

the remaining atoms as incorporated in *SHELX76* (Sheldrick, 1976). All calculations on SUN4/280 computer system. Atomic parameters are given in Table 1, selected interatomic parameters in Table 2* and numbering scheme used is shown in Fig. 1 [drawn with *ORTEP* (Johnson, 1971) at 15% probability levels].

Related literature. Dimeric ring structures similar to that observed in [Ag{Ph₂P(CH₂)₃PPh₂}NO₃]₂ are well known in Ag chemistry for compounds of the general formula [Ag(P—P)X]₂ where X is a uninegative anion. Eight-membered rings have been characterized crystallographically in two bis(diphenylphosphino)methane (dppm) complexes, *i.e.* [Ag(dppm)NO₃]₂ (Ho & Bau, 1983; Tiekink, 1990) and in the bis(dimethylphosphino)methane analogue [Ag(dmpm)NO₃]₂ (Karsch & Schubert, 1982).

The Australian Research Council is thanked for support.

* Lists of structure factors, anisotropic thermal parameters, non-H atom bond lengths and angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53051 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

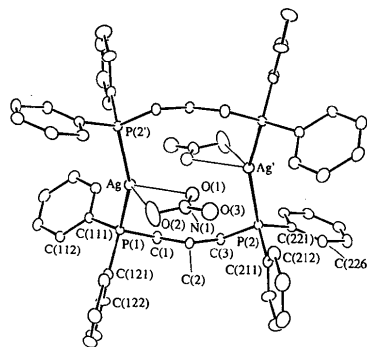


Fig. 1. Molecular structure and crystallographic numbering scheme employed for [Ag{Ph₂P(CH₂)₃PPh₂}NO₃]₂ (Johnson, 1971).

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Acta Cryst. (1990). **C46**, 1934–1936

Structure of a Nucleoside Analogue, 3'-Deoxy-2'-fluorothymidine*

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(Received 17 November 1989; accepted 28 March 1990)

Abstract. 1-(2,3-Dideoxy-2-fluoro-β-D-erythro-pentofuranosyl)thymine, C₁₀H₁₃FN₂O₄, *M_r* = 244.22, monoclinic, *P*2₁, *a* = 10.6001 (7), *b* = 6.1847 (1), *c* = 16.9227 (4) Å, β = 98.040 (3)°, *V* = 1098.52 (8) Å³, *Z* = 4, *D_m* = 1.47, *D_x* = 1.477 Mg m⁻³, Ni-filtered

Cu Kα radiation, λ = 1.54178 Å, μ = 1.040 mm⁻¹, *F*(000) = 512, *T* = 298 K, final *R* = 0.033 for 1868 unique observed reflections. The asymmetric unit contains two molecules (*A* and *B*). For molecule *A*: the *N*-glycosidic torsion angle χ has a value of -155.2 (2)° in the *anti* range; the sugar pucker is ³*T*₂ with *P* = 12 (1)° and ψ_{*m*} = 35 (1)° and the C4'—C5' conformation is +*sc* with γ = 55.1 (3)°. For molecule *B*: the *N*-glycosidic torsion angle χ has a value

* Structural Studies of Modified Nucleosides. Part III. Part II: Everaert, Peeters, Blaton & De Ranter (1990).

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of $-168.0(2)^\circ$ in the *anti* range; the sugar pucker is 3T_2 with $P = 8(1)^\circ$ and $\psi_m = 35(1)^\circ$ and the C4'—C5' conformation is +*sc* with $\gamma = 49.7(3)^\circ$. The conformational parameters are in accordance with the IUPAC-IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280] guidelines. There is little difference in sugar puckering (P and ψ_m) between molecules *A* and *B*. The main difference lies in the *N*-glycosidic torsion angle χ .

