BL40B2 Structural Biology II

The beamline is dedicated to macromolecular crystallography in monochromatic X-ray (PX) mode and small-angle X-ray scattering (SX) mode from non-crystalline materials.
The experimental station has two experiment modes, the PX mode and SX mode, in the energy range of 7 ~ 18 keV. In both modes, detector choice between an imaging plate detector (RIGAKU R-AXIS IV++) and a CCD detector (ADSC Quantum 4R) is available.

Area of research

Macromolecular crystallography, Small angle X-ray (solution) scattering

Keywords

Scientific field

Macromolecular crystallography, Small angle X-ray (solution) scattering (SAXS), Structural biology, Protein, Polymer, Fiber, Diffraction

Equipment

Imaging plate, CCD, Imaging intensifier + CCD, Cryostat (cooled nitrogen or helium stream generator)

Source and optics

The light source is a bending magnet that has its magnetic field at 0.679 T and shows critical energy of 28.9 keV. The generated white X-rays are monochromatized using a fixed-exit double crystal monochromator and focused by a 1-m-long rhodium-coated bent-cylinder mirror. The glancing angle of 3.2 mrad is adjusted for the optimum focusing at the detector position. The photon flux is 10^{11} at 12 keV. The energy resolution ($\Delta E/E$) is in the order of 10^{-4}.

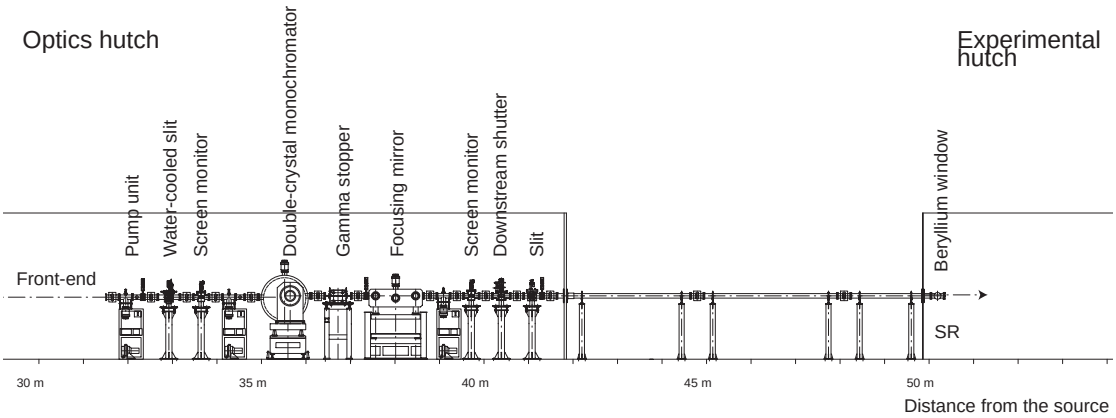
X-rays at sample

Energy range	6 ~ 17.5 keV
Energy resolution	$\Delta E/E = 10^{-4}$
Photon flux	10^{11} at 12 keV monochromatic X-rays
Spot size	horizontally 250 μm, vertically 200 μm (@ 12.4 keV (λ = 1.0 Å)) at CCD detector position

Experimental stations

Protein crystallography

The total system of monochromatic data collection for routine macromolecular crystallography has been installed completely. Its LabVIEW software system contains to change wavelength and to align experimental table. The detector for protein crystallography is set on camera stage, which camera length of 80 mm ~ 470 mm is available. A detector choice between CCD and Imaging Plate is easily available using off-line exchange system (Fig.1). Especially, an anomalous dispersion measurement for MAD (Multi-wavelength Anomalous Diffraction) is easily done and a routine MAD data collection can be performed automatically. For diffraction data processing, an academic license of HKL2000 software is installed in some workstations.



