stable than (Ib), while structure (IIa) is 3.0 kcal mol⁻¹ (12.6 kJ mol⁻¹) more stable than (Iib). For (I), the presence of an intermolecular hydrogen bond can easily explain that (Ib), while higher in energy, is the preferred structure in the crystalline state. For compound (II), (IIa), which is lower in energy, corresponds to the X-ray structure. No major interactions are found in the crystal of (II).

References

- ALTONA, C., GEISE, H. J. & ROMERS, C. (1968). *Tetrahedron*, **24**, 13–32.
- DOUGLAS, S. P., SAWYER, J. F. & YATES, P. (1987). *Acta Cryst.* **C43**, 1372–1375.
- GABE, E. J., LEE, F. L. & LEPAGE, Y. (1985). *The NRCVAX Crystal Structure System*. In *Crystallographic Computing 3; Data Collection, Structure Determination, Proteins and Databases*, edited by G. M. SHELDRIK, C. KRÜGER & R. GODDARD, pp. 167–174. Oxford: Clarendon Press.
- LARSON, A. C. (1967). *Acta Cryst.* **23**, 664–665.
- RUEL, R. & P. DESLONGCHAMPS. (1990). *Can. J. Chem.* **68**, 1917–1922.
- Tripos Associates Inc. (1989). *SYBYL. Molecular Modeling Software*, Version 5.22. Tripos Associates Inc., St. Louis, Missouri, USA.
- WEISNER, K. & TSAI, T. Y. R. (1986). *Pure Appl. Chem.* pp. 799–810.
- ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1991). **C47**, 1693–1697

Structures of the 1-(2-Deoxy-2-fluoro- β -D-arabinopyranosyl)thymine–Water Complex and 1-(2-Deoxy-2-fluoro- β -D-arabinopyranosyl)-5-ethyluracil*

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Abstract. (I) 1-(2-Deoxy-2-fluoro- β -D-arabinopyranosyl)thymine–water complex, C₁₀H₁₃FN₂O₅·H₂O, M_r = 278.24, monoclinic, $P2_1$, a = 8.669 (4), b = 6.395 (3), c = 10.713 (7) Å, β = 103.73 (5)°, V = 576.9 (6) Å³, Z = 2, D_m = 1.60, D_x = 1.602 Mg m⁻³, graphite-monochromated Mo $K\alpha$ radiation, λ = 0.71069 Å, μ = 0.134 mm⁻¹, $F(000)$ = 292, room temperature, final R = 0.030 for 1307 unique observed [$F \geq 4\sigma(F)$] reflections. (II) 1-(2-Deoxy-2-fluoro- β -D-arabinopyranosyl)-5-ethyluracil, C₁₁H₁₅FN₂O₅, M_r = 274.25, hexagonal, $P6_5$, a = 10.108 (7), c = 19.48 (1) Å, V = 1724 (2) Å³, Z = 6, D_m = 1.54, D_x = 1.585 Mg m⁻³, graphite-monochromated Mo $K\alpha$ radiation, λ = 0.71069 Å, μ = 0.127 mm⁻¹, $F(000)$ = 864, room temperature, final R = 0.026 for 694 unique observed [$F \geq 4\sigma(F)$] reflections. The geometries of molecules (I) and (II) show a close similarity. In both structures the sugar ring adopts a

slightly distorted chair conformation and for both molecules the fluorine and one of the hydroxyl substituents are placed in an axial position on the ring. The N -glycosidic torsion angle χ between the pyranose ring and the pyrimidine base is oriented $-ac$ for both molecules. The terminal C of the ethyl group in (II) deviates from the base least-squares plane. The packing in both crystals is determined by intermolecular hydrogen bonds and base-stacking forces. In (I) the solvent water is kept from being disordered by a dense network of strong hydrogen bonds. In (II) the molecules are packed in such a way that a single stranded left-handed helix is formed which resembles the structure of the 6₁ poly(2'- O -methylcytidine) single stranded helix. No intramolecular hydrogen bonds are present. The conformational parameters are in accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature (1983) guidelines.

