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Structure of 3-(3-Azido-2,3-dideoxy-β-D-*erythro*-pentofuranosyl)cytosine Hydrogen Chloride

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Abstract. $C_9H_{13}N_6O_3^+.Cl^-$, $M_r = 288{\cdot}7$, monoclinic, $P2_1$, $a = 6{\cdot}308$ (3), $b = 9{\cdot}336$ (5), $c = 10{\cdot}945$ (6) Å, $\beta = 97{\cdot}40$ (4)°, $V = 639$ Å^3, $Z = 2$, $D_x = 1{\cdot}500$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 3{\cdot}1$ cm^{-1}, $F(000) = 300$, $T = 293$ K. $R = 0{\cdot}068$ for 931 unique observed $[F > 4\sigma(F)]$ reflections. The N-glycosidic torsion angle χ has a value of -130 (1)°, in the *anti* range. The sugar pucker is ${}_1T^2$ with $P = 135$ (1)° and $\psi = 38$ (1)°. The C4′–C5′ conformation is *sc* with $\gamma = 48$ (1)°. There are two hydrogen bonds in the structure: O5′···Cl1($-1-x$, $0{\cdot}5+y$, $-1-z$), 3·03 (2) Å and N3···O5′(x, $-0{\cdot}5-y$, z), 2·79 (2) Å. In each case the first atom is the donor.

Introduction. This structure was determined as part of an ongoing investigation of potentially anti-viral nucleoside analogues, with particular reference to possible anti-AIDS compounds.

Experimental. The compound was kindly supplied by Dr J. Rideout of Burroughs Wellcome Co., Research Triangle Park, NC 27709, USA. Crystals were obtained from aqueous solution. Space group and initial cell dimensions were obtained from Weissenberg photographs. Data were collected on a Nicolet *P*3 (four-circle) diffractometer in Aberdeen by RAH.

The crystal had dimensions $0{\cdot}4 \times 0{\cdot}2 \times 0{\cdot}2$ mm. Cell parameters were measured on the diffractometer using the $22\bar{2}$, 004, $20\bar{3}$ and 220 and symmetry-related reflections which have 2θ in the range 15 to 17°. Range of indices: $0 \le h \le 9$; $0 \le k \le 13$; $-15 \le l \le 15$. Data measured using $\omega/2\theta$ scans in the range

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$0 < 2\theta < 55°$. Standard reflections were measured every 50 reflections on each layer. No changes greater than 2σ from the means in the intensities of these reflections were found throughout data collection. Lorentz and polarization factors were applied. No corrections were made for absorption or secondary extinction. 1583 independent reflections measured, 931 observed $[F > 4\sigma(F)]$ reflections used in the refinement. The structure was solved using the program *SHELXS*86 (Sheldrick, 1986), the position of the Cl atom being obtained from a Harker section. This atom was then put into *PATSEE* (Egert, 1985) as an atom of known position along with model coordinates for a cytidine base. The set of coordinates with the lowest R_e value obtained from *PATSEE* were then put into the program *SHELXS*86 and a tangent expansion carried out. All atoms except those in the azido group were located. These latter atoms were located after Fourier refinement using the program *SHELX*76 (Sheldrick, 1976). A difference synthesis revealed the positions of the H atoms attached to atoms N3, N4 and O5′. All other H atoms were included at calculated positions, C—H = 1·08 Å. The H atoms were given fixed isotropic temperature factors approximately 1·5 times that of the parent atom and allowed to ride on their parent atoms except for those located in the difference synthesis which were held in fixed positions. All other atoms were refined anisotropically. Blocked full-matrix refinement (on F) was carried out using the program *SHELX*76. The refinement converged at $R = 0{\cdot}068$, $wR = 0{\cdot}059$, $w = 1{\cdot}7555[\sigma^2(F) + 0{\cdot}000167F^2]^{-1}$. 171 refined parameters; average shift/e.s.d. = 0·001; max. shift/e.s.d. < 0·003; max. difference peak 0·34; min. difference peak −0·36 e Å^{-3}. The chirality of the molecule was not determined.