Schematic view of beamline

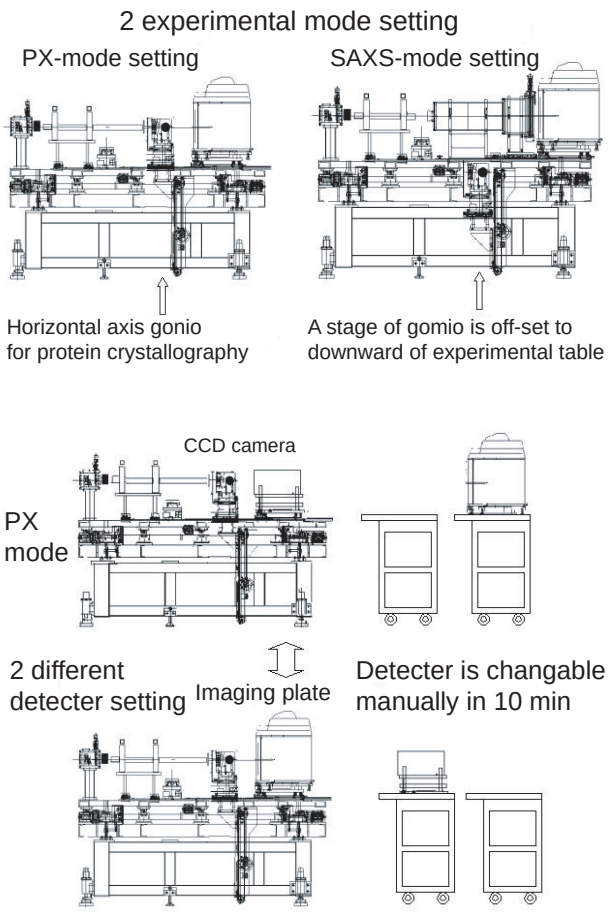


Fig.1. protein crystallography

Facilities

- CCD detector for protein crystallography (Quantum 4R (Area Detector System Corporation) : Fig.2) :
  2 × 2 array of detector modules, a total active area of 188 mm × 188 mm and 82 μm pixels in a 2300 grid. mm^2
- Imaging plate detector for structural biology (R-AXIS IV^{++} (RIGAKU)) : a total active area of 300 mm × 300 mm and 100 μm pixels in a 3000 grid. mm^2
- Horizontal axis goniometer
- Cryostat 1 (Nitrogen type) : temperature control range 80 ~ 350 K
- Cryostat 2 (Helium type) : temperature control range 30 ~ 295 K
- 3 quadrant slit
- 400 mm vacuum path with 300 mm radius window
- Gas-flow type ionization chamber
- Si PIN photodiode detector for fluorescence measurement
- Multi-channel analyzer of fluorescence measurement
- Microscope
- Cryo Xe siter
- Incubator
- Freezer and refrigerator

LabVIEW software system is installed to change wavelength and angle of goniometer, align experimental table, measure XAFS of heavy atoms in protein crystal for MAD, calculate anomalous dispersion.

Linux system is adopted for data collection control using ADSC Quantum 4R including automatically changing wavelength for MAD data collection. An academic license of HKL2000 software for processing diffraction data is installed in a Linux workstation. Total 2.3 TB RAID system mounted on workstations is set for diffraction data backup.

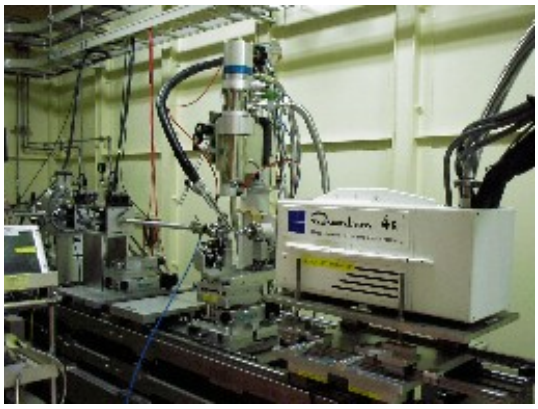


Fig.2.

Typical data

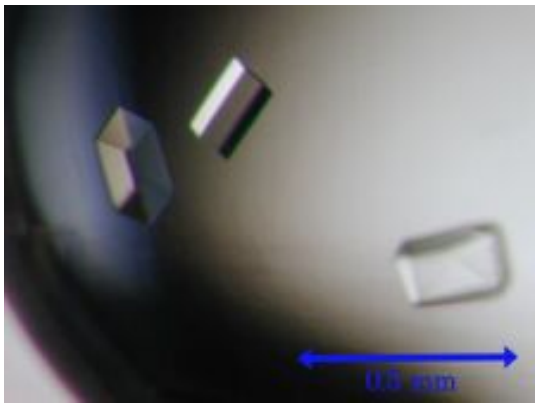


Fig.3.

