Development of Electronic Excited State Crystallography by Imaging Plate Detector

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Introduction. Crystallographic studies on excited-state molecular crystals provide direct geometrical information of those of light-induced structural changes or transition states in solid-state chemical reactions. For achievement of the excited-state crystallography, a very high performance detector which can detect small changes of Bragg peaks both in positions and intensities, must be required. SR x-ray beam with low divergence and high-energy resolution can give good contrast diffraction images, which are especially useful for diffuse streak. .

Experimental and Results. We have made x-ray diffraction experiments with flat IP detector, using 30.0 keV X-ray source from BL02B1 bending magnet beam line, monochromatized by Si(111) monochromator. Crystals were mounted on a HUBER 7-axis diffractometer, under N_2 gas-flow type cryostat employed for ϕ-axis rotation photographs.

Diffuse streaks of halogen-bridged 1-D Pt complex: X-ray oscillation photographs of a $[Pt_2(EtCS_2)_4I]$ single crystal along 1-D chain (*b*) axis were taken at 300, 350, and 380 K (Fig.1). At 300 K, additional 2×*b* and 3*b* satellite layers were observed. The 2×*b* spots were changed to diffuse streaks in 350 K photograph. The 3×*b* spots were dimmed with temperature increase, and disappeared at 380 K. Those structural changes are caused by valence ordering and disordering of Pt atoms which affects electronic properties.

X-ray diffraction of tetra-copper complex crystal under UV illumination : A single crystal of $[Cu_4I_4(py)_4]$ (*py* = C_5H_5N) with the size of 100×50×25 μm^3 was used for X-ray diffraction measurements. The crystal was illuminated periodically by high-pressure Hg lamp (250 W). In order to record diffraction spots under both light-on and -off stages on the same IP, the IP plate was shifted 2.0 mm along the rotation axis during light-on periods. Light-on and -off periods were repeated five times for each oscillation range($\Delta\phi$ = 8 deg). Indexing and integration in both light-on and -off images were made separately. Further data reductions and structure analyses have been in progress.

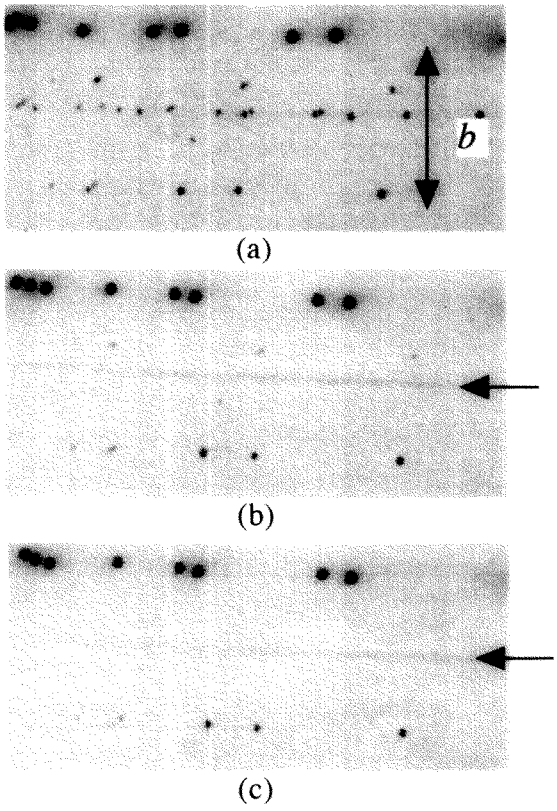

Fig. 1. Oscillation Photograph
(a) 300 K, (b) 350 K, (c) 380 K