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Structure of N^6-Methyl-2′-deoxyadenosine

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Abstract. $C_{10}H_{15}N_5O_3$, $M_r = 265{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}246\,(1)$, $b = 22{\cdot}686\,(2)$, $c = 5{\cdot}233\,(1)$ Å, $V = 1216{\cdot}4\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}448$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}12$ mm^{-1}, $F(000) = 560$, room temperature, final $R = 0{\cdot}037$ for 1490 observed reflections. The molecule has a C(1′)-*exo*—C(2′)-*endo* $({}^{2}_{1}T)$ sugar associated with an *anti* base: O(4′)—C(1′)—N(9)—C(8) = 67·2 (3)°. The conformation about the C(4′)—C(5′) bond is *gauche–trans*; C(3′)—C(4′)—C(5′)—O(5′) = −170·7 (2)°. The orientation of the methyl substituent at N(6) is *anti* with respect to the purine ring. C(1′) is appreciably displaced from the imidazole ring [0·254 (2) Å]. There is an intermolecular short contact between C(8)—H and N(3).

Introduction. As part of a series of crystallographic investigations of nucleic-acid constituents the X-ray analysis of the title compound was carried out.

Experimental. Crystals grown from methanol/hexane solution by vapor diffusion. D_m not measured. Colorless plate, 0·08 × 0·35 × 0·35 mm. Rigaku AFC-5 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters refined by least-squares method (25 reflections, $30 < 2\theta < 37°$). ω scan ($2\theta \le 30°$), ω–2θ scan ($2\theta > 30°$), $2\theta_{\max} = 55°$; h 0 to 13, k 0 to 29, l 0 to 6. Three standard reflections: no variation. 1649 unique reflections, 1490 with $I \ge 0{\cdot}75\sigma(I)$ considered observed. No absorption correction. Structure solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), H atoms located from difference map. Block-diagonal least-squares refinement. Anisotropic temperature factors for non-H atoms, isotropic for H atoms. No correction for secondary extinction. $\sum w\Delta^2$ minimized, $\Delta = |F_o| - |F_c|$, $w = 1/\sigma^2(F_o)$ for $|F_c| \ge 3\sigma(F_o)$, $w = 0$ for $|F_c| < 3\sigma(F_o)$ or $|\Delta| \ge 3\sigma(F_o)$, $\sigma(F_o) = [\sigma_1{}^2(F_o) + 0{\cdot}00040|F_o|^2]^{1/2}$, $\sigma_1(F_o)$ = e.s.d. based on counting errors (Grant, Killean & Lawrence, 1969). $R = 0{\cdot}037$, $wR = 0{\cdot}042$, $S = 1{\cdot}07$. $(\Delta/\sigma)_{\max} = 0{\cdot}1$, $(\Delta\rho)_{\max} = 0{\cdot}4$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations performed on a FACOM-M34OR computer.

Discussion. Final atomic parameters are in Table 1.* Bond distances and angles are listed in Table 2. A perspective view of the molecule is shown in Fig. 1.

The deoxyribose ring is puckered in a C(1′)-*exo*—C(2′)-*endo* $({}^{2}_{1}T)$ twist form. The pseudorotation coordinates of the five-membered ring (Sato, 1983) are $\Pi = 34{\cdot}5\,(2)°$ and $\Phi = 237{\cdot}9\,(4)°$. The conformation about the glycosidic bond is *anti*. This structure involves a short sugar–base contact between C(2′) and C(8) [3·090 (3) Å]. The conformation about the C(4′)—C(5′) bond is *gauche–trans:* C(5′)—O(5′) is *gauche* to C(4′)—O(4′) and *trans* to C(4′)—C(3′). As in other N^6-alkylated adenines (*e.g.* Takeda, Ohashi, Sasada & Kakudo, 1976), the orientation of the methyl substituent at N(6) is *anti* with respect to the purine ring, torsion angle C(5)—C(6)—N(6)—C(10) being −169·7 (2)°.

Compared with the bond distances and angles of the unsubstituted adenine base (Taylor & Kennard, 1982), those of the present structure are quite normal except for the exocyclic bond angles involving N(6): the angle N(1)—C(6)—N(6) is 1·8° larger than the standard value probably owing to a steric hindrance of the methyl group. The purine ring is slightly buckled: the N(1), C(2), N(3) and C(6) atoms are displaced by 0·025 (2), −0·025 (2), −0·024 (2) and 0·075 (2) Å, respectively, from the mean plane of the imidazole ring,

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* Lists of structure factors, anisotropic temperature factors and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42344 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates and isotropic thermal parameters*

