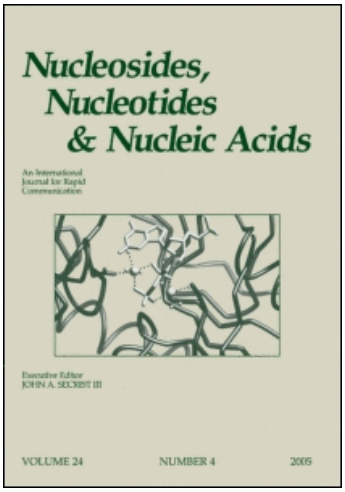

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STRUCTURE OF ETHYL HYDROGEN P(R),5'-ANHYDRO-2',3'-O-ISOPROPYLIDENE-ADENOSINE-8-PHOSPHONATE HEMIETHYLACETATE SOLVATE

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Abstract: The crystal and molecular structure of the title compound [8,5'-P-cyclo-A(R)] was determined by the X-ray diffraction method. It crystallizes in space group $C222_1$ with unit-cell dimensions a=14.222(3), b=13.879(4), c=43.612(8) Å. There are two 8,5'-P-cyclo-A(R) molecules and one ethyl acetate molecule per asymmetric unit. The two cyclonucleosides have a very similar overall conformation; the glycosidic torsion angle is in the anti range, the hetero-cyclic ring between the base and sugar moieties being in the endo form, while the sugar ring conformation is C(3')-exo. The two independent cyclonucleoside molecules form a hydrogen bonded dimer N(1)---H-N(6) [3.07(2) Å] and N(6)-H---O(6)(the free oxygen atom of the phosphonate group) [2.99(2) Å] .

INTRODUCTION

Cyclonucleosides are not only useful intermediates for the transformation of the base and sugar portions of nucleosides, but also important model compounds for studies on nucleoside conformation. A variety of cyclonucleosides has already been synthesized in which the oxygen, nitrogen, sulfur or carbon atom could form a bridge between base and sugar moieties.[1] As a part of our studies concerning the synthesis of cyclonucleosides, we have recently reported the synthesis of P, 5'-anhydroadenosine-8-phosphonic acid,[2] which is the first example of a novel type of cyclonucleoside bridged with the phosphorus atom between the base and sugar portions.

Photo-reaction of diethyl 8-bromo-2',3'-O-isopropylidene-adenosine-5'-phosphite has resulted in the preparation of two

TABLE 1. Crystal data

$C_{15}H_{20}N_5O_6P.1/2C_4H_8O_2$	FW 441.4
Orthorhombic	Space group $C222_1$
a=14.222(3) Å	Z=16
b=13.879(4) Å	F(000)=3712
c=43.612(8) Å	V=8608(3) Å^3
λ(Cu Kα)=1.54178 Å	D_x=1.36 Mg m^{-3}
μ=1.54 mm^{-1}	

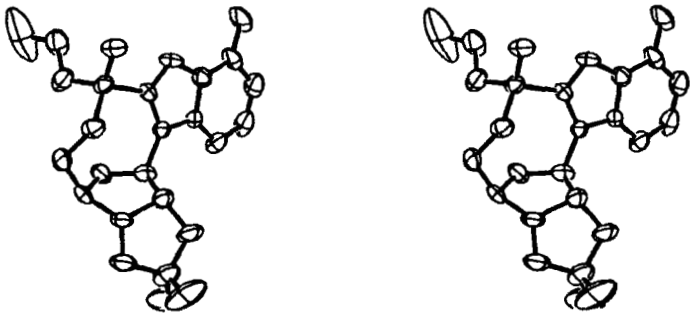
TABLE 2. Final atomic parameters with their standard deviations

