 MLF Experimental Report	提出日 Date of Report 2011.1.12
課題番号 Project No. 2010A0029 実験課題名 Title of experiment Neutron diffraction analysis of disodium uridine 5'-mono-phosphate heptahydrate in low temperature phase 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 2010/10/22 – 2010/10/24

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, $C_9H_{11}N_2O_9PN_{a_2} \cdot 7H_2O$

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.
<p>In X-ray diffraction of disodium uridine 5'-monophosphate (Na_2UMP) hydrate, diffused scattering was observed at room temperature. It would be originated in the disorder of crystal water and sodium ion sites. Around 200K it changed to Bragg spots and the unit cell enlarged. The phenomenon causes attention because ordering of hydration water around biomolecules at low temperature is known to correlate the function and the glass transition of proteins. Under such background, we carried out the preliminary neutron data collection at 123K.</p>
<p>Two crystals with approximate dimensions of $3 \times 1.6 \times 1 \text{ mm}^3$ (crystal A) and $3.5 \times 2.5 \times 2.0 \text{ mm}^3$ (crystal B) were prepared. Crystal A was small but its quality was better than that of crystal B. Neutron intensity data were collected using an IBARAKI biological diffractometer (iBIX) at BL-03 in MLF at 123K. For each crystal, data collection of two crystal orientations, where the beam was set to be parallel to the <i>b</i> and <i>c</i> axes, was planned. Data collection of crystal A was finished, but that of crystal B was interrupted due to the trouble of the moderator. Data reduction was carried</p>

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

out using STARGazer.

One example of the observed data was show in Figure 1. Crystallographic data of the room temperature phase were: space group $P2_12_12_1$, $a = 22.99(5)$, $b = 8.92(2)$, $c = 58.45(12)$ Å. Comparing the X-ray reflections, intensities of the superlattice reflections corresponding to the cell doubling along the a axis were rather weak, which indicated that the ordering of sodium ion sites mainly contributed to them. At the same time, super lattice reflections at unpredicted positions were observed. They could be indexed if the cell parameter b tripled, and might reflect ordering of hydrogen positions of crystal water molecules.

In the next experiments, we are going to carry out the neutron intensity measurements of deuterated Na_2UMP hydrate ($\text{C}_9\text{H}_8\text{D}_3\text{N}_2\text{O}_9\text{P} \cdot \text{Na}_2 \cdot 7\text{D}_2\text{O}$) at 123K in order to confirm the superlattice of the low temperature phase.

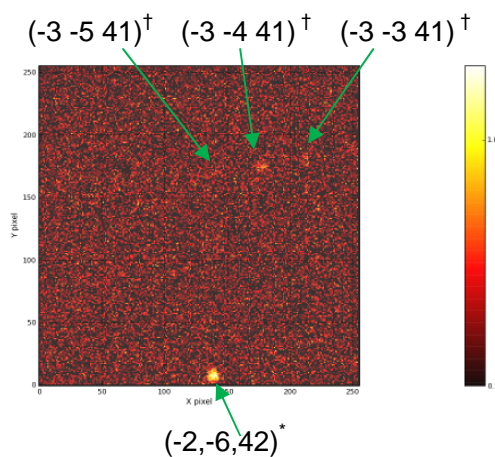


Figure 1 One example of the observed reflections with the Miller indices based on the tentative cell parameters ($a = 22.99 \times 3$, $b = 8.92 \times 2$, $c = 58.45 \text{Å}$, α, β, γ approximately 90°) at 123K. Beam power 120kW; exposure time 6 hours; * fundamental-lattice reflections; † superlattice reflections.