The Crystal Structure of the Dipotassium Salt of Uridine 5'-Diphosphate

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Abstract

$C_9H_{12}N_2O_{12}P_2^{2-} \cdot 2K^+ \cdot 3H_2O$ is orthorhombic, $P2_12_12_1$, with $a = 18 \cdot 977$ (5), $b = 22 \cdot 597$ (6), $c = 8 \cdot 995$ (2) Å, $Z = 8$. The structure was refined to $R = 0 \cdot 059$ for 2587 observed reflexions. The two molecules of the asymmetric unit have very similar conformations with a 2'-*endo* sugar pucker and a folded pyrophosphate chain. They form a dimer, coordinated by the K^+ ions but without direct bridging between the base and the pyrophosphate within each individual molecule. One uracil base has the keto–enol and the other the diketo form. The extended structure shows alternating hydrophobic and hydrophilic regions.

Introduction

We report here the structure of uridine 5'-diphosphate (UDP) as obtained from the crystal structure analysis of its dipotassium salt. UDP and UDP–sugar coenzymes are key intermediates in several biosynthetic reactions in carbohydrate metabolism. The present investigation is part of our studies on the conformation of phosphate coenzymes. Conformational differences between these molecules are of interest in our understanding of the different mechanisms in the group-transfer reactions involving the coenzymes. The binding of K^+ ions to phosphate anions is also of interest as many enzymatic reactions involving the phosphate coenzymes require univalent ions like K^+ in addition to divalent metal ions for activation.

Experimental

The crystals of the dipotassium salt of UDP (from Boehringer–Mannheim Biochemicals) were prepared by slow diffusion of ethanol into aqueous solutions of

the compound at neutral pH. They were flat plates with well-developed faces. To preserve the crystals, they were sealed in Lindemann capillary tubes with a small quantity of the mother liquor.

The space group and approximate cell dimensions were determined from oscillation and Weissenberg photographs. The cell constants were refined by least-squares calculations from 15 reflexions measured on a Syntex $P2_1$ diffractometer with monochromatized Cu radiation.

Crystal data

Dipotassium uridine 5'-diphosphate trihydrate, $C_9H_{12}N_2O_{12}P_2^{2-} \cdot 2K^+ \cdot 3H_2O$. $M_r = 534 \cdot 38$. Orthorhombic, $a = 18 \cdot 977$ (5), $b = 22 \cdot 597$ (6), $c = 8 \cdot 995$ (2) Å, $U = 3857 \cdot 3$ Å^3, $D_x = 1 \cdot 84$ Mg m^{-3}, $Z = 8$. Space group $P2_12_12_1$ from absences; μ(Cu $K\alpha$) $= 6 \cdot 63$ mm^{-1}. $F(000) = 2192$.

A crystal, $0 \cdot 1 \times 0 \cdot 1 \times 0 \cdot 3$ mm, was used to collect data through the range $0 \leq 2\theta \leq 130°$ for the hkl and $0 \leq 2\theta \leq 120°$ for the $\bar{h}kl$ octants. Measurements were made in the θ–2θ scan mode with variable speed ($1 \cdot 8$–$29 \cdot 3°$ min^{-1}) dependent on a pre-scan estimate of the peak intensity. Background counts totalling $0 \cdot 6$ of the scan time were recorded at each limit. During the data collection three reflexions were monitored for crystal and instrument stability. 7845 lattice points were measured which yielded 2587 independent reflexions $[F_o > 3\sigma(F_o)]$. These were corrected for Lorentz and polarization factors and for absorption.

