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Structure of 3,*N*⁴-Ethenocytidinium Dihydrogenphosphate

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Abstract. 5-Oxo-6-β-D-ribofuranosyl-1*H*⁺-imidazol[1,2-*c*]pyrimidinium dihydrogenphosphate, C₁₁H₁₄N₃O₅⁺·H₂PO₄⁻, *M_r* = 365.2, orthorhombic, *P*2₁2₁2₁, *a* = 6.767 (2), *b* = 13.770 (3), *c* = 16.093 (3) Å, *V* = 1499.7 (6) Å³, *Z* = 4, *D_x* = 1.62 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 2.3 cm⁻¹, *F*(000) = 760, *T* = 291 K, *R* = 0.052 for 991 observed reflections. The ribose conformation is characterized by *P* = 164.5 (6)° (²*E*) and τ_{*m*} = 38.2 (4)°. The glycoside torsion angle [O(4')—C(1')—N(1)—C(2)—128.0 (7)°] is in the *anti* region and the side chain has the preferred *gauche*⁺ conformation [γ = 55 (1)°]. The H₂PO₄⁻ groups are arranged in a helical pattern in which a single O atom of each anion accepts two strong hydrogen bonds from two different OH donors of the neighboring H₂PO₄⁻ units. The fourth O atom of the H₂PO₄⁻ anion is an acceptor of hydrogen bonds from both the protonation site [N(4)H⁺] and the ribose of the cation. The cations are also linked directly, *via* an O(3')H⋯O(5') hydrogen bond.

Introduction. This work continues a series of structural studies of phosphate salts of nucleosides in which the two main components of the nucleotide coexist as separate ions in the crystal. The first report in this series (cytidinium dihydrogenphosphate, CydH⁺·H₂PO₄⁻; Jaskólski, 1989) revealed a pattern of infinite ⋯phosphate⋯sugar⋯ chains in which the

dihydrogenphosphate anions are hydrogen bonded, *via* their POH donors, to O(3') and O(5') of neighboring nucleosides. One interesting aspect of the hydrogen bonds along these chains is that they mimic the O(3')—P—O(5') ester bonds in the backbone of a real polynucleotide. Secondly, as the chains do not involve the ribose O(2')H function, an identical pattern of nucleoside⋯phosphate association could be maintained in the crystals of 2'-deoxycytidinium dihydrogenphosphate (Jaskólski, 1991). In the present paper, which describes the structure of 3,*N*⁴-ethenocytidinium dihydrogenphosphate, our aim is to verify the conclusion drawn from the previous structures that the driving force determining the packing mode in nucleoside phosphates is the phosphate anion. Additionally, the ethenocytidine system (*ε*Cyd) is an interesting subject for study in itself. This derivative of cytidine was first introduced by Barrio, Sotssangi, Gruber, Damman & Leonard (1976) as a useful fluorescent anchor in the studies of nucleic acids. To date, the structure of *ε*Cyd has been described in the neutral form (Jaskólski, Krzyżosiak, Sierzputowska-Gracz & Wiewiórowski, 1981) and in the *N*⁴-protonated form as *ε*CydH⁺·Cl⁻ (Wang, Barrio & Paul, 1976). The effects of etheno bridging and protonation in the Cyd/*ε*Cyd system have been analyzed in detail by Jaskólski *et al.* (1981), Krzyżosiak, Jaskólski, Sierzputowska-Gracz & Wiewiórowski (1982) and Kozerski, Sierzputowska-Gracz, Krzyżosiak, Bratek-Wiewiórowska, Jaskólski & Wiewiórowski (1984).

