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Structures of a Pair of Diastereomeric Salts of Ephedrine and Fluoro-Substituted Cyclic Phosphoric Acid

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Abstract. (1) Salt of (*S*)-(−)-5,5-dimethyl-4-*o*-fluorophenyl-2-hydroxy-1,3,2-dioxaphosphorinane 2-oxide and (1*S*,2*R*)-(+)-α-[(1-methylamino)ethyl]benzyl alcohol, (+)-$C_{10}H_{16}NO^+$.(−)-$C_{11}H_{13}FO_4P^-$, $M_r = 425.436$, monoclinic, $P2_1$, $a = 10.122\,(1)$, $b = 7.826\,(1)$, $c = 13.715\,(1)$ Å, $\beta = 92.99\,(1)°$, $V = 1085.0\,(2)$ Å^3, $Z = 2$, $D_x = 1.302$ Mg m^{-3}, Mo $K\alpha$ radiation (graphite monochromator), $\lambda = 0.71073$ Å, $\mu = 0.1599$ mm^{-1}, $F(000) = 452$, $T = 130$ K, final conventional $R = 0.040$, $wR = 0.042$ for 3487 observed unique reflections and 272 variables. (2) Salt of (*S*)-(−)-5,5-dimethyl-4-*o*-fluorophenyl-2-hydroxy-1,3,2-dioxaphosphorinane 2-oxide and (1*R*,2*S*)-(−)-α-[(1-methylamino)ethyl]benzyl alcohol, (−)-$C_{10}H_{16}NO^+$.(−)-$C_{11}H_{13}FO_4P^-$, $M_r = 425.436$, monoclinic, $P2_1$, $a = 9.679\,(1)$, $b = 8.115\,(1)$, $c = 13.797\,(1)$ Å, $\beta = 98.14\,(2)°$, $V = 1072.8\,(2)$ Å^3, $Z = 2$, $D_x = 1.317$ Mg m^{-3}, Mo $K\alpha$ radiation (graphite monochromator), $\lambda = 0.71073$ Å, $\mu = 0.1617$ mm^{-1}, $F(000) = 452$, $T = 130$ K, final conventional $R = 0.041$, $wR = 0.043$ for 3117 observed unique reflections and 272 variables. For both (1) and (2), the phosphorinane ring is in the usual chair conformation and the ephedrine is in the usual extended form. The crystal packings of (1) and (2) can be regarded as chains parallel to the *b* axis consisting of phosphorinane and ephedrine linked by hydrogen bonds. (1) and (2) are isostructural; only minor differences in the packing of the ephedrine phenyl moieties are observed.

Table 1. *Crystal and experimental data*

	(1)	(2)
Crystal dimensions (mm)	0.30 × 0.25 × 0.25	0.35 × 0.25 × 0.20
No. of reflections to determine lattice parameters	25	25
θ range (°)	9.2–15.6	9.2–15.6
Maximum $(\sin\theta)/\lambda$ (Å^{-1})	0.745	0.807
hkl range		
h	0–15	0–15
k	0–11	0–13
l	−20–20	−22–22
No. of standard reflections	3	3
Drift correction range	0.983–1.054	1.000–1.093
Total data measured	3992	3299
Unique data [$I > 3\sigma(I)$]	3495	3120
Data used in the refinement	3487	3117
Parameters refined	272	272
R	0.040	0.041
wR [$w = (\sigma^2F + gF^2)^{-1}$]	0.042	0.043
Weighting scheme parameter g	0.001	0.001
S	1.26	1.63
Maximum shift/e.s.d. in last cycle		
for non-H atoms	0.15	0.09
for H atoms	1.03	0.95
$(\Delta\rho)_{\max}$ (e Å^{-3})	0.44	0.52
$(\Delta\rho)_{\min}$ (e Å^{-3})	−0.37	−0.40

Experimental. Colorless block shaped crystals were prepared from equimolar amounts of (−)-phosphorinane for both (1) and (2), and (+)-ephedrine for (1) and (−)-ephedrine for (2) dissolved in acetone/water (1/1), by slow evaporation. Data sets were collected on an Enraf–Nonius CAD-4F κ-controlled diffractometer using graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å) and the θ–2θ scan technique [$0 < \theta < 32°$ for (1); $0 < \theta < 35°$ for (2)] with a scan angle of $(0.75 + 0.35\tan\theta)°$ and a variable scan rate with a maximum scan time of 1.5 s per reflection at 130 K (van Bolhuis, 1971; van Nes & van Bolhuis, 1978). Profile analysis was not performed. Normal Lorentz–polarization corrections were applied, but no absorption corrections were made. Data pertinent to the intensity data collection and refinement are given in Table 1.

