

Structure of Disodium Uridine 5'-Phosphate Heptahydrate

BY T. P. SESHADRI AND M. A. VISWAMITRA

ICMR Centre on Genetics and Cell Biology, Physics Department, Indian Institute of Science,
Bangalore-560012, India

AND G. KARTHA

Centre for Crystallographic Research, Roswell Park Memorial Institute, Buffalo, NY 14623, USA

(Received 1 November 1979; accepted 6 December 1979)

Abstract. $\text{Na}_2[\text{C}_9\text{H}_{11}\text{N}_2\text{O}_9\text{P}]\cdot 7\text{H}_2\text{O}$, $M_r = 494.0$, orthorhombic, $C222_1$, $a = 22.880$ (7), $b = 8.877$ (3), $c = 19.592$ (9) Å, $Z = 8$, $V = 3979.2$ Å³. The $\text{Cu } K\alpha$ intensity data consisted of 1005 unique reflections. Final $R = 14.5\%$. This nucleotide shows no unusual conformational features. The uracil base has an *anti* conformation about the glycosidic bond ($\varphi_{\text{OO}} = 44.4^\circ$). The furanose ring conformation is *C(2')-endo, gauche-gauche* with $\varphi_{\text{OO}} = -75.5^\circ$ and $\varphi_{\text{OC}} = 49.6^\circ$.

Introduction. An investigation of uridine 5'-phosphate as its disodium salt ($5'\text{-UMPNa}_2\cdot 7\text{H}_2\text{O}$) was made to determine the differences between its geometry and that of $5'\text{-dUMPNa}_2$ (Viswamitra, Seshadri & Post, 1975) when the sugar moiety is changed from deoxyribose to ribose. The present analysis shows that $5'\text{-UMPNa}_2$ has the common nucleotide conformational features, unlike $5'\text{-dUMPNa}_2$.

The sample ($0.88 \times 0.06 \times 0.05$ mm) was obtained from Sigma Chemical Co., USA, as the disodium salt. It was crystallized from acetone/water mixtures by a diffusion technique (Seshadri & Viswamitra, 1974). The crystal data (see *Abstract*) were obtained from

preliminary X-ray studies. Systematic absences were: hkl for $h + k$ odd, $hk0$ for $h + k$ odd, $0kl$ for k odd and $h0l$ for h odd. Data were collected on an XRD-490 diffractometer. Density measurements with carbon tetrachloride/bromoform mixtures ($d_m = 1.64$, $d_c = 1.64$ Mg m⁻³) indicated the presence of seven water molecules, assuming two Na^+ ions per nucleotide molecule as indicated by the supplier. The numbering of the atoms is shown in Fig. 1.

The structure was obtained from Patterson, successive structure factor and electron-density calculations. Block-diagonal least-squares refinement with isotropic temperature factors gave $R = 28\%$ for the nucleotide molecule alone. A difference map computed at this stage had too many peaks of the same height compared to the number of water O and Na atoms present in the structure. Several of them were found to be unsatisfactory as they showed abnormal temperature factors and low occupancies on refinement. In view of this it was not possible to locate the Na and O atoms individually and calculations were carried out finally with O atom scattering factors only. Similar problems concerning water molecules had been encountered in $5'\text{-UMPBa}$ (Shefter & Trueblood, 1965) and $5'\text{-IMPNa}$ (Rao & Sundaralingam, 1969). The final R with anisotropic thermal parameters was 14.5%. The H atoms of the molecules were fixed wherever possible from their expected geometry.

Discussion. The positional parameters of non-hydrogen atoms are listed in Table 1 and those of the H atoms in Table 2.* The bond lengths and angles involving the non-hydrogen atoms and H atoms are listed in Tables 3 and 4 respectively. The average e.s.d.'s in bond distances are $\sigma(\text{P-O}) = 0.019$ and $\sigma(\text{C-N, O, C}) = 0.037$ Å; those in bond angles vary from 1.0 to 3.1° .