Structure solution and refinement

Although K_2UDP crystallized in a primitive cell the structure was pseudo C-centred as indicated by the fact that reflexions with $h + k$ odd were weak and the Patterson function had a strong peak at $\frac{1}{2},\frac{1}{2},0$. As a result, many attempts at structure solution employing multisolution tangent refinement techniques had to be

made before a fortuitous origin choice yielded an E map from which the positions of the pyrimidine rings in the two molecules and parts of sugar fragments could be located. Two successive structure factor and difference synthesis calculations were then required to establish the positions of all 60 non-hydrogen atoms in the asymmetric unit. Refinement by full-matrix least-squares calculations with isotropic thermal parameters reduced R to 18%. Further anisotropic refinement reduced R to 14% when it became evident that the pseudosymmetry (pseudo space group $C222_1$) was affecting the convergence. A weighting scheme was chosen to smooth the variance *versus* F_o curve; this effectively up-weighted the weak reflections. H atoms were introduced in calculated positions and refined in a constrained mode, with an overall isotropic thermal parameter ($U = 0.045$ Å^2), to $R = 7.1$%. One of the four hydroxyl H atoms (H3'O) was located from a difference map. Refinement finally converged to $R = 5.9$%. In the final cycles the weighting scheme was: $w = 11 - 0.2F_o$ for $F_o < 35.0$, $w = 5.48 - 0.042F_o$ for $35.0 < F_o < 120.0$, $w = 0.6 - 0.0017F_o$ for $F_o < 120.0$. The function minimized was $\sum w(|F_o| - |F_c|)^2$. Scattering factors were taken from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965).

The positional parameters of the heavy atoms are given in Table 1, those of H atoms in Table 2, bond lengths and angles in Tables 3 and 4, and torsion angles

Table 2. *Final positional and thermal parameters for the hydrogen atoms* ($\times 10^3$)

	x	y	z	U (Å^2)
H(3A)	388	322	125	45
H(5A)	272	168	−55	45
H(6A)	186	226	54	45
H(1'A)	195	382	122	45
H(2'A)	105	310	−73	45
H(3'A)	9	331	66	45
H(4'A)	66	336	338	45
H(5'1A)	1	253	286	45
H(5'2A)	79	230	345	45
H(3B)	366	−310	136	45
H(5B)	251	−161	22	45
H(6B)	167	−222	−83	45
H(1'B)	182	−380	−111	45
H(2'B)	84	−307	65	45
H(3'OB)	10	−453	−174	45
H(4'B)	44	−338	−339	45
H(5'1B)	−21	−26	−287	45
H(5'2B)	55	−236	−355	45
H(3'B)	−11	−335	−65	45

Table 1. *Final positional parameters for the non-hydrogen atoms* ($\times 10^4$)

E.s.d.'s are in parentheses.