The structure of (2) was solved by direct methods (*SDP/PDP* software, Enraf–Nonius, 1985) and

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Table 2. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *of non*-H *atoms*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor. The first lines give values for (1), the second lines for (2).

	x	y	z	U_{eq}
P(2)	0.24521 (5)	0.27000	0.55621 (3)	0.0164 (2)
	0.25439 (5)	0.27270	0.54797 (3)	0.0141 (2)
F(1)	0.55199 (15)	−0.1724 (2)	0.73905 (12)	0.0338 (8)
	0.55391 (19)	−0.2100 (2)	0.69966 (11)	0.0509 (9)
O(1)	0.35952 (14)	0.4109 (2)	0.55865 (11)	0.0170 (6)
	0.38297 (16)	0.3971 (2)	0.55246 (11)	0.0157 (6)
O(3)	0.26690 (13)	0.1810 (2)	0.66119 (10)	0.0148 (6)
	0.28677 (14)	0.1798 (2)	0.65152 (10)	0.0127 (6)
O(21)	0.26512 (16)	0.1428 (2)	0.47726 (11)	0.0291 (7)
	0.12465 (17)	0.3709 (2)	0.54867 (13)	0.0160 (7)
O(22)	0.11455 (14)	0.3606 (2)	0.55553 (11)	0.0167 (6)
	0.25389 (17)	0.1498 (2)	0.46724 (11)	0.0261 (8)
C(4)	0.39922 (18)	0.1167 (3)	0.68688 (14)	0.0160 (8)
	0.4215 (2)	0.0979 (3)	0.67016 (14)	0.0135 (8)
C(5)	0.50020 (17)	0.2657 (3)	0.68595 (14)	0.0136 (7)
	0.5383 (2)	0.2293 (3)	0.67327 (15)	0.0142 (8)
C(6)	0.4916 (2)	0.3440 (3)	0.58290 (16)	0.0148 (8)
	0.5195 (2)	0.3184 (3)	0.57340 (15)	0.0134 (8)
C(41)	0.39172 (14)	0.02634 (19)	0.78501 (8)	0.0177 (8)
	0.42109 (16)	−0.00343 (18)	0.76406 (8)	0.0178 (9)
C(42)	0.47358 (14)	−0.11398 (19)	0.80532 (8)	0.0260 (10)
	0.49224 (16)	−0.15338 (18)	0.77284 (8)	0.0284 (11)
C(43)	0.46905 (14)	−0.19793 (19)	0.89482 (8)	0.0386 (13)
	0.49120 (16)	−0.24983 (18)	0.85643 (8)	0.0443 (14)
C(44)	0.38266 (14)	−0.14156 (19)	0.96399 (8)	0.0431 (14)
	0.41902 (16)	−0.19632 (18)	0.93124 (8)	0.0416 (14)
C(45)	0.30079 (14)	−0.00124 (19)	0.94368 (8)	0.0366 (13)
	0.34787 (16)	−0.04637 (18)	0.92245 (8)	0.0308 (11)
C(46)	0.30532 (14)	0.08271 (19)	0.85419 (8)	0.0243 (10)
	0.34890 (16)	0.05007 (18)	0.83886 (8)	0.0246 (10)
C(51)	0.4723 (2)	0.4020 (3)	0.76143 (18)	0.0283 (10)
	0.5357 (3)	0.3534 (3)	0.75602 (17)	0.0243 (10)
C(52)	0.6408 (2)	0.1963 (3)	0.70416 (18)	0.0139 (8)
	0.6817 (2)	0.1430 (4)	0.68343 (16)	0.0144 (9)
O(10)	0.14343 (16)	0.1605 (3)	0.29736 (12)	0.0249 (7)
	0.16458 (16)	0.1135 (3)	0.27301 (12)	0.0132 (7)
N(2)	−0.07014 (16)	0.2133 (2)	0.42488 (12)	0.0176 (7)
	−0.06936 (17)	0.1896 (3)	0.40985 (12)	0.0149 (7)
C(1)	0.08079 (19)	0.3211 (3)	0.30428 (14)	0.0151 (8)
	0.0209 (2)	0.0716 (3)	0.26375 (15)	0.0168 (8)
C(2)	−0.06513 (19)	0.2958 (3)	0.32615 (14)	0.0167 (8)
	−0.0693 (2)	0.2095 (3)	0.30174 (14)	0.0154 (8)
C(3)	−0.1409 (2)	0.1866 (3)	0.25011 (16)	0.0249 (10)
	−0.0233 (3)	0.3817 (3)	0.27810 (18)	0.0293 (11)
C(11)	0.09146 (15)	0.4278 (2)	0.21201 (8)	0.0168 (8)
	−0.03337 (15)	0.0397 (2)	0.15735 (8)	0.0198 (9)
C(12)	0.04427 (15)	0.5953 (2)	0.21060 (8)	0.0216 (10)
	−0.15916 (15)	−0.0454 (2)	0.13534 (8)	0.0260 (11)
C(13)	0.05298 (15)	0.6949 (2)	0.12681 (8)	0.0289 (12)
	−0.21909 (15)	−0.0660 (2)	0.03804 (8)	0.0279 (12)
C(14)	0.10889 (15)	0.6270 (2)	0.04443 (8)	0.0428 (15)
	−0.15324 (15)	−0.0016 (2)	−0.03724 (8)	0.0497 (17)
C(15)	0.15608 (15)	0.4595 (2)	0.04585 (8)	0.0529 (18)
	−0.02746 (15)	0.0835 (2)	−0.01524 (8)	0.0581 (19)
C(16)	0.14737 (15)	0.3599 (2)	0.12964 (8)	0.0372 (13)
	0.03248 (15)	0.1041 (2)	0.08206 (8)	0.0342 (12)
C(20)	−0.1936 (2)	0.2491 (3)	0.47521 (18)	0.0257 (9)
	−0.1860 (3)	0.2768 (3)	0.44794 (16)	0.0284 (10)