Atom	x	y	z	$B_{eq/iso}$(Å^2)^a
molecule(a)				
P(1)	0.0698(3)	0.9370(3)	0.8097(1)	5.0(1)
N(1)	0.2830(11)	0.5678(9)	0.8497(3)	7.1(5)
C(2)	0.3399(15)	0.6004(12)	0.8283(3)	7.5(6)
N(3)	0.3322(10)	0.6764(9)	0.8113(3)	6.2(4)
C(4)	0.2546(10)	0.7269(9)	0.8166(3)	3.9(4)
C(5)	0.1883(12)	0.7016(11)	0.8377(3)	5.8(5)
C(6)	0.2028(13)	0.6209(11)	0.8558(3)	6.4(5)
N(7)	0.1176(8)	0.7733(9)	0.8380(2)	5.4(4)
C(8)	0.1434(11)	0.8347(10)	0.8173(3)	5.0(4)
N(9)	0.2272(8)	0.8095(7)	0.8032(2)	4.2(3)
N(6)	0.1392(13)	0.5921(10)	0.8763(3)	8.8(6)
O(6)	-0.0260(7)	0.9093(8)	0.8159(2)	6.5(3)
O(7)	0.1071(8)	1.0241(8)	0.8275(2)	7.0(4)
C(9)	0.1046(16)	1.0236(13)	0.8619(4)	9.1(7)
C(10)	0.1178(36)	1.1015(20)	0.8767(6)	25.1(23)
C(1')	0.2828(10)	0.8582(11)	0.7788(3)	5.1(4)
C(2')	0.2399(14)	0.8328(11)	0.7461(4)	6.3(5)
C(3')	0.2332(11)	0.9323(12)	0.7306(3)	6.2(5)
C(4')	0.2349(13)	1.0075(10)	0.7561(3)	6.0(5)
O(4')	0.2787(7)	0.9581(7)	0.7822(2)	5.7(3)
O(2')	0.3045(9)	0.7796(9)	0.7282(2)	8.3(4)
O(3')	0.3223(9)	0.9365(9)	0.7140(2)	8.3(4)
O(5')	0.0847(7)	0.9727(7)	0.7760(2)	5.8(3)
C(5')	0.1443(14)	1.0478(11)	0.7660(4)	7.6(6)
C(6')	0.3473(13)	0.8434(14)	0.7050(4)	7.7(6)
C(7')	0.2966(21)	0.8145(24)	0.6766(5)	15.8(13)
C(8')	0.4474(15)	0.8367(25)	0.7045(5)	15.0(13)

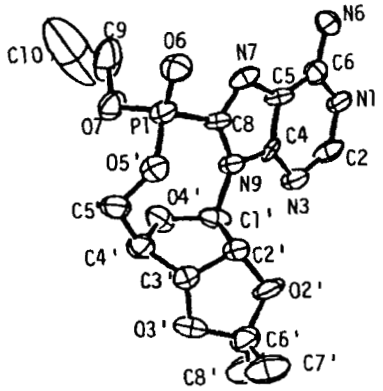
TABLE 2. (cont.)

molecule(b)				
P(1)	0.5244(3)	0.9362(3)	0.9411(1)	5.7(1)
N(1)	0.8893(9)	0.7341(9)	0.8994(3)	5.6(4)
C(2)	0.8623(13)	0.6697(12)	0.9206(4)	6.8(6)
N(3)	0.7862(9)	0.6709(10)	0.9390(3)	6.3(4)
C(4)	0.7364(9)	0.7518(10)	0.9337(3)	4.6(4)
C(5)	0.7583(12)	0.8252(10)	0.9125(3)	5.3(4)
C(6)	0.8384(11)	0.8120(11)	0.8941(3)	5.3(5)
N(7)	0.6871(10)	0.8952(8)	0.9129(2)	5.5(4)
C(8)	0.6278(11)	0.8649(10)	0.9347(3)	4.9(4)
N(9)	0.6539(9)	0.7792(8)	0.9470(2)	5.0(3)
N(6)	0.8672(9)	0.8792(11)	0.8744(3)	6.6(4)
O(6)	0.5450(8)	1.0340(7)	0.9347(2)	7.1(4)
O(7)	0.4436(8)	0.8881(9)	0.9217(2)	7.6(4)
C(9)	0.4495(15)	0.8907(22)	0.8892(4)	12.9(11)
C(10)	0.3910(31)	0.8668(51)	0.8749(7)	40.1(39)
C(1')	0.6122(12)	0.7224(10)	0.9720(3)	6.2(5)
C(2')	0.6312(11)	0.7670(12)	1.0049(3)	5.9(5)
C(3')	0.5338(11)	0.7656(11)	1.0195(3)	5.4(4)
C(4')	0.4657(11)	0.7593(13)	0.9940(3)	6.2(5)
O(4')	0.5140(8)	0.7189(8)	0.9679(2)	7.0(4)
O(2')	0.6858(9)	0.7000(10)	1.0209(2)	9.1(5)
O(3')	0.5384(9)	0.6753(8)	1.0357(2)	7.4(4)
O(5')	0.4929(8)	0.9178(7)	0.9753(2)	6.3(3)
C(5')	0.4222(13)	0.8520(13)	0.9844(3)	7.0(6)
C(6')	0.6320(13)	0.6577(15)	1.0446(4)	8.3(7)
C(7')	0.6516(19)	0.7107(16)	1.0750(4)	10.9(9)
C(8')	0.6483(20)	0.5566(15)	1.0461(5)	12.7(10)
ethylacetate				
C(11)	0.327(2)	0.5	0.	9.9(8)
C/O(12)*	0.277(3)	0.447(3)	-0.016(1)	9.8(12)
C/O(13)*	0.340(3)	0.558(3)	-0.014(1)	9.7(12)
O(14)*	0.346(3)	0.489(3)	0.030(1)	10.4(12)
C(15)*	0.314(3)	0.439(3)	0.046(1)	10.4(12)
C(16)*	0.378(3)	0.450(4)	0.062(1)	12.1(14)
C(21)	0.	0.343(2)	0.25	10.5(9)
C/O(22)*	-0.068(3)	0.324(3)	0.270(1)	7.8(9)
C/O(23)*	-0.001(3)	0.396(3)	0.218(1)	10.4(12)
O(24)*	0.052(3)	0.360(3)	0.270(1)	7.7(9)
C(25)*	0.067(3)	0.328(3)	0.300(1)	10.1(12)
C(26)*	0.076(5)	0.395(5)	0.315(1)	17.2(21)

