

BL45XU RIKEN Structural Biology I

BL45XU consists of two experimental stations, protein crystallography (PX) and small-angle x-ray scattering (SAXS). Both experiments can be carried out simultaneously, with tandem vertical undulators. Only SAXS station is open to the public. For application to PX station, please contact to the office of Division of Bio-Crystallography Technology, RIKEN Harima Institute.

The main characteristic of BL45XU-SAXS is its idealistic optics for scattering experiments from non-crystalline biological samples as well as polymer materials. Two-crystal monochromator (diamond, (111) plane) followed by K-B mirrors (vertical and horizontal) are used in order to reduce parasitic scattering. The change of camera length in limited beamtime is strongly avoided. We contribute 20% of beam time for public use. As for biological sample we also accept extra external users in collaboration base. Before submitting the application to JASRI for the first time, please talk with contact person.

Area of research

Time-resolved structures of non-crystalline biological materials such as protein, nucleic acid solutions, membrane, muscle, and micelle system under various conditions, are studied by using small-angle scattering and diffraction technique.

Keywords

Scientific field

Small-angle X-ray scattering, Macromolecular crystallography (closed for RIKEN use)

Equipment

High-resolution small-angle scattering camera, Stopped-flow mixer, Temperature jump cell, High-pressure cell for protein solutions

Source and optics

Specification of tandem vertical undulators

| | |
|------------------|---------------------------------|
| Type | In vacuum, vertically polarized |
| Period length | 37 mm |
| Number of period | 37 + 37 |

SAXS optics

The beam is splitted by a transparent double crystal monochromator. The transmitted beam is used for PX. The optics of PX branch is described in the reference (M. Yamamoto *et al.*, *J. Synchrotron Rad.*, **5**, 222-225, (1998)). The SAXS monochromator can tune from 6.5 keV to 14 keV, though it is usually fixed at 13.8 keV. Laue geometry of (100) faced synthetic diamond with $8 \times 7 \times 0.3 \text{ mm}^3$ (horizontal \times vertical \times thickness) is used. If one wants to use different energy other than 13.8 keV, please consult to contact person and notify in the application form. The focusing system consists of a vertical and a horizontal mirror, which is located at 40.5 or 41.73 m from the source, respectively. The focus is formed at 57.5 m from the source. Therefore, the focusing ratio of the mirrors is 2.48 to 1. The mirrors are rhodium-coated and have 4 mrad of glancing angle where 82% of reflectivity is expected at 12 keV. In

order to reduce parasitic scattering, four slits are installed; down-stream of the monochromator (slit 1), of the mirror (slit 2), 2 m up stream of the sample (slit 3), and immediate up stream of the sample (slit 4). More detailed information is written in the reference (T. Fujisawa *et al.*, *J. Appl. Cryst.*, **33**, 797-800 (2000)).

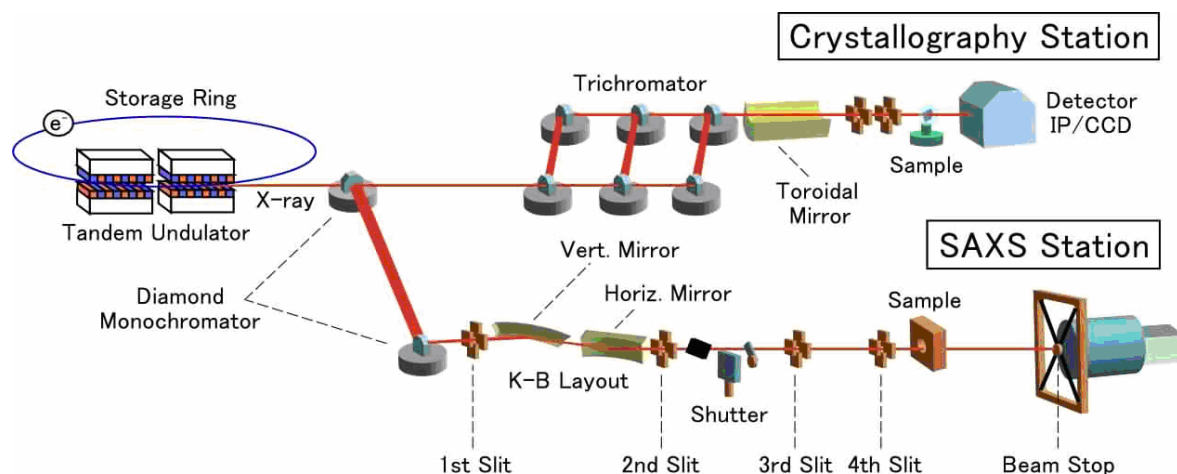
X-rays at sample

(i) SAXS station

| | |
|-----------------------|--|
| Energy range | 6.8 ~14.0 keV (tunable) 13.8 keV (fixed) |
| Energy resolution | $\Delta E/E < 10^{-4}$ |
| Photon flux | $\sim 3 \times 10^{11}$ photons/sec |
| Beam size (typically) | 0.4 mm (horizontal) \times 0.2 mm (vertical) |

(ii) Crystallography station (closed for RIKEN use)

| | |
|-----------------------|--|
| Energy range | 7.5 ~14.0 keV (tunable) |
| Energy resolution | $\Delta E/E < 10^{-4}$ |
| Photon flux | $\sim 10^{11}$ photons/sec |
| Beam size (typically) | 0.1 mm (horizontal) \times 0.1 mm (vertical) |



Optics of BL45

Experimental stations

SAXS station

The length of the camera can be changed by sliding a detector wagon on which a vacuum chamber for beam stop and a detector stage are equipped. The choice of camera length is discrete; 0.6 m, 1.0 m, 1.8 m, and 2.2 m and 3.6 m. The more short camera lengths are available upon request. Please ask contact person. The table below indicates the relation ship between camera length and S-range. The change of camera length during limited user time should be avoided.

