

Development of Electronic Excited State Crystallography by Imaging Plate Detector

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Introduction. Crystallographic studies on excited-state molecular crystals can provide direct geometrical information of those of light-induced structural changes or transition states in solid-state chemical reactions. For achievement of the electronic excited-state crystallography, a very high performance detector which can detect small changes in Bragg peak positions and intensities must be required. The vacuum IP camera at BL02B1 can detect X-ray diffraction in low background without air-diffracted noises. This detector will be useful for micron-size small crystal diffraction studies and for detecting small changes in intensities diffracted from a crystal under external perturbations.

Experimental and Results. We have made several preliminary experiments with the vacuum camera in order to evaluate the ability of this detector, using 29.2 keV X-ray source from BL02B1 bending magnet beam line, monochromatized by Si(311) monochromator. IP plate was attached to the cylindrical chamber, and X-ray irradiations were made under vacuum. Crystals were mounted on a HUBER 7-axis diffractometer employed for ϕ -axis rotation photographs.

Structure analysis of Ru complex:

Diffractions from a single crystal of $[\text{Ru}(\text{en})_2(\text{NO})(\text{OH}_2)]\text{Cl}_3$ (en = diaminoethane)

with the size of $86 \times 60 \times 60 \mu\text{m}^3$ were collected by the vacuum IP camera. Cell parameter determination, integration of intensities, and data reductions were performed by using DENZO program. Although the crystal was deteriorated during exposure due to loss of solvent molecules under vacuum, structure was successfully solved and refinement was converged to $R=0.11$ for 61 parameters and 495 unique reflections in the range of $4^\circ < 2\theta < 30^\circ$ at present stage (Fig.1).

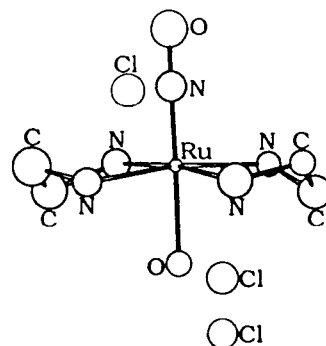


Fig. 1. Structure of $[\text{Ru}(\text{en})_2(\text{NO})(\text{OH}_2)]\text{Cl}_3$

Structure analysis of Pd dimeric complex :

A single crystal of $\text{Pd}_2(\text{dtbu})_4$ [dtbu = dithio-butanoate] with the size of $100 \times 50 \times 25 \mu\text{m}^3$ was used for X-ray diffraction experiments. Cell parameters determination, intensity data processing were performed by DENZO program. Further structure analyses are in progress.