difference Fourier syntheses were used to find the remaining non-H atoms. The structure of (1) was solved using the fractional coordinates of the phosphorinane moiety of (2). Difference Fourier syntheses revealed all remaining non-H atoms. Full-matrix least-squares refinement based on F (Sheldrick, 1976), with H atoms at calculated positions (except for the H10 atom in both structures, which was located from difference Fourier syntheses). Anisotropic thermal parameters for all non-H atoms, H atoms refined isotropically. Positional parameters and temperature factors are given

Table 3. *Selected bond lengths* (Å), *bond angles* (°) *and torsion angles* (°) *between non*-H *atoms*

	(1)	(2)
P(2)—O(1)	1.5974 (15)	1.5969 (16)
P(2)—O(3)	1.6042 (15)	1.6065 (15)
P(2)—O(21)	1.4922 (16)	1.4884 (17)
P(2)—O(22)	1.5002 (15)	1.4946 (16)
F(1)—C(42)	1.319 (2)	1.326 (2)
O(1)—C(6)	1.458 (3)	1.459 (3)
O(3)—C(4)	1.457 (2)	1.454 (3)
C(4)—C(5)	1.551 (3)	1.550 (3)
C(4)—C(41)	1.526 (2)	1.535 (2)
C(5)—C(6)	1.539 (3)	1.544 (3)
C(5)—C(51)	1.523 (3)	1.525 (3)
C(5)—C(52)	1.532 (3)	1.543 (3)
O(10)—C(1)	1.413 (3)	1.420 (3)
N(2)—C(2)	1.503 (3)	1.500 (3)
N(2)—C(20)	1.485 (3)	1.490 (3)
C(1)—C(2)	1.535 (3)	1.555 (3)
C(1)—C(11)	1.525 (2)	1.510 (2)
C(2)—C(3)	1.524 (3)	1.516 (3)
O(1)—P(2)—O(3)	102.46 (8)	102.08 (8)
O(1)—P(2)—O(21)	110.52 (9)	108.35 (9)
O(1)—P(2)—O(22)	108.13 (8)	111.78 (9)
O(3)—P(2)—O(21)	110.14 (8)	107.70 (9)
O(3)—P(2)—O(22)	106.87 (8)	109.22 (8)
O(21)—P(2)—O(22)	117.59 (9)	116.64 (10)
P(2)—O(1)—C(6)	114.30 (13)	114.20 (14)
P(2)—O(3)—C(4)	116.62 (12)	115.40 (12)
O(3)—C(4)—C(5)	109.65 (17)	108.83 (18)
O(3)—C(4)—C(41)	106.62 (14)	106.55 (15)
C(5)—C(4)—C(41)	114.76 (15)	115.73 (16)
C(4)—C(5)—C(6)	107.53 (16)	107.70 (16)
C(4)—C(5)—C(51)	112.04 (16)	112.68 (18)
C(4)—C(5)—C(52)	109.83 (18)	109.5 (2)
C(6)—C(5)—C(51)	109.99 (19)	110.3 (2)
C(6)—C(5)—C(52)	107.37 (16)	106.53 (17)
C(51)—C(5)—C(52)	109.95 (17)	109.97 (19)
O(1)—C(6)—C(5)	111.09 (16)	111.76 (16)
C(4)—C(41)—C(42)	118.99 (13)	118.71 (13)
C(4)—C(41)—C(46)	121.01 (14)	121.27 (14)
F(1)—C(42)—C(41)	120.51 (12)	120.41 (12)
F(1)—C(42)—C(43)	119.44 (14)	119.47 (14)
