# Highly Accurate Measurement of Electron Density Distribution in Transition-Metal Complexes with SR and Vacuum Camera

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**1. Introduction** The results of our experiments for $KNiF_3$ using the vacuum camera are reported. The vacuum camera is an evacuated cylinder putting an imaging plate(IP) on the inside wall. The camera radius is 75mm and the size of the IP is 440x220mm$^2$. It enables to measure structure factors under vacuumwith extremely low noise. Since the electron density of $KNiF_3$ has been extensively studied, it is a good sample to assess the quality of the X-ray beams of BL02B1 and test the performance of the vacuum camera.

**2. Experimental** Incident beam slit of 0.5 mm wide was used. X-rays with energies 50 kev monochromated by Si(311) were utilized with the SR beam current from 20mA to 10mA. 50keV SR beam were employed to make the extinction effect minimum. XAFS from a Zr foils were measured to determine the wavelenghs. The vacuum camera is mounted on a off-center 7-axis Huber diffractometer and $\phi$-axis permits the rotation of the crystal. The deviation of the orientation of the camera from zero $2\theta$-position was adjusted by the $\omega$-axis rotation of the diffractometer. The necessary adjustment ranges from 0.1 to 0.3°. Incident X-rays passing through slits 0.5 mm$\phi$ or 1.0 mm$\phi$which comes into the camera passing through a beam paths 50mm long and 3mm$\phi$ at the frontal and rear sides were used. Once we adjusted the beam position, we did not need to adjust it again. The direct beam-stooper was located outside the camera to avoid high background. The oscillation photographs were taken with the overlap of the rotation angle to do the scaling between IP plates. A total of nine photographs were taken by rotating the crystal by 10° sixty times. The scan speed 10°/min was selected so that single scanning is accomplished within a minute to avoid the error due to decay of the incident SR beam intensity. The rotation angle were overlapped by 3° for the scaling of intensities recorded on separate IP plates. Accordingly photographs of rotation angle between 35 and 109° were taken.

**3. Results** The position of X-ray beam was stable. The background intensity was reduced one hundredth of the measured one without evacuating the camera. Even zero background area were found on a significant part of an IP plate, when $KNiF_3$ crystal with a diameter of 60$\mu$m was attached on a glass capillary with a diameter of 10$\mu$m. The orientation matrix was obtained by DENZO using the phtograph rotating $\phi$-axis from 0° to 5°. The intensity data is now being analyzed by HIPPO98 and the detailed results will be presented elsewhere.