[a] The equivalent isotropic temperature factors for 8,2'-P-cyclo-A molecules were computed using the expression: $B_{eq}=4/3\Sigma_i\Sigma_j\beta_{ij}a_ia_j$.
* Occupancy factors are 0.5.



(a)



(b)

FIG 1. Molecular conformations of two independent 8,5'-P-cyclo-A(R) molecules. (a); Stereoview of molecule (a), (b); Molecule (b) with atomic numberings.

diastereoisomers of ethyl hydrogen P,5'-anhydro-2',3'-O-isopropylidene-adenosine-8-phosphonate.[2] The present paper deals with the X-ray structure determination and molecular conformation of the ethyl acetate solvate of the R-isomer [8,5'-P-cyclo-A(R)], the minor product.

EXPERIMENTAL

8,5'-P-cyclo-A(R) was synthesized as described in the previous paper[2] and crystallized as its ethyl acetate solvate. A crystal with dimensions 0.2x0.2x0.2 mm was used for data collection on an automatic four-circle diffractometer (Rigaku Rota 300 AFC-5R-FOS) graphite-monochromated Cu Kα radiation (40 kV 240 mA). The cell parameters were determined by a least-squares procedure based on the 2θ values of 25 reflections. The crystallographic data are summarized in TABLE 1. A total of 3535 independent reflections was collected up to a $\sin\theta/\lambda$ maximum of 0.58 $\mathring{A}^{-1}$ using the ω-2θ scan mode. Of those, 2275 with $|Fo| > 3\sigma|Fo|$ were used for the structure analysis. Three reflections were monitored during data collection and their fluctuations were within 2 %. The data were corrected for Lorentz and polarization factors, but not for absorption.

The structure was solved by direct methods using the program MULTAN 78[3] and refined by the block-diagonal least-squares method with anisotropic temperature factors for non-hydrogen atoms of 8,5'-P-cyclo-A(R) molecules and with isotropic temperature factors for non-hydrogen atoms of ethyl acetate molecules, because of their disordered arrangement. All hydrogen atoms of 8,5'-P-cyclo-A(R) molecules obtained by geometrical criteria were fixed and included in the structure factor calculations. The final R factor was 0.084. The highest peak height in the final difference Fourier map was 0.7 $e\mathring{A}^{-3}$. The maximum ratio of parameter shift to e.s.d. after refinement was 0.6 [B_{33} of C(10) of molecule(a)] for 8,5'-P-cyclo-A(R) and 2.0 [B_{iso} of C(22)] for the ethyl acetate molecule. The large thermal ellipsoids of both C(10) atoms suggest overlapping of some slightly different orientations of C(10) atoms. All numerical calculations were carried out on an ACOS 850 computer at the Crystallographic Research Center, Institute for Protein Research, Osaka University, by using the modified programs of The Universal Crystallographic Computing System-Osaka (1979).[4] The atomic scattering factors used were those cited in International Tables for X-ray Crystallography.[5]

RESULTS AND DISCUSSION

The final atomic parameters for non-hydrogen atoms are listed in TABLE 2.

