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The crystal structures of barium inosine-5'-phosphate and disodium inosine-5'-phosphate. By NOBUYA NAGASHIMA, Central Research Laboratories, Ajinomoto Co., Inc., Kawasaki, Japan, and YOICHI IITAKA Faculty of Pharmaceutical Sciences, University of Tokyo, Hongo, Tokyo, Japan

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The crystal structures of barium inosine-5'-phosphate ( $C_{10}H_{11}O_8N_4P$ .Ba.6H<sub>2</sub>O) and disodium inosine-5'-phosphate (C10H11O8N4P.Na2.7.5H2O) have been determined. The barium salt contains two crystallographically independent molecules in the structure of the space group  $P2_12_12_1$ . The structure of the sodium salt (space group, C2221) has been deduced from that of the barium salt. The structural parameters defining the conformations of the molecules of the nucleotides are given as a preliminary report.

The crystal structures of barium inosine-5'-phosphate (Ba-IMP) and disodium inosine-5'-phosphate (Na-IMP) have been determined. The two structures are not isomorphous but they are homologous. Therefore, the result of the structure determination of Ba-IMP makes it possible to elucidate the structure of a Na-IMP.

	Table 1. Crystal	data		
	$\begin{array}{c} Ba-IMP\\ (C_{10}H_{11}O_8N_4P.Ba.6H_2O)\end{array}$	$\begin{array}{c} Na-IMP \\ (C_{10}H_{11}O_8N_4P.Na_2.7.5H_2O) \end{array}$		
System	Orthorhombic	Orthorhombic		
a b c	21·45 Å 8·85 21·75 4128.9 Å 3	23·06 Å 8·64 21·92 43674 Å 3		
Z Space group	8 $P2_{1}2_{1}2_{1}2_{1}$	8 C2221		
$D_m$ $D_x$	1.935 g.cm <sup>-3</sup> 1.928	1.62 g.cm <sup>-3</sup> 1.616		

Number of independent observed reflexions used for the structure determination. 2819 2165

Nucleotido			Conformation about the	- (C)	Displaced	m (P)
Nucleotide	$\varphi_{00}$	$\varphi_{\rm OC}(\omega)$	C(4) = C(5) bond	$\varphi_{\rm CN}^{(0)}$	sugar atomor	$\varphi_{OP}$
5'-TMP(Ca)	63°	57°	gg(b)	-48°	C(3') endo (deoxyribose)	156°
5'-AMP	78	40	88	18	C(3') endo	177
5'-UMP(Ba)	54	70	88	-43	C(2') endo	176
3'-CMP, orthorhombic form	74.7	43.8	gg	$-42.1^{(d)}$	C(2') endo	
3'-CMP, monoclinic form	73.7	45.5	88	39·3(d)	C(2') endo	
3'-AMP	56.7	171.7	gt	$- 3.9^{(d)}$	C(3') endo	
AUP			Ū			
adenosine residue	73	45	88	- 55	C(2') endo	
uridine residue	62	57	88	-5	C(3') endo	
5'-IMP(Ba)						
molecule I	58	49	88	$-46^{(d)}$	C(2') endo	170
molecule II	57	51	88	$-34^{(d)}$	C(2') endo	160
5'-IMP(2Na)	61	56	88	$-43^{(d)}$	C(2') endo	172
	Nucleotide 5'-TMP(Ca) 5'-AMP 5'-UMP(Ba) 3'-CMP, orthorhombic form 3'-CMP, monoclinic form 3'-AMP AUP adenosine residue uridine residue 5'-IMP(Ba) molecule I molecule II 5'-IMP(2Na)	Nucleotide $\varphi_{00} o^{(a)}$ 5'-TMP(Ca) $63^{\circ}$ 5'-AMP785'-UMP(Ba)543'-CMP, orthorhombic form74·73'-CMP, monoclinic form73·73'-AMP56·7AUPadenosine residueadenosine residue73uridine residue625'-IMP(Ba)molecule I5857Smolecule II575'-IMP(2Na)61	Nucleotide $\varphi_{00}(a)$ $\varphi_{0C}(a)$ 5'-TMP(Ca) $63^{\circ}$ $57^{\circ}$ 5'-AMP $78$ $40$ 5'-UMP(Ba) $54$ $70$ 3'-CMP, orthorhombic form $74 \cdot 7$ $43 \cdot 8$ 3'-CMP, monoclinic form $73 \cdot 7$ $45 \cdot 5$ 3'-AMP $56 \cdot 7$ $171 \cdot 7$ AUPadenosine residue $62$ $57$ s'-IMP(Ba)molecule I $58$ $49$ molecule I $58$ $49$ molecule II $57$ $51$ 5'-IMP(2Na) $61$ $56$	Nucleotide $\varphi_{00}^{(a)}$ $\varphi_{0c}^{(a)}$ Conformation about the $\varphi_{0c}^{(a)}$ C(4')-C(5') bond5'-TMP(Ca)63°57° $gg(b)$ 5'-AMP63°57° $gg(b)$ 5'-LMP(Ba)5470 $gg$ 3'-CMP, orthorhombic form74·743·8 $gg$ 3'-CMP, monoclinic form73·745·5 $gg$ 3'-AMP56·7171·7 $gt$ AUP $gg$ 57 $gg$ adenosine residue7345 $gg$ 5'-IMP(Ba) $gg$ $57$ $gg$ $S'-IMP(Ba)$ $gg$ $57$ $51$ molecule I5849 $gg$ $S'-IMP(2Na)$ 6156 $gg$	Nucleotide $\varphi_{OO}^{(a)}$ $\varphi_{OC}^{(a)}$ C(A')-C(5') bond $\varphi_{CN}^{(c)}$ 5'-TMP(Ca)63°57° $gg(b)$ -48°5'-AMP63°57° $gg(b)$ -48°5'-AMP7840 $gg$ -185'-UMP(Ba)5470 $gg$ -433'-CMP, orthorhombic form74·743·8 $gg$ -42·1(a)3'-CMP, monoclinic form73·745·5 $gg$ -39·3(a)3'-AMP56·7171·7 $gt$ - 3·9(a)AUPadenosine residue6257 $gg$ -55uridine residue6257 $gg$ -55of -IMP(Ba)molecule I5849 $gg$ -46(a)molecule II5751 $gg$ -34(a)5'-IMP(2Na)6156 $gg$ -43(a)	Conformation about theDisplacedNucleotide $\varphi_{00}(a)$ $\varphi_{00}(a)$ $C(4')-C(5')$ bond $\varphi_{CN}(e)$ sugar atom(f)5'-TMP(Ca) $63^{\circ}$ $57^{\circ}$ $gg(b)$ $-48^{\circ}$ $C(3')$ endo (deoxyribose)5'-AMP7840 $gg$ $-18$ $C(3')$ endo (deoxyribose)5'-AMP7840 $gg$ $-43$ $C(2')$ endo5'-UMP(Ba)5470 $gg$ $-43$ $C(2')$ endo3'-CMP, orthorhombic form $74\cdot7$ $43\cdot8$ $gg$ $-42\cdot1(a)$ $C(2')$ endo3'-CMP, monoclinic form $73\cdot7$ $45\cdot5$ $gg$ $-39\cdot3(a)$ $C(2')$ endo3'-AMP $56\cdot7$ $171\cdot7$ $gt$ $-3\cdot9(a)$ $C(3')$ endoAUP $a$ $a$ $62$ $57$ $gg$ $-55$ $C(2')$ endoadenosine residue $73$ $45$ $gg$ $-55$ $C(2')$ endo $S'-IMP(Ba)$ $gg$ $-46(a)$ $C(2')$ endo $S'-IMP(Ba)$ molecule I $57$ $51$ $gg$ $-34(a)$ $C(2')$ endo $5'-IMP(2Na)$ $61$ $56$ $gg$ $-43(a)$ $C(2')$ endo