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Introduction. The crystal structures of the title compounds have been determined as part of a continuing program of investigation of potentially anti-viral

Table 1. *Experimental data*

	(I)	(II)
Space group	<i>P</i> 2 ₁	<i>P</i> 6 ₃
Crystal dimensions (mm)	0.1 × 0.4 × 0.6	0.3 × 0.3 × 0.5
Lattice parameters		
No. of reflections	34	24
2θ range (°)	20 ≤ 2θ ≤ 30	20 ≤ 2θ ≤ 26
Scan technique	ω/2θ	ω/2θ
Maximum 2θ (°)	55	50
<i>hkl</i> range	−11 ≤ <i>h</i> ≤ 11 0 ≤ <i>k</i> ≤ 8 −14 ≤ <i>l</i> ≤ 14	−12 ≤ <i>h</i> ≤ 12 0 ≤ <i>k</i> ≤ 12 0 ≤ <i>l</i> ≤ 23
Standard reflections	005, 040	451, 511
No. of measured reflections	2890	3306
No. of unique reflections	1445	1055
No. of unique observed reflections [<i>F</i> ≥ 4σ(<i>F</i>)]	1307	694
<i>R</i> _{int} on <i>F</i> for equivalent observed reflections	0.015	0.025
<i>R</i> , <i>wR</i>	0.030, 0.037	0.026, 0.030
Goodness-of-fit <i>S</i>	1.41	0.90
Weighting scheme	<i>w</i> = 1/[σ(<i>F</i> _o)] ²	<i>w</i> = 1/[σ(<i>F</i> _o)] ²
Max. shift/e.s.d.	0.01	0.01
Min. and max residual electron density (e Å ^{−3})	−0.25, +0.20	−0.16, +0.12
No. of refined parameters	215	170
No. of reflections per refined parameter	6.1	4.1
Absorption coefficient μ (mm ^{−1})	0.134	0.127

modified nucleosides. The synthesis of (I) and (II) will be described elsewhere.

Experimental. Transparent colourless crystals obtained at room temperature, (I) from methanol-water and (II) from methanol. 1-(2-Deoxy-2-fluoro-β-D-arabinopyranosyl)thymine, (I), crystallized with one water molecule per asymmetric unit. Density measured by flotation in *n*-heptane/CCl₄. Space group determined from systematic absences. X-ray diffraction data collected on a Stoe Stadi-4 four-circle diffractometer, cell constants by least-squares refinement of the setting angles of 34 (I) and 24 (II) reflections respectively. Experimental details are set out in Table 1. Standard reflections measured every two hours show only statistical fluctuations. Data reduction with a locally modified version of Stoe & Co. (1985) *REDU4* program, Lorentz and polarization corrections were applied, absorption corrections were unnecessary owing to the small absorption coefficients. Scattering factors were taken from Cromer & Waber (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Table 2.2B) and for H atoms from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were included for all non-H atoms (Ibers & Hamilton, 1964). Both structures solved with *MULTAN82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) which revealed all the non-H atoms of the pyrimidine ring for (I) and 17 of the 19 non-H atoms of molecule (II). Positions of the remaining non-H atoms were located stepwise by subsequent Fourier syntheses. Refinement on *F* by full-matrix least squares, first with isotropic temperature factors and

then anisotropically. Reflection 100 of (I), badly affected by extinction, was eliminated from the refinement. For both structures all H atoms were found in a difference synthesis. For (I) they were included in the refinement with fixed temperature factors *B*, 1.3 × *B*_{eq} (of parent atoms). For (II) most of the H atoms were placed at their calculated positions with a riding distance of 0.95 Å, in order to keep the number of reflections per refined parameter sufficiently high; only H(4), H(6), H(12), H(13) and H(14) (these are the H atoms for which the positions could not be calculated theoretically) were placed at their experimental positions and kept riding. For all H atoms the temperature factor *B* was kept fixed at 1.3 × *B*_{eq} (of parent atoms).

All calculations were performed on a Digital PDP-11/73 and MicroVAX 2000 microcomputer using *SDP* (Enraf-Nonius, 1985) and *PARST* (Nardelli, 1983).

Discussion. The structural formulas of the title compounds are shown in the scheme. *PLUTO* (Motherwell & Clegg, 1978) views of the title compounds with the atomic numbering schemes are shown in Fig. 1. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 2.* Bond lengths, bond angles and selected torsion angles are given in Table 3. All bond lengths and bond angles of both nucleosides are within the normal range (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The O(5')—C(1') bond lengths in both molecules are 0.020 (3) (I) and 0.028 (7) Å (II) shorter than the C(5')—O(5') distances.