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Experimental. Suitable crystals from water-methanol solution, crystal $0.1 \times 0.3 \times 0.4$ mm, Syntex $P2_1$ diffractometer, graphite monochromator, Mo $K\alpha$ radiation. Cell parameters from least-squares treatment of setting angles of 15 reflections ($10 < 2\theta < 18^\circ$). $\theta:2\theta$ profiles measured for 1368 unique $+h+k+l$ reflections $2\theta < 49^\circ$ [max. $(\sin\theta/\lambda) = 0.584 \text{ \AA}^{-1}$] and with $0 \leq h \leq 7$, $0 \leq k \leq 16$, $0 \leq l \leq 18$; profile analysis according to Lehmann & Larsen (1974). Random intensity variation (within 3.1%) of two standard reflections measured every 1.5 h. No absorption or extinction correction. 991 observed reflections with $F \geq 2\sigma(F)$. Structure solved using the direct-methods procedure of *SHELXS86* (Sheldrick, 1986). Full-matrix least-squares refinement on $F_o \cdot w^{-1} = \sigma^2(F_o) + 0.0001F_o^2$. H atoms at N(4), C(7), C(8) and O atoms located in successive ΔF maps. H(7) and H(8) riding on C(7) and C(8), respectively; positional parameters of O(N)H hydrogen atoms included in the refinement; ribose O—H distances restrained at 0.98 (5) Å; remaining H atoms generated geometrically and fixed. Each H atom given (fixed) B_{iso} one unit greater than that of its carrier. Final $R = 0.052$, $wR = 0.052$, $S = 2.39$, $(\Delta/\sigma)_{\text{max}} = 0.63$; electron density in final ΔF map between -0.35 and 0.31 e \AA^{-3} . Computer programs: *SHELXS86* (Sheldrick, 1986), *SHELX76* (Sheldrick, 1976) and local programs (Jaskólski, 1982). Molecular illustrations drawn using local version of *ORTEP* (Johnson, 1976). Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. Atomic coordinates are listed in Table 1.* Bond distances and angles are given in Table 2 and a thermal-ellipsoid representation of the cation is shown in Fig. 1.

The dimensions of the ethenocytosinium cation are consistent with those reported for $\epsilon\text{CydH}^+\cdot\text{Cl}^-$ (Wang *et al.*, 1976). The protonation site is N(4) but the pattern of geometrical changes accompanying the protonation ($\epsilon\text{Cyd} \rightarrow \epsilon\text{CydH}^+$) reveals that the positive charge is distributed within the base fragment and is delocalized over N(4), N(3) and (to a lesser degree) N(1) (Jaskólski *et al.*, 1981). Both the pyrimidine and the imidazole rings of the ϵCydH^+ system are planar ($\chi^2 = 9.15$ and 4.25 , respectively), but they form a 'V' shape with a dihedral angle of $2.5(2)^\circ$, slightly more pronounced than in $\epsilon\text{CydH}^+\cdot\text{Cl}^-$ [$1.4(1)^\circ$; Wang *et al.*, 1976]. The geometry of the ribose fragment agrees, within experimental error, with the data found in other β -ribofuranosides. The

Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
P(1)	-0.1709 (3)	0.8332 (2)	0.0243 (1)	0.0316 (6)
O(1P)	-0.1635 (9)	0.9249 (3)	-0.0264 (3)	0.038 (2)
O(2P)	-0.3520 (8)	0.8339 (5)	0.0857 (4)	0.039 (2)
O(3P)	-0.1743 (9)	0.7417 (4)	-0.0286 (3)	0.039 (2)
O(4P)	0.0045 (8)	0.8298 (5)	0.0878 (4)	0.040 (2)
N(1)	0.054 (1)	0.7495 (5)	0.3709 (4)	0.034 (2)
C(2)	-0.040 (1)	0.8016 (6)	0.4313 (5)	0.032 (3)
O(2)	-0.2077 (8)	0.7913 (4)	0.4556 (3)	0.030 (2)
N(3)	0.081 (1)	0.8765 (4)	0.4633 (4)	0.039 (2)
C(4)	0.273 (1)	0.8935 (6)	0.4395 (5)	0.035 (3)
N(4)	0.337 (1)	0.9658 (5)	0.4865 (5)	0.040 (2)
C(7)	0.190 (1)	0.9945 (6)	0.5418 (5)	0.042 (3)
C(8)	0.031 (1)	0.9387 (5)	0.5283 (5)	0.036 (3)
C(5)	0.357 (1)	0.8370 (6)	0.3758 (5)	0.039 (3)
C(6)	0.245 (1)	0.7658 (6)	0.3437 (5)	0.035 (3)
C(1')	-0.061 (1)	0.6680 (5)	0.3340 (5)	0.032 (3)
C(2')	-0.110 (1)	0.6818 (5)	0.2436 (5)	0.030 (3)
O(2')	-0.2894 (9)	0.7376 (4)	0.2399 (4)	0.045 (2)
C(3')	-0.130 (1)	0.5770 (6)	0.2138 (6)	0.041 (3)
O(3')	-0.3170 (9)	0.5376 (4)	0.2372 (5)	0.056 (2)
C(4')	0.027 (1)	0.5236 (6)	0.2636 (6)	0.039 (3)
O(4')	0.0609 (9)	0.5824 (4)	0.3389 (4)	0.037 (2)
C(5')	0.221 (1)	0.5073 (7)	0.2221 (5)	0.042 (3)
O(5')	0.304 (1)	0.5977 (4)	0.1927 (4)	0.046 (2)

