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Structure of 1-(β-D-Arabinofuranosyl)-6-methylcytosine

BY KENTARO YAMAGUCHI, GO MATSUMURA, MIHO SHIMIZU, HIROMICHI TANAKA AND TADASHI MIYASAKA

School of Pharmaceutical Sciences, Showa University 1–5–8, *Hatanodai, Shinagawa-ku, Tokyo* 142, *Japan*

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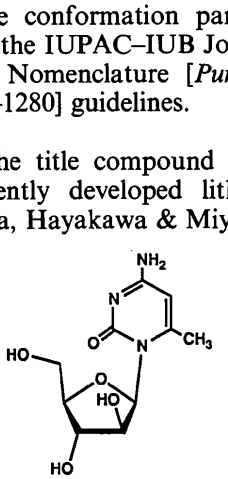
Abstract. $C_{10}H_{15}N_3O_5$, $M_r = 257.25$, orthorhombic, $P2_12_12_1$, $a = 10.235$ (2), $b = 10.833$ (1), $c = 10.129$ (3) Å, $V = 1123.0$ (3) Å^3, $Z = 4$, $D_x = 1.521$ Mg m^{-3}, λ(Cu $K\alpha_1$) = 1.54050 Å, $\mu = 1.002$ mm^{-1}, $F(000) = 544$, $T = 295$ K, final $R = 0.035$ for 950 reflections. The sugar conformation and puckering parameters are ^3E with $P = 18.5°$ and $\psi_m = 21.4°$. The *N*-glycosidic torsion angle χ has a value of -72.7 (3)° in the *syn* range. The C4′—C5′ side-chain conformation is $-ap$ with $\gamma = -174.5$ (4)°. The conformation parameters are in accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280] guidelines.

Experimental. The title compound (I) was synthesized by a recently developed lithiation method (Shimizu, Tanaka, Hayakawa & Miyasaka, 1990).



(I)

Crystal dimensions 0.30 × 0.10 × 0.40 mm, by recrystallization from CH_3OH, having m.p. 492–493 K. Rigaku AFC-5 four-circle diffractometer used with ω–2θ-scan method, ω-scan width $(1.3 + 0.14\tan\theta)°$ and scan speed 16° min^{-1}. Lattice parameters obtained from least-squares analysis of 20 reflections with 2θ values ranging from 57 to 60°. Of 1026 reflections scanned (within index range h 0–11, k 0–12, l 0–11 up to $\sin\theta/\lambda < 0.56$ Å^{-1}), 986 unique reflections [$F > \sigma(F)$] classified as observed. Three standard reflections measured every 150 reflections, intensity variation <3%. Intensities corrected for Lorentz and polarization factors, but absorption correction not applied. Structure solved using program package *SAPI*85 (Yao, Zheng, Qian, Han, Gu & Fan, 1985). The refinement was carried out by the full-matrix least-squares method with anisotropic temperature factors for non-H atoms. The function minimized was $\sum w[|(|F_o|)^2 - (|F_c|)^2|]^2$ with $w = 1/[\sigma^2(F_o) + 0.02(F_c)]$, $\sigma(F_o)$ was determined from counting statistics. All H atoms located from the difference map and theoretical calculations were refined, initial thermal parameters set at equivalent isotropic thermal parameter of each bonded atom. Final discrepancy indices $R = 0.035$, $wR = 0.044$, $S = 1.535$ for 298 variables and 950 reflections with $F > 2.5\sigma(F)$. Maximum $\Delta/\sigma = 0.37$ in final least-squares cycle. Final difference Fourier excursions 0.14 and -0.21 e Å^{-3}. All major computations performed on PANAFACOM computer with *RCRYSTAN* (Rigaku Corporation, 1985) X-ray analysis program system. The atomic scattering factors were

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = \frac{1}{3}\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$.

	x	y	z	B_{eq}
N1	0.5731 (2)	1.0912 (2)	0.3109 (2)	2.22 (6)
C2	0.4637 (3)	1.0449 (3)	0.3791 (3)	2.42 (8)
N3	0.4594 (2)	1.0433 (2)	0.5113 (2)	2.76 (7)
C4	0.5595 (3)	1.0911 (3)	0.5785 (3)	2.59 (8)
C5	0.6671 (3)	1.1468 (3)	0.5142 (3)	2.61 (8)
C6	0.6721 (3)	1.1476 (3)	0.3817 (3)	2.29 (7)
O2	0.3716 (2)	1.0032 (2)	0.3116 (2)	3.15 (6)
N4	0.5547 (3)	1.0840 (3)	0.7093 (3)	3.60 (9)
C16	0.7839 (3)	1.2083 (4)	0.3117 (4)	3.31 (10)
O4′	0.4875 (2)	1.2003 (1)	0.1288 (2)	2.97 (5)
C1′	0.5696 (3)	1.1018 (3)	0.1663 (3)	2.27 (7)
C2′	0.5262 (3)	0.9886 (2)	0.0851 (3)	2.21 (7)
C3′	0.3908 (3)	1.0201 (3)	0.0327 (3)	2.27 (8)
C4′	0.3846 (3)	1.1608 (3)	0.0411 (3)	2.56 (8)
O2′	0.5399 (2)	0.8739 (2)	0.1503 (2)	3.00 (6)
O3′	0.3772 (2)	0.9761 (2)	−0.0996 (2)	2.99 (6)
C5′	0.2572 (4)	1.2083 (3)	0.0948 (4)	3.51 (10)
O5′	0.2615 (2)	1.3393 (2)	0.1122 (3)	4.26 (8)