A least-squares fit procedure with the program *BMFIT* (Nyburg, 1974) on all the non-H atoms of both molecules [except the water molecule in (I) and C(8) in (II)] showed a close similarity in geometry between molecules (I) and (II) (r.m.s. deviation = 0.16 Å).

* 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 53860 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Using the method of Cremer & Pople (1975), we calculated as phase angles $\varphi_2 = 82$ (5) (I) and 109 (3) $^\circ$ (II) and $\theta_2 = 177.3$ (3) (I) and 172.6 (4) $^\circ$ (II), with total puckering amplitudes $Q = 0.570$ (3) (I) and 0.571 (3) Å (II) for the sequences $C(1')-C(2')-C(3')-C(4')-C(5')-O(5')$. These puckering parameters describe slightly distorted chair conformations, with the total puckering amplitudes Q almost equal for both molecules and similar to comparable pyranosyl rings such as in 4-amino-1-[4-amino-2-oxo-1(2*H*)-pyrimidinyl]-1,4-dideoxy- β -D-glucopyranuronic acid (*C*-Substance) (Swaminathan, McAlister & Sundaralingam, 1980) and in 1-(2,3-dideoxy-*erythro*- β -D-hexopyranosyl)thymine (De Winter, Blaton, Peeters, De Ranter, Van Aerschot & Herdewijn, 1991).

Of the four ring substituents in the two molecules, F(22') and O(33') are placed in axial positions while N(1) and O(44') are in equatorial positions. The relative orientation of the base with respect to the sugar ring is given by the *N*-glycosidic torsion angle

Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) plot (a) of (I) and (b) of (II) with atom-numbering schemes.

Table 2. Fractional atomic coordinates and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^4$) with *e.s.d.*'s in parentheses

	$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
(I)				
N(1)	0.6036 (2)	0.7948 (3)	0.7367 (1)	229 (4)
C(2)	0.4492 (2)	0.7827 (3)	0.6685 (2)	228 (4)
O(2)	0.3373 (2)	0.7932 (3)	0.7178 (1)	316 (4)
N(3)	0.4309 (2)	0.7599 (3)	0.5397 (1)	250 (4)
C(4)	0.5487 (2)	0.7539 (3)	0.4736 (2)	252 (4)
O(4)	0.5126 (2)	0.7469 (3)	0.3567 (1)	362 (4)
C(5)	0.7084 (2)	0.7536 (4)	0.5519 (2)	261 (4)
C(6)	0.7277 (2)	0.7763 (4)	0.6778 (2)	253 (4)
C(7)	0.8440 (3)	0.7362 (5)	0.4900 (2)	392 (5)
C(1')	0.6348 (2)	0.8571 (3)	0.8705 (2)	211 (4)
C(2')	0.6900 (2)	0.6769 (3)	0.9614 (2)	229 (4)
F(22')	0.8249 (1)	0.589*	0.9334 (1)	324 (3)
C(3')	0.7341 (2)	0.7531 (3)	1.0993 (2)	236 (4)
O(33')	0.5910 (1)	0.8182 (3)	1.1277 (1)	308 (4)
C(4')	0.8500 (2)	0.9334 (3)	1.1088 (2)	225 (4)
O(44')	0.8934 (2)	1.0196 (3)	1.2332 (1)	304 (4)
C(5')	0.7834 (2)	1.1004 (3)	1.0120 (2)	257 (4)
O(5')	0.7498 (2)	1.0151 (3)	0.8857 (1)	265 (3)
O(W)	0.8523 (2)	0.2413 (3)	0.6543 (1)	445 (4)
(II)				
N(1)	0.0498 (3)	0.7713 (2)	0.7477 (2)	270 (8)
C(2)	0.1332 (3)	0.9230 (3)	0.7624 (2)	249 (9)
O(2)	0.2644 (2)	0.9834 (2)	0.7825 (2)	336 (5)
N(3)	0.0595 (2)	1.0015 (3)	0.7524 (2)	272 (8)
C(4)	-0.0890 (3)	0.9445 (3)	0.7310 (2)	274 (9)
O(4)	-0.1365 (2)	1.0322 (2)	0.7218 (2)	417 (8)
C(5)	-0.1748 (3)	0.7820 (3)	0.7202 (2)	272 (9)
C(6)	-0.1020 (3)	0.7042 (3)	0.7282 (2)	304 (10)
C(7)	-0.3369 (4)	0.7152 (3)	0.6988 (2)	389 (10)
C(8)	-0.4069 (4)	0.5608 (4)	0.6679 (3)	348 (13)
C(1')	0.1257 (3)	0.6812 (3)	0.7516 (2)	270 (9)
C(2')	0.0859 (4)	0.5843 (3)	0.8143 (3)	234 (10)
F(22')	-0.0702 (2)	0.4889 (2)	0.818*	522 (6)
C(3')	0.1611 (3)	0.4867 (3)	0.8135 (2)	353 (10)
O(33')	0.3186 (3)	0.5893 (2)	0.8246 (2)	477 (8)
C(4')	0.1332 (3)	0.4065 (3)	0.7461 (2)	309 (9)
O(44')	0.2264 (2)	0.3369 (2)	0.7448 (2)	364 (6)
C(5')	0.1732 (3)	0.5167 (3)	0.6872 (2)	353 (10)
O(5')	0.0850 (2)	0.5915 (2)	0.6919 (2)	349 (6)

