# Single crystal structure analysis using the large cylindrical Image-Plate camera

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## 1. Outline of lecture

In this lecture we study to observe the X-ray diffraction of single crystal using the large cylindrical Image-Plate (IP) camera at BL02B1. Thus, we perform the data reduction from image data and the determination of crystal structure.

## 2. The advantage of 2-D detector for data collection of single crystal X-ray diffraction

In general, there are two kind of two dimensional (2-D) detector for data collection of single crystal X-ray diffraction, which one is IP, the other is a charge coupled device (CCD) detector. The advantages of 2-D detector are as follows;

1) A number of diffraction spots can be observed at one X-ray shot. It means total measurement time is reduced.
2) Because the measured image is the projection image of reciprocal space, we can easily understand a diffuse scattering or spots based on super-structure.
3) It is easy to re-perform data reduction from measured image after data collection because all diffraction spots are recorded on images.

These advantages are very important factor even for synchrotron X-ray diffraction experiment, which is limited experiment time.

## 3. Image Plate (IP)

IP of two dimension detector is the plastic film coated by the phosphor; $BaFBr:Eu^{2+}$ ($Eu^{2+}$ doped BaFBr). It is possible for IP to record X-ray diffraction as a distribution of 'color centers' in phosphor, which can proportionally estimate the intensity of X-ray. 'Color centers' generated by X-ray diffraction are read out by counting the intensity fluorescence ($\lambda \approx 390$ nm), which stimulated by scanning the surface of IP by He-Ne laser ($\lambda \approx 633$ nm) beam. After reading out data, color centers are erased by the visible light exposure, therefore, we can reuse the same IP for next measurement.

The advantages of IP are high dynamic range, large active area, good linearity of detected X-ray intensity and good efficiency for the wide X-ray wavelength. On the other hand, the mechanical scanning of large area causes longer read-out time than CCD detector which directly read out by electronic signals. For the details of IP and CCD detector, refer the papers [1, 2].

## 4. Large cylindrical IP camera

In order to the development of the investigation of relationship between structure and

function, the large cylindrical image-plate camera was installed in BL02B1 beamline since 2007 (Figure 1) [3]. The features of this camera are as follows,

1) Large size image plate detector (350 x 683 mm)

2) Two types of goniometers (single axis goniometer and three axes 1/4χ goniometer)

3) Helium flow type low temperature measurement system (20~400 K)

The details of this camera are tabled in Table 1.

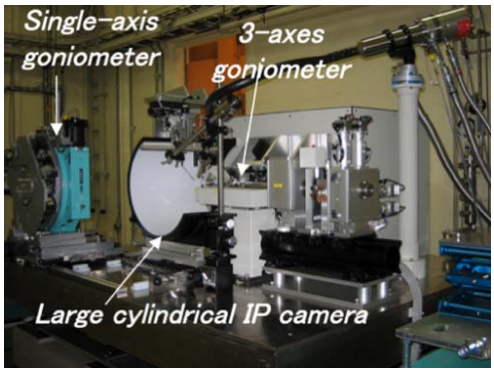


Figure 1 Large cylindrical IP camera

Table 1

Specifications of large cylindrical IP camera

| | |
|---|---|
| Size of IP | 350 mm x 683 mm |
| Pixel size | 100 μm x 100 μm |
| Camera radius | 191.3 mm |
| Resolution on 2θ | 0.030°/pixel |
| 2θ coverage | -60° ～ +145° |

For the structure and electron density analysis, we must collect the X-ray intensity data set with high accuracy. The advantages described in section 3 satisfy the requirements for the structure and electron density analysis. The large camera radius (191.3 mm) realized to collect the high resolution data using high energy X-ray beam and to install the equipments for the external field response experiment.

There are two goniometers on the large cylindrical IP camera (Figure 1). The 1/4 χ -axis goniometer can rotate the crystal to collect the complete data set with high angle 2θ. On the other hand, the single-axis goniometer can be installed the large and/or heavy equipment to adapt for the various experiment conditions such as the low- and high- temperature cryostat, the humidity controller chamber, the gas absorption control system and so on.

The data collection and reduction software are prepared for the external user, which have almost same interface as that of conventional instrument. Basically, it is easy to understand to perform the data collection and reduction with the large cylindrical IP camera.

## 5. Observation of a reciprocal lattice map and three-dimensional structure determination of organic compound

In this lecture, we will carry out the sequential procedure of single crystal structure analysis with a single crystal of organic compound. The contents are observation of reciprocal lattice map, extraction of the diffraction intensity data from IP images and

determination of three-dimensional structure. In this lecture, the experiment procedure is as follows,

1) Brief introduction of the large cylindrical IP camera.
2) Hold the single crystal on the top of the glass capillary by a glue.
3) Mount the goniometer head to the large cylindrical IP camera.
4) For centering of the crystal, adjust the crystal position to the center of goniometer with screw driver so that the crystal stays on the rotation center of goniometer, which the crystal remains in X-ray beam during measurement.
5) Set the collimator and beam stop to the IP camera. The light and TV monitor should be turned off. The light guide should move not to make a shade of X-ray diffractions.
6) Close the experimental hutch (Exit sequence) and open down-stream shutter (DSS) to induce the synchrotron radiation into experimental hutch.
7) Input the measurement conditions (ω, Δω, φ, χ and exposure time) to the measurement software. For evaluation of the crystal, a few diffraction images (ω=0, 45, 90°) are measured to index to the diffraction spots.
8) Check the shape of diffraction spots.
9) For indexing, in order to know the orientation of measurement crystal, indexes of diffraction spot (*hkl*) are assigned with the data reduction software. If you success the indexing, the three types of boxes (green, yellow and red boxes) are covered on the diffraction spots. 'Right-click' on the box indicates the *hkl* index of that diffraction spot. If you find a series of *hkl* index (*h00*, *0k0* or *00l*), you can understand a rule between the index and diffraction spot.
10) If you success the indexing correctly, you get a primary lattice parameter and lattice volume. You can approximately estimate the number of molecule in unit cell ($Z$) with the equation, $Z=V/(20\times N)$, where $V$ is lattice volume $N$ is the number of the non-hydrogen atom in molecule.
11) Input the measurement conditions (ω, Δω, φ, χ and exposure time) to collect the whole diffraction spots in the measurement software.
12) During the measurement, the detail of the compound and measurement are explained.
13) For integration, diffraction intensities are extracted with the data reduction software after measurement. The resolution is set to 0.7Å.
14) For merge data, intensities of diffraction spot extracted from each diffraction image are merged. The data reduction software provides the crystal information file and the intensity file (*texray.inf* and *f2plus.dat*).
15) With two files and structure determination software (shelxs-97 [4]), the crystal structure is solved. The atom position and thermal parameters are refined with

the structure refinement software (shelxl-97 [4]).

16) Discussions on the determined structure.

## 6. Summary

In this lecture, we have determined the three dimensional (3-D) crystal structure of organic compound using the large cylindrical IP camera at BL02B1. The single crystal structure analysis can also determine the absolute structure as well as the 3-D structure with a precision of 0.001Å. In pharmaceutical field, the determination of absolute structure is essential to take a patent of a new medicine.

The visualization of 3-D structure gives one of the most important information to investigate the relations between the crystal structure and the functions, even if they are induced by small change of electronic state. In BL02B1 beamline, the large cylindrical IP camera will lead the precise structure analysis of single crystal such as investigation of relationship between structure and function.

## 7. References


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4. G. M. Sheldrick, (2008). *Acta Cryst.* A**64**, 112–122.