to 54 keV. Using this light source, highly brilliant X-rays are available from 9 keV to 38 keV.

X-rays from the undulator are led to the monochromator after reducing unnecessary heat loads by the front end elements. X-rays are monochromatized with a rotated-inclined double crystal monochromator (RIDCM) using Si(111) crystals. This monochromator has two features to avoid a heat load of highly brilliant undulator light; one is the so called pin-post structure of the first crystal for water cooling, and the other is a rotation mechanism that enables crystals to maintain a constant length of foot print of the undulator beam on the surface of the crystals.

In order to focus the X-ray beam at the sample position, two mirrors are utilized in a KB configuration. The first mirror (300 mm length) is for focusing in the vertical direction and the second (700 mm length) is for the horizontal direction. These two mirrors are made from silicon crystal and fused quartz and coated with three different materials (Pt, Rh and supermirror) in three independent stripes on the same surface. By selecting the stripes, users can use an X-ray beam of any energy in a wide range without any loss of high brilliance.

All of these components are fully controlled by a computer. A database for tuning parameters of the components and a controlling program for them has also been completed. By just inputting a value for wavelength or energy of X-ray, this program can arrange all components automatically to their optimal positions in a few minutes.

In 1998, the current of the storage ring increased from 20 to 70 mA, and the minimum gap of the undulator was restricted to 9.6 mm. Consequently, X-rays in the energy range from 17.5 to 19.0 keV could not be used. A higher heat load caused by increasing ring current made the monochromator crystals distorted and the monochromatized X-ray beam dispersed. The beam profile at sample position, therefore, increased to about 200 micron. Overall characteristics of the X-ray beam under 70 mA of ring current as follows; The flux at 12.4 keV is about 2×10^{12} photons/sec. and the energy resolution is in the order of 10^{-4}.

3. Experimantal Station

The experimental station is equipped with an automated diffractometer consisting of a goniometer and detectors.

The goniometer has a phi axis for crystal rotaion in the horizontal direction and two manual arcs orthogonal to each other for arranging the crystal axis to the phi axis. The phi axis can be driven for high speed rotation (max. 10 deg./sec.). A collimator is installed on the goniometer, and the minimum size of the collimator is 50 × 50 micron. The goniometer has several axes to align itself to an X-ray beam, and these axes are fully and automatically controlled by a computer program that only requires the wavelength and camera length to be input.

Three different types of detectors are installed in the experimantal station. The Rigaku R-AXIS IV is a default detector in the bio-crystallography beamline. It has IPs with a size of 300 × 300 mm and can read out 11 IPs per hour. A flat-type manual cassette for large format IPs has been prepared. The maximum size is 800 × 800 mm with two of the large format IPs (400 × 800 mm). This cassette can move in horizontal and vertical directions for the Weissenberg method. There are 20 sheets of large format IPs and two drum-scanner type IP readers to use them. The on-line IP detector specially fabricated for the diffractometer has two sheets of IPs (400 × 500 mm) which can be read out with a line laser and a CCD. This system is still under construction. The final tuning of the readout mechanism is on-going. All three of the detectors mentioned above can be used for high energy X-rays as high as 38 keV for the MIR-OAS routine analysis. The camera length is variable for each detector. Minimum lengths are common at 260 mm, and the maximums are as follows; R-AXIS IV 780 mm, flat-type casette 1,100 mm and on-line IP detector 1,500 mm.

Because of the increment of ring current to 70 mA, a full data collection from one crystal is not realistic for almost all of the protein crystals in the non-cryogenic condition. The cryogenic cooler has been changed from a product of Oxford Cryosystems to that of Rigaku Co. The Rigaku cryogenic cooler does not need liquid nitorogen and is much more convenient for continuous experiments over a long time.

To apply the optimized anormalous scattering effect (OAS) to crystallographic analysis, it is indispensable to measure an X-ray absorption spectrum of the sample. For this purpose, the measurement system prepared for X-ray absorption consists of a NaI(Tl) scintillation counter and discriminator.

Cryo-Xe-Siter (MSC Co.) is installed in the measurement preparation laboratory. It is used to dope Xe gas into protein crystals to be used as heavy atom derivatives. Because the energy of X-rays up to 38 keV is available in the bio-crystallography beamline, Xe-doped crystals can be utilized not only as heavy atom derivatives but also in the multiple anormalous dispersion (MAD) method.