* Parameters kept fixed during refinement.

$\chi[\text{O}(5')-C(1')-N(1)-C(2)]$, which is 229.2 (2) ($-ac$) for (I) and 226.3 (3) $^\circ$ ($-ac$) for (II). The sugar-base $C(1')-N(1)$ glycosyl bond lengths of 1.450 (2) (I) and 1.457 (5) Å (II) are significantly shortened compared with the mean value of 1.491 (4) Å found in the corresponding pyrimidine furanosyl nucleosides (Lin, Sundaralingam & Arora, 1971).

The pyrimidine heterocycles are essentially planar [maximum deviation 0.036 (2) and 0.021 (4) Å for C(4') of (I) and (II) respectively]. However, the heterocyclic substituents deviate more pronouncedly, with C(1') lying 0.277 (2) (I) and 0.118 (4) Å (II) above the plane. The C(4)-C(5)-C(7)-C(8) torsion angle in compound (II) is 161.9 (4) $^\circ$, leading to a C(8) out-of-plane deviation of 0.341 (5) Å.

The crystal packing of both molecules is determined by intermolecular hydrogen bonds

Table 3. Bond lengths (Å), bond angles (°) and selected torsion angles (°) with *e.s.d.*'s in parentheses

(I)									
N(1)	C(2)	1.367 (3)	C(1')	C(2')	1.512 (3)				
N(1)	C(6)	1.376 (2)	C(1')	O(5')	1.403 (2)				
N(1)	C(1')	1.450 (2)	C(2')	F(22')	1.392 (2)				
C(2)	O(2)	1.212 (2)	C(2')	C(3')	1.516 (2)				
C(2)	N(3)	1.359 (2)	C(3')	O(33')	1.409 (2)				
N(3)	C(4)	1.375 (3)	C(3')	C(4')	1.518 (3)				
C(4)	O(4)	1.217 (2)	C(4')	O(44')	1.408 (2)				
C(4)	C(5)	1.438 (2)	C(4')	C(5')	1.505 (3)				
C(5)	C(6)	1.326 (2)	C(5')	O(5')	1.423 (2)				
C(5)	C(7)	1.485 (4)							
N(1)	C(2)	O(2)	123.2 (1)	C(6)	N(1)	C(1')	119.7 (1)		
N(1)	C(2)	N(3)	114.4 (2)	C(6)	C(5)	C(7)	122.8 (2)		
N(1)	C(6)	C(5)	123.6 (1)	C(1')	C(2')	F(22')	108.8 (1)		
N(1)	C(1')	C(2')	112.6 (2)	C(1')	C(2')	C(3')	110.6 (2)		
N(1)	C(1')	O(5')	105.9 (1)	C(1')	O(5')	C(5')	111.5 (1)		
C(2)	N(1)	C(6)	121.5 (1)	C(2')	C(1')	O(5')	111.5 (1)		
C(2)	N(1)	C(1')	118.2 (2)	C(2')	C(3')	O(33')	105.9 (1)		
C(2)	N(3)	C(4)	127.2 (1)	C(2')	C(3')	C(4')	108.6 (2)		
O(2)	C(2)	N(3)	122.4 (1)	F(22')	C(2')	C(3')	108.0 (1)		
N(3)	C(4)	O(4)	119.4 (2)	C(3')	C(4')	O(44')	112.6 (2)		
N(3)	C(4)	C(5)	115.5 (1)	C(3')	C(4')	C(5')	110.7 (1)		
C(4)	C(5)	C(6)	117.7 (2)	O(33')	C(3')	C(4')	111.3 (2)		
C(4)	C(5)	C(7)	119.6 (2)	C(4')	C(5')	O(5')	110.0 (2)		
O(4)	C(4)	C(5)	125.3 (2)	O(4')	C(4')	C(5')	110.3 (2)		