The sample table can be movable, which offers great freedom in applying a various sample cell holder.

The details of experimental information can be seen in beamline web page.

(<http://www.riken.go.jp/lab-www/biochemi/Beamline/index.html>)

Detectors

For most purposes we use XR-II + CCD detectors are used (Amemiya *et al.*, *Rev. Sci. Instrum.* 2290, 2 (1995)).

We have R-AXIS IV⁺⁺ for data collection with wide dynamic range.

Description of Rigaku R-Axis IV⁺⁺

| | |
|---------------|---------------------------------|
| Manufacture | Rigaku R-Axis IV ⁺⁺⁺ |
| IP material | BAS-MS |
| IP size | 300 mm × 300 mm |
| Pixel size | 50 / 100 / 200 μm |
| Pixel depth | |
| Type | double photomultiplier |
| ADC | 14 bit+12 bit (× 256) |
| Total | 10 ⁶ counts |
| Exposure time | 1 sec or more |
| Readout time | ca. 180 sec |
| Erase time | 60 sec (recommended) |

| Camera Length | S-range (Å ⁻¹) | | q-range (Å ⁻¹) | | spacing (Å) | | angle (deg.) | |
|--|----------------------------|-------|----------------------------|-------|-------------|------|--------------|-------|
| Normal setup with II+CCD | | | | | | | | |
| 600 mm | 0.0044 | 0.132 | 0.0276 | 0.829 | 7.6 | 227 | 0.23 | 6.77 |
| 1000 mm | 0.0026 | 0.079 | 0.0166 | 0.498 | 12.6 | 379 | 0.14 | 4.08 |
| 1800 mm | 0.0011 | 0.044 | 0.0069 | 0.276 | 22.7 | 909 | 0.06 | 2.23 |
| 2300 mm | 0.0009 | 0.033 | 0.0055 | 0.207 | 30.3 | 1136 | 0.05 | 1.70 |
| 3600 mm | 0.0007 | 0.022 | 0.0042 | 0.142 | 44.2 | 1400 | 0.04 | 1.14 |
| Medium Camera with IP | | | | | | | | |
| Long part (1 m) | 0.0015 | 0.088 | 0.0099 | 0.553 | 11.4 | 649 | 0.08 | 4.53 |
| Short part (150 mm) | 0.33 | 0.825 | 2.0735 | 5.183 | 1.2 | 3 | 16.53 | 36.5 |
| Very short camera with IP. w/o vacuum path or sample in vacuum. N/A VMIC | | | | | | | | |
| 300 mm | 0.0143 | 0.605 | 0.0902 | 3.802 | 1.7 | 70 | 0.73 | 28.52 |

Feature of available CCD

| Feature | Slow CCD | | Fast CCD | | Full controlled CCD | |
|--|---------------------------|-----------|---|-----------------|---|---------------|
| Manufacture | Hamamatsu C4880-10-14A | | Hamamatsu C4880-80-14A | | Hamamatsu C7300-12-NR | |
| Type | Cooled CCD (-50°C) | | Cooled CCD (-10°C) | | Interline cooled CCD (-10°C) | |
| Mode | Slow read | Fast read | Slow read | Fast read | Full frame | Binning 2 × 2 |
| Pixel size | 0.15 mm | | 0.25 mm | | 0.125 mm | 0.25 mm |
| Image size | 1000 × 1028 | | 655 × 493 | | 1280 × 1024 | 640 × 512 |
| Area | 150 mm × 150 mm | | 160 mm × 120 mm | | 150 mm × 140 mm | |
| Pixel depth | 14 bit | | 14 bit | 10 bit | 12 bit | |
| Saturation charge | 60000 electrons | | 30000 electrons | | 60000 electrons | |
| Exposure time | 150 msec ~ 60 sec | | 1 sec ~ 20 sec | 30 msec ~ 1 sec | 75 msec ~ 5 sec | 35 msec ~ |
| Minimum time slice | 6 sec | 1 sec | 3 sec | 30 msec | 75 msec | 35 msec |
| CCD shutter control | Mechanical | | Optical | | Optical | |
| X-ray shutter control | Via software | | Manual or external trig. | | Client mode. Full-controlled via software | |
| External Trigger | No supported | | Supported (start of sequential read) | | Full supported | |
| Recommended aperture size (without defocusing) | # 5 or more | | # 4 or more | | # 5 or more | # 4 or more |



Side view of XR-II+CCD

The references on characteristics of these detectors for the use of synchrotron solution x-ray scattering are;

XR-II + CCD : T. Fujisawa *et al.*, *J. Synchrotron Rad.*, 6, 1106-1114 (1999).

R-AXIS IV++ : T. Fujisawa *et al.*, *J. Appl. Cryst.*, 36, 535-539, (2003).

The detailed information for the practical usage of these detectors is also available in our web page.

(<http://www.riken.go.jp/lab-www/biochemi/Beamline/BeamlineInformation.html>)

PX station

PX station is closed only for RIEN use. More information on PX station is available in the web page of Division of Bio-Crystallography Technology.

(<http://bioxtal.harima.riken.jp>.)

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