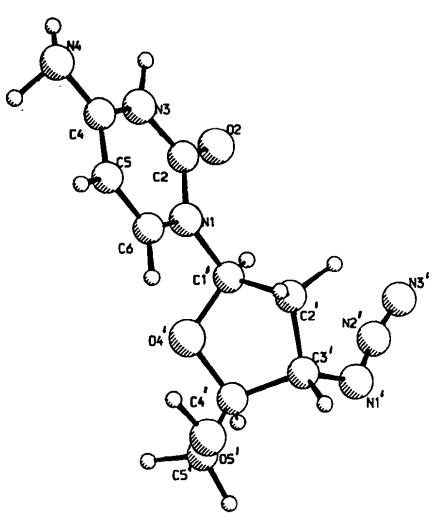

Fig. 1. Perspective view of the molecule.

Table 1. *Coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\times 10^3$) *for non*-H *atoms with e.s.d.'s in parentheses*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}(Å^2)
Cl1	−2377 (3)	−20†	−4609 (2)	50 (1)
N1	−4974 (12)	1138 (8)	−7538 (7)	40 (2)
C2	−4612 (15)	−328 (8)	−7613 (8)	36 (3)
O2	−3189 (11)	−835 (7)	−8126 (7)	56 (2)
N3	−5972 (12)	−1172 (8)	−7048 (7)	34 (2)
C4	−7583 (15)	−702 (11)	−6443 (9)	39 (3)
N4	−8747 (11)	−1601 (8)	−5892 (7)	47 (2)
C5	−8008 (16)	798 (11)	−6455 (8)	45 (3)
C6	−6638 (15)	1652 (10)	−6968 (9)	44 (3)
C1′	−3487 (15)	2119 (10)	−8069 (9)	46 (3)
C2′	−4440 (16)	2947 (12)	−9170 (9)	61 (4)
C3′	−3011 (19)	4257 (11)	−9128 (10)	70 (4)
N1′	−1339 (28)	4154 (18)	−9925 (14)	176 (8)
N2′	−703 (18)	3128 (15)	−10290 (11)	85 (5)
N3′	−168 (27)	2205 (18)	−10790 (21)	206 (11)
C4′	−2171 (15)	4398 (9)	−7775 (8)	43 (3)
C5′	−2890 (15)	5724 (10)	−7159 (9)	51 (3)
O5′	−5127 (10)	5939 (7)	−7438 (6)	54 (2)
O4′	−2856 (10)	3162 (7)	−7156 (5)	52 (2)

† Held constant to define origin.

Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The program packages *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978) were also used. All calculations were carried out on the Dundee University PRIME computer.

Discussion. The atomic numbering is shown in the perspective drawing (Fig. 1). Tables 1 and 2 give the atomic parameters, bond lengths and angles.* The nomenclature in the following discussion is that of the IUPAC–IUB Joint Commission on Biochemical Nomenclature (1983). The bonds and angles agree well with those of similar nucleosides. In particular it is interesting to note that the C2—N3—C4 angle of 126 (1)° is greater than 122·4° which, according to Taylor & Kennard (1982), suggests that the base is protonated at N(3), as is indeed the case. The *erythro*-thymidine analogue, the anti-AIDS drug AZT, has two molecules in its asymmetric unit with χ of −126 and −172° and sugar ring puckers P = 171 and 213° respectively (Dyer, Low, Tollin, Wilson & Howie, 1988). The *erythro*-uracil analogue (Low & Tollin, 1989) has $\chi = -160°$ and pucker $P = 174°$. Thus the present compound with $\chi = -130$ (1)° and $P = 135$ (1)° has a markedly different conformation from these azido nucleosides. It also has a different conformation from the anti-AIDS drug 2′,3′-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52221 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic distances* (Å) *and angles* (°)