C(2)	C(1')	O(5')	C(5')	-61.2 (2)	C(4')	C(3')	C(2')	F(22')	66.8 (2)
O(5')	C(1')	C(2')	C(3')	56.4 (3)	C(4')	C(3')	O(33')	H(6)	-93 (2)
O(5')	C(1')	C(2)	F(22')	-62.0 (3)	C(3')	C(4')	C(5')	O(5')	-58.6 (3)
C(1')	C(2)	C(3')	C(4')	-52.1 (3)	C(3')	C(4')	O(44')	H(4)	-69 (2)
C(2)	C(3')	C(4')	O(44')	177.8 (2)	O(44')	C(4')	C(5')	O(5')	176.1 (2)
C(2)	C(3')	C(4')	C(5')	53.8 (3)	C(5')	C(4')	O(44')	H(4)	55 (2)
C(2)	C(3')	O(33')	H(6)	149 (2)	C(4')	C(5')	O(5')	C(1')	62.0 (3)
(II)									
N(1)	C(2)	1.360 (3)	C(7)	C(8)	1.482 (6)				
N(1)	C(6)	1.384 (4)	C(1')	C(2')	1.490 (5)				
N(1)	C(1')	1.457 (5)	C(1')	O(5')	1.403 (5)				
C(2)	O(2)	1.215 (4)	C(2')	F(22')	1.379 (3)				
C(2)	N(3)	1.347 (5)	C(2')	C(3')	1.517 (6)				
N(3)	C(4)	1.377 (4)	C(3')	O(33')	1.416 (3)				
C(4)	O(4)	1.214 (5)	C(3')	C(4')	1.494 (6)				
C(4)	C(5)	1.440 (5)	C(4')	O(44')	1.431 (5)				
C(5)	C(6)	1.328 (6)	O(4')	C(5')	1.507 (5)				
C(5)	C(7)	1.486 (5)	C(5')	O(5')	1.431 (5)				
N(1)	C(2)	O(2)	122.7 (3)	C(6)	N(1)	C(1')	120.7 (2)		
N(1)	C(2)	N(3)	114.8 (3)	C(6)	C(5)	C(7)	125.2 (3)		
N(1)	C(6)	C(5)	123.3 (3)	C(1')	C(2')	F(22')	109.7 (3)		
N(1)	C(1')	C(2')	113.6 (3)	C(1')	C(2')	C(3')	111.1 (3)		
N(1)	C(1')	O(5')	106.6 (3)	C(1')	O(5')	C(5')	109.6 (3)		
C(2)	N(1)	C(1')	118.0 (2)	C(2')	C(1')	O(5')	111.0 (2)		
C(2)	N(1)	C(6)	121.3 (3)	C(2')	C(3')	O(33')	105.5 (2)		
C(2)	N(3)	C(4)	127.3 (3)	C(2')	C(3')	C(4')	110.0 (3)		
O(2)	C(2)	N(3)	122.4 (3)	F(22')	C(2')	C(3')	108.5 (2)		
N(3)	C(4)	O(4)	119.0 (3)	C(3')	C(4')	O(44')	107.0 (3)		
N(3)	C(4)	C(5)	115.5 (3)	C(3')	C(4')	C(5')	111.1 (3)		
C(4)	C(5)	C(6)	117.5 (3)	O(33')	C(3')	C(4')	111.7 (4)		
C(4)	C(5)	C(7)	117.2 (3)	C(4')	C(5')	O(5')	109.5 (3)		
O(4)	C(4)	C(5)	125.5 (3)	O(44')	C(4')	C(5')	110.5 (4)		
C(5)	C(7)	C(8)	115.5 (4)						
C(2)	C(1')	O(5')	C(5')	-64.4 (4)	C(4')	C(3')	C(2')	F(22')	71.3 (4)
O(5')	C(1')	C(2')	C(3')	56.8 (4)	C(4')	C(3')	O(33')	H(6)	-54.6 (4)
O(5')	C(1')	C(2)	F(22')	-63.1 (4)	C(3')	C(4')	C(5')	O(5')	-58.1 (4)
C(1')	C(2)	C(3')	C(4')	-49.3 (4)	C(3')	C(4')	O(44')	H(4)	-171.8 (3)
C(2')	C(3')	C(4')	O(44')	171.0 (3)	O(44')	C(4')	C(5')	O(5')	-176.6 (3)
C(2')	C(3')	C(4')	C(5')	50.4 (4)	C(5')	C(4')	O(44')	H(4)	-50.7 (4)
C(2')	C(3')	O(33')	H(6)	-174.1 (3)	C(4')	C(5')	O(5')	C(1')	64.6 (4)