	x	y	z		x	y	z
N(1A)	2366 (3)	3060 (3)	284 (9)	O(4B)	3695 (3)	−2023 (3)	1433 (10)
C(2A)	2940 (4)	3341 (4)	−252 (11)	C(5B)	2586 (5)	−2061 (4)	225 (13)
O(2A)	3005 (3)	3896 (3)	−193 (8)	C(6B)	2105 (5)	−2410 (4)	−343 (12)
N(3A)	3438 (4)	3006 (3)	−873 (9)	C(1'B)	1604 (4)	−3385 (4)	−979 (11)
C(4A)	3418 (5)	2393 (4)	−1065 (13)	O(1'B)	1379 (3)	−3124 (3)	−2320 (7)
O(4A)	3896 (4)	2143 (3)	−1685 (10)	C(2'B)	939 (4)	−3426 (4)	−3 (11)
C(5A)	2789 (5)	2129 (4)	−482 (13)	C(3'B)	353 (5)	−3494 (4)	−1086 (11)
C(6A)	2300 (5)	2459 (4)	146 (14)	O(3'B)	280 (3)	−4109 (3)	−1530 (11)
C(1'A)	1776 (4)	3399 (4)	1001 (10)	C(4'B)	609 (4)	−3160 (4)	−2471 (13)
O(1'A)	1577 (3)	3101 (3)	2270 (7)	C(5'B)	315 (5)	−2559 (4)	−2671 (11)
C(2'A)	1127 (5)	3444 (4)	−8 (11)	O(5'B)	431 (3)	−2209 (3)	−1350 (8)
O(2'A)	1195 (4)	3927 (3)	−978 (8)	P(1B)	200 (1)	−1533 (1)	−1324 (3)
C(3'A)	542 (4)	3474 (4)	1105 (12)	O(11B)	459 (4)	−1264 (3)	50 (9)
O(3'A)	421 (4)	4077 (3)	1583 (9)	O(12B)	−551 (3)	−1490 (3)	−1654 (10)
C(4'A)	816 (4)	3137 (4)	2453 (11)	O(6'B)	580 (5)	−1277 (3)	−2772 (9)
C(5'A)	531 (5)	2512 (4)	2617 (11)	P(2B)	1130 (2)	−768 (1)	−3042 (4)
O(5'A)	633 (3)	2194 (3)	1237 (7)	O(21B)	1271 (13)	−709 (7)	−4576 (16)
P(1A)	463 (1)	1501 (1)	1195 (3)	O(22B)	935 (6)	−255 (3)	−2246 (11)
O(11A)	669 (4)	1269 (3)	−273 (8)	O(23B)	1782 (6)	−1039 (7)	−2319 (30)
O(12A)	−267 (3)	1399 (3)	1703 (8)	K(1)	2481 (1)	4654 (1)	−2372 (3)
O(6'A)	1002 (4)	1269 (3)	2460 (9)	K(2)	1158 (1)	−997 (1)	2431 (3)
P(2A)	1335 (2)	642 (1)	2681 (5)	K(3)	1260 (1)	892 (1)	−2677 (3)
O(21A)	1656 (10)	660 (5)	4141 (18)	K(4)	−33 (1)	11 (1)	−87 (3)
O(22A)	867 (4)	187 (3)	2189 (10)	W(1)	−1288 (4)	3 (3)	−1634 (9)
O(23A)	2025 (5)	635 (6)	1647 (23)	W(11)	3315 (4)	4978 (3)	−4851 (9)
N(1B)	2161 (4)	−3017 (3)	−319 (10)	W(12)	3657 (4)	5359 (3)	−1385 (9)
C(2B)	2740 (5)	−3289 (4)	270 (12)	W(0)	98 (7)	11 (5)	−5185 (13)
O(2B)	2806 (3)	−3825 (3)	333 (9)	W(2)	2198 (9)	−529 (6)	588 (31)
N(3B)	3230 (4)	−2913 (3)	861 (10)	W(3)	2415 (7)	615 (6)	−1193 (28)
C(4B)	3212 (5)	−2297 (4)	859 (13)				

Table 3. *Bond lengths* (Å)

	Molecule A	Molecule B		Molecule A	Molecule B		Molecule A	Molecule B
N(1)—C(2)	1·350 (11)	1·367 (12)	C(1')—C(2')	1·534 (13)	1·539 (12)	O(5')—P(1)	1·600 (6)	1·590 (6)
C(2)—N(3)	1·333 (12)	1·367 (12)	C(2')—C(3')	1·498 (14)	1·487 (13)	P(1)—O(6')	1·617 (8)	1·597 (9)
N(3)—C(4)	1·397 (12)	1·393 (12)	C(3')—C(4')	1·524 (13)	1·536 (14)	P(1)—O(11)	1·474 (8)	1·462 (8)
C(4)—C(5)	1·434 (14)	1·422 (14)	C(4')—O(1')	1·455 (10)	1·470 (10)	P(1)—O(12)	1·477 (7)	1·459 (7)
C(5)—C(6)	1·318 (15)	1·311 (14)	C(1')—O(1')	1·378 (11)	1·408 (12)	P(2)—O(6')	1·566 (7)	1·572 (9)
C(6)—N(1)	1·370 (12)	1·376 (12)	C(2')—O(2')	1·403 (11)	1·427 (12)	P(2)—O(21)	1·448 (18)	1·412 (15)
C(2)—O(2)	1·262 (11)	1·218 (11)	C(3')—O(3')	1·447 (11)	1·451 (11)	P(2)—O(22)	1·429 (8)	1·411 (9)
C(4)—O(4)	1·206 (12)	1·220 (12)	C(4')—C(5')	1·518 (12)	1·479 (13)	P(2)—O(23)	1·606 (15)	1·528 (16)
N(1)—C(1')	1·501 (12)	1·470 (12)	C(5')—O(5')	1·448 (12)	1·444 (12)			