TABLE 3. Conformational parameters (°)

		molecule(a)	molecule(b)
χ	O(4')-C(1')-N(9)-C(4)	-142.4(12)	-140.2(12)
	O(4')-C(1')-N(9)-C(8)	36.3(18)	45.4(23)
	C(1')-N(9)-C(8)-P(1)	2.0(21)	-8.0(23)
	N(9)-C(8)-P(1)-O(5')	25.2(15)	30.9(15)
	C(8)-P(1')-O(5')-C(5')	-96.2(13)	-97.6(12)
	P(1)-O(5')-C(5')-C(4')	95.4(14)	97.0(14)
	O(5')-C(5')-C(4')-O(4')	-60.6(16)	-62.3(16)
	C(5')-C(4')-O(4')-C(1')	111.4(13)	110.2(14)
	C(4')-O(4')-C(1')-N(9)	-117.8(12)	-120.8(14)
τ_0	C(4')-O(4')-C(1')-C(2')	2.0(15)	2.0(16)
τ_1	O(4')-C(1')-C(2')-C(3')	11.5(15)	11.6(15)
τ_2	C(1')-C(2')-C(3')-C(4')	-20.2(15)	-20.5(15)
τ_3	C(2')-C(3')-C(4')-O(4')	21.7(15)	22.7(16)
τ_4	C(3')-C(4')-O(4')-C(1')	-14.8(15)	-15.5(16)
P	phase angle of pseudorotation	203.5	204.4
τ_m	maximum amplitude of pseudorotation	22.0	22.5
ψ	C(3')-C(4')-C(5')-O(5')	57.8(18)	58.5(18)

A minor product, one of the two diastereoisomers observed from the photo-reaction of diethyl 8-bromo-2',3'-O-isopropylideneadenosine-5'-phosphite, takes the R-configuration around its P atom. The molecular conformations of the two crystallographically independent cyclonucleosides are shown in FIG 1, and the selected torsion angles and pseudorotational parameters[6] are given in TABLE 3. The overall conformations of the two cyclonucleosides are very similar to each other. The glycosidic torsion angle [C(4')-C(1')-N(9)-C(4)] is -142.4(12)° for molecule (a) and -140.2(12)° for molecule (b); both are in the anti conformation. TABLE 4 shows the comparison of the glycosidic and sugar conformations for several 8,5'-cyclonucleosides with a covalent linkage between C(8) in the purine base and C(5') in the sugar moiety directly or through the oxygen or sulfur atom. It is seen that the conformation around the glycosidic bond and sugar ring are closely related to the number of atoms in the cyclic ring.

TABLE 4. Comparison of conformations for 8,5'-cyclonucelosides

Compounds	No. of atoms in the cyclic ring	χ(°)	P(°)	τm(°)	References
8,5'-P-cyclo-A(R)(a)	8	-142	203($_3$E)	22	This work
(b)	8	-140	204($_3$E)	22	
8,5'-S-cyclo-A(E)*	7	-115	255($^4_0$T)	36	11
8,5'-S-cyclo-A(I)**	7	-110	263($_0$E)	33	11,12
8,5'-O-cyclo-A***	7	-128	255($^4_0$T)	42	13
8,5'-cyclo-A****	6	-155	289($^1_0$T)	48	14

Abbreviations; *8,5'-anhydro-8-mercapto-2',3'-O-ethoxy-methylideneadenosine, **8,5'-anhydro-8-mercapto-2',3'-O-iso-propylideneadenosine, ***8,5'-anhydro-8-hydroxyadenosine, ****8,5'-anhydroadenosine.

TABLE 5. Bond lengths (Å) of 8,5'-P-cyclo-A(R)