C(2)—N(2)—C(20)	113.97 (16)	113.99 (17)
O(10)—C(1)—C(2)	109.76 (18)	112.76 (19)
O(10)—C(1)—C(11)	112.33 (15)	109.27 (16)
C(2)—C(1)—C(11)	110.08 (16)	108.45 (16)
N(2)—C(2)—C(1)	108.02 (15)	109.69 (18)
N(2)—C(2)—C(3)	109.64 (17)	110.77 (19)
C(1)—C(2)—C(3)	113.18 (17)	113.25 (18)
C(1)—C(11)—C(12)	119.15 (13)	117.98 (13)
C(1)—C(11)—C(16)	120.86 (15)	121.81 (14)
C(6)—O(1)—P(2)—O(3)	53.42 (15)	53.99 (14)
C(6)—O(1)—P(2)—O(21)	−63.92 (15)	167.48 (14)
C(6)—O(1)—P(2)—O(22)	166.06 (13)	−62.64 (15)
C(4)—O(3)—P(2)—O(1)	−53.50 (15)	−56.90 (16)
C(4)—O(3)—P(2)—O(21)	64.11 (16)	−170.88 (14)
C(4)—O(3)—P(2)—O(22)	−167.09 (14)	61.55 (16)
C(5)—C(4)—O(3)—P(2)	59.67 (18)	63.09 (18)
C(41)—C(4)—O(3)—P(2)	−175.53 (11)	−171.47 (12)
O(3)—C(4)—C(5)—C(6)	−58.6 (2)	−59.4 (2)
O(3)—C(4)—C(5)—C(51)	62.4 (2)	62.5 (2)
O(3)—C(4)—C(5)—C(52)	−175.11 (16)	−174.80 (16)
C(41)—C(4)—C(5)—C(6)	−178.52 (16)	−179.25 (16)
C(41)—C(4)—C(5)—C(51)	−57.5 (2)	−57.4 (2)
C(41)—C(4)—C(5)—C(52)	64.9 (2)	65.3 (2)
O(3)—C(4)—C(41)—C(42)	147.98 (15)	145.97 (15)
O(3)—C(4)—C(41)—C(46)	−32.4 (2)	−32.6 (2)
C(5)—C(4)—C(41)—C(42)	−90.4 (2)	−92.9 (2)
C(5)—C(4)—C(41)—C(46)	89.18 (19)	88.5 (2)
C(4)—C(5)—C(6)—O(1)	60.9 (2)	59.2 (2)
C(51)—C(5)—C(6)—O(1)	−61.4 (2)	−64.2 (2)
C(52)—C(5)—C(6)—O(1)	178.99 (19)	176.53 (19)
C(5)—C(6)—O(1)—P(2)	−62.1 (2)	−60.2 (2)
C(4)—C(41)—C(42)—F(1)	−3.0 (2)	−2.8 (2)
C(4)—C(41)—C(42)—C(43)	179.59 (14)	−178.63 (15)
C(46)—C(41)—C(42)—F(1)	177.40 (14)	175.84 (15)
C(4)—C(41)—C(46)—C(45)	−179.58 (15)	178.60 (15)
F(1)—C(42)—C(43)—C(44)	−177.43 (14)	−175.89 (15)
O(10)—C(1)—C(2)—N(2)	64.2 (2)	−86.3 (2)
O(10)—C(1)—C(2)—C(3)	−57.4 (2)	38.0 (2)
C(11)—C(1)—C(2)—N(2)	−171.68 (15)	152.53 (16)
C(11)—C(1)—C(2)—C(3)	66.7 (2)	−83.2 (2)
O(10)—C(1)—C(11)—C(12)	−174.62 (15)	162.89 (18)
O(10)—C(1)—C(11)—C(16)	5.5 (2)	−22.4 (3)

Table 3 (*cont.*)

	(1)	(2)
C(2)—C(1)—C(11)—C(12)	62.8 (2)	−73.8 (2)
C(2)—C(1)—C(11)—C(16)	−117.17 (18)	100.88 (19)
C(1)—C(2)—N(2)—C(20)	154.16 (18)	−160.99 (19)
C(3)—C(2)—N(2)—C(20)	−82.1 (2)	73.3 (2)
C(1)—C(11)—C(12)—C(13)	−179.9 (2)	174.78 (16)
C(1)—C(11)—C(16)—C(15)	179.9 (2)	−174.58 (16)