Table 4. Geometry of intermolecular hydrogen bonds with *e.s.d.*'s in parentheses (distances in Å, angles in °)

(I)				
<i>X</i> —H... <i>Y</i>		<i>d</i> (H... <i>Y</i>)	<i>d</i> (<i>X</i> ... <i>Y</i>)	<i>X</i> —H... <i>Y</i>
O(33')—H(6)...O(4)	(1)	2.03 (3)	2.735 (3)	160 (3)
N(3)—H(9)...O(<i>W</i>)	(2)	1.99 (2)	2.817 (3)	165 (2)
C(7)—H(10)...O(44')	(3)	2.47 (3)	3.405 (3)	159 (3)
O(<i>W</i>)—H(1 <i>W</i>)...O(5')	(4)	2.50 (3)	3.173 (3)	140 (3)
O(44')—H(4)...O(2)	(5)	2.04 (3)	2.799 (2)	165 (3)
C(2')—H(7)...O(33')	(6)	2.40 (2)	3.317 (3)	169 (2)
O(<i>W</i>)—H(2 <i>W</i>)...O(44')	(7)	2.01 (3)	2.867 (3)	175 (3)

Symmetry code: (1) *x*, *y*, *z* + 1; (2) 1 - *x*, *y* + $\frac{1}{2}$, 1 - *z*; (3) *x*, *y*, *z* - 1; (4) *x*, *y* - 1, *z*; (5) 1 - *x*, *y* + $\frac{1}{2}$, 2 - *z*; (6) 1 - *x*, *y* - $\frac{1}{2}$, 2 - *z*; (7) 2 - *x*, *y* - $\frac{1}{2}$, 2 - *z*.

(II)				
<i>X</i> —H... <i>Y</i>		<i>d</i> (H... <i>Y</i>)	<i>d</i> (<i>X</i> ... <i>Y</i>)	<i>X</i> —H... <i>Y</i>
O(33')—H(6)...O(5')	(1)	2.494 (3)	3.326 (4)	140.3 (2)
C(3')—H(5)...O(4)	(2)	2.273 (3)	3.159 (5)	154.8 (2)
O(44')—H(4)...O(2)	(3)	1.843 (3)	2.748 (4)	158.0 (2)
N(3)—H(9)...O(44')	(4)	2.029 (2)	2.940 (4)	159.9 (2)

Symmetry code: (1) *y*, *y* - *x*, *z* + $\frac{1}{6}$; (2) *y* - 1, *y* - *x* - 1, *z* + $\frac{1}{6}$; (3) *x* - *y* + 1, *x*, *z* - $\frac{1}{6}$; (4) *x*, *y* + 1, *z*.

Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) plot of the packing in crystals of (a) (I) and (b) (II), along *b* and *c* respectively. Hydrogen bonds are not marked for clarity.

