der Haest, Wynberg, Leusen & Bruggink (1990). Previous crystal structure determinations of related compounds include Kok, Wynberg, Smits, Beurskens & Parthasarathi (1987); Smits, Beurskens, Kok & Wynberg (1987); Smits, Beurskens, Parthasarathi, Rijk, Kok & Wynberg (1987); Kok, Wynberg, Parthasarathi, Smits & Beurskens (1987); and Bruins Slot, Leusen, van der Haest & Smits, 1992). Salt (1) may be called FINAM, and salt (2) FINAP.* The resolving power of the phosphorinane agents in these compounds is described by van der Haest *et al.* (1990).

A detailed description of the crystal packing, to identify interactions which determine the differences in resolution efficiency of the resolving agent for a pair of diastereomeric salts, has been published (Leusen, Bruins Slot, Noordik, van der Haest, Wynberg & Bruggink, 1991). A second paper on this subject will be published shortly by the same authors.

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* These names are composed of the general code INA, used for all compounds in the mentioned series, followed by M or P, indicating the two diastereomers of a pair (minus and plus rotation of plane polarized light). Eventual substituents at the aromatic moiety of the acid are indicated by prefixes, like F.

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Structure of a Nucleoside Analogue 2′,3′-Dideoxy-3′,5-difluorouridine*

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Abstract. 1-(2,3-Dideoxy-3-fluoro-β-D-*erythro*-pentofuranosyl)-5-fluorouracil, $C_9H_{10}F_2N_2O_4$, $M_r = 248.19$, monoclinic, $P2_1$, $a = 5.8653$ (2), $b = 11.1843$ (7), $c = 15.9975$ (12) Å, $\beta = 93.308$ (4)°, $V = 1047.7$ (1) Å^3, $Z = 4$, $D_m = 1.58$, $D_x = 1.574$ Mg m^{-3}, Ni-filtered Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å, $\mu = 1.250$ mm^{-1}, $F(000) = 512$, $T = 298$ K, final $R = 0.049$ for 1685 unique observed reflections [$F > 6\sigma(F)$]. The asymmetric unit contains two molecules *A* and *B*. For molecule *A*, the *N*-glycosidic torsion angle χ has a value of -167.6 (4)° in the *anti* range. Since the terminal CH_2OH group attached to atom

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* Structural Studies of Modified Nucleosides. XV. Part XIV: De Winter, De Ranter, Blaton, Peeters, Van Aerschot & Herdewijn (1992).

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C4′*A* of the sugar moiety is found to be disordered, all data concerning sugar puckering and C4′—C5′ conformation should be considered with great caution. The sugar pucker is ${}_3T^4$ with $P = 208\ (1)°$ and $\psi_m = 29\ (1)°$. Atom O5′*A* has two different positions (O51′*A* and O52′*A*); for O51′*A* the C4′—C5′ conformation is +*sc* with $\gamma = 62.2\ (9)°$ and for O52′*A* +*ac* with $\gamma = 140.6\ (7)°$. For molecule *B*, the *N*-glycosidic torsion angle χ has a value of $-125.1\ (4)°$ in the *anti* range; the sugar pucker is ${}^2T_3$ with $P = 169.3\ (1)°$ and $\psi_m = 29\ (1)°$ and the C4′—C5′ conformation is +*sc* with $\gamma = 49.1\ (5)°$. All conformational parameters are in accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280] guidelines. Base-pair formation occurs in the crystal structure.

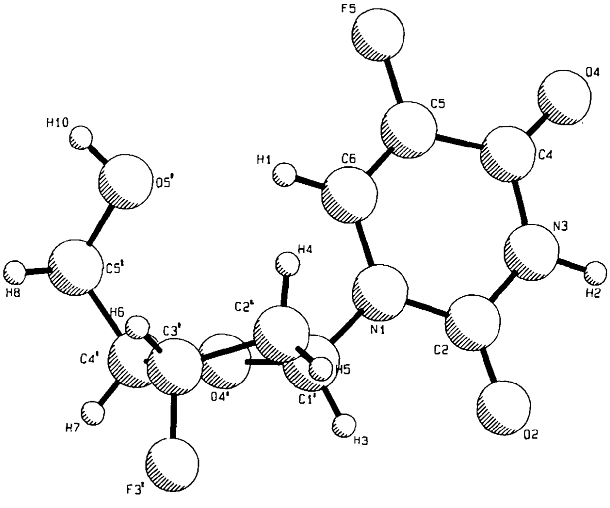

Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) plot of molecule *B* with the atomic numbering scheme.

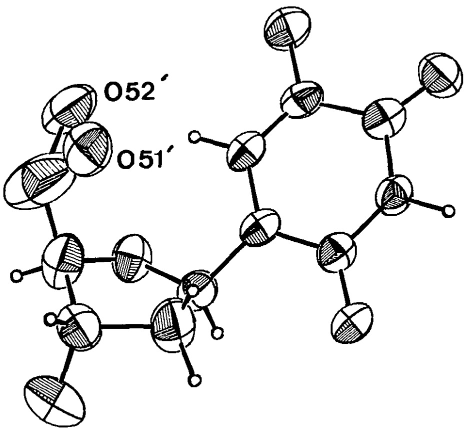

Fig. 2. *ORTEP* (Johnson, 1976) view of molecule *A* with 50% probability anisotropic displacement ellipsoids for the non-H atoms.