Table 4. *Bond angles* (°)

	Molecule A	Molecule B		Molecule A	Molecule B		Molecule A	Molecule B
C(1')—N(1)—C(2)	121·1 (8)	118·7 (7)	N(1)—C(1')—C(2')	112·2 (8)	113·1 (8)	O(11)—P(1)—O(5')	108·4 (4)	108·6 (4)
C(1')—N(1)—C(6)	118·4 (7)	120·1 (8)	C(4')—O(1')—C(1')	109·9 (6)	110·9 (7)	O(12)—P(1)—O(11)	118·1 (4)	118·3 (4)
C(6)—N(1)—C(2)	120·5 (8)	121·2 (8)	O(2')—C(2')—C(1')	110·3 (7)	109·1 (7)	O(12)—P(1)—O(5')	109·5 (4)	109·2 (4)
O(2)—C(2)—N(1)	122·1 (8)	123·2 (8)	C(3')—C(2')—C(1')	101·7 (8)	104·2 (8)	O(6')—P(1)—O(5')	99·9 (3)	102·2 (4)
N(3)—C(2)—N(1)	117·0 (8)	114·7 (8)	C(3')—C(2')—O(2')	116·6 (8)	115·0 (7)	O(6')—P(1)—O(11)	110·3 (4)	112·8 (5)
N(3)—C(2)—O(2)	120·9 (8)	122·0 (9)	O(3')—C(3')—C(2')	111·1 (7)	110·6 (7)	O(6')—P(1)—O(12)	109·0 (4)	104·5 (5)
C(4)—N(3)—C(2)	126·6 (8)	127·2 (8)	C(4')—C(3')—C(2')	104·8 (7)	104·1 (7)	P(2)—O(6')—P(1)	129·6 (5)	133·7 (5)
O(4)—C(4)—N(3)	120·1 (9)	119·2 (9)	C(4')—C(3')—O(3')	106·8 (8)	106·1 (8)	O(21)—P(2)—O(6')	105·0 (6)	110·3 (9)
C(5)—C(4)—N(3)	113·0 (8)	113·3 (9)	C(3')—C(4')—O(1')	106·1 (7)	105·5 (8)	O(22)—P(2)—O(6')	111·2 (5)	110·4 (6)
C(5)—C(4)—O(4)	126·9 (9)	127·3 (9)	C(5')—C(4')—O(1')	108·2 (7)	109·7 (7)	O(22)—P(2)—O(21)	124·2 (7)	117·9 (8)
C(6)—C(5)—C(4)	120·4 (9)	120·9 (9)	C(5')—C(4')—C(3')	114·8 (7)	115·5 (8)	O(23)—P(2)—O(6')	105·3 (6)	100·3 (7)
C(5)—C(6)—N(1)	122·5 (9)	122·6 (9)	O(5')—C(5')—C(4')	109·3 (8)	110·2 (8)	O(23)—P(2)—O(21)	100·5 (10)	107·4 (13)
N(1)—C(1')—O(1')	108·2 (7)	109·1 (7)	P(1)—O(5')—C(5')	118·6 (5)	119·7 (6)	O(23)—P(2)—O(22)	108·7 (7)	109·0 (9)
C(2')—C(1')—O(1')	107·6 (7)	105·5 (7)						

Table 5. *Torsion angles* (°) *involving non-hydrogen atoms*

E.s.d.'s are in the range 0·05–0·15°.