	molecule(a)	molecule(b)		molecule(a)	molecule(b)
P(1)-C(8)	1.795(15)	1.794(14)	O(7)-C(9)	1.501(22)	1.420(26)
P(1)-O(6)	1.441(11)	1.416(11)	C(9)-C(10)	1.275(40)	1.092(53)
P(1)-O(7)	1.532(12)	1.576(12)	C(1')-C(2')	1.594(22)	1.586(22)
P(1)-O(5')	1.563(10)	1.580(11)	C(1')-O(4')	1.396(17)	1.408(19)
N(1)-C(2)	1.316(22)	1.342(21)	C(2')-C(3')	1.539(23)	1.525(21)
N(1)-C(6)	1.384(21)	1.321(19)	C(2')-O(2')	1.412(21)	1.398(20)
C(2)-N(3)	1.294(21)	1.347(21)	C(3')-C(4')	1.523(21)	1.476(22)
N(3)-C(4)	1.328(18)	1.348(18)	C(3')-O(3')	1.461(19)	1.441(18)
C(4)-C(5)	1.365(20)	1.411(20)	C(4')-O(4')	1.469(18)	1.443(19)
C(4)-N(9)	1.342(16)	1.364(18)	C(4')-C(5')	1.469(23)	1.488(23)
C(5)-C(6)	1.385(22)	1.405(21)	O(2')-C(6')	1.478(21)	1.413(22)
C(5)-N(7)	1.415(19)	1.403(19)	O(3')-C(6')	1.396(22)	1.408(22)
C(6)-N(6)	1.334(22)	1.333(20)	O(5')-C(5')	1.413(19)	1.414(20)
N(7)-C(8)	1.294(18)	1.337(18)	C(6')-C(7')	1.485(33)	1.545(29)
C(8)-N(9)	1.386(18)	1.358(18)	C(6')-C(8')	1.427(32)	1.423(30)
N(9)-C(1')	1.488(17)	1.470(19)			

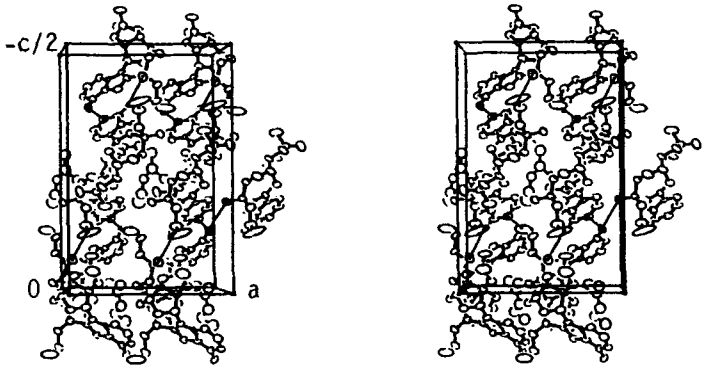


FIG 2. Stereoview of molecular packing along the b axis. Thin lines indicate the hydrogen bonding.

Structural consideration suggested that 8,5'-P-cyclo-A(R) may adopt two conformations: the endo form ($\chi \approx -140°$) with O(5') positioned over the center of the sugar ring or the exo form ($\chi \approx -100°$) with O(5') above O(4'). This result indicates that the endo form is preferable for 8,5'-P-cyclo-A(R), because the exo form causes the hetero-cyclic ring to introduce the short contact between two oxygen atoms, O(4') and O(5'). ^{1}H NMR data[2] indicate that 8,5'-P-cyclo-A(R) takes the endo form in solution as found in the crystal.

The sugar rings of 8,5'-P-cyclo-A(R) take a normal C(3')-exo conformation. Other 8,5'-cyclonucleosides exhabit an unusual C(4')-endo or O(4')-exo conformation as shown in TABLE 4. These observations indicate that the eight-membered ring formed by the cyclization between C(8) in the base and O(5') in the sugar moiety through a P atom causes little alteration in the sugar conformation, because each bond in eight-membered ring is rather flexible. The isopropylidene group causes flattering of the sugar puckering as indicated in the previous paper.[7]

The conformation about the exocyclic C(4')-C(5') bond is gauche-gauche, which is usually found in normal nucleoside structures and is favorable for the endo form of the cyclic eight-membered ring.

The bond lengths and angles are listed in TABLE 5. Although their standard deviations are somewhat larger than those of the related

TABLE 6. Bond angles (°) of 8,5'-P-cyclo-A(R)