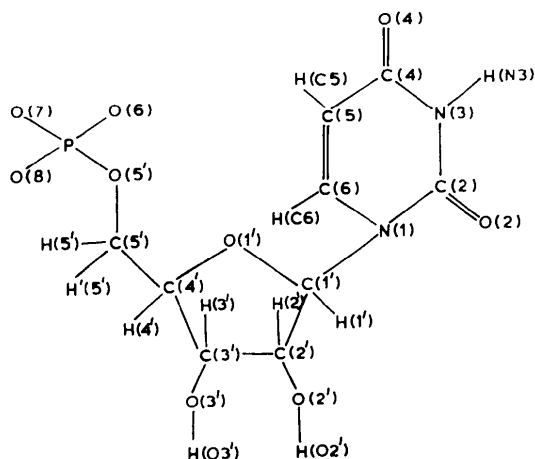


Fig. 1. Numbering in uridine 5'-phosphate.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34953 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates ($\times 10^4$) of the heavy atoms with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>
P	1249 (3)	1256 (8)	2328 (4)
O(6)	1062 (7)	2908 (17)	2244 (9)
O(7)	878 (8)	186 (24)	1926 (12)
O(8)	1263 (7)	824 (20)	3073 (9)
O(5')	1891 (8)	1205 (19)	2037 (9)
C(5')	2180 (10)	-238 (25)	2019 (13)
C(4')	2785 (12)	-154 (34)	1778 (16)
C(3')	3192 (11)	987 (29)	2087 (14)
O(3')	3777 (7)	323 (21)	2091 (10)
C(2')	3240 (13)	2188 (32)	1502 (15)
O(2')	3738 (8)	3034 (22)	1478 (10)
C(1')	3172 (11)	1260 (27)	856 (13)
O(1')	2771 (8)	62 (22)	1011 (10)
N(1)	2921 (9)	2166 (24)	274 (11)
C(2)	3283 (13)	2440 (37)	-274 (13)
O(2)	3843 (9)	2028 (23)	-267 (9)
N(3)	3070 (11)	3308 (25)	-796 (12)
C(4)	2501 (13)	3702 (42)	-812 (15)
O(4)	2316 (9)	4431 (24)	-1302 (10)
C(5)	2135 (16)	3229 (43)	-266 (17)
C(6)	2395 (14)	2527 (31)	270 (16)
W(1)	5487 (17)	0	0
W(2)	5000	850 (40)	7500
W(3)	9835 (11)	677 (38)	1336 (20)
W(4)	5282 (12)	144 (66)	5958 (26)
W(5)	6176 (10)	296 (28)	8907 (14)
W(6)	5507 (12)	2250 (36)	1891 (23)
W(7)	9703 (12)	1993 (69)	9357 (19)
W(8)	8996 (18)	46 (57)	5630 (20)

Table 2. Fractional coordinates ($\times 10^3$) of the H atoms with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>
H(5')	195 (2)	-117 (6)	169 (2)
H'(5')	224 (2)	-67 (5)	254 (2)
H(4')	210 (2)	368 (6)	323 (3)
H(3')	313 (2)	134 (6)	265 (3)
H(2')	281 (2)	308 (7)	155 (3)
H(1')	360 (1)	73 (3)	71 (1)
H(N3)	340 (2)	342 (6)	-116 (3)
H(C5)	170 (3)	378 (7)	-24 (3)
H(C6)	205 (2)	225 (5)	69 (2)

This nucleotide shows no unusual conformational features, unlike 5'-dUMPNa₂.

The orientation of the uracil base relative to the sugar is *anti* ($\chi_{\text{CN}} = 44.4^\circ$) which is close to the 43.0° in 5'-UMPBa.

C(2') deviates from the plane defined by C(1')-C(3')-C(4')-O(1') by 0.53 Å on the same side as C(5'). The conformation is, therefore, C(2')-*endo* (Table 5). However, when referred to the three-atom plane the ribose conformation is C(2')-*endo*, C(3')-*endo*. The conformation of the ribose moiety is the same as in 5'-UMPBa, but in 5'-dUMPNa₂ one of the sugars had C(1')-*exo* puckering.