Experimental. Colourless prismatic crystals obtained at room temperature from an acetone–hexane solution, dimensions $\sim 0.5 \times 0.3 \times 0.2$ mm. Density measured by flotation in *n*-heptane/ CCl_4 . Hilger & Watts computer-controlled four-circle diffractometer, Ni-filtered $\text{Cu K}\alpha$ radiation, $\omega/2\theta$ scan technique ($2\theta_{\text{max}} = 130^\circ$, $-12 \leq h \leq 12$, $-7 \leq k \leq 0$, $-20 \leq l \leq 20$). Cell dimensions by least-squares refinement of the setting angles of 24 reflections with $19 < 2\theta < 50^\circ$, space group $P2_1$ from systematic absences $0k0$ for k odd. Four standard reflections (422, 125, 120, $3\bar{1}5$) monitored after every 50 reflections showed no significant decrease in intensity per hour, 3997 reflections measured, 2011 unique reflections ($R_{\text{int}} = 0.007$) of which 1872 were considered observed [$F > 6\sigma(F)$]. Four reflections (004, 011, 200, 211) badly affected by extinction were eliminated. Lorentz-polarization corrections, absorption corrections by the method of North, Phillips & Mathews (1968) based on one reflection (040) with values between 0.9965 and 0.8838, scattering factors from *International Tables for X-ray Crystallography* (1974) and Stewart, Davidson & Simpson (1965) (for H). The structure was solved by *MULTAN82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The *E* map calculated from the solution with the best figure of merit revealed 17 of the 34 non-H atoms in the asymmetric unit. The remaining atoms were obtained from a subsequent Fourier synthesis. A difference synthesis revealed the

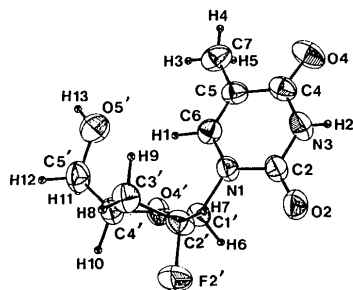


Fig. 1. A view of the title compound with 50% probability anisotropic displacement ellipsoids for the non-H atoms and atomic numbering scheme.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters with *e.s.d.*'s of the refined parameters in parentheses

$$B_{\text{eq}} = (4/3) \sum_i \beta_i a_i^2$$

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
N1A	9228 (2)	6140*	2627 (1)	3.24 (4)
C2A	9842 (2)	7627 (4)	3142 (1)	3.20 (4)
O2A	10432 (2)	9161 (3)	2932 (1)	4.27 (4)
N3A	9750 (2)	7228 (4)	3934 (1)	3.51 (4)
C4A	9141 (2)	5519 (4)	4237 (1)	3.35 (4)
O4A	9164 (2)	5376 (4)	4973.2 (9)	4.54 (4)
C5A	8510 (2)	4032 (5)	3662 (1)	3.38 (5)
C6A	8587 (2)	4419 (5)	2887 (1)	3.43 (5)
C7A	7799 (3)	2133 (5)	3923 (2)	4.60 (6)
C1'A	9350 (3)	6422 (5)	1763 (1)	3.77 (5)
C2'A	10526 (2)	5293 (6)	1565 (1)	4.59 (6)
F2'A	10953 (2)	6435 (4)	932 (1)	7.73 (5)
C3'A	10040 (2)	3136 (6)	1259 (2)	4.42 (6)
C4'A	8730 (2)	3674 (5)	840 (1)	3.72 (5)
O4'A	8309 (2)	5459 (3)	1292.8 (9)	3.85 (3)
C5'A	7755 (3)	1887 (6)	775 (2)	4.81 (6)
O5'A	7664 (2)	1082 (4)	1542 (1)	6.43 (5)
N1B	4865 (2)	5186 (4)	2405 (1)	3.50 (4)
C2B	5433 (2)	6399 (5)	1866 (1)	3.68 (5)
O2B	6084 (2)	7974 (4)	2076 (1)	4.73 (4)
N3B	5186 (2)	5722 (4)	1095 (1)	4.19 (4)
C4B	4472 (2)	3973 (5)	815 (1)	4.09 (5)
O4B	4316 (2)	3588 (4)	87 (1)	5.79 (5)
C5B	3938 (2)	2721 (5)	1401 (2)	3.94 (5)
C6B	4150 (2)	3383 (5)	2170 (2)	3.66 (5)
C7B	3198 (3)	734 (6)	1141 (2)	5.68 (7)
C1'B	5134 (2)	5858 (5)	3255 (1)	3.40 (5)
C2'B	4424 (3)	7897 (4)	3407 (1)	3.79 (5)
F2'B	5151 (2)	8951 (3)	4049 (1)	5.93 (4)
C3'B	3225 (3)	7057 (5)	3670 (2)	4.05 (5)
C4'B	3667 (2)	5051 (5)	4130 (1)	3.74 (5)
O4'B	4716 (2)	4239 (3)	3735.5 (9)	3.75 (3)
C5'B	2694 (3)	3299 (6)	4155 (2)	4.93 (6)
O5'B	2074 (2)	2840 (4)	3377 (1)	6.04 (5)

