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Crystal Structure and Humidity-Induced Phase Transition of Disodium Salt of Adenosine 5′-Monophosphate

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CRYSTAL STRUCTURE AND HUMIDITY-INDUCED PHASE TRANSITION OF DISODIUM SALT OF ADENOSINE 5'-MONOPHOSPHATE

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Abstract Crystal structure of disodium adenosine 5'-monophosphate dodecahydrate ($Na_2AMP.12H_2O$) was determined. Crystal data are $C222_1$, a= 6.8768(6), b=19.287(3), c=41.099(6) Å, and Z=8. Bases are stacked to make a columnar structure along the a axis. There exist water layers parallel to the ab and ac planes, and molecular columns are isolated by the water layers . Sodium ions are coordinated by six water molecules, and columnar structure of hydrated sodium ions runs parallel to the molecular column. Phase transition of Na_2AMP undergoes between 0 to 90 % relative humidity by four steps. Coupled with increase or decrease of the number of water molecules, cell parameters b and c seem to change.

INTRODUCTION

We are carrying out crystallographic and spectroscopic analyses of humidity-induced phase transitions of a series of nucleoside and nucleotide crystals, e.g. guanosine and disodium adenosine 5'-triphosphate (Na_2ATP).[1-3] In these crystals, the number of crystal water molecules is a function of relative humidity, and reversible phase transitions take place. The transition accompanies reconstruction of hydrogen-bonding networks and conformational changes of nucleoside and nucleotide molecules. Phase transition of disodium adenosine 5'-monophosphate (Na_2AMP) undergoes between 0 to 90 % relative humidity by four steps.[4] Recently, we successfully determined the original crystal

structure of $Na_2AMP.12H_2O$.

RESULTS AND DISCUSSION

Crystals of Na_2AMP were obtained from a water-ethanol solution. The crystals crack when they are taken out from a mother liquor. Therefore data collection was carried out using a crystal sealed in a capillary. Crystal data are: orthorhombic, $C222_1$, a= 6.8768(6), b=19.287(3), c=41.099(6) Å, and Z=8. The structure was determined by direct methods using SHELXS-86,[5] and refined by the full-matrix least-squares method with isotropic temperature factors. An R value is 0.12 for 2983 independent reflections at the present stage. The ribose moiety and two water molecules near the ribose group are highly disordered probably due to partial loss of the crystal water molecules.

The crystal structure of $Na_2AMP.12H_2O$ is shown in Figure 1. The base moieties of AMP are stacked with a spacing of 3.4 Å to form a molecular column along the a axis (Figure 2a). Water-intermediated hydrogen bonds exist between the columns. Each of the two symmetrically independent sodium ions, Na1 and Na2, is coordinated by six crystal water molecules. The neighboring octahedra share two water molecules, and columnar structure of hydrated sodium ions (Figure 2b) runs parallel to the molecular column. There are water regions which are parallel to the *ab*

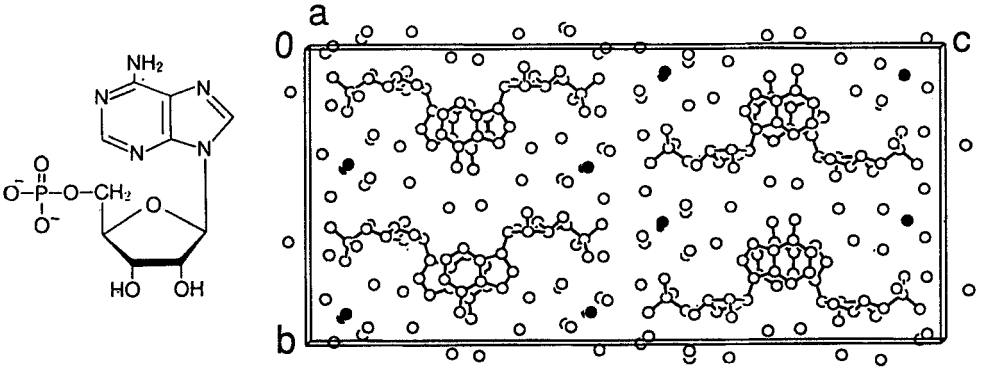

FIGURE 1 Structural formula of AMP and crystal structure of $Na_2AMP.12H_2O$. Sodium ions and crystal water molecules are indicated by black and white circles, respectively.

