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Structure of Ethyl 2-Cyanomethyl-1-β-D-arabinofuranosyl-1*H*-pyrrole-3-carboxylate

By Steven B. Larson, Nabih S. Girgis, Howard B. Cottam and Roland K. Robins

Nucleic Acid Research Institute, 3300 Hyland Avenue, Costa Mesa, California 92626 and Department of Chemistry, Brigham Young University, Provo, Utah 84602, USA

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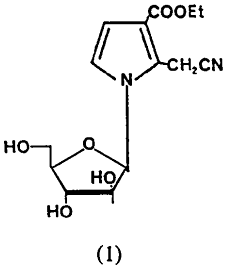
Abstract. $C_{14}H_{18}N_2O_6$, $M_r = 310\cdot31$, orthorhombic, $P2_12_12_1$, $a = 4\cdot8660\,(2)$, $b = 14\cdot7729\,(11)$, $c = 21\cdot4296\,(14)$ Å, $V = 1540\cdot4\,(2)$ Å^3, $Z = 4$, $D_x = 1\cdot338$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1\cdot5418$ Å, $\mu = 8\cdot518$ cm^{-1}, $F(000) = 656$, room temperature, $R = 0\cdot0453$ for 1411 reflections ($F \geq 4\sigma_F$). The β anomeric configuration is confirmed. The sugar ring is C(3′)-*endo* ($^3T_4$ form) with a phase angle of pseudorotation of 23·8° and an amplitude of pucker of 36·7° [Altona & Sundaralingam (1972). *J. Am. Chem. Soc.* **94**, 8205–8212]. The C(5′)—O(5′) side chain is in the *gauche–trans* conformation. The nucleoside conformation can be considered as *anti* with a torsion angle about the glycosidic bond [χ = O(1′)—C(1′)—N(1)—C(5)] of 43·0 (5)°. All atoms of the base ring are within 0·012 Å of its mean plane. The ester moiety [excluding C(13)] is fairly planar as well. The dihedral angle between these planes is 7 (1)°. The cyanomethyl mean plane makes an angle of 95·6 (1)° with the base ring. The hydroxyl hydrogens are involved in intermolecular hydrogen bonding.

Experimental. The title compound (1) was prepared as previously described (Girgis, Cottam, Larson & Robins, 1987*b*). Crystals grew as colorless, transparent flat needles. A crystal was mounted on the end of a glass fiber with epoxy. Details of data collection and structural refinement are given in Table 1.



(1)

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Table 1. *Summary of data collection and structural refinement*

Data collection (295 K)	
Mode	ω scan
Scan range (°)	0·80 + 0·15tan*θ*
Background (°)	scan 0·25 times scan range before and after scan
Scan rate (° min^{-1})	1·27–5·5
Exposure time (h)	26·7
Stability correction range on *I*	1·000–1·010
2*θ* range (°)	3·0–150·0
Total refls: measd, unique	1875, 1875
Range in *hkl*: min.	0,0,0
max.	6,18,26
Crystal dimensions (mm)	0·33 × 0·20 × 0·09
Crystal volume (mm^3)	0·00594
Transmission factor range:	0·794–0·928
Structure refinement	
Instability factor *p*	0·02
Reflections used, *m* ($I \geq 2\sigma_I$)	1411
No. of variables, *n*	226
Goodness of fit, *S*	2·364
R, *wR*	0·0453, 0·0531
R (all data)	0·0908
Max. shift/e.s.d.	0·01
Max., min. density in $\Delta\rho$ map (e Å^{-3})	0·15, −0·18

Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $32\cdot90 \leq 2\theta \leq 53\cdot22°$.
Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used. Data reduction was performed with the *SDP-Plus* software (Frenz, 1985). Crystal and instrument stability was monitored by remeasurement of three check reflections every hour. A linear fit of the intensities of these reflections was used to correct the data.
Function minimized was $\sum w(F_o - F_c)^2$, where $w = \sigma_F^{-2}$. $\sigma_F = F\sigma_I/2I$; $\sigma_I = [N_{\rm pk} + N_{\rm bg1} + N_{\rm bg2} + (pI)^2]^{1/2}$.