	Molecule A	Molecule B
C(2)—N(1)—C(1')—O(1)	−136·66	−136·91
C(2)—N(1)—C(1')—C(2')	105·71	105·74
C(6)—N(1)—C(1')—O(1')	45·33	41·29
C(6)—N(1)—C(1')—C(2)	−72·29	−76·05
N(1)—C(1')—C(2')—O(2')	86·63	−87·53
O(1')—C(1')—C(2')—C(3')	31·56	29·76
C(1')—C(2')—C(3')—C(4')	−28·96	−30·14
C(2')—C(3')—C(4')—O(1')	18·01	20·52
C(3')—C(4')—O(1')—C(1')	2·20	−1·85
O(1')—C(4')—C(5')—O(5')	−66·48	−65·17
C(3')—C(4')—C(5')—O(5')	52·29	53·98
O(3')—C(3')—C(4')—C(5')	140·52	142·52
C(4')—C(5')—O(5')—P(1)	172·35	176·51
C(5)—O(5')—P(1)—O(6')	−59·17	−52·79
C(5')—O(5')—P(1)—O(13)	55·31	57·32
C(5')—O(5')—P(1)—O(11)	−174·76	−172·40
O(5')—P(1)—O(6')—P(2)	−155·95	−121·22
O(11)—P(1)—O(6')—P(2)	−42·10	−4·46
O(13)—P(1)—O(6')—P(2)	89·18	125·13
P(1)—O(6')—P(2)—O(21)	−170·55	180·00
P(1)—O(6')—P(2)—O(22)	−31·64	−46·22
P(1)—O(6')—P(2)—O(23)	85·02	68·68
O(1')—C(1')—C(2')—O(2')	155·55	153·10
C(1')—C(2')—C(3')—O(3')	85·99	83·23
C(4')—C(3')—C(2')—O(2')	−148·61	−149·63
O(1')—C(4')—C(3')—O(3')	−99·54	−96·06

in Table 5. Fig. 1 shows the chemical structure and numbering scheme.*

Results and discussion

Molecular conformation and geometry

The conformations of the two UDP molecules of the asymmetric unit are similar. They form a dimer coordinated by K⁺ ions, similar to that in Na₂ATP (Kennard *et al.*, 1971). Fig. 2 shows two views of the dimer down **a** and **c**.

Uracil base

The uracil base is *anti* with respect to the sugar moiety in both molecules, with the glycosidic torsion angle χ_{CN} 45·3 and 41·3° in molecules A and B respectively. These values fall within the range $36 \leq \chi_{CN} \leq 65°$, which is the favoured region for β-pyrimidine nucleotides (Sundaralingam, 1973). The uracil base is

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34186 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
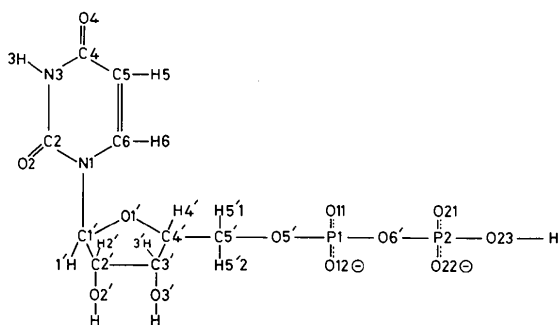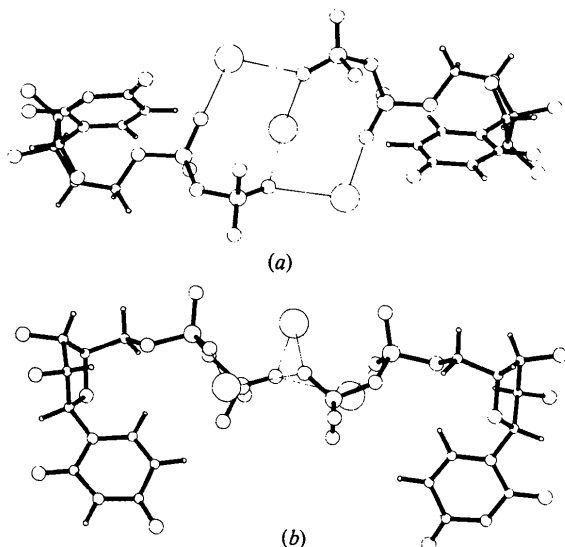
Fig. 1. UDP chemical structure and numbering scheme.

