Neutron Diffraction Analysis of Hydrogen-Bonding Networks of the Inosine 5'-Monophosphate Hydrate Stable in Medium Humidity Range II

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Disodium inosine 5′-monophosphate (Na_2IMP) hydrates undergo phase transitions depending on temperature and relative humidity conditions. We have carried out a series of neutron diffraction analyses [1], and clarified reconstruction of hydrogen-bonding networks accompanied by the phase transitions. Besides, nuclear distributions of oxygen and hydrogen atoms indicated fluctuating figures of crystal water molecules which are hydrated to biomolecules. In the case of the heptahydrate, which is stable in the relative humidity range 40 - 60 %, there were uncertain sites of a part of sodium ions and water molecules [2]. Under such circumstances, we reinvestigated neutron diffraction analysis of the heptahydrate.

A crystal with approximate dimensions 11.4 × 7.7 × 1.4 mm^3 was sealed in a quartz tube. The crystal volume was about 2.5 times larger than that of the previous experiment. Data collection was carried out using a four-circle diffractometer, FONDER, at T2-2 of JRR-3 in JAERI (Tokai). With the neutron wavelength of 1.24 Å, 1563 unique reflections up to 0.76 Å resolution (2θ = 110 °) were measured at room temperature. Two crystal settings were used due to the limitation of the diffractometer. There remained inconsistency in intensities of a few reflections after the absorption correction.

Initial difference Fourier maps were calculated based on non-hydrogen atoms of IMP molecule determined by X-ray analysis. In a series of full-matrix least squares refinement cycles fixing non-hydrogen atoms of IMP molecule followed by difference Fourier syntheses, sodium ions, O and H atom sites of water molecules were assigned and refined. Then the sites of IMP molecule and those of sodium ions and water molecules were refined alternatively. All hydrogen atoms of the IMP molecule and 13 hydrogen sites of seven crystal water were determined (Fig. 1). The R factor is 0.075 for 1385 observed reflections ($|F_o| > 4\sigma(F_o)$) at the present stage. The crystal structure is essentially the same to that of the previous study except the atomic displacement parameters of the uncertain sites of sodium ion (Na3) and water molecule (W7).

References [1] Sugawara et al., ISSP-NSL Research Highlights 2005, 38-39(2005). [2] Sugawara et al., ISSP Activity Report on Neutron Scattering Research, **10**, 86-87(2003).

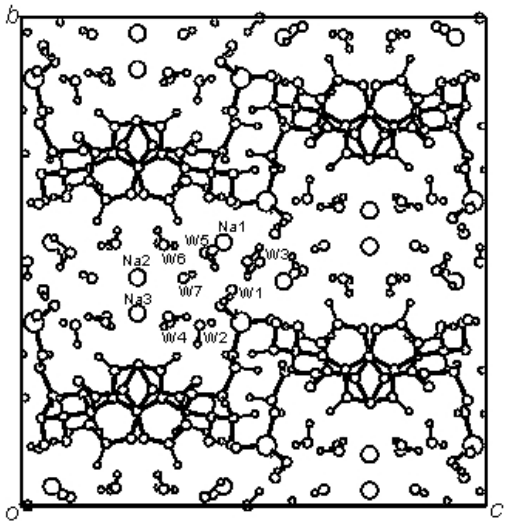

Fig. 1. Crystal structure of Na_2IMP.$7H_2O$ viewed along the *a* axis.