21 non-hydrogen atoms were located with *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982); the remaining non-hydrogen atom was located in an electron density difference map. H atoms were located in difference

maps; methyl and methylene H atoms were idealized. The structure was refined by full-matrix least squares (Frenz, 1985) to $R = 0{\cdot}0453$. Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). All computations were performed with the *SDP-Plus* program package (Frenz, 1985).

Atomic coordinates are listed in Table 2;* bond lengths and bond angles are in Table 3. Fig. 1 is a perspective *ORTEP*II (Johnson, 1976) drawing of the molecule with atom labels indicated. Fig. 2 is the packing diagram indicating the intermolecular hydrogen bonding listed in Table 4.

Table 2. *Atomic positions and thermal parameters*

	x	y	z	U/U_{eq}*(Å^2)
N(1)	0·3838 (6)	0·8596 (2)	0·90408 (11)	0·0391 (8)
C(2)	0·2963 (8)	0·9273 (2)	0·94238 (13)	0·0394 (10)
C(3)	0·4466 (9)	0·9208 (2)	0·99714 (14)	0·0498 (12)
C(4)	0·6263 (9)	0·8451 (2)	0·9917 (2)	0·0532 (11)
C(5)	0·5870 (9)	0·8103 (2)	0·9336 (2)	0·0501 (11)
C(6)	0·0688 (8)	0·9915 (2)	0·92545 (14)	0·0463 (11)
C(7)	0·1567 (10)	1·0688 (2)	0·8900 (2)	0·0654 (14)
N(8)	0·2156 (13)	1·1313 (2)	0·8618 (2)	0·117 (2)
C(9)	0·4317 (11)	0·9787 (3)	1·0530 (2)	0·068 (2)
O(10)	0·5569 (11)	0·9641 (3)	1·09940 (13)	0·136 (2)
O(11)	0·2772 (9)	1·0512 (2)	1·04563 (12)	0·0972 (14)
C(12)	0·259 (2)	1·1130 (3)	1·0972 (2)	0·124 (3)
C(13)	0·213 (2)	1·1980 (4)	1·0767 (3)	0·174 (4)
C(1′)	0·2815 (8)	0·8434 (2)	0·84144 (14)	0·0426 (10)
C(2′)	0·4788 (8)	0·8736 (2)	0·78947 (14)	0·0408 (10)
C(3′)	0·6183 (9)	0·7865 (2)	0·7695 (2)	0·0464 (11)
C(4′)	0·3918 (9)	0·7168 (2)	0·7782 (2)	0·0463 (11)
C(5′)	0·4916 (10)	0·6217 (2)	0·7899 (2)	0·0581 (13)
O(1′)	0·2472 (6)	0·74775 (15)	0·83315 (10)	0·0492 (7)
O(2′)	0·6581 (6)	0·94157 (13)	0·80992 (9)	0·0485 (8)
O(3′)	0·7147 (7)	0·7944 (2)	0·70767 (11)	0·0702 (10)
O(5′)	0·2771 (7)	0·55699 (15)	0·78663 (11)	0·0697 (10)
H(O2′)	0·696 (10)	0·973 (2)	0·776 (2)	0·053
H(O3′)	0·755 (10)	0·746 (3)	0·686 (2)	0·070
H(O5′)	0·193 (10)	0·565 (3)	0·823 (2)	0·069

* For non-hydrogen atoms, U_{eq} is calculated as

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* A_{ij},$$

where A_{ij} is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

Table 3. *Bond lengths* (Å) *and bond angles* (°)