Table 3. Bond distances (Å) and angles ($^\circ$) involving non-hydrogen atoms

P-O(6)	1.52 (2)	C(2')-O(2')	1.37 (3)
P-O(7)	1.50 (2)	C(1')-O(1')	1.44 (3)
P-O(8)	1.51 (2)	C(1')-N(1)	1.51 (3)
P-O(5')	1.58 (2)	N(1)-C(2)	1.38 (4)
O(5')-C(5')	1.44 (3)	N(1)-C(6)	1.25 (4)
C(5')-C(4')	1.47 (4)	C(2)-O(2)	1.33 (4)
C(4')-C(3')	1.50 (4)	C(2)-N(3)	1.37 (4)
C(4')-O(1')	1.51 (4)	N(3)-C(4)	1.35 (4)
C(3')-O(3')	1.46 (3)	C(4)-O(4)	1.23 (4)
C(3')-C(2')	1.57 (4)	C(4)-C(5)	1.42 (5)
C(2')-C(1')	1.52 (4)	C(5)-C(6)	1.36 (5)
O(6)-P-O(7)	113.1 (1.1)	C(2')-C(1')-O(1')	106.9 (2.0)
O(6)-P-O(8)	110.6 (1.0)	O(1')-C(1')-N(1)	108.1 (1.9)
O(6)-P-O(5')	104.3 (1.0)	C(2')-C(1')-N(1)	112.3 (2.1)
O(7)-P-O(8)	111.1 (1.1)	C(1')-O(1')-C(4')	106.9 (1.9)
O(7)-P-O(5')	108.7 (1.1)	C(2)-N(1)-C(6)	122.0 (2.5)
O(8)-P-O(5')	108.8 (1.0)	C(2)-N(1)-C(1')	117.0 (2.1)
C(5')-O(5')-P	117.4 (1.5)	C(6)-N(1)-C(1')	120.7 (2.3)
C(4')-C(5')-O(5')	113.3 (2.1)	N(1)-C(2)-O(2)	121.4 (2.5)
C(3')-C(4')-C(5')	119.4 (2.3)	N(1)-C(2)-N(3)	117.9 (2.5)
C(3')-C(4')-O(1')	109.1 (2.1)	O(2)-C(2)-N(3)	120.2 (2.5)
O(1')-C(4')-C(5')	107.8 (2.2)	C(2)-N(3)-C(4)	120.4 (2.5)
C(2')-C(3')-C(4')	102.0 (2.2)	N(3)-C(4)-C(5)	118.3 (2.9)
C(2')-C(3')-O(3')	102.4 (2.0)	N(3)-C(4)-O(4)	119.0 (2.8)
C(4')-C(3')-O(3')	107.4 (2.1)	O(4)-C(4)-C(5)	122.7 (3.0)
C(1')-C(2')-C(3')	103.4 (2.2)	C(4)-C(5)-C(6)	117.4 (3.1)
C(1')-C(2')-O(2')	110.8 (2.3)	C(5)-C(6)-N(1)	123.1 (2.9)
C(3')-C(2')-O(2')	117.3 (2.3)		

Table 4. Bond distances (Å) and angles ($^\circ$) involving H atoms

C(5')-H(5')	1.19 (5)	C(1')-H(1')	1.13 (3)
C(5')-H'(5')	1.09 (7)	N(3)-H(N3)	1.03 (6)
C(4')-H(4')	1.06 (6)	C(5)-H(C5)	1.10 (7)
C(3')-H(3')	1.15 (6)	C(6)-H(C6)	1.15 (6)
C(2')-H(2')	1.26 (6)		
H(5')-C(5')-O(5')	113 (3)	H(1')-C(1')-C(2')	110 (2)
H(5')-C(5')-C(4')	108 (3)	H(1')-C(1')-O(1')	108 (2)
H(5')-C(5')-H'(5')	107 (4)	H(1')-C(1')-N(1)	112 (2)
H'(5')-C(5')-O(5')	109 (3)	H(N3)-N(3)-C(2)	108 (4)
H'(5')-C(5')-C(4')	104 (3)	H(N3)-N(3)-C(4)	131 (4)
H(4')-C(4')-C(5')	101 (3)	H(C3)-C(5)-C(4)	116 (4)
H(4')-C(4')-C(3')	120 (3)	H(C5)-C(5)-C(6)	124 (4)
H(3')-C(3')-C(4')	119 (3)	H(C6)-C(6)-C(5)	110 (3)
H(3')-C(3')-C(2')	122 (3)	H(C6)-C(6)-N(1)	126 (3)
H(2')-C(2')-C(3')	109 (3)		
H(2')-C(2')-C(1')	109 (3)		

The conformation about the exocyclic C(4')-C(5') bond is *gauche-gauche* with the torsion angles $\varphi_{\text{OO}} = -75.5^\circ$ and $\varphi_{\text{OC}} = 49.6^\circ$ which are close to those observed in 5'-UMPBa (-67.4 and 55.2°) and significantly different from those found in 5'-dUMPNa₂ (171.1 , -64.7° , molecule *A*; 172.2 , -65.9° , molecule *B*).

The uracil base is planar (Table 5). C(5) is displaced by 0.07 Å from this plane. The bond distances and angles are close to those observed in other uridine compounds.

Table 5. Deviations (Å) of atoms from the least-squares planes through the uracil base and ribose moiety, and torsion angles (°) about the furanose ring bonds

Atoms not used in the calculation of the plane are indicated by asterisks.

$$\text{Plane (I)} \quad -0.7503X + 0.6555Y + 0.0857Z = 12.6052$$

$$\text{Plane (II)} \quad -0.2438X - 0.8577Y - 0.4526Z = 2.0957$$

	Plane (I)		Plane (II)
C(1')	-0.007	N(1)	-0.039
C(2')	0.525*	C(2)	0.037
C(3')	0.006	N(3)	-0.043
C(4')	-0.010	C(4)	-0.011
C(5')	1.021*	C(5)	0.068
O(1')	0.001	C(6)	-0.017
r.m.s. (Δ)	0.009	O(2)	0.040*
σ (r.m.s. Δ)	0.002	O(4)	-0.028
		r.m.s. (Δ)	0.041
		σ (r.m.s. Δ)	0.002

E.s.d.'s in the torsion angles are $\sim 3^\circ$.

