

BL40B2 SAXS BM

1. Introduction

BL40B2 beamline is used for small-angle X-ray scattering (SAXS) for soft materials such as proteins, synthetic polymers, and lipids. We are facilitating the automation of measurement systems to improve user convenience, and as part of this effort, we installed a liquid chromatography system with an automatic sampler. Furthermore, we upgraded the vacuum chamber system for wide-angle X-ray scattering (WAXS) to improve its performance and also installed interlock equipment for the WAXS detector in the vacuum chamber to ensure stable operation. The beamline name was changed from “Structural Biology II” to “SAXS BM” on 1 May 2022.

2. Liquid chromatography with automatic sampler

Liquid chromatography is a separation technique used to obtain, for example, size-separated flows from dissolved biological and synthetic macromolecules. By connecting this equipment to the SAXS measurement system, Size Exclusion Chromatography SAXS (SEC-SAXS), which allows for structural analysis while isolating the target sample from a mixed one, becomes possible.

An ultrahigh-performance liquid chromatograph (Prominence, Shimadzu Co.) was installed to the beamline as a size-exclusion chromatograph system with an automatic sampler (see Fig. 1). Two sets of liquid feeding and degassing units were installed, allowing the precise control of flow rates for each analysis line and cleaning line. The analysis line is equipped with an

automatic sampler, a fluorescence detector and/or a differential refractive index detector. Two sets of columns are available, one placed between the automatic sampler and the detector on the analysis line and the other in the cleaning line. It is possible to switch these columns as needed. To measure SAXS, the flow is connected to a flow cell on the temperature-adjustable TS62 (Instec Inc. CO), which is placed downstream of, for example, the differential refractive index detector. This equipment is expected to reduce the burden on SEC-SAXS users.



Fig. 1. Liquid chromatography system and SAXS measurement system at BL40B2.

3. Interlock system for WAXS vacuum chamber

The WAXS vacuum chamber was installed in FY2021 ^[1] and is widely used by users who want to quickly switch between SAXS and WAXS measurements. During measurement, the vacuum level in the vacuum chamber is maintained below 1 Pa to prevent electrical discharge that could lead to damage within the detector. Since the vacuum level

may deteriorate owing to human error during setup or breakage of the vacuum window during user operation, we installed an interlock system that immediately cuts off the power supply to the detector when a drop in the vacuum level is detected. This is expected to minimize user-time loss.

4. Upgrade WAXS detector for vacuum chamber

When simultaneous SAXS and WAXS measurements are performed using the two detectors, there is a gap between respective images where scattered X-rays cannot be detected. In the WAXS detector placed near the sample, a frame surrounding its image sensor absorbs X-rays. This X-ray shadow is then projected onto a SAXS detector placed far from the sample. To reduce the loss of information from SAXS to WAXS, it is necessary to reduce the gap.

The WAXS detector was upgraded to have a newly designed frame and housing, as shown in Fig. 2. This allows the image sensor to be exposed outside the frame and the housing of the detector by tilting the detector by 45 degrees. Therefore, in the simultaneous measurements of SAXS and WAXS, missing information between SAXS and WAXS can be reduced, and improved modeling accuracy can be expected.



Fig. 2. WAXS detector with newly designed frame and housing.

Ohta Noboru, Gan Hongyi, and Sekiguchi Hiroshi
Scattering and Imaging Division, JASRI

Reference:

- [1] Ohta, N. & Sekiguchi, H. (2021). *SPring-8/SACLA Annual Report FY2021*, 74.