(a)

(b)

Fig. 2. View of the UDP dimer along (a) the a and (b) the c axes.

essentially planar with the exocyclic O(2) showing the maximum deviation from the best plane (−0·052 and 0·22 Å in A and B respectively). The geometry around C(2) is significantly different in the two molecules with C(2)−O(2) = 1·262, C(2)−N(3) = 1·333 Å and N(3)−C(2)−N(1) = 117·0° in A, and 1·218, 1·367 Å and 114·7° in B. The other ring dimensions are closely similar. The resonant structures of the two uracil rings appear therefore to be different, with A predominantly in the keto–enol form. This tautomer was also observed in BaUMP-5' (Shefter & Trueblood, 1965) but there C(4)−O(4) was significantly longer (1·29 Å) than in the present structure (1·220 Å). B has the diketo structure as in NaUMP-3' (Viswamitra, Reddy, James & Williams, 1972). The best least-squares planes through the bases and the atom deviations are given in Table 6.

Ribose

In both molecules the ribose shows C(2')-*endo* puckering in contrast to Na₂ATP (Kennard *et al.*,

Table 6. *Least-squares planes and deviations of atoms* (Å)

E.s.d.'s in the atom deviations are *ca* 0·005 Å.

Uracil

Equations of planes
Molecule A: $0.4468x - 0.1155y + 0.8872z = 1.4273$
Molecule B: $-0.4588x - 0.0320y + 0.8880z = -1.9263$

	Molecule A	Molecule B		Molecule A	Molecule B
N(1)	0·007*	0·006*	O(4)	−0·025	0·004
C(2)	−0·010*	−0·008*	C(5)	−0·003*	0·010
O(2)	−0·052	0·022	C(6)	−0·001*	−0·007*
N(3)	0·006*	0·012*	C(1')	−0·008	−0·005
C(4)	0·001*	−0·012*			

Ribose

Equations of planes
Molecule A: $0.0863x + 0.8522y + 0.5161z = 7.2978$
Molecule B: $-0.1057x + 0.8735y + 0.4751z = -7.4253$

	Molecule A	Molecule B		Molecule A	Molecule B
C(1')	0·008*	0·006*	C(3')	−0·007*	−0·006*
O(1')	−0·012*	−0·010*	C(4')	0·011*	0·010*
C(2')	−0·491	0.472	O(2')	0·002	−0·052

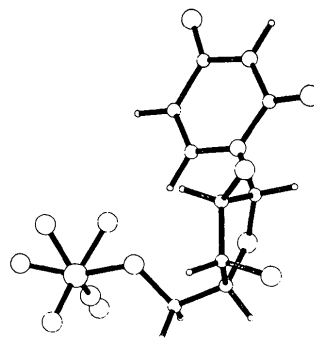* Atoms used to calculate mean plane.

Fig. 3. A view of the molecule showing the staggered geometry of the pyrophosphate group.

1971) and ADP free acid (Viswamitra & Hosur, 1977) where one of the independent molecules is in the C(2')-*endo* and the other in the C(3')-*endo* conformation. The best least-squares planes for the ribose rings are given in Table 6. The conformation about the exocyclic C(4')−C(5') bond is *gauche–gauche* ($\varphi_{OO} = -66.5$ and $-65.2°$, $\varphi_{OC} = 52.3$ and 54·0°). This conformation is the one most commonly observed for nucleotides in the solid state. The uncommon *gauche–trans* and *trans–gauche* conformers were, however, observed in two uracil nucleotides: azauridine 5'-phosphate (Saenger & Suck, 1973) and deoxyuridine 5'-phosphate (Viswamitra, Seshadri & Post, 1975). dGMP-5' also has the uncommon *gauche–trans* conformation in its crystal structure (Young, Tollin & Wilson, 1974; Viswamitra & Seshadri, 1974).