(a)  $\varphi_{00}$ : torsion angle O(1')-C(4)-C(5')-O(5') and  $\varphi_{00}$ : torsion angle C(3')-C(4')-C(5')-O(5'), defined by Shefter & Trueblood (1965).

(b) Abbreviation of the torsion angles  $\varphi_{00}$  and  $\varphi_{0c}$ , indicating the relative positions of the sugar and O(5') atom. g and t denote the gauche and trans conformations, respectively.

(c)  $\varphi_{CN}$ : torsion angle about the glycosidic bond defined by Donohue & Trueblood (1960) and

(a) by Sundaralingam & Jensen (1965).
 (b) φ<sub>OP</sub>: torsion angle C(4')-C(5')-O(5')-P.

(f) Sugars are ribofranose except 5'-TMP(Ca).

- 1. Calcium thymidylate (Trueblood, Horn & Luzzati, 1961).

- Adenosine-5'-phosphate (Kraut & Jensen, 1963).
  Barium uridine-5'-phosphate (Shefter & Trueblood, 1965).
  Orthorhombic form of cytidine-3'-phosphate (Sundaralingam & Jensen, 1965).
- 5.
- 6.
- Monoclinic form of cytidine-3'-phosphate (Bugg & Marsh, 1967). Adenosine-3'-phosphate (Sundaralingam, 1966). β-Adenosine-2'-β-uridine-5'-phosphoric acid (Shefter, Barlow, Sparks & Trueblood, 1964). 7.
- Barium inosine-5'-phosphate (present work). 8.
- Disodium inosine-5'-phosphate (present work). 9



O(6) C(6) N(7) C(5 O(II) N(1) O(2′) C(8) C(2) C(2') N(3) N(9) C(1') C(3' O(3 0(1) o c/2 Ō(1') C(5') C(4')

(b)

Fig. 1. Molecular packing viewed along the *a* axis. Metal ions and water molecules are omitted. Broken lines indicate intermolecular hydrogen bonds. (*a*) Ba-IMP (space group  $P2_12_12_1$ ). Crosses (×) represent pseudo twofold rotation axes between molecules I and II. (*b*) Na-IMP (space group  $C222_1$ ).

The unit-cell dimensions determined from precession photographs are shown in Table 1 together with other crystal data. Intensities were measured by visual estimation of the equiinclination Weissenberg photographs taken with Cu  $K\alpha$  radiation. The structure of Ba-IMP determined by the heavy atom method was refined by block-matrix leastsquares calculations, with anisotropic temperature factors, to an R value of 0.12. The *a*-axis projection of the structure is shown in Fig. 1(a). It is seen that the two crystallographically independent molecules (I and II) in the asymmetric unit take very similar conformations. However, they differ somewhat in the internal rotation angles such as those around the glycosidic bond ( $\varphi_{CN}$ ) and the C(5')–O(5') bond  $(\varphi_{OP})$ . By inspection of Fig.1 and disregarding the metal ions and water molecules, it is expected that a slight displacement of molecule I relative to molecule II would produce a twofold rotation axis (parallel to a) between them. If the molecules I and II moved so that the rotation axis passing through the point (0,0,0) was generated, then the space group of the structure would become  $C222_1$ . A gross feature of the structure of Na-IMP found in this way has been refined by difference Fourier syntheses followed by several cycles of block-matrix least-squares calculations. The R value at the present stage is 0.12. In Fig.1(b) is shown the *a*-axis projection of the structure of Na-IMP.

Some of the well known structural parameters obtained for nucleotides are summarized in Table 2. A full account of the present work will be published elsewhere.

## References

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