Table 2. Bond distances (Å) and angles ($^\circ$)

N(1)—C(2)	1.363 (10)	C(2')—O(2')	1.438 (9)
C(2)—O(2)	1.211 (10)	C(2')—C(3')	1.527 (11)
C(2)—N(3)	1.412 (9)	C(3')—O(3')	1.426 (10)
N(3)—C(4)	1.374 (10)	C(3')—C(4')	1.519 (11)
C(4)—N(4)	1.323 (10)	C(4')—C(5')	1.495 (12)
N(4)—C(7)	1.392 (11)	C(5')—O(5')	1.445 (10)
C(7)—C(8)	1.340 (11)	C(4')—O(4')	1.475 (10)
C(8)—N(3)	1.395 (10)	O(4')—C(1')	1.442 (9)
C(4)—C(5)	1.407 (11)	P(1)—O(1P)	1.504 (5)
C(5)—C(6)	1.343 (11)	P(1)—O(2P)	1.574 (6)
C(6)—N(1)	1.384 (10)	P(1)—O(3P)	1.522 (5)
N(1)—C(1')	1.489 (9)	P(1)—O(4P)	1.567 (6)
C(1')—C(2')	1.505 (11)		

O(1P)—P(1)—O(2P)	111.2 (4)	N(4)—C(7)—C(8)	107.9 (7)
O(1P)—P(1)—O(3P)	113.1 (3)	C(7)—C(8)—N(3)	106.1 (7)
O(1P)—P(1)—O(4P)	110.7 (4)	C(4)—C(5)—C(6)	117.1 (8)
O(2P)—P(1)—O(3P)	110.2 (3)	C(5)—C(6)—N(1)	121.7 (8)
O(2P)—P(1)—O(4P)	100.4 (3)	N(1)—C(1')—C(2')	113.8 (6)
O(3P)—P(1)—O(4P)	110.6 (4)	N(1)—C(1')—O(4')	107.1 (6)
C(6)—N(1)—C(2)	124.9 (7)	C(2)—C(1')—O(4')	106.3 (6)
C(6)—N(1)—C(1')	119.0 (7)	C(1')—C(2')—O(2')	106.9 (6)
C(1')—N(1)—C(2)	116.1 (7)	C(1')—C(2')—C(3')	101.7 (6)
N(1)—C(2)—O(2)	127.1 (8)	O(2')—C(2')—C(3')	114.5 (6)
N(1)—C(2)—N(3)	112.1 (7)	C(2')—C(3')—O(3')	110.9 (7)
O(2)—C(2)—N(3)	120.7 (8)	C(2')—C(3')—C(4')	103.2 (7)
C(2)—N(3)—C(4)	124.7 (8)	O(3')—C(3')—C(4')	107.2 (7)
C(2)—N(3)—C(8)	125.5 (7)	C(3')—C(4')—C(5')	116.9 (7)
C(4)—N(3)—C(8)	109.6 (7)	C(3')—C(4')—O(4')	106.1 (6)
N(3)—C(4)—C(5)	119.4 (8)	C(5')—C(4')—O(4')	108.1 (7)
N(3)—C(4)—N(4)	106.1 (8)	C(4')—O(4')—C(1')	108.3 (6)
C(5)—C(4)—N(4)	134.5 (8)	C(4')—C(5')—O(5')	111.1 (7)
C(4)—N(4)—C(7)	110.3 (7)		

conformation of the ribose ring is $2'$ -endo (2E). In terms of pseudorotation (Altona & Sundaralingam, 1972) the ribose conformation is characterized by $P = 164.5(6)^\circ$ and $\tau_m = 38.2(4)^\circ$ (Jaskólski, 1984). The glycosidic torsion angle, O(4')—C(1')—N(1)—C(2), which describes the relative orientation of the base

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53726 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

with respect to the sugar is in the *anti* region [$-128.0(7)^\circ$]. The torsion angles $O(5')-C(5')-C(4')-C(3')$ (γ) and $O(5')-C(5')-C(4')-O(4')$ [$55(1)$ and $-64.8(8)^\circ$, respectively] indicate that the side chain has the preferred *gauche*⁺ conformation with the $C(5')-O(5')$ bond rotated only 5° from the ideal staggered orientation towards $C(4')-C(3')$. At these orientations of the base and the side chain a short $C(6)-H\cdots O(5')$ intramolecular contact is formed (Table 3). This weak interaction is not uncommon in pyrimidine nucleosides and, in particular, has also been found in ϵ CydH⁺.Cl⁻ (Wang *et al.*, 1976). Another short intramolecular contact exists between $O(3')H$ and $O(2')$. This contact is very weak as a hydrogen bond due to the unfavorable $O-H\cdots O$ angle (Table 3).