1	2	3	1–2	1–2–3
C(2)	N(1)	C(5)	1·362 (4)	109·5 (3)
C(5)	N(1)	C(1′)	1·382 (5)	125·7 (3)
C(1′)	N(1)	C(2)	1·451 (4)	124·8 (3)
C(3)	C(2)	C(6)	1·386 (5)	129·5 (3)
C(3)	C(2)	N(1)		107·1 (3)
C(6)	C(2)	N(1)	1·502 (5)	123·3 (3)
C(4)	C(3)	C(9)	1·425 (5)	123·5 (3)
C(4)	C(3)	C(2)		108·0 (3)
C(9)	C(3)	C(2)	1·473 (5)	128·5 (4)
C(5)	C(4)	C(3)	1·360 (5)	106·6 (3)
N(1)	C(5)	C(4)		108·7 (3)
C(7)	C(6)	C(2)	1·437 (5)	114·2 (3)
N(8)	C(7)	C(6)	1·140 (5)	177·2 (6)
O(10)	C(9)	O(11)	1·186 (5)	122·8 (4)
O(10)	C(9)	C(3)		123·4 (4)
O(11)	C(9)	C(3)	1·319 (6)	113·7 (3)
C(12)	O(11)	C(9)	1·436 (5)	117·3 (4)
C(13)	C(12)	O(11)	1·349 (8)	110·6 (4)
C(2′)	C(1′)	O(1′)	1·536 (5)	105·6 (2)
C(2′)	C(1′)	N(1)		114·1 (3)
O(1′)	C(1′)	N(1)	1·434 (4)	108·4 (2)
C(3′)	C(2′)	O(2′)	1·517 (5)	114·7 (3)
C(3′)	C(2′)	C(1′)		103·8 (3)
O(2′)	C(2′)	C(1′)	1·401 (4)	111·7 (2)
C(4′)	C(3′)	O(3′)	1·520 (6)	114·4 (3)
C(4′)	C(3′)	C(2′)		102·4 (3)
O(3′)	C(3′)	C(2′)	1·410 (4)	110·1 (3)
C(5′)	C(4′)	O(1′)	1·508 (5)	108·4 (3)
C(5′)	C(4′)	C(3′)		114·7 (4)
O(1′)	C(4′)	C(3′)	1·447 (4)	103·8 (3)
O(5′)	C(5′)	C(4′)	1·417 (5)	112·5 (4)
C(1′)	O(1′)	C(4′)		110·9 (2)

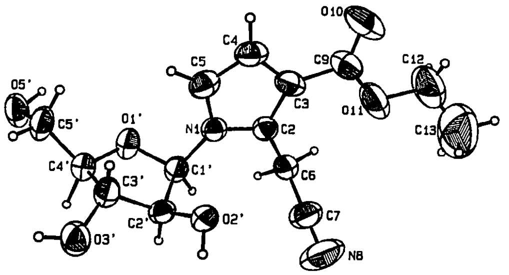

Fig. 1. Perspective drawing of (1) indicating atom labeling. Thermal ellipsoids are drawn at the 50% probability level. The large thermal ellipsoids and short C(12)–C(13) bond suggest disorder.

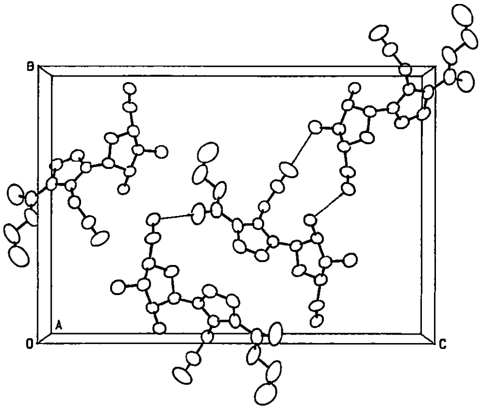

Fig. 2. Perspective drawing of the molecular packing as viewed down the *c* axis. The hydrogen bonding is indicated by thin lines.