Table 2. *Bond lengths* (Å) *and angles* (°)

N1—C6	1.382 (4)	N1—C2	1.409 (4)
N1—C1′	1.470 (4)	C2—O2	1.247 (4)
C2—N3	1.340 (4)	N3—C4	1.333 (4)
C4—N4	1.328 (4)	C4—C5	1.417 (5)
C5—C6	1.343 (5)	C6—C16	1.500 (5)
O4′—C1′	1.409 (4)	O4′—C4′	1.440 (4)
C1′—C2′	1.541 (4)	C2′—O2′	1.415 (4)
C2′—C3′	1.521 (4)	C3′—O3′	1.428 (3)
C3′—C4′	1.528 (5)	C4′—O4′	1.440 (4)
C4′—C5′	1.505 (5)	C5′—O5′	1.430 (4)

C6—N1—C2	119.1 (2)	C6—N1—C1′	119.9 (2)
C2—N1—C1′	119.9 (2)	O2—C2—N3	121.0 (3)
O2—C2—N1	117.5 (2)	N3—C2—N1	121.5 (3)
C4—N3—C2	118.5 (3)	N4—C4—N3	117.1 (3)
N4—C4—C5	121.0 (3)	N3—C4—C5	121.9 (3)
C6—C5—C4	119.7 (3)	C5—C6—N1	119.0 (3)
C5—C6—C16	120.5 (3)	N1—C6—C16	120.5 (3)
C1′—O4′—C4′	112.1 (2)	O4′—C1′—N1	109.9 (2)
O4′—C1′—C2′	106.7 (2)	N1—C1′—C2′	118.4 (2)
O2′—C2′—C3′	116.7 (2)	O2′—C2′—C1′	114.9 (2)
C3′—C2′—C1′	105.6 (2)	O3′—C3′—C2′	109.8 (2)
O3′—C3′—C4′	112.5 (2)	C2′—C3′—C4′	104.0 (2)
O4′—C4′—C5′	108.0 (2)	O4′—C4′—C3′	107.5 (2)
C5′—C4′—C3′	113.4 (2)	O5′—C5′—C4′	110.9 (3)

taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atomic parameters are listed in Table 1.* Bond lengths and angles are in Table 2. Fig. 1 shows an *ORTEP*II drawing (Johnson, 1976) of the molecule with the atom labels.

Related literature. Glycosidic conformations of cytidine and 6-methylcytidine have been studied comparatively by NMR spectroscopy (Schweizer, Banta, Witkowski & Robins, 1973). As confirmed in the case of 6-methyluridine (Suck & Saenger, 1972), 6-methylcytidine was found to exist preferentially in the *syn* conformation due to the presence of the C-6 substituent. The *anti* conformation of arabinofuranosylcytosine, an anti-leukemic nucleoside, was determined by X-ray crystallography (Chuwang & Sundaralingam, 1973). Several conformational characteristics of the arabinofuranosyl moiety have been pointed out (Dalton, George, Hruska, McGaig, Ogilvie, Peeling & Wood, 1977). The structure of a similar compound, 1-(β-D-arabinosyl)-5-methylcytosine, a potent anti-viral nucleoside, has also been reported (Birnbaum & Gentry, 1983).

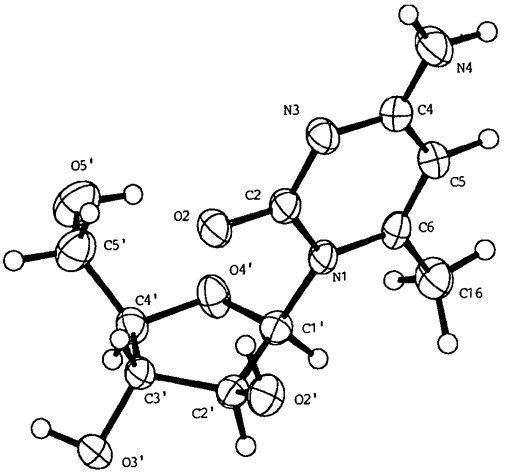

Fig. 1. *ORTEP* drawing of (I). Ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

* Tables of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54474 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0513]

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