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

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

	x	y	z	B_{eq} (Å^2)
N1*A*	3724 (6)	7929*	4075 (2)	3.45 (6)
C2*A*	5165 (7)	7772 (4)	3442 (2)	3.32 (7)
O2*A*	6863 (5)	7136 (3)	3524 (2)	4.28 (6)
N3*A*	4556 (6)	8358 (3)	2708 (2)	3.49 (7)
C4*A*	2668 (7)	9071 (4)	2535 (3)	3.64 (8)
O4*A*	2259 (6)	9510 (3)	1841 (2)	4.67 (7)
C5*A*	1290 (7)	9196 (4)	3252 (3)	3.75 (8)
F5*A*	− 603 (5)	9866 (3)	3142 (2)	5.73 (7)
C6*A*	1773 (7)	8632 (4)	3959 (3)	3.69 (8)
C1′*A*	4147 (8)	7183 (4)	4846 (3)	4.19 (9)
C2′*A*	6268 (8)	7567 (7)	5352 (3)	5.8 (1)
C3′*A*	5524 (9)	7587 (5)	6247 (3)	5.0 (1)
F3′*A*	5944 (9)	6490 (4)	6615 (2)	9.8 (1)
C4′*A*	3004 (8)	7784 (6)	6136 (3)	5.1 (1)
O4′*A*	2295 (5)	7271 (3)	5337 (2)	4.14 (6)
C5′*A*	2250 (10)	9020 (8)	6222 (5)	10.3 (2)
O51′*A*†	2880 (10)	9652 (7)	5761 (4)	5.6 (2)
O52′*A*†	440 (10)	9525 (8)	5692 (5)	7.1 (2)
N1*B*	− 580 (6)	11851 (3)	− 850 (2)	3.32 (6)
C2*B*	855 (7)	12049 (3)	− 1481 (3)	3.28 (8)
O2*B*	2617 (5)	12651 (3)	− 1388 (2)	4.09 (6)
N3*B*	208 (6)	11559 (3)	− 2246 (2)	3.51 (7)
C4*B*	− 1717 (7)	10881 (4)	− 2450 (3)	3.59 (8)
O4*B*	− 2136 (6)	10497 (3)	− 3147 (2)	4.65 (7)
C5*B*	− 3091 (7)	10726 (4)	− 1738 (3)	3.70 (8)
F5*B*	− 5035 (5)	10081 (3)	− 1877 (2)	5.40 (6)
C6*B*	− 2557 (7)	11185 (4)	− 993 (3)	3.54 (8)
C1′*B*	− 28 (8)	12326 (4)	− 12 (3)	3.61 (8)
C2′*B*	− 1760 (10)	13228 (4)	283 (3)	4.8 (1)
C3′*B*	− 1799 (9)	12968 (4)	1206 (3)	4.7 (1)
F3′*B*	− 108 (8)	13632 (3)	1632 (2)	8.52 (9)
C4′*B*	− 1198 (7)	11654 (4)	1297 (3)	3.59 (8)
O4′*B*	− 56 (5)	11349 (3)	544 (2)	3.65 (5)
C5′*B*	− 3226 (8)	10852 (5)	1391 (3)	4.26 (9)
O5′*B*	− 4955 (5)	11113 (4)	766 (2)	5.27 (8)

* Parameter kept fixed for origin definition.
† Atom O5′*A* is disordered.

Experimental. Colorless prismatic crystals obtained at room temperature from an ethanol–dioxane solution, dimensions ~0.60 × 0.15 × 0.10 mm. Density measured by flotation in *n*-heptane/CCl$_4$. Hilger & Watts computer-controlled four-circle diffractometer, Ni-filtered Cu $K\alpha$ radiation, $\omega/2\theta$ scan technique, $2\theta_{max} = 130°$, $-7 \le h \le 0$, $-13 \le k \le 13$, $-19 \le l \le 19$. Cell dimensions by least-squares refinement of the setting angles of 24 reflections with $37 < 2\theta < 53°$, space group $P2_1$ from systematic absences, 0*k*0 for *k* odd. Four standard reflections ($\bar{4}00$, 020, 002, $\bar{2}1\bar{4}$) monitored after every 50 reflections showed no significant decrease in intensity per hour. 3805 reflections measured, 1822 unique reflections ($R_{int} = 0.032$) of which 1687 were considered observed [$F > 6\sigma(F)$]. Two reflections (012, 121) badly affected by extinction were eliminated. Lorentz–polarization corrections, no absorption corrections, scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) and for H atoms from Stewart, Davidson & Simpson (1965). Structure solved with *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The *E* map calculated from the solution with