Table 4. *Hydrogen bonding for* (1)

D–H···A	Symmetry of A relative to D	$d(D\cdots A)$ (Å)	$d(\mathrm{H}\cdots A)$ (Å)	$\angle(D\text{–H}\cdots A)$ (°)
O(2′)–H(O2′)···O(5′)	$1-x, 0{\cdot}5+y, 1{\cdot}5-z$	2·699 (3)	1·83 (4)	168 (4)
O(3′)–H(O3′)···N(8)	$1-x, y-0{\cdot}5, 1{\cdot}5-z$	2·854 (4)	1·98 (4)	171 (4)
O(5′)–H(O5′)···O(10)	$x-0{\cdot}5, 1{\cdot}5-y, 2-z$	2·685 (4)	1·84 (4)	158 (4)

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* Tables of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43773 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. A fused pyrrolo-arabinofuranosyl-nucleoside has recently been reported (Girgis, Cottam, Larson & Robins, 1987*a*). Nucleoside and nucleotide structural parameters have been discussed by Altona & Sundaralingam (1972).

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9-Acetyloxymethyl-1,3,4-trifluoro-7-methyl-11*H*-pyrido[4,3-*c*]benzo[1,2]diazepine

By R. G. Pritchard

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

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Abstract. $C_{16}H_{12}F_3N_3O_2$, $M_r = 335{\cdot}3$, monoclinic, $P2_1/c$, $a = 13{\cdot}591\,(4)$, $b = 4{\cdot}823\,(3)$, $c = 24{\cdot}930\,(4)$ Å, $\beta = 115{\cdot}60\,(2)°$, $V = 1473{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}51$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}084$ mm^{-1}, $F(000) = 688$, $T = 293$ K, $R = 0{\cdot}042$ for 1313 unique reflexions $[F > 3\sigma(F)]$. The ring skeleton is composed of a boat-shaped diazepine nucleus flanked by benzene and pyridine rings, whose planes intersect at an angle of 113·6 (6)°.

Experimental. The title compound was obtained in admixture with 1,3,4-trifluoro-7,9-dimethyl-11*H*-pyrido[4,3-*c*]benzo[1,2]diazepine by heating a solution of 2,3,5,6-tetrafluoro-4-(2,4,6-trimethylphenylazo)-pyridine in glacial acetic acid containing fused potassium acetate at 392 K for 4 h. The mixture was separated chromatographically followed by recrystallization from aqueous ethanol, m.p. 415 K. Crystal dimensions 0·2 × 0·2 × 0·1 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centred reflexions ($4{\cdot}4 < \theta < 9{\cdot}1°$), ω–2θ scan mode used to measure 1905 reflexions with $I > 0$, ω-scan width of $0{\cdot}40° + 0{\cdot}35°\tan\theta$ and scan speed ranging from 0·6 to 5° min^{-1} according to the intensity gathered in a pre-scan, $-14 \le h \le 14$, $0 \le k \le 5$, $0 \le l \le 28$, $0 \le \theta \le 25°$, 1313 unique structure amplitudes with $F \ge 3\sigma(F)$, negligible drift in three intensity standards ($\bar{2}06$, $\bar{3}12$, 114) measured every 2 h, Lorentz and polarization corrections but absorption ignored, *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) used to solve the phase problem by direct methods, all non-H atoms found in a Fourier map, H atoms from difference Fourier map, full-matrix least-squares refinement based on F using *SHELX*76 (Sheldrick, 1976), final $R = 0{\cdot}042$, $wR = 0{\cdot}044$, $w = 1{\cdot}5022/[\sigma^2(F_o) + 0{\cdot}000272F_o^2]$, anisotropic thermal parameters for heavier atoms, isotropic for H. Maximum fluctuation in final difference map in range −0·15 to +0·18 e Å^{-3}, maximum LS shift-to-e.s.d. ratio 0·058 [y, H(20)].

Scattering factors from *International Tables for X-ray Crystallography* (1974), computations carried out on the joint CDC 7600/Amdahl 470 system of the University of Manchester Regional Computing Centre.

Fractional atomic coordinates and vibrational parameters are listed in Table 1,* selected bond lengths in Table 2. The molecule, including labelling scheme, is displayed in Fig. 1.

Related literature. Details of relevant structures can be found in Alty, Banks, Fishwick, Pritchard & Thompson (1984) and Pritchard (1987).

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* Lists of structure factors, anisotropic vibrational parameters, H-atom parameters, all bond lengths and angles, torsion angles and normalized least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43690 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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