(summarized in Table 4) and base stacking forces. The bases are stacked almost parallel with average interplanar spacings of 3.2 (3) (I) and 3.4 (4) Å (II), respectively, and with the normals to their planes

nearly parallel to the 2₁ or 6₅ axis [dihedral angle between adjacent base least-squares planes is 11.59 (7) (I) and 16.8 (1)° (II) respectively]. It thus appears that the base-stacking properties of nucleosides as described earlier by Bugg & Thomas (1971) are not only found in furanosyl nucleosides but also in pyranosyl nucleosides, although the latter consists of a larger and more rigid six-membered sugar ring instead of the smaller and more flexible five-membered ring. In both compounds only partial base overlap is found with a polar substituent like O(4) (I) or O(2) (II) positioned above the adjacent base (see Fig. 2). The result is that (II) is packed as a single stranded left-handed helix with six molecules per complete turn, an axial rise of 3.2 Å and a pitch of 19.48 (1) Å (for nomenclature see Saenger, 1984). This resembles more or less the structure of the 6₁ poly(2'-*O*-methylcytidine) single stranded helix (Leslie & Arnott, 1978), the latter with an axial rise of 3.2 Å and a pitch height of 18.9 Å. In contrast, however, the poly(2'-*O*-methylcytidine) helix is right-handed and the nucleosides are covalently bonded to each other by a sugar-phosphate backbone.

Many crystal structures in which solvent molecules are included are characterized by larger thermal parameters for those solvent molecules. The small thermal vibrations of the water molecule in (I) are a consequence of a dense hydrogen-bond network involving the water molecule and which consists of two extremely strong hydrogen bonds between N(3)—H(9) and the water O atom and from H(2*W*) towards O(44'), and a third somewhat weaker hydrogen bond between H(1*W*) and O(5'). The result is a neatly ordered water molecule with small thermal vibrations and normal geometry [mean O(*W*)—H(*W*) distance = 0.84 (4) Å and H(1*W*)—O(*W*)—H(2*W*) angle = 101 (3)°]. No intramolecular hydrogen bonds are present.

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References

- ALLEN, F. H., KENNARD, O., WATSON, D. G., BRAMMER, L., ORPEN, A. G. & TAYLOR, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
- BUGG, C. E. & THOMAS, J. M. (1971). *Biopolymers*, **10**, 175–219.
- CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- DE WINTER, H. L., BLATON, N. M., PEETERS, O. M., DE RANTER, C. J., VAN AERSCHOT, A. & HERDEWIJN, P. (1991). *Acta Cryst.* **C47**, 838–842.
- Enraf-Nonius (1985). *Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.
- EVERAERT, D. H., PEETERS, O. M., BLATON, N. M., DE RANTER, C. J., VAN AERSCHOT, A. & HERDEWIJN, P. (1991). *Acta Cryst.* **C47**, 898–902.
- IBERS, J. A. & HAMILTON, W. C. (1964). *Acta Cryst.* **17**, 781–782.
- IUPAC-IUB JOINT COMMISSION ON BIOCHEMICAL NOMENCLATURE (1983). *Pure Appl. Chem.* **55**, 1273–1280.
- LESLIE, A. G. W. & ARNOTT, S. (1978). *J. Mol. Biol.* **119**, 399–414.
- LIN, G. H.-Y., SUNDARALINGAM, M. & ARORA, S. K. (1971). *J. Am. Chem. Soc.* **93**, 1235–1241.
- MAIN, P., FISKE, S., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
- NYBURG, S. C. (1974). *Acta Cryst.* **B30**, 253–254.
- SAENGER, W. (1984). *Principles of Nucleic Acid Structure*, ch. 2. New York: Springer-Verlag.
- Stoe & Co. (1985). *REDU4. Data Reduction Program*. Stoe & Co., Darmstadt, Germany.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- SWAMINATHAN, P., MCALISTER, J. & SUNDARALINGAM, M. (1980). *Acta Cryst.* **B36**, 878–885.

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N-[(1-Butyl-2-pyrrolidiny)methyl]-2-methyl-5-sulfamoyl-2,3-dihydrobenzofuran-7-carboxamide

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Abstract. C₁₉H₂₇N₃O₄S, *M_r* = 393.5, monoclinic, *P*2₁, *a* = 12.955 (2), *b* = 35.050 (8), *c* = 9.3196 (7) Å, β = 94.548 (8)°, *V* = 4218 (1) Å³, *Z* = 8, *D_m* = 1.235, *D_x* = 1.239 Mg m⁻³, Cu *K*α, λ = 1.5418 Å, μ = 1.58 mm⁻¹, *F*(000) = 1680, *T* = 298 K, *R* = 0.067 for

4899 observed reflections. An intramolecular N...O hydrogen bond between the amide group and benzofuran ring makes these two parts of the molecule flat. These two parts and the sulfamoyl groups of the four unique molecules form a layer by two kinds of