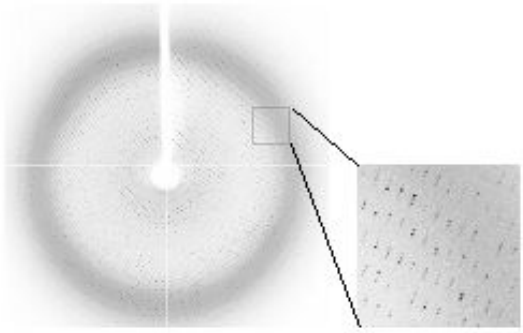


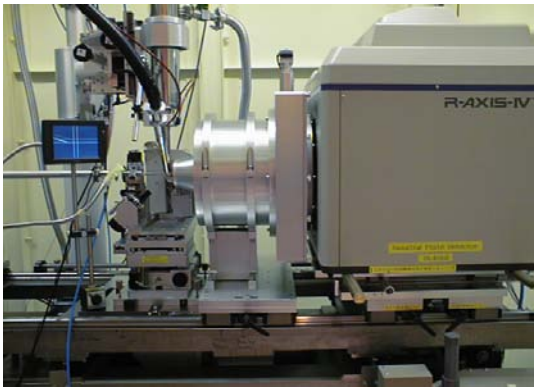
Fig.4.

Crystal of protein (311 amino acids, MW 34KDa) (Fig.3)
Space Group : $P4_12_12$
Unit cell dimension : $a = b = 146.7$ Å, $c = 369.2$ Å
Resolution limit : 2.7 Å (Diffraction image (Fig.4))

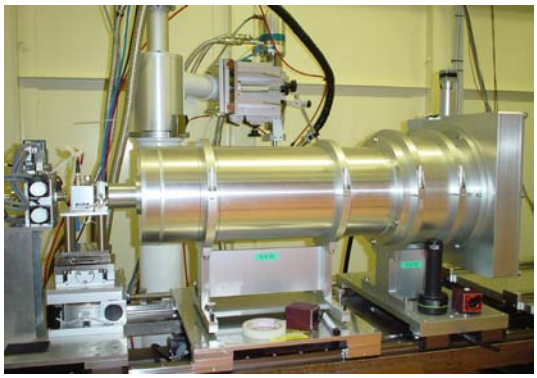
Small angle scattering

In the small angle scattering mode, the X-ray beam is orthopediated by an extra quadrant slit in the beamline optics and excess scattering is removed by quadrant slit and circular collimator in the experimental station. Two fixed length vacuum paths allow camera lengths of 400 mm and 1000 mm for scattering experiments possible with wide ranges of resolution.

Using 3 mm radius beam stopper, a diffraction data with range of 6.72 ~ 800 Å is collected when 1000 mm length vacuum path is set. Sample temperature is controlled in the range of 280 ~ 320 K using water cooling chiller. For fiber diffraction a specified sample holder with slits is prepared.



short camera (400 mm)



long camera (1000 mm)

Facilities

- Imaging plate detector for structural biology (R-AXIS IV $^{++}$ (RIGAKU)) : a total active area of 300 mm × 300 mm and 100 μm pixels in a 3000 grid. mm^2
- 2 quadrants slit
- 400 mm vacuum path with 300 mm radius window
- 1000 mm vacuum path with 300 mm radius window
- Gas-flow type ionization chamber
- Si PIN photodiode detector for fluorescence measurement
- Multi-channel analyzer of fluorescence measurement
- On-line controlled sample stage
- Sample cell for SX, temperature control range 280 ~ 320 K
- Sample holder for fiber diffraction with slits
- Microscope

Experimental table control system including changing its height, translation, tilt, rotation. LabVIEW software system is installed to change wavelength, align experimental table, on-line control of 3 quadrant slits.

Original softwares for calculation of SAXS data are installed in a SGI workstation and a Windows PC. Total 600 GB RAID system mounted on workstations is set for diffraction data backup.

Typical data

SX-example

Fig.5. shows representative scattering patterns of protein I and a complex between protein I and protein II. The scattering curves of complexes are different from that of native protein I.

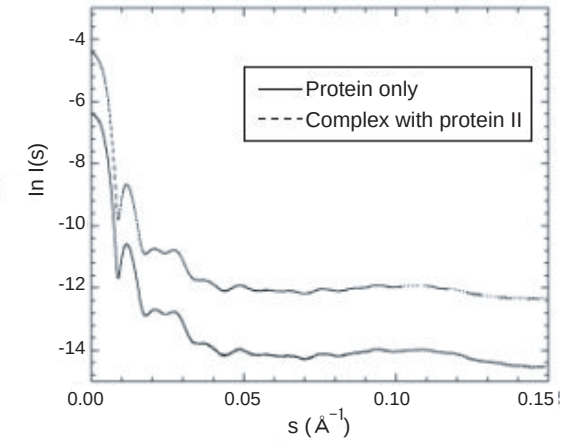


Fig.5.

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