	molecule(a)	molecule(b)
C(8)-P(1)-O(6)	107.8(7)	109.2(7)
C(8)-P(1)-O(7)	109.1(6)	106.3(6)
C(8)-P(1)-O(5')	110.3(6)	106.9(6)
O(6)-P(1)-O(7)	116.2(6)	116.8(7)
O(6)-P(1)-O(5')	113.0(6)	113.6(6)
O(7)-P(1)-O(5')	100.3(6)	103.4(6)
C(2)-N(1)-C(6)	117.4(14)	120.6(13)
N(1)-C(2)-N(3)	129.4(16)	129.2(15)
C(2)-N(3)-C(4)	113.8(13)	109.3(13)
N(3)-C(4)-C(5)	123.7(13)	126.7(13)
N(3)-C(4)-N(9)	128.1(12)	127.7(12)
C(5)-C(4)-N(9)	108.2(12)	105.6(12)
C(4)-C(5)-C(6)	119.4(14)	117.3(13)
C(4)-C(5)-N(7)	108.4(12)	109.3(12)
C(6)-C(5)-N(7)	132.0(14)	133.1(13)
N(1)-C(6)-C(5)	116.4(14)	116.8(13)
N(1)-C(6)-N(6)	121.8(14)	121.2(14)
C(5)-C(6)-N(6)	121.6(15)	121.7(14)
C(5)-N(7)-C(8)	104.8(12)	104.3(11)
P(1)-C(8)-N(7)	119.0(11)	117.0(10)
P(1)-C(8)-N(9)	128.3(10)	130.2(11)
N(7)-C(8)-N(9)	112.7(12)	112.6(12)
C(4)-N(9)-C(8)	105.9(10)	108.1(11)
C(4)-N(9)-C(1')	122.9(11)	120.9(12)
C(8)-N(9)-C(1')	131.2(11)	130.8(12)
P(1)-O(7)-C(9)	119.7(10)	118.8(12)
O(7)-C(9)-C(10)	120.0(21)	121.2(32)
N(9)-C(1')-C(2')	109.8(11)	113.2(12)
N(9)-C(1')-O(4')	110.8(11)	108.9(12)
C(2')-C(1')-O(4')	107.3(11)	107.3(12)
C(1')-C(2')-C(3')	102.6(12)	102.5(12)
C(1')-C(2')-O(2')	111.2(13)	106.7(12)
C(3')-C(2')-O(2')	105.6(13)	106.8(12)
C(2')-C(3')-C(4')	107.2(13)	106.5(12)
C(2')-C(3')-O(3')	101.4(12)	100.1(11)
C(4')-C(3')-O(3')	108.7(12)	110.4(12)
C(3')-C(4')-O(4')	104.6(11)	107.7(12)
C(3')-C(4')-C(5')	117.5(13)	115.7(14)
O(4')-C(4')-C(5')	108.7(12)	108.1(13)
C(1')-O(4')-C(4')	113.6(10)	111.1(11)
C(2')-O(2')-C(6')	109.4(12)	109.9(13)
C(3')-O(3')-C(6')	109.0(12)	109.2(12)
P(1)-O(5')-C(5')	127.3(10)	124.8(10)
C(4')-C(5')-O(5')	109.7(13)	110.0(13)
O(2')-C(6')-O(3')	104.8(13)	103.9(14)
O(2')-C(6')-C(7')	102.0(16)	109.4(15)
O(2')-C(6')-C(8')	112.4(17)	110.9(16)
O(3')-C(6')-C(7')	111.2(17)	108.9(15)
O(3')-C(6')-C(8')	108.6(17)	109.8(16)
C(7')-C(6')-C(8')	117.1(19)	113.6(17)

nucleosides, the P-C(8) bond length of 1.79(1) Å for both molecules are in agreement with the average values for the P-C (aromatic carbon) bond lengths.[8] The bond angles around the N(9) atom are typical for the anti conformer as found in non-cyclonucleosides.[9]

FIG. 2 shows the crystal structure viewed along to the b axis. A pronounced structural feature is the formation of a hydrogen bonded dimer as shown in FIG. 2, that is, N(6) of molecule (a) is hydrogen bonded to O(6) of molecule (b) with an N---O distance of 2.99(2) Å, and N(6) of molecule (b) is hydrogen bonded to N(1) of molecule (a) with an N---N distances of 3.07(2) Å. The hydrogen-bonded self-pairings between adenine moieties have been found in several neutral adenine derivatives, of which, the pairing scheme with N(6)-H--N(7) and N(1)---H-N(6)[10] is common. In this crystal, O(6) in place of N(7) accepts the hydrogen of N(6), because the ability of the free oxygen atom O(6) in the phosphonate group as a hydrogen-bond acceptor may be stronger than that of the ring nitrogen atom. Another hydrogen bonding is observed between N(6) of molecule (b) and O(6) of molecule (a) [N(6)-H---O(6); 3.00(2) Å].

Both disordered ethyl acetate molecules lie nearly on the two-fold axes, and each ethyl acetate interacts with 8,5'-P-cyclo-A(R) molecules by van der Waals contacts.

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