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

		A	*B*
N1—C2		1.369 (5)	1.369 (5)
N1—C6		1.393 (5)	1.387 (6)
N1—C1′		1.498 (6)	1.461 (5)
C2—O2		1.226 (5)	1.235 (5)
C2—N3		1.374 (6)	1.373 (5)
N3—C4		1.380 (5)	1.384 (5)
C4—O4		1.225 (5)	1.207 (5)
C4—C5		1.447 (6)	1.445 (6)
C5—F5		1.342 (5)	1.357 (6)
C5—C6		1.312 (6)	1.318 (7)
C1′—C2′		1.506 (7)	1.526 (7)
C1′—O4′		1.380 (6)	1.409 (5)
C2′—C3′		1.521 (7)	1.505 (7)
C3′—F3′		1.378 (7)	1.386 (6)
C3′—C4′		1.495 (7)	1.517 (7)
C4′—O4′		1.441 (6)	1.452 (5)
C4′—C5′		1.46 (2)	1.504 (6)
C5′—O5′	(O51′)	1.11 (1)	1.414 (5)
	(O52′)	1.44 (1)	
C2—N1—C6		120.8 (3)	120.6 (3)
C2—N1—C1′		117.3 (3)	120.3 (3)
C6—N1—C1′		121.4 (3)	119.1 (3)
N1—C2—O2		122.0 (4)	123.0 (4)
N1—C2—N3		115.5 (3)	116.2 (3)
O2—C2—N3		122.6 (4)	120.8 (4)
C2—N3—C4		127.8 (4)	127.5 (4)
N3—C4—O4		121.8 (4)	122.3 (4)
N3—C4—C5		112.0 (4)	111.3 (3)
O4—C4—C5		126.2 (4)	126.5 (4)
C4—C5—F5		116.5 (4)	115.9 (4)
C4—C5—C6		122.3 (4)	123.5 (4)
F5—C5—C6		121.1 (5)	120.6 (4)
N1—C6—C5		121.5 (4)	120.7 (4)
N1—C1′—C2′		112.1 (5)	114.1 (4)
N1—C1′—O4′		109.3 (3)	106.7 (3)
C2′—C1′—O4′		109.1 (4)	106.6 (4)
C1′—C2′—C3′		103.7 (4)	102.9 (4)
C2′—C3′—F3′		109.7 (5)	109.1 (3)
C2′—C3′—C4′		103.1 (4)	105.4 (4)
F3′—C3′—C4′		109.5 (5)	108.5 (4)
C3′—C4′—O4′		106.2 (4)	105.3 (3)
C3′—C4′—C5′		115.6 (6)	114.0 (4)
O4′—C4′—C5′		112.7 (5)	110.4 (3)
C1′—O4′—C4′		109.7 (3)	111.3 (3)
C4′—C5′—O5′	(O51′)	115.5 (9)	110.2 (4)
	(O52′)	122.4 (7)	
C2—N1—C1′—O4′		−167.6 (4)	−125.1 (4)
C2′—′C1′—O4′—C4′		4.4 (6)	−14.9 (4)
O4′—C1′—C2′—C3′		13.7 (6)	26.9 (5)
C1′—C2′—C3′—C4′		−25.4 (6)	−28.6 (5)
C2′—C3′—C4′—O4′		28.7 (6)	20.5 (5)
C3′—C4′—O4′—C1′		−21.3 (6)	−3.4 (4)
C3′—C4′—C5′—O5′	(O51′)	62.2 (9)	49.1 (4)
	(O52′)	140.6 (7)	

the best figure of merit revealed 18 of the 34 non-H atoms in the asymmetric unit. All remaining atoms (except O5′*A*) were obtained from a subsequent Fourier synthesis. O5′*A* was found to be disordered (O51′*A* and O52′*A*, 50% each). A difference synthesis revealed the position of atom H10*B* (attached to atom O5′*B*). All other H atoms except those bonded to atoms C5′*A* and O5′*A* were included at calculated positions (C—H and N—H distances 0.95 Å). H atoms were given fixed isotropic temperature factors 1.3 times that of their parent atom. All other atoms were refined anisotropically on *F* by full-matrix least squares. Refinement converged at $R = 0.049$, $wR = 0.066$, $S = 2.703$, $w = [\sigma(F_o)^2 + 0.0004(F_o)^2]^{-1}$. 317 refined parameters, maximum shift/e.s.d. = 0.03, min. and max. electron density −0.316 and 0.214 e Å^{-3}. Number of reflections per refined variable was 1685/317 = 5.3. All calculations were performed on a PDP 11/73 microcomputer using *SDP* (B. A. Frenz & Associates, Inc., 1982) and *PARST* (Nardelli, 1983).

A *PLUTO* (Motherwell & Clegg, 1978) plot of molecule *B* with the atomic numbering scheme is shown in Fig. 1. An *ORTEP* (Johnson, 1976) view of molecule *A* with 50% probability anisotropic displacement ellipsoids for the non-H atoms is shown in Fig. 2. Final fractional atomic coordinates are given in Table 1.* Bond lengths, bond angles and selected torsion angles are given in Table 2. Except for the disordered terminal CH_2OH group of molecule *A*, all values are regarded as normal.

Related literature. The method of preparation has been described by Van Aerschot, Herdewijn, Balzarini, Pauwels & De Clercq (1989).

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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 54662 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0282]