（ ※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 2014/12/
課題番号 Project No. 2013B0032 実験課題名 Title of experiment Neutron crystallographic analysis of human farnesyl pyrophosphate synthase complexed with bisphosphonate 実験責任者名 Name of principal investigator Takeshi Yokoyama 所属 Affiliation University of Toyama	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 2014/3/4 – 2014/3/16

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 No. 1. Farnesyl pyrophosphate synthase (C1806H(D)2804N464O524S12,D2O, NaCl,CD3COOD, CD3COONa, MgCl2), single crystal (3.5 mm3) in capillary tube.

Sample No. 2. Farnesyl pyrophosphate synthase (C1806H(D)2804N464O524S12,D2O, NaCl,CD3COOD, CD3COONa, MgCl2), single crystal (4.5 mm3) in capillary tube.

Sample No. 3. Farnesyl pyrophosphate synthase (C1806H(D)2804N464O524S12,D2O, NaCl,CD3COOD, CD3COONa, MgCl2), single crystal (3.5 mm3) in capillary tube.

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.

TOF-Diffraction experiments of the sample No. 1 and 2 were tested with 2.7-6.2 Å of the wavelength range and we considered that the sample No. 1 was more suitable for the complete data collection (Figure 1. Therefore, we sellected the sample No. 1 for the diffraction collection. Twenty-three data sets were collected at RT with 2.7-6.2 Å wavelength range and 8 h exposure time (Figure 2).

Figure 1.
Crystal picture of the sample No. 1.

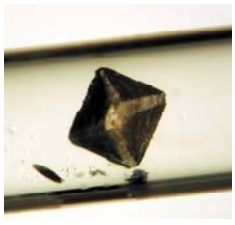
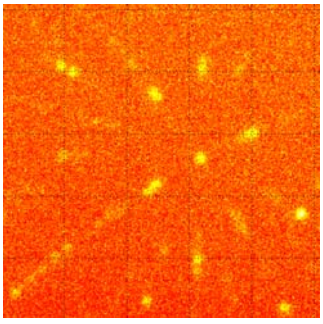


Figure 2.
TOF-diffraction image of the detector No. 14. The ranges of the resolution and wavelength are 2.7 – 6.3 and 3.1 – 5.8 Å, respectively.



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

Diffraction spots were integrated and scaled up to 2.1 Å resolution using STARGazer program which have been developed for the processing iBIX data. X-N joint refinement was carried out using PHENIX.REFINE and the atomic model, which include hydrogens and deuterium, was refined, and final Rfactor and Rfree for the X-ray data were 17.1 and 20.4%, and those for the neutron data were 29.8 and 33.6%, respectively. Neutron structure analysis revealed that

Figure 3. Protonation state of the nitrogen atom of RIS bound to FPPS. D-fourier map of neutron omitted deutrium atoms are shown as green mesh (4.0 σ).

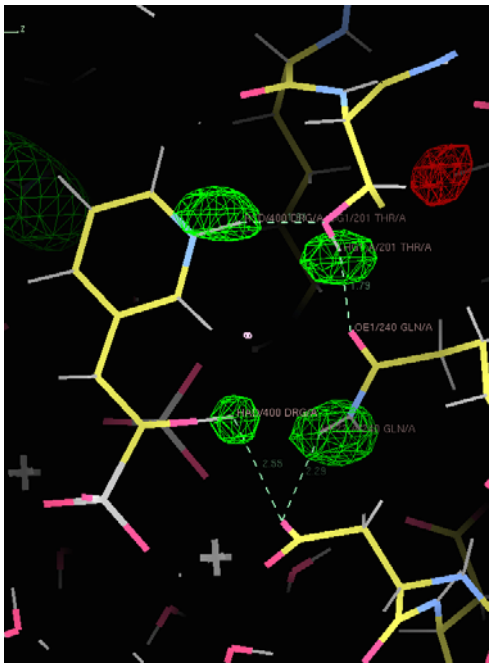


Figure 4. Water molecules around RIS. RIS. D-Fourier maps of X-ray omitted the whole water molecules are indicated as blue mesh (4.0 σ) and neutron omitted deuterium atoms as green mesh (4.0 σ).