* Parameter kept fixed for origin definition.

Table 2. Bond lengths (\AA), bond angles ($^\circ$) and selected torsion angles ($^\circ$) with *e.s.d.*'s in parentheses

	A	B	A	B	
N1—C2	1.366 (3)	1.381 (3)	C5—C7	1.495 (4)	1.491 (4)
N1—C6	1.370 (3)	1.376 (3)	C1'—C2'	1.507 (4)	1.509 (4)
N1—C1'	1.496 (3)	1.486 (3)	C1'—O4'	1.400 (3)	1.401 (3)
C2—O2	1.217 (3)	1.217 (3)	C2'—F2'	1.409 (3)	1.401 (3)
C2—N3	1.380 (3)	1.360 (4)	C3'—C3'	1.496 (5)	1.496 (4)
N3—C4	1.374 (3)	1.367 (4)	C3'—C4'	1.506 (3)	1.504 (4)
C4—O4	1.246 (3)	1.243 (4)	C4'—O4'	1.450 (3)	1.465 (4)
C4—C5	1.434 (3)	1.437 (4)	C4'—C5'	1.506 (4)	1.501 (4)
C5—C6	1.347 (3)	1.352 (4)	C5'—O5'	1.406 (4)	1.416 (3)
C2—N1—C6	122.2 (2)	121.6 (2)	N1—C6—C5	123.4 (2)	122.3 (2)
C2—N1—C1'	116.6 (2)	116.4 (3)	N1—C1'—C2'	110.5 (2)	111.5 (2)
C6—N1—C1'	121.3 (2)	122.0 (3)	N1—C1'—O4'	109.7 (2)	109.2 (3)
N1—C2—O2	123.8 (3)	121.6 (2)	C2—C1'—O4'	106.7 (3)	107.0 (2)
N1—C2—N3	114.0 (2)	115.0 (2)	C1'—C2'—F2'	107.5 (2)	106.7 (2)
O2—C2—N3	122.3 (2)	123.4 (2)	C1'—C2'—C3'	103.6 (2)	103.0 (2)
C2—N3—C4	126.9 (3)	126.9 (3)	F2'—C2'—C3'	108.4 (2)	109.2 (3)
N3—C4—O4	118.9 (2)	119.1 (3)	C2'—C3'—C4'	102.5 (2)	102.9 (3)
N3—C4—C5	116.1 (2)	116.1 (2)	C3'—C4'—O4'	104.5 (2)	104.3 (2)
O4—C4—C5	125.1 (2)	124.9 (3)	C3'—C4'—C5'	116.8 (3)	116.4 (2)
C4—C5—C6	117.4 (2)	118.2 (2)	O4'—C4'—C5'	109.8 (2)	109.5 (2)
C4—C5—C7	120.6 (2)	118.8 (2)	C1'—O4'—C4'	110.4 (2)	110.0 (2)
C6—C5—C7	121.9 (2)	122.9 (3)	C4'—C5'—O5'	109.1 (2)	110.6 (2)
C2—N1—C1'—O4'	-155.24 (0.19)		-168.02 (0.20)		
C2'—C1'—O4'—C4'	3.79 (0.26)		6.14 (0.25)		
O4'—C1'—C2'—C3'	-24.14 (0.27)		-26.07 (0.25)		
C1'—C2'—C3'—C4'	34.07 (0.27)		35.07 (0.25)		
C2'—C3'—C4'—O4'	-32.14 (0.26)		-31.81 (0.24)		
C3'—C4'—O4'—C1'	18.01 (0.26)		16.22 (0.25)		
C3'—C4'—C5'—O5'	55.07 (0.33)		49.70 (0.33)		

