


(※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。)

 <b>MLF Experimental Report</b>	提出日 Date of Report
課題番号 Project No. 2012A0093 実験課題名 Title of experiment Structural Analysis of a [NiFe]Hydrogenase Model Complex by Neutron Powder Diffraction 実験責任者名 Name of principal investigator Seiji Ogo 所属 Affiliation Kyushu University	装置責任者 Name of responsible person Toru Ishigaki 装置名 Name of Instrument/(BL No.) iMATERIA (BL No. 20) 実施日 Date of Experiment 2012/06/12, 9:00 a.m. ~ 2012/06/13, 9.00 a.m.

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.

Sample; [NiFe] hydride complex 1 (see the figures 1 and 2).

Formula;  $C_{53}H_{90}BFeN_2O_9NiP_3S_2$ .

Lattice parameters;

$a = 19.314(4)$ ,  $b = 14.483(3)$ ,  $c = 21.771(5)$ ,

$\alpha = 90$ ,  $\beta = 103.400(3)$ ,  $\gamma = 90$ ,

$V = 5923(2)$ , Space group;  $P2_1/n$ .

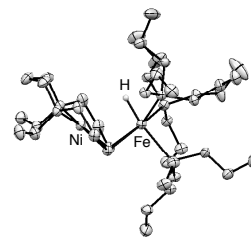
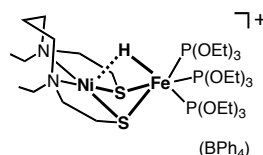


Figure 1. Structure of 1. Figure 2. An ORTEP drawing of 1.

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.

The powder sample of a [NiFe] hydride complex,  $[Ni(N_2S_2)FeH\{P(OEt)_3\}_3](BPh_4)$  [1]( $BPh_4$ ), was enclosed in a vanadium cylindrical cell ( $\phi 6 \times 50$  mm) under a He atmosphere and was measured by a neutron powder diffractometer (iMATERIA). The sample was cooled off using a spare freezer because the planned 4 K cryostat was not usable by the trouble of the He transfer tube. In order to shorten the cooling time, the sample and cold head were cooled by using liquid nitrogen and were set into a vacuum chamber. The measurement temperature was 120 K.

The neutron powder diffraction measurement was performed with a double frame mode for 16 hours. Figure 3 shows a neutron powder diffraction pattern for the hydride complex 1 at a back scattering detector bank (the vertical axis is the diffraction intensity and the horizontal axis is the Time-of-Flight (TOF) [ $\mu\text{sec}$ ]). Although the broad peaks assigned to be the Bragg reflections were observed in low-Q domain, the sharp peaks to be seen by normal diffraction patterns were not able to observe in this measurement.

## 2. 実験方法及び結果(つづき) Experimental method and results (continued)

At this moment, we think the reasons for this unexpected result as follows:

- (1) Since the sample is air-sensitive, crystal characteristics have decreased through the sample preparation.
- (2) Therefore, the Bragg reflections become broad and are buried among the backgrounds derived from the hydrogen atoms of the sample.

Further investigation is needed to make the details clear.

Since we were not able to observe clear Bragg reflections by this measurement, the Rietveld analysis using Z-Code is difficult. Therefore, we are now considering how we can extract the information from this diffraction pattern.

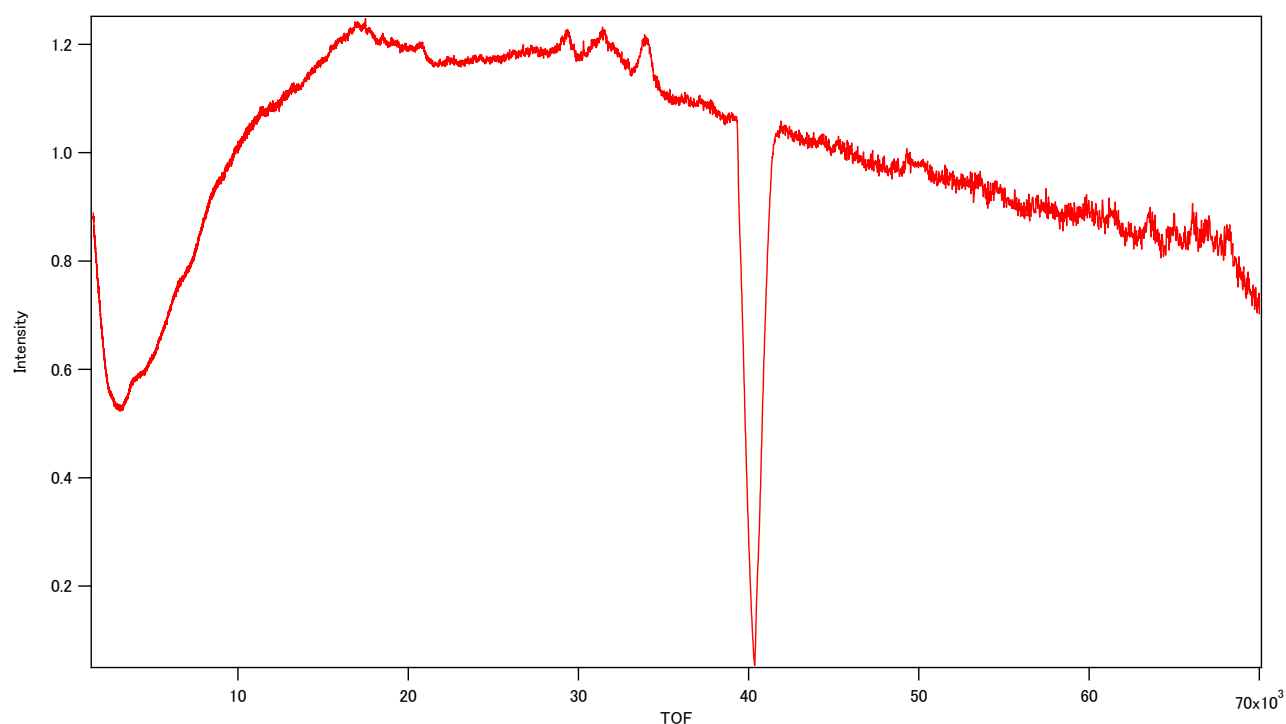


Figure 3. Neutron powder diffraction pattern for [NiFe]hydride complex 1 (Back scattering detector bank).