The P—OH bonds of the dihydrogenphosphate anion are significantly longer than the remaining two P—O bonds. Also, the O—P—O angles reflect the electronic state of the phosphate O atoms and the distribution of the negative charge over $O(1P)$ and $O(3P)$, since the $O(1P)-P-O(3P)$ angle is the widest and $HO(2P)-P-O(4P)H$ the smallest (Table 2). The difference between the P—O(1P) and P—O(3P) bond distances, although barely significant, is consistent with the different pattern of hydrogen-bond interactions of these O atoms: the atom accepting stronger hydrogen bonds [$O(3P)$, Table 3] has a longer P—O distance. Such correlations between the P—O bond length and the degree to which the O atom shares the protons through which it is hydrogen bonded have been observed before and apply to the P—OH bonds as well (Jaskólski, 1989). The sum

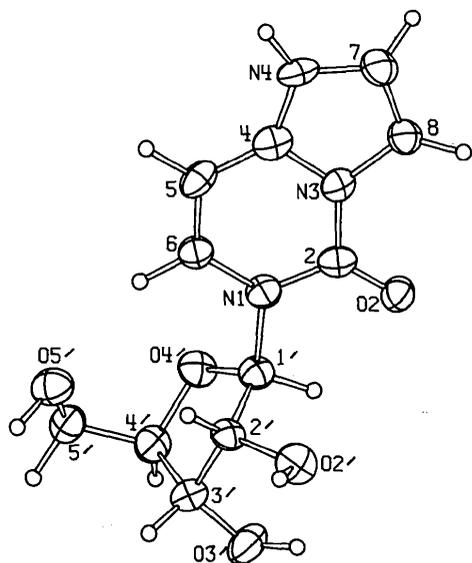


Fig. 1. Thermal-ellipsoid representation of the 3,*N*⁴-ethenocytidinium cation drawn at the 50% probability level. H-atom spheres are arbitrarily sized.

Table 3. Geometry of the hydrogen bonds (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O(2P)-H(2P)\cdots O(3P^i)$	0.83 (9)	1.76 (9)	2.585 (8)	170 (9)
$O(4P)-H(4P)\cdots O(3P^{ii})$	0.97 (8)	1.60 (8)	2.569 (8)	172 (8)
$N(4)-H(4)\cdots O(1P^{iii})$	0.96 (8)	1.74 (8)	2.683 (9)	170 (7)
$O(2')-H(2O')\cdots O(2P)$	0.92 (6)	1.95 (6)	2.846 (8)	164 (8)
$O(5')-H(5O')\cdots O(1P^{iv})$	1.03 (5)	1.68 (5)	2.702 (8)	173 (8)
$O(3')-H(3O')\cdots O(5^{iv})$	0.97 (8)	2.08 (8)	2.786 (9)	128 (6)
$O(3')-H(3O')\cdots O(2')$	0.97 (8)	2.28 (8)	2.759 (8)	110 (5)
$C(6)-H(6)\cdots O(5')$	1.08	2.36	3.381 (10)	157

Symmetry codes: (i) $x-0.5, 1.5-y, -z$; (ii) $x+0.5, 1.5-y, -z$; (iii) $0.5-x, 2-y, z+0.5$; (iv) $x-1, y, z$.

of the P—O distances, which is constant in any phosphate (Cruickshank, 1961), is $6.17(1)\text{\AA}$, in agreement with the value of 6.184\AA given by Math-eja & Degens (1971).