C2—N1	1·392 (10)	C6—N1	1·374 (10)
C1′—N1	1·482 (11)	O2—C2	1·214 (10)
N3—C2	1·369 (10)	C4—N3	1·355 (10)
N4—C4	1·312 (11)	C5—C4	1·425 (12)
C6—C5	1·350 (12)	C2′—C1′	1·492 (13)
O4′—C1′	1·415 (11)	C3′—C2′	1·517 (14)
N1′—C3′	1·456 (14)	C4′—C3′	1·513 (14)
N2′—N1′	1·131 (16)	N3′—N2′	1·097 (17)
C5′—C4′	1·507 (11)	O4′—C4′	1·433 (9)
O5′—C5′	1·419 (10)		
C6—N1—C2	120·7 (8)	C1′—N1—C2	117·8 (8)
C1′—N1—C6	121·4 (7)	O2—C2—N1	123·2 (8)
N3—C2—N1	114·9 (8)	N3—C2—O2	121·9 (8)
C4—N3—C2	126·0 (8)	N4—C4—N3	121·1 (8)
C5—C4—N3	117·7 (9)	C5—C4—N4	121·1 (9)
C6—C5—C4	117·0 (10)	C5—C6—N1	123·3 (9)
C2′—C1′—N1	115·3 (8)	O4′—C1′—N1	106·4 (7)
O4′—C1′—C2′	105·1 (7)	C3′—C2′—C1′	102·6 (8)
N1′—C3′—C2′	113·7 (9)	C4′—C3′—C2′	103·4 (8)
C4′—C3′—N1′	113·7 (12)	N2′—N1′—C3′	125·8 (14)
N3′—N2′—N1′	170·8 (21)	C5′—C4′—C3′	114·9 (8)
O4′—C4′—C3′	107·7 (7)	O4′—C4′—C5′	108·8 (6)
O5′—C5′—C4′	111·6 (8)	C4′—O4′—C1′	107·1 (7)

dideoxycytidine (J. N. Low, unpublished work; Silverton, Quinn, Haugwitz & Todaro, 1988), in which $\chi = -156°$ and $P = 205°$. The χ and P values place the molecule at the lower P edge of the C2′-*endo* minimum, *i.e.* close to the potential barrier between the C2′-*endo* and C3′-*endo* conformations for 2′-deoxynucleoside molecules discussed by Low, Tollin & Wilson (1982), and to this extent the conformation is unusual. The azido group, as with the other azido-substituted nucleosides referred to above, is not involved in hydrogen bonding; it does have two short contacts less than 3·5 Å, N1′···N3′($-x$, $0·5+y$, $-2-z$), 3·07 (2) Å and O2···N3′($-x$, $-0·5+y$, $-2-z$), 3·14 (2) Å. The atoms of the azido group have high thermal parameters, a common feature discussed by Low, Tollin, Howie & Wilson (1988).

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A Novel β-Adrenergic Receptor Antagonist MY336-a: Structure of the Tetraacetylated Compound

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Abstract. (1*R**,3*S**)-1,2,3,4-Tetrahydro-8-acetoxy-1,3-bis(acetoxymethyl)-2-acetyl-7-methoxy-6-methylisoquinoline, $C_{21}H_{27}NO_8$, $M_r = 421·45$, monoclinic, $P2_1$, $a = 11·892$ (2), $b = 11·067$ (2), $c = 8·4866$ (8) Å, $\beta = 92·12$ (1)°, $V = 1116·1$ (5) Å^3, $Z = 2$, $D_x = 1·255$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·54184$ Å, $\mu = 7·702$ cm^{-1}, $F(000) = 448$, $T = 293$ K, $R = 0·048$, $wR = 0·072$ for 2178 observed reflections. The tetrahydropyridine ring adopts a half-boat conformation. The bond lengths and angles are within the normal ranges. There are no intermolecular contacts less than the sum of the van der Waals radii.

Introduction. MY336-a (I) was isolated from the culture broth of *Streptomyces gabonae* KY2234 and characterized as a new specific β-adrenergic antagonist based on the profile of the receptor binding assay and pharmacological properties (Kase, Fujita, Nakamura, Hashizume, Goto, Kubo & Shuto, 1986). (I) is the first microbial metabolite known to act on

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