Folding of the pyrophosphate chain

The pyrophosphate groups have a characteristic staggered conformation (Fig. 3) as in RbADP (Viswamitra, Hosur, Shakked & Kennard, 1976). In RbADP there is a difference of $0 \cdot 1$ Å in the length of the bridging bonds in the pyrophosphate linkage [P(1)–O(6') = 1·54 and P(2)–O(6') = 1·64 Å]. This difference is less pronounced, though still significant, in K_2UDP [P(1)–O(6') = 1·617 and 1·597 Å, P(2)–O(6') = 1·566 and 1·572 Å]. Further work on related compounds is required to interpret these differences. Both the UDP molecules are in the folded conformation but without any direct metal-ion or water bridge between the pyrophosphate chain and the uracil base of the same molecule. A similar conformation was found in other nucleoside 5'-diphosphate coenzymes including ADP free acid and its salts and CDP choline (Viswamitra, Seshadri, Post & Kennard, 1975). It is possible that the folded shape represents a characteristic low-energy state of these molecules with any metal chelation further stabilizing the folded conformation. This possibility has been suggested earlier for
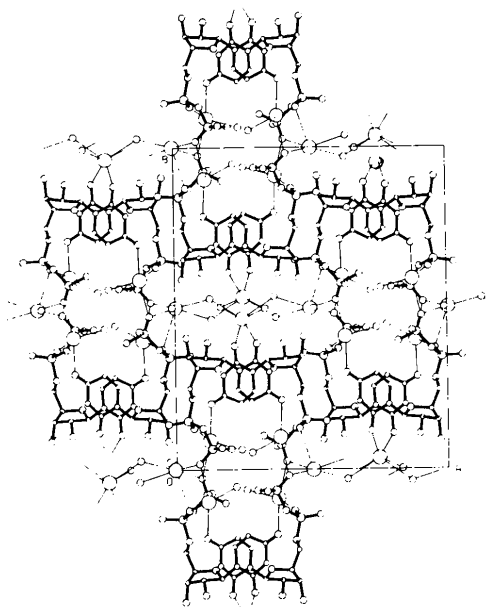

Fig. 4. Projection of the extended crystal structure on the ab plane.

Table 7. *Potassium and water coordination, and hydrogen bonding*

E.s.d.'s are in the range 0·010–0·015 Å.

Potassium coordination (Å)		Symmetry code		Water coordination (Å)		Symmetry code		Hydrogen bonding (Å) Molecule A		Symmetry code	
K(1)···O(2A)	2·786	1	0,0,0	W(1)···K(4)	2·834	1	0,0,0				
K(1)···O(2B)	2·841	2	0,0,0	W(1)···O(2A)	2·793	3	0,0,0				
K(1)···W(11)	2·848	2	0,1,−1	W(1)···O(3'B)	2·861	4	0,−1,−1	N(3)–H···O(13A)	2·899	3	−1,0,0
K(1)···W(11)	2·831	1	0,0,0	W(1)···K(1)	2·835	4	0,0,0	O(2)···W(1)	2·793	3	−1,0,0
K(1)···W(12)	2·882	1	0,0,0					O(2')–H···O(13B)	2·629	4	0,−1,−1
K(1)···W(1)	2·835	4	0,−1,−1	W(11)···K(1)	2·831	1	0,0,0	O(2')···W(11)	2·833	2	0,1,−1
				W(11)···O(2'A)	2·833	2	0,1,0	O(3')–H··· W(0)	2·646	4	0,−1,−1
K(2)···O(22A)	2·739	1	0,0,0	W(11)···O(2'B)	2·845	2	0,0,0	O(3')···W(12)	2·834	2	0,1,−1
K(2)···O(11B)	2·590	1	0,0,0					O(13)···O(2'B)	2·589	4	0,−1,0
K(2)···O(21B)	2·778	1	0,0,−1	W(12)···O(3'A)	2·834	2	0,1,0	O(21)···W(2)	2·553	2	0,0,−1
K(2)···W(2)	2·786	1	0,0,0	W(12)···O(2B)	2·898	1	0,1,0	O(22)···W(0)	2·805	1	0,0,−1
K(2)···O(4A)	2·711	2	0,0,−1	W(12)···K(1)	2·882	1	0,0,0	O(23)–H···O(23B)	2·612	2	0,0,−1
K(2)···W(3)	3·101	2	0,0,−1	W(12)···K(4)	2·938	3	−1,0,0	O(23)···W(3)	2·660	1	0,0,0
								O(23)···W(2)	2·816	1	0,0,0
K(3)···O(11A)	2·580	1	0,0,0	W(0)···O(22A)	2·805	1	0,0,1				
K(3)···O(22B)	2·692	1	0,0,0	W(0)···O(21B)	2·811	1	0,0,0	Molecule B			
K(3)···O(4B)	2·680	2	0,0,0	W(0)···O(3'A)	2·646	4	0,0,−1				
K(3)···O(21B)	3·005	1	0,0,1	W(0)···O(3'B)	2·616	4	0,−1,−1	N(3)–H···O(13B)	2·771	3	−1,−1,0
K(3)···O(3'B)	3·008	4	0,−1,−1					O(2)···W(12)	2·898	1	0,0,0
K(3)···W(3)	2·641	1	0,0,0	W(2)···O(23A)	2·816	1	0,0,0	O(2')–H···O(13A)	2·589	4	0,0,0
				W(2)···O(23B)	2·965	1	0,0,0	O(2')···W(11)	2·845	2	0,0,−1
K(4)···O(22A)	2·695	1	0,0,0	W(2)···O(21A)	2·553	2	0,0,0	O(3')–H··· W(0)	2·618	4	0,0,−1
K(4)···O(22B)	2·741	1	0,0,0	W(2)···K(2)	2·786	1	0,0,0	O(3')···W(1)	2·861	4	0,0,−1
K(4)···O(11A)	3·145	1	0,0,0					O(13)···O(2'A)	2·629	4	0,0,−1
K(4)···O(11B)	3·030	1	0,0,0	W(3)···O(23A)	2·660	1	0,0,0	O(21)···W(0)	2·811	1	0,0,0
K(4)···W(1)	2·834	1	0,0,0	W(3)···O(21B)	2·895	2	0,0,−1	O(21)···W(3)	2·894	2	0,0,0
K(4)···W(12)	2·938	3	0,0,0	W(3)···K(3)	2·641	1	0,0,0	O(23)···O(23A)	2·612	2	0,0,0
				W(3)···K(2)	3·101	2	0,0,−1				

