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 $(B_{eq} = 4 \text{ Å}^2)$. 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 Å^{-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.

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* 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).

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

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Abstract. 1-(2,3-Dideoxy-3-fluoro-β-D-*erythro*-pentofuranosyl)-5-iodouracil, $C_9H_{10}FIN_2O_4$, $M_r = 356.09$, orthorhombic, $B22_12$, $a = 7.917$ (4), $b = 16.962$ (8), $c = 17.694$ (9) Å, $V = 2377$ (2) Å^3, $Z = 8$, $D_m = 1.98$, $D_x = 1.990$ Mg m^{-3}, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 2.680$ mm^{-1}, $F(000) = 1376$, $T = 290$ 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 ^3_2T 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

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Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280] guidelines. Base-pair formation occurs between symmetry-related molecules.

Experimental. Colourless prismatic crystals obtained at room temperature from a water–ethanol solution, dimensions $\sim 0.4 \times 0.2 \times 0.2$ mm. Density measured by flotation in n-heptane/CCl_4. Stadi-4 computer-controlled four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique ($2\theta_{max} = 50°$, $0 \le h \le 9$, $-20 \le k \le 20$, $0 \le l \le 21$). Cell dimensions by least-squares refinement of the setting angles of 50 reflections with $20 < 2\theta < 23°$, space group $B22_12$ from systematic absences hkl for $h + l$ odd and $0k0$ for k odd. Two standard reflections (004, 220) monitored every 120 min showed no significant decrease in intensity per hour, 4689 reflections measured, 1209 unique reflections of which 1107 were considered observed [$F > 4\sigma(F)$]. One reflection (004) badly affected by extinction was eliminated. Lorentz–polarization corrections, no absorption corrections, scattering factors from *International Tables for X-ray Crystallography* (1974) and Stewart, Davidson & Simpson (1965) (for H). $R_{int} = 0.024$. The position of the I atom was obtained from a sharpened Patterson synthesis. The position of this atom served as input for a Fourier synthesis which revealed the position of the remaining non-H atoms. A difference synthesis revealed the position of all H atoms. All H atoms were refined with fixed isotropic temperature factors (1.3 times that of the parent atom). All other atoms were refined anisotropically on F by full-matrix least squares. The refinement converged at $R = 0.022$, $wR = 0.028$, $S = 1.384$, $w = 1/\sigma^2(F_o)$. 184 refined parameters, maximum shift/e.s.d. $= 0.03$, minimum and maximum electron density -0.444 and 0.657 e Å^{-3}. The number of reflections per refined variable was $1106/184 = 6.0$. All calculations were performed on a PDP 11/73 micro-computer using *SDP/PDP* (Enraf–Nonius, 1982) and *PARST* (Nardelli, 1983). An *ORTEP* view (Johnson, 1976) of the title compound with the atomic
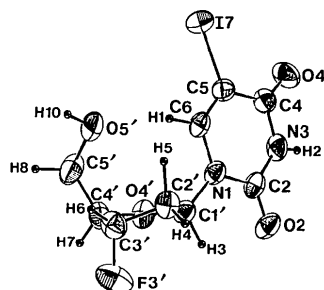
Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters with e.s.d.'s of the refined parameters in parentheses*

$$B_{eq} = (4/3)\Sigma_i \Sigma_j \beta_{ij} \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	B_{eq}(Å²)
N1	1555 (5)	3986 (2)	1673 (2)	3·22 (7)
C2	1556 (6)	4411 (3)	1006 (2)	3·44 (9)
O2	1260 (4)	5117 (2)	983 (2)	4·13 (6)
N3	1917 (6)	3978 (2)	376 (2)	3·83 (8)
C4	2360 (6)	3196 (3)	324 (3)	3·79 (9)
O4	2748 (6)	2909 (2)	-278 (2)	5·68 (9)
C5	2346 (6)	2796 (2)	1048 (3)	3·43 (8)
C6	1986 (6)	3197 (2)	1676 (2)	3·12 (7)
I7	3017·5 (4)	1609·1 (2)	1075·0 (2)	4·297 (6)
C1'	1273 (6)	4401 (2)	2394 (2)	3·12 (8)
C2'	-1 (6)	4005 (3)	2920 (2)	3·51 (9)
C3'	756 (7)	4144 (3)	3695 (3)	4·0 (1)
F3'	226 (4)	4892 (2)	3965 (2)	6·02 (7)
C4'	2645 (6)	4189 (3)	3560 (2)	3·72 (9)
O4'	2818 (4)	4437 (2)	2783 (2)	3·72 (6)
C5'	3584 (7)	3433 (3)	3683 (3)	5·0 (1)
O5'	2703 (6)	2770 (2)	3358 (2)	5·49 (9)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