The $H_2PO_4^-$ anions are arranged in a helical, hydrogen-bonded pattern around the screw axis along *a*. Each anion uses its $O(2P)-H$ and $O(4P)-H$ donors to form two strong hydrogen bonds with the $O(3P)$ atoms of its nearest $H_2PO_4^-$ neighbors within the helix (Table 3, Fig. 2) and, at the same time, accepts, at the $O(3P)$ atom, the complementary hydrogen bonds from those same neighbors. Viewed in a different way, the hydrogen-bonding interactions within the infinite phosphate \cdots phosphate chains can be characterized as two helical systems, $O(2P)H\cdots O(3P)-P-O(2P)H\cdots$ and $O(4P)H\cdots O(3P)-P-O(4P)H\cdots$ running in opposite directions. A consequence of this self association of the $H_2PO_4^-$ anions is their exclusive acceptor function in the hydrogen-bonding interactions with the nucleoside cations. This is in striking contrast to the situation in cytidinium dihydrogenphosphate (Jaskólski, 1989) and 2'-deoxycytidinium dihydrogenphosphate (Jaskólski, 1991) where the dominating motif of anion \cdots cation association consists of infinite $-O(3')\cdots HOPO_2OH\cdots O(5')\cdots O(3')\cdots$ chains in which the phosphate anion mimics the oligonucleotide backbone formation by donating both its O—H functions to $O(3')$ and $O(5')$ of adjacent (2'-deoxy)ribose units. In the present structure, the hydrogen bonds donated by the ϵ CydH⁺ cation are accepted primarily by the $O(1P)$ atom of the $H_2PO_4^-$ anion. This atom is promoted for such a role as the P—O(1P) bond is normal to a [$88.1(2)^\circ$] and sticks out from the phosphate \cdots phosphate helix (Fig. 2). The main strong hydrogen bond is from the $N(4)$ protonation site in the base fragment while the ribose interacts through medium strong hydrogen bonds with a c-screw-related phosphate \cdots phosphate helix. $O(5')$ donates a hydrogen bond to the $O(1P)$ acceptor while $O(2')$ interacts with $O(2P)$ of the next unit in the phosphate \cdots phosphate chain (Fig. 2). This arrangement of ribose \cdots phosphate hydrogen bonds leads to helical $-O(2')H\cdots OHP(O_2H)O\cdots HO(5')\cdots O(2')H\cdots$ chains in which the 2'-OH group is an

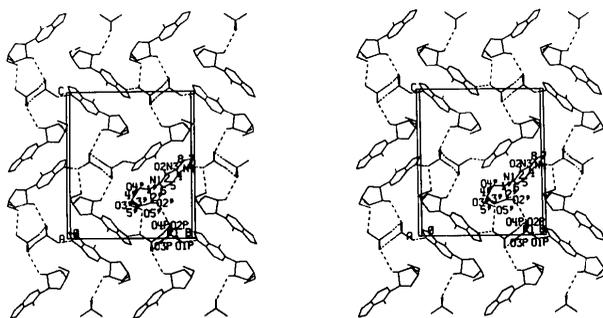


Fig. 2. Stereoscopic view of the crystal packing. H atoms have been omitted. Hydrogen bonds are indicated by broken lines.

essential link. The ribose...phosphate backbone reported for cytidinium dihydrogenphosphate does not include the 2'-hydroxyl group (Jaskólski, 1989) and therefore can be preserved in identical form in 2'-deoxycytidinium dihydrogenphosphate (Jaskólski, 1991). *a*-Translated sugar units within the present sugar...phosphate chains are linked through a direct O(3')H...O(5') hydrogen bond. This bond is the only intermolecular interaction in which the nucleoside cation plays the role of the acceptor. The hydrogen-bond network described above extends in three dimensions. The ethenocytosinium system does not form any stacking pattern in the crystal.

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Structure of 1,5-Diselenoniabicyclo[3.3.0]octane Bis(tetrafluoroborate) Acetonitrile Solvate

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Abstract. $C_6H_{12}Se_2^{2+} \cdot 2BF_4^- \cdot CH_3CN$, $M_r = 456.74$, monoclinic, $P2_1/a$, $a = 24.053(6)$, $b = 6.955(2)$, $c = 9.416(3)$ Å, $\beta = 100.47(2)^\circ$, $V = 1549.0(7)$ Å³, $Z = 4$, $D_x = 1.959$ Mg m⁻³, $T = 295$ K, $\lambda(Mo K\alpha) =$

0.71069 Å, $\mu = 4.796$ mm⁻¹, $F(000) = 880$, $R = 0.069$ for 1688 observed reflections. The Se⁺—Se⁺ distance is 2.382(2) Å. The conformation of the eight-membered ring is a chair-boat form, while that in 1,5-dithioniabicyclo[3.3.0]octane bis(trifluoromethanesulfonate) is a distorted chair-chair form.

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