position of two H atoms (attached to atoms O5'A and O5'B). All the other H atoms were included at calculated positions (C—H and N—H distance = 0.95 Å). All H atoms were refined with fixed isotropic temperature factors ($B_{\text{eq}} = 4 \text{ \AA}^2$). All other atoms were refined anisotropically on F by full-matrix least squares. The refinement converged at $R = 0.033$, $wR = 0.041$, $S = 0.264$, $w = (C_0 + C_1|F_o| + C_2|F_o|^2 + C_3|F_o|^3)^{-1}$, where $C_0 = 1000$, $C_1 = 1$, $C_2 = 0.001$, $C_3 = 0.000015$. 385 refined parameters, maximum shift/e.s.d. = 0.03, minimum and maximum electron density -0.183 and 0.129 e \AA^{-3} . The number of reflections per refined variable was $1868/385 = 4.9$. All calculations were performed on a PDP 11/73 microcomputer using *SDP/PDP* (Enraf-Nonius, 1982) and *PARST* (Nardelli, 1983). An *ORTEP* view (Johnson, 1976) of the title compound with the atomic numbering scheme is shown in Fig. 1.* The final fractional atomic coordinates are given in Table 1. Bond lengths, bond angles and some selected torsion angles are given in Table 2.

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, least-squares planes, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52902 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The method of preparation of this possible anti-AIDS compound has been described by Van Aerschot, Herdewijn, Balzarini, Pauwels & De Clercq (1989).

The authors wish to thank J. P. Van Cuyck for his help in preparing the figure.

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Acta Cryst. (1990). **C46**, 1936–1938

Structure of a Nucleoside Analogue, 2',3'-Dideoxy-3'-fluoro-5-iodouridine*

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(Received 23 November 1989; accepted 28 March 1990)

Abstract. 1-(2,3-Dideoxy-3-fluoro- β -D-erythro-pentofuranosyl)-5-iodouracil, $\text{C}_9\text{H}_{10}\text{FIN}_2\text{O}_4$, $M_r = 356.09$, orthorhombic, $B22_12$, $a = 7.917$ (4), $b = 16.962$ (8), $c = 17.694$ (9) Å, $V = 2377$ (2) Å³, $Z = 8$, $D_m = 1.98$, $D_x = 1.990 \text{ Mg m}^{-3}$, graphite-monochro-

mated Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 2.680 \text{ mm}^{-1}$, $F(000) = 1376$, $T = 290 \text{ K}$, final $R = 0.022$ for 1106 unique observed reflections. The N -glycosidic torsion angle χ has a value of -107.0 (4)° in the *anti* range; the sugar pucker is 2_3T with $P = 184$ (1)° and $\psi_m = 29$ (1)° and the C4'—C5' conformation is $+sc$ with $\gamma = 43.8$ (6)°. The conformational parameters are in accordance with the IUPAC-IUB Joint Commission on Biochemical

* Structural Studies of Modified Nucleosides. Part IV. Part III: Everaert, Peeters, Blaton, De Ranter, Van Aerschot & Herdewijn (1990).

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