N1—C2	1·384 (6)	C5—I7	2·084 (4)
N1—C6	1·381 (5)	C1'—C2'	1·528 (6)
N1—C1'	1·475 (5)	C1'—O4'	1·405 (6)
C2—O2	1·221 (5)	C2'—C3'	1·515 (6)
C2—N3	1·366 (6)	C3'—F3'	1·419 (6)
N3—C4	1·375 (6)	C3'—C4'	1·517 (7)
C4—O4	1·211 (6)	C4'—O4'	1·443 (6)
C4—C5	1·451 (6)	C4'—C5'	1·499 (7)
C5—C6	1·333 (7)	C5'—O5'	1·444 (7)
C2—N1—C6	120·5 (4)	N1—C6—C5	123·0 (4)
C2—N1—C1'	119·3 (3)	N1—C1'—C2'	114·7 (4)
C6—N1—C1'	119·9 (3)	N1—C1'—O4'	108·2 (4)
N1—C2—O2	122·7 (4)	C2'—C1'—O4'	107·2 (3)
N1—C2—N3	114·6 (4)	C1'—C2'—C3'	102·8 (4)
O2—C2—N3	122·8 (4)	C2'—C3'—F3'	109·1 (4)
C2—N3—C4	128·9 (4)	C2'—C3'—C4'	104·8 (4)
N3—C4—O4	120·8 (4)	F3'—C3'—C4'	107·4 (4)
N3—C4—C5	112·9 (4)	C3'—C4'—O4'	105·0 (4)
O4—C4—C5	126·2 (5)	C3'—C4'—C5'	115·0 (4)
C4—C5—C6	120·0 (4)	O4'—C4'—C5'	110·0 (4)
C4—C5—I7	118·0 (3)	C1'—O4'—C4'	111·8 (3)
C6—C5—I7	121·9 (3)	C4'—C5'—O5'	111·6 (4)

C2—N1—C1'—O4'	-106·99 (0·43)
C2'—C1'—O4'—C4'	-7·17 (0·47)
O4'—C1'—C2'—C3'	22·51 (0·46)
C1'—C2'—C3'—C4'	-28·75 (0·46)
C2'—C3'—C4'—O4'	25·21 (0·47)
C3'—C4'—O4'—C1'	-11·36 (0·48)
C3'—C4'—C5'—O5'	43·75 (0·59)

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.

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* 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 52905 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

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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Structure of 2,2',6,6'-Tetrakis[(dimethylammonium)methyl]biphenyl Tetrachloride n.Hydrate (n ≃ 1·72)

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Abstract. 2,2',6,6'-Biphenyltetrayltetrakis[methyl(dimethylammonium)] tetrachloride 1·72-hydrate, $C_{24}H_{42}N_4^{4+}.4Cl^-.1·72H_2O$, $M_r = 559·42$, monoclinic, $I2/a$, $a = 13·121$ (16), $b = 11·030$ (7), $c = 20·807$ (9) Å, $\beta = 102·43$ (6)°, $V = 2941$ (4) Å³, $Z = 4$, $D_x = 1·263$, $D_m = 1·29$ (2) Mg m⁻³, $\lambda(Cu\,K\alpha) = 1·54184$ Å, $\mu = 39·3$ cm⁻¹, $F(000) = 1196·8$, $T = 295$ K, $R = 0·131$ for 1160 unique observed diffractometer data $[I \geq 3\sigma(I)]$. Only crystals with a flaky habit, of poor quality were available, but the resulting moderate quality of the data is sufficient to secure the conformational and packing features of the title compound. The cation and an ordered water molecule are situated on a crystallographic twofold axis. The two phenyl rings are approximately perpendicular [83·3 (8)°] due to the fourfold *ortho* substitution. N—H···Cl and O—H···Cl hydrogen bonds link the ions and ordered H_2O into infinite chains running in the **a** direction. Voids in this structure contain additional disordered solvate molecules.

Experimental. The density was determined by flotation in an ethanol/CCl₄ mixture. The crystals crack and turn white after drying and were therefore mounted in glass capillaries. The crystals have a flaky habit causing broad reflection profiles. The crystals diffract poorly at higher θ values. An optimum specimen was selected by photographic techniques. Data were collected on an Enraf–Nonius CAD-4F diffractometer for a transparent, colorless crystal (0·24 × 0·06 × 1·4 mm). The cell parameters of the primitive triclinic unit cell [$a = 11·030$ (7), $b = 12·350$ (5), $c = 13·121$ (16) Å, $\alpha = 69·46$ (7), $\beta = 102·43$ (6), $\gamma = 63·37$ (4)°] were calculated by least squares from the SET4 setting angles of 25 reflections with $14 \leq \theta \leq 17°$. The metrical symmetry for this cell is *I*-centered monoclinic (Spek, 1988). Data set collected in the triclinic setting. 5847 reflections were scanned [$h - 13:13$, $k - 14:15$, $l\,0:15$; $\theta \leq 70°$; $\omega/2\theta$-scan mode; $\Delta\omega = (1·50 + 0·14\tan\theta)°$; Ni-filtered Cu $K\alpha$ radiation]. One reference reflection (124) showed a linear increase of 10% during 114 h of X-ray exposure time. The data were corrected for Lp and for absorption [*ABSORB* (Spek, 1983); correction factors 1·24–3·71] and transformed to monoclinic, resulting in the unique set of 1326 reflections [$I > 3\sigma(I)$] used in the structure determination. $\sigma^2(I) = \sigma_{cs}^2(I) + (pI)^2$ (McCandlish, Stout & Andrews, 1975) with $p = 0·031$. All non-H atoms were found by direct methods followed by peak optimization (*SHELXS*86; Sheldrick, 1986). The structure was refined on F by full-matrix least-squares procedures using anisotropic thermal parameters for all non-H atoms (*SHELX*76; Sheldrick, 1976). All H atoms