

実験報告書様式(一般利用課題・成果公開利用)

(※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。)

	提出日 Date of Report 2013.1.31
課題番号 Project No. 2012A0054 実験課題名 Title of experiment Neutron diffraction analysis of a crystalline-state 3-1 photo-isomerization of cobaloxime complexes 実験責任者名 Name of principal investigator Takashi OHHARA 所属 Affiliation CROSS-Tokai	装置責任者 Name of responsible person Takashi OHHARA 装置名 Name of Instrument/(BL No.) SENJU (BL18) 実施日時 Date and time of Experiment Jun. 24, 2012, 9:00 ~ Jul. 2, 2012, 9:00

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form. Some alkyl groups in cobaloxime complexes, model compounds of the Vitamin B12, are well known to isomerize on exposure to visible light with retention of its single crystal form. In this proposal, the applicant carried out a single crystal neutron diffraction measurement of a photo-irradiated cobaloxime complex, (3-cyanopropyl- d_{α},d_{α})[(R)-phenylethylamine] cobaloxime, to observe deuterium-migration accompanied by the photo-isomerization of the alkyl group and finally to understand the behavior of the cobalt atom in the intermediate stage of the photo-isomerization.
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2. 実験方法及び結果 (実験がうまくいかなかった場合、その理由を記述してください。) Experimental method and results. If you failed to conduct experiment as planned, please describe reasons. <u>Method</u> A 3.5 x 2.0 x 0.8 mm single crystal of (3-cyanopropyl- d_{α},d_{α})[(R)-phenylethylamine] cobaloxime was attached to a aluminum stick using epoxy glue and mounted to the goniometer at the end of 4K-cryostat. Neutron diffraction intensities were collected on the following conditions. ----- Crystal orientation: 8 Exposure time: 8 hrs. / orientation Temperature: 70 K -----
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2. 実験方法及び結果(つづき) Experimental method and results (continued)

Result

In this experiment, low temperature diffraction measurement by using the 4K-cryostat was performed to reduce thermal motion of the cobaloxime complex molecule. However, the 2-axes goniometer at the end of the 4K-cryostat was unstable and both axes were unexpectedly moved during the measurement. So, we adjusted parameters of goniometer controller, temperature of 4K-cryostat and wiring of the heat-path around the sample position, and tried low-temperature measurement again and again. Finally, we successfully obtained 8 diffraction images (8 crystal orientations). The measurement temperature was 70K.

Figure 1 shows a diffraction image of the cobaloxime complex. Although quality of the crystal was not so good and consequently each Bragg spot was broad, minimum value of d-spacing was 0.5 Å, relatively good for organometallic complex crystal.

Data processing was performed by software STARGazer. Obtained cell parameters were $a=9.209(2)$ Å, $b=13.831(6)$ Å, $c=9.016(3)$ Å, $\beta=99.79(5)$ ° (monoclinic). These values well agreed with those of X-rays. After the data processing, intensities of 20298 reflections were obtained. Structure analysis by using the neutron diffraction data was now under progress.

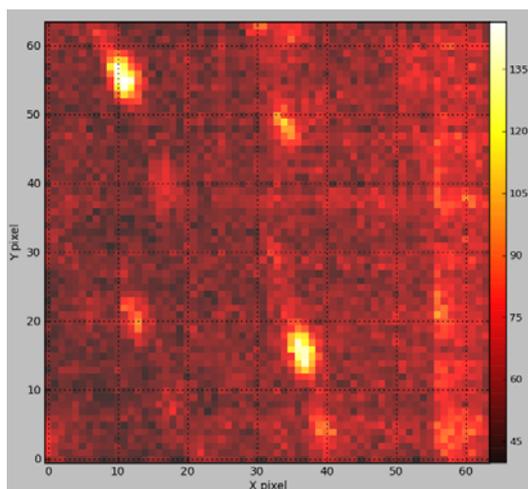


Figure 1 Diffraction image of the cobaloxime complex observed on the No.11 detector (the highest 2θ angle). Minimum value of d-spacing was less than 0.5 Å.