MLF Experimental Report	提出日 Date of Report
課題番号 Project No. 2009A0044 実験課題名 Title of experiment Neutron diffraction analysis of disodium uridine 5'-monophosphate heptahydrate 実験責任者名 Name of principal investigator Yoko Sugawara 所属 Affiliation Kitasato University, School of Science	装置責任者 Name of responsible person Ichiro Tanaka 装置名 Name of Instrument/(BL No.) IBARAKI biological diffractometer (iBIX) / BL No.03 実施日 Date of Experiment 2009/12/12 – 2009/12/21

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.

Disodium uridine 5'-monophosphate heptahydrate, $C_9H_{11}N_2O_9PNa_2.7H_2O$

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.

An as-grown crystal of disodium urisine 5′-monophosphate (Na_2UMP) heptahydrate with approximate dimensions of 3 × 3 × 1 mm^3 was sealed in a quartz capillary tube. Neutron intensity data were collected using a IBARAKI biological diffractometer (iBIX) at BL-03 in MLF over 8 days at room temperature.

Data reduction was carried out using STARGazer. Neutron intensities of 56184 reflections up to 0.7 Å were collected. Excluding reflections whose intensities were inconsistent among the equivalents, refinements were carried out using 12851 unique reflections up to 0.8 Å.

Crystal data are: orthorhombic, $P2_12_12_1$, a=8.942(1), b=23.057(9), c=58.55(1)Å, Z=6. Hydrogen positions were determined by iteration of difference Fourier syntheses starting from the coordinates of non-hydrogen atoms of UMP molecules determined by X-ray analysis. All atoms except sodium ions were refined with isotropic atomic displacement parameters. Sodium ions were partly disordered and fixed at the X-ray positions. At the present stage, an R value is 0.136 for 7776 observed reflections ($|F_o| > 4\sigma(F_o)$). Absorption and extinction corrections are in progress.

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

There are six UMP molecules, twelve sodium ions, and 42 crystal water molecules in an asymmetric unit. A part of water molecules and sodium ions are disordered. All 66 hydrogen positions of six independent UMP molecules and 68 hydrogen sites of 44 crystal water sites were determined.

The nuclear distribution, obtained by the maximum entropy method (MEM), is shown in Figure 1(a). Light blue cages indicate the negative peaks which correspond to hydrogen atoms. The hydrogen bonding networks were determined based on the hydrogen positions (Figure 1(b)). Fluctuation of crystal water molecules will be discussed based on the refined structure from the viewpoint of humidity and temperature dependent phase transitions.

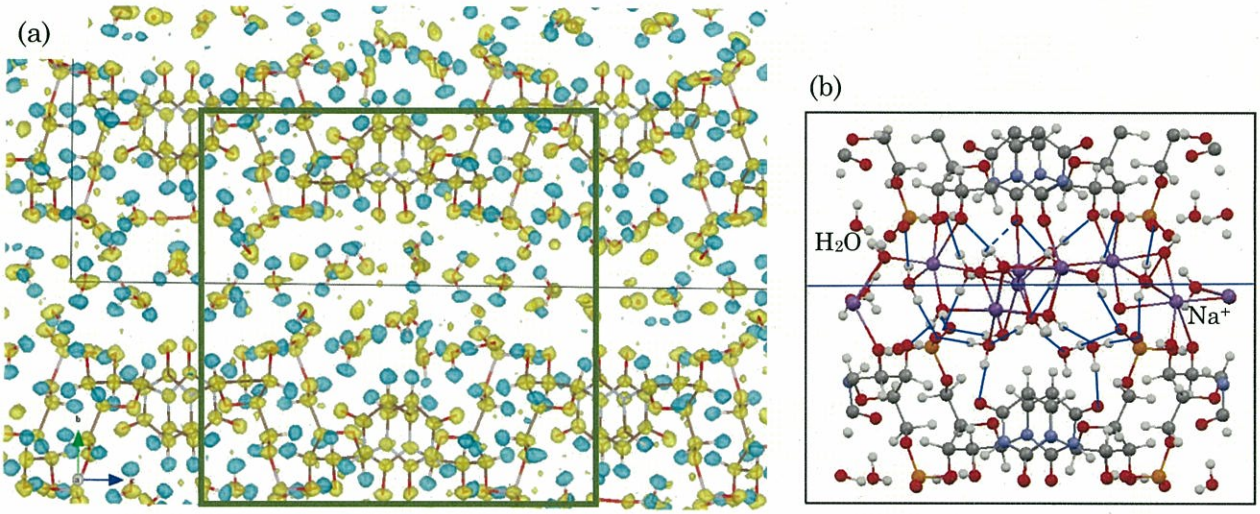

Fig. 1 Crystal structure of disodium uridine 5'-monophosphate heptahydrate

(a) The nuclear distribution obtained by the maximum entropy methods (MEM) using a program ENIGMA (Tanaka et al., J. Appl. Crystallogr., 35 (2002) 282-286.) and drawn by a program VESTA (Momma & Izumi, J. Appl. Crystallogr., 41 (2008)653-658.). Yellow and light blue cages indicate positive peaks of non-hydrogen atoms and negative peaks of hydrogen atoms, respectively.

(b) A crystal structure of the disordered region indicated by a green box in Fig. 1(a). Sodium coordination schemes and hydrogen bonding networks are shown by purple-red and blue lines, respectively.