C(4')-O(1')-C(1')-C(2')	-22.7
O(2')-C(1')-C(2')-C(3')	34.2
C(1')-C(2')-C(3')-C(4')	-31.3
C(2')-C(3')-C(4')-O(1')	18.6
C(3')-C(4')-O(1')-C(1')	1.9

The molecular packing viewed down b is shown in Fig. 2. The molecules pack as chains running parallel to c with waters situated between the chains. There is partial overlap of bases belonging to molecules related by the twofold axis parallel to a . The degree of overlap is similar to that in uracil (Stewart & Jensen, 1967).

The amino N(3) atom of the base forms a hydrogen bond with the phosphate O(8) atom at a distance of 2.8 Å. The O(2') and O(3') hydroxyl atoms of the ribose moiety form hydrogen bonds with the phosphate O(8) and O(6) atoms respectively.

Acta Cryst. (1980). B36, 927-930

Structure of Tetrakis[dichloroacetato- μ -(2-dimethylaminoethanolato)-copper(II)]

BY U. TURPEINEN, R. HÄMÄLÄINEN AND M. AHLGRÉN

Department of Inorganic Chemistry, University of Helsinki, SF-00100 Helsinki 10, Finland

(Received 25 September 1979; accepted 14 December 1979)

Abstract. $C_{24}H_{44}Cl_8Cu_4N_4O_{12}$, $[Cu_4(C_2HCl_2O_2)_4(C_4H_{10}NO)_4]$, monoclinic, $C2/c$, $a = 18.675$ (9), $b = 15.109$ (8), $c = 16.403$ (8) Å, $\beta = 101.33$ (7)°, $V = 4538$ Å³, $M_r = 1118.4$, $Z = 4$, $D_x = 1.638$ Mg m⁻³, μ (Mo $K\alpha$) = 2.44 mm⁻¹. R is 0.050 for 1779 reflexions. The complex has a tetrameric cubane-type structure formed by four Cu and four bridging

0567-7408/80/040927-04\$01.00

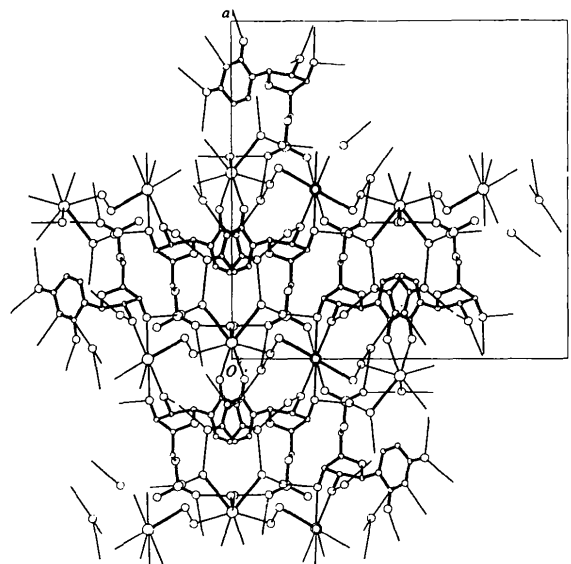


Fig. 2. Molecular packing viewed along b .

TPS and MAV thank the DAE and DST for financial support. TPS thanks the Indian Institute of Science, Bangalore, for the award of a Senior Research Fellowship.

References

- RAO, S. T. & SUNDARALINGAM, M. (1969). *J. Am. Chem. Soc.* **91**, 1210-1217.
 SESHADRI, T. P. & VISWAMITRA, M. A. (1974). *Curr. Sci.* **43**, 111-112.
 SHEFTER, E. & TRUEBLOOD, K. N. (1965). *Acta Cryst.* **18**, 1067-1077.
 STEWART, R. F. & JENSEN, L. H. (1967). *Acta Cryst.* **23**, 1102-1105.
 VISWAMITRA, M. A., SESHADRI, T. P. & POST, M. L. (1975). *Nature (London)*, **258**, 542-544.

ethanolato O atoms with Cu-Cu distances of 3.124, 3.256, 3.609 and 3.935 Å.

Introduction. Blue crystals of the title compound were prepared by slow evaporation of an ethanol solution containing copper(II) dichloroacetate and 2-dimethylaminoethanol in a molar ratio of approximately 1:1.

© 1980 International Union of Crystallography