Symmetry code
(1) x,y,z (3) $\frac{1}{2} + x, \frac{1}{2} - y, -z$
(2) $-x, \frac{1}{2} + y, \frac{1}{2} - z$ (4) $\frac{1}{2} - x, -y, \frac{1}{2} + z$.

The numbers following the symmetry code indicate cell translations of the atom.

ATP on the basis of potential-energy calculations (Perahia, Pullman & Saran, 1972).

Potassium and water coordinates

Each of the four non-equivalent K$^+$ ions is six-coordinated but with significant differences (Table 7). K(2), K(3) and K(4) are coordinated by O atoms of the α and β phosphate groups forming the UDP dimer (Fig. 2). K(1) is coordinated by two O(2) atoms of the uracil, and O atoms of four water molecules. The water molecules are either three- or four-coordinated (Table 7) and provide a hydrogen-bonded network. As a result the structure has large hydrophilic cavities containing water and K$^+$ ions, alternating with hydrophobic regions (Fig. 4).

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The Crystal Structure of Bis(tetraethylammonium) Tetrachlorodioxouranate(VI): Correction from P$\bar{1}$ to P2₁/n

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Abstract

Bois, Nguyen & Rodier [*Acta Cryst.* (1976). B32, 1541–1544] refined the structure of [(C₂H₅)₄N$^+$]₂- (UCl₄O₂)$^{2-}$ in space group P$\bar{1}$, Z = 2, to R = 0·048, but it very nearly has symmetry P2₁/n. We find that refinement *in* P2₁/n hardly changes R and renders both ions substantially more symmetrical. Including (but not refining) the hydrogen positions and isotropic Gaussian amplitudes, letting all the heavy-atom Gaussians become anisotropic, and accounting for secondary extinction reduces R to 0·040 and further regularizes the ions. Finally, allowing for the presence of two minor twin components (4·4% and 7·1%) brings R to 0·035, still further regularizes the bond lengths and angles, and