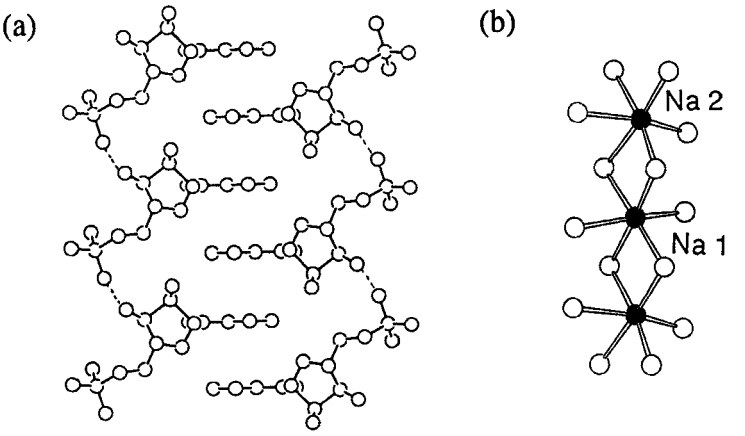

FIGURE 2 Columnar structures of AMP molecules (a) and hydrated sodium ions (b). Dotted lines indicate hydrogen bonds between AMP, and white bonds indicate coordinations from crystal water molecules to the sodium ions.

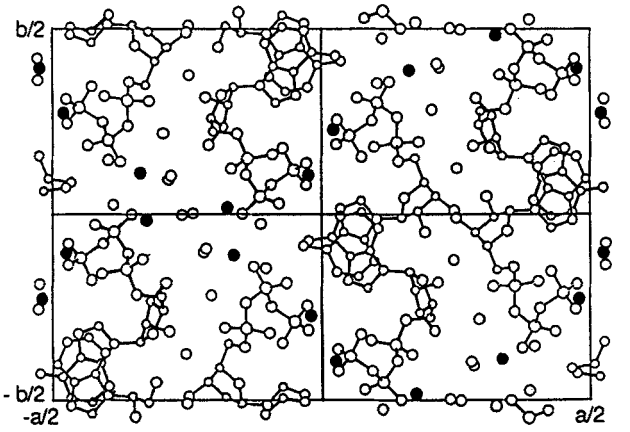

FIGURE 3 Crystal structure of $Na_2ATP.3H_2O$. Sodium ions and crystal water molecules are indicated by black and white circles, respectively.

and *ac* planes, and they isolate the molecular columns.

We have reported the humidity-induced phase transition of nucleoside and nucleotide crystals, i.e. guanosine, Na_2ATP, and disodium cytidine 5'-monophosphate (Na_2CMP). In these crystals, the base moieties of the molecules are stacked to form a columnar structure as is the case of Na_2AMP. In addition, neighboring molecular columns interact

through coordinations to common sodium ions and/or direct hydrogen bonds, and molecular layers are formed. There are water region between the molecular layers (Figure 3). Coupled with increase or decrease of crystal water molecules, the dimension of one axis which is perpendicular to the water layers lengthens or shortens. For example in the case of Na_2ATP, the cell parameter *a* of the dihydrate and the trihydrate are 27.572(5) and 30.254(13) Å, respectively. The variations of cell parameters *b* and *c* are less than 1 %. The situation is a little different in the case of Na_2AMP. In the original crystals of $Na_2AMP.12H_2O$, the molecular columns are completely isolated by the water layers which extend to two directions (Figure 1). We pursued the humidity-induced phase transition of Na_2AMP by means of X-ray powder diffraction analysis.[4] Determination of cell parameters of the dehydrated forms from powder diffraction data is in progress. Both the cell parameters *b* and *c* seem to change with the dehydration process.

As previously mentioned, in the crystal of Na_2AMP the ribose moiety and two water molecules near the ribose are disordered. It is indicated that the conformational change of the ribose moiety would be accompanied by the loss of these water molecules as is the case of Na_2ATP and guanosine.[1,2] The columnar structure made of hydrated sodium ions would play a role in retention of the crystal lattice.

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