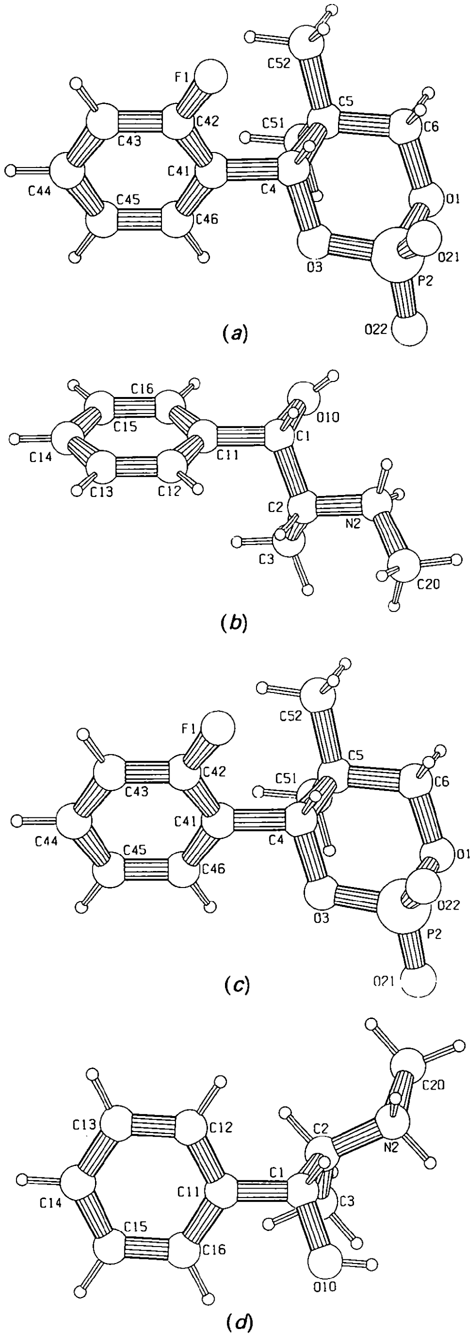
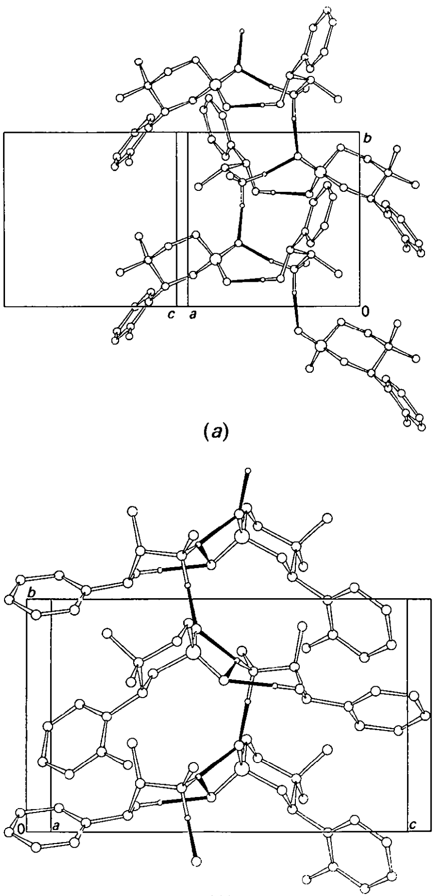


Fig. 1. Perspective drawings of the (−)-phosphorinane moieties of (*a*) salt (1) and (*c*) salt (2), and (*b*) the (+)-ephedrine moiety of (1) and (*d*) the (−)-ephedrine moiety of (2) showing the atomic numbering of non-H atoms.

in Table 2.* Selected bond lengths, angles and torsion angles are given in Table 3. Perspective drawings of the molecules are shown in Fig. 1 and packing diagrams in Fig. 2. Scattering factors as in *SHELX76* (Sheldrick, 1976). Geometry calculations and pictures obtained using the *EUCLID* package (Spek, 1982). Calculations were performed on a PDP11/23 and VAX 3800.

Related literature. These crystal structure determinations were undertaken as part of an ongoing research project to gain insight into the processes involved in the resolution of enantiomers *via* diastereomeric salt formation. The synthesis of the chiral acidic resolving phosphorinane agent is described by van



(*b*)

Fig. 2. Molecular packing, showing the hydrogen chains along the *b* axes: (*a*) salt (1) and (*b*) salt (2).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and full lists of bond lengths, angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54673 (50 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0244]

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, β = 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, μ = 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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