


(※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。)

 MLF Experimental Report	提出日 Date of Report
課題番号 Project No. 2014B0312 実験課題名 Title of experiment Neutron diffraction study of [NiFe] hydrogenase 実験責任者名 Name of principal investigator Taro Tamada 所属 Affiliation Japan Atomic Energy Agency	装置責任者 Name of responsible person Katsuhiro Kusaka 装置名 Name of Instrument/(BL No.) iBIX/BL-03 実施日 Date of Experiment Mar.21~Apr.01

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.
<p>[NiFe] hydrogenase ($C_{3956}H_{6099}N_{1067}O_{1140}S_{24}Fe_{12}MgNi$)</p>

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.
<p>Large crystals of [NiFe] hydrogenase were obtained using a deuterated 2-methyl-2,4-pentanediol as a precipitant. The crystals were mounted on LithoLoops mode of polyimide and they were flash frozen in a nitrogen-gas stream at 100 K. The neutron diffraction experiments were performed at the BL03 beamline (iBIX) with the beam power of 400 kW. The diffraction intensities were measured by 30 detectors using a ZnS type scintillator with wavelength shift fiber. Several crystals were used for diffraction data collection with exposure times of 2-4 hours to check diffraction power of the crystals. Finally, diffraction data sets were collected using a large crystal. The wavelength range was set to 1.8-5.8 Å and exposure time for each data set was 10.5 hours. Total of 24 data sets were collected at different orientations of the crystal in 11 experimental days. Diffraction spots were observed up to 2.0 Å resolution (Figure 1). Diffraction intensities were integrated using the <i>STAR</i>Gazer program. The data sets were merged to 2.0 Å resolution (Table 1). The crystal belongs to the space group $P2_12_12_1$ with $a = 98.5$ Å, $b = 126.8$ Å, $c = 66.6$ Å.</p>

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

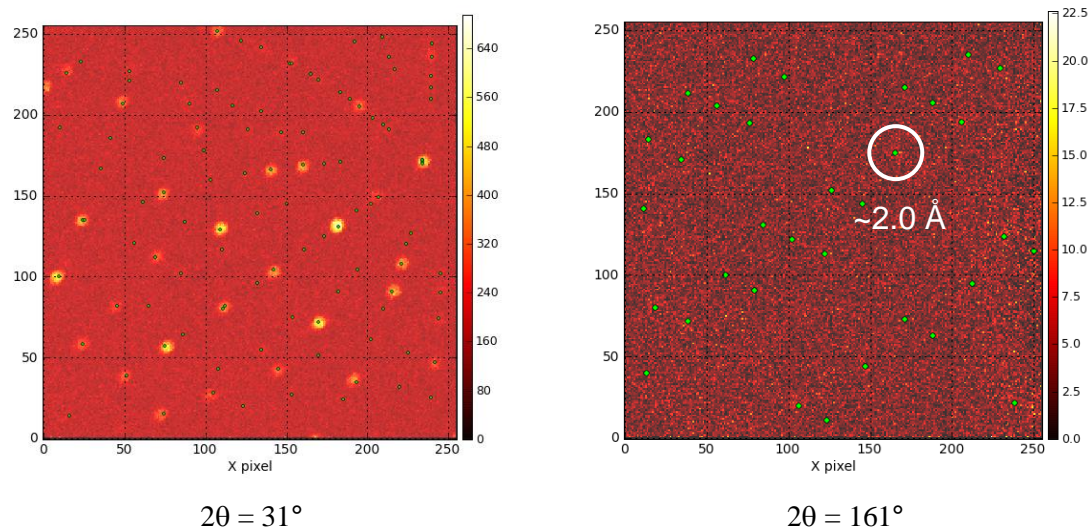


Figure 1 Neutron diffraction images from the crystal of [NiFe] hydrogenase.

Table 1 Diffraction data statistics

Resolution (Å)	20-2.0 (2.07-2.00)
Space group	$P2_12_12_1$
a, b, c (Å)	98.5, 126.8, 66.6
Completeness (%)	96.2 (94.0)
$I/\sigma(I)$	4.0 (1.6)
R_{int} (%)	23.7 (44.9)