Crystallographic analysis of micro crystals II

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Crystal structure determination with laboratory X-ray source requires the size of single crystals about a few hundred μm. The high flux synchrotron radiation from SPring-8 is particularly useful for a compound which does not form larger crystals. In this study, single crystal diffraction measurements of small organic crystals, which did not diffract enough reflections by the laboratory X-ray source, were carried out on the branched experimental hutch A of Hyogo beamline (BL24XU).

There are several technological problems that must be solved before SR experiment, which include the selection of a small single crystal and transfer to a glass fiber of dimensions comparable to the crystal. Micromanipulators and micro pipette pullers, developed mainly for biotechnology applications, were used for this task.

A fine needle crystal ($C_{25}H_{33}NO_2$), 0.005 ×0.010×0.25 mm, was mounted on an Imaging Plate diffractometer Rigaku R-AXIS4 implemented in the experimental Hutch A of BL24XU. The wavelength of the SR was set to 0.834 Å. 36 oscillation photograph, an oscillation angle of 6° with 1° overlap, 12 min. exposure, with crystal-to-detector distance 80 mm and the beam size 0.3mm, were taken within 9 hours. The indexing of peaks and the integration was carried out using the program PROCESS. 8294 reflections up to 2θ value of 60° were integrated (completeness : 84.9%). The crystal belonged to the space group $P2_12_12_1$, Z = 4. The crystal structure was solved by using SHELXS86 and refined using SHELXL93. Current R1 value is 0.105 for 1414 reflections ($I > 2\sigma(I)$). Further analysis of diffraction data is in progress.