$$B_{\rm eq}=\tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
N(1)	0·0816 (2)	0·1399 (1)	−0·1020 (4)	2·59 (4)
C(2)	0·0020 (2)	0·1865 (1)	−0·0637 (4)	2·60 (5)
N(3)	0·0151 (2)	0·2311 (1)	0·0975 (4)	2·38 (4)
C(4)	0·1236 (2)	0·2254 (1)	0·2408 (4)	2·12 (4)
C(5)	0·2115 (2)	0·1792 (1)	0·2299 (4)	2·38 (4)
C(6)	0·1915 (2)	0·1359 (1)	0·0415 (4)	2·41 (4)
N(7)	0·3104 (2)	0·1865 (1)	0·4070 (4)	3·00 (4)
C(8)	0·2819 (2)	0·2361 (1)	0·5188 (5)	2·82 (5)
N(9)	0·1695 (2)	0·2626 (1)	0·4292 (4)	2·33 (4)
N(6)	0·2771 (2)	0·0924 (1)	0·0036 (4)	3·09 (4)
C(10)	0·2720 (3)	0·0524 (1)	−0·2133 (6)	3·80 (6)
O(4′)	0·2187 (2)	0·3614 (1)	0·3602 (3)	2·95 (4)
C(1′)	0·1272 (2)	0·3227 (1)	0·4826 (4)	2·18 (4)
C(2′)	0·1313 (2)	0·3399 (1)	0·7622 (4)	2·52 (4)
C(3′)	0·1513 (2)	0·4063 (1)	0·7532 (4)	2·43 (4)
C(4′)	0·2265 (2)	0·4153 (1)	0·5015 (4)	2·38 (4)
O(3′)	0·0273 (2)	0·4345 (1)	0·7470 (4)	3·44 (4)
C(5′)	0·3682 (3)	0·4311 (1)	0·5381 (6)	3·82 (6)
O(5′)	0·4267 (2)	0·4480 (1)	0·3019 (5)	4·84 (6)

Table 2. *Molecular dimensions*

Bond distances (Å)			
N(1)—C(2)	1·350 (3)	N(9)—C(1′)	1·458 (3)
N(1)—C(6)	1·356 (3)	N(6)—C(10)	1·454 (4)
C(2)—N(3)	1·324 (3)	O(4′)—C(1′)	1·435 (3)
N(3)—C(4)	1·347 (3)	O(4′)—C(4′)	1·431 (3)
C(4)—C(5)	1·383 (3)	C(1′)—C(2′)	1·515 (3)
C(4)—N(9)	1·380 (3)	C(2′)—C(3′)	1·521 (3)
C(5)—C(6)	1·407 (3)	C(3′)—C(4′)	1·540 (3)
C(5)—N(7)	1·383 (3)	C(3′)—O(3′)	1·423 (3)
C(6)—N(6)	1·335 (3)	C(4′)—C(5′)	1·508 (4)
N(7)—C(8)	1·301 (3)	C(5′)—O(5′)	1·426 (4)
C(8)—N(9)	1·381 (3)		
Bond angles (°)			
C(2)—N(1)—C(6)	118·1 (2)	C(4)—N(9)—C(1′)	127·4 (2)
N(1)—C(2)—N(3)	129·2 (2)	C(8)—N(9)—C(1′)	126·2 (2)
C(2)—N(3)—C(4)	111·4 (2)	C(6)—N(6)—C(10)	123·6 (2)
N(3)—C(4)—C(5)	126·0 (2)	C(1′)—O(4′)—C(4′)	109·2 (2)
N(3)—C(4)—N(9)	128·3 (2)	N(9)—C(1′)—O(4′)	107·0 (2)
C(5)—C(4)—N(9)	105·7 (2)	N(9)—C(1′)—C(2′)	114·7 (2)
C(4)—C(5)—C(6)	117·6 (2)	O(4′)—C(1′)—C(2′)	104·8 (2)
C(4)—C(5)—N(7)	111·0 (2)	C(1′)—C(2′)—C(3′)	103·2 (2)
C(6)—C(5)—N(7)	131·3 (2)	C(2′)—C(3′)—C(4′)	103·0 (2)
N(1)—C(6)—C(5)	117·5 (2)	C(2′)—C(3′)—O(3′)	109·0 (2)
N(1)—C(6)—N(6)	120·8 (2)	C(4′)—C(3′)—O(3′)	111·6 (2)
C(5)—C(6)—N(6)	121·6 (2)	O(4′)—C(4′)—C(3′)	107·5 (2)
C(5)—N(7)—C(8)	103·9 (2)	O(4′)—C(4′)—C(5′)	108·8 (2)
N(7)—C(8)—N(9)	114·3 (2)	C(3′)—C(4′)—C(5′)	113·9 (2)
C(4)—N(9)—C(8)	105·1 (2)	C(4′)—C(5′)—O(5′)	111·0 (2)

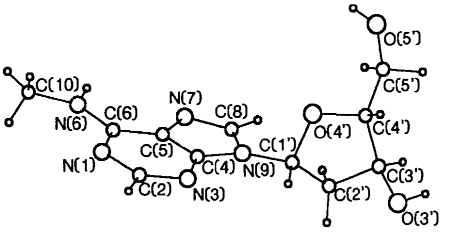

Fig. 1. A perspective view of the molecule, showing the atom numbering.

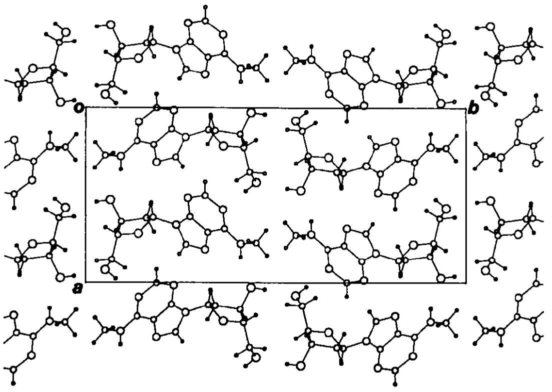

Fig. 2. A projection of the structure viewed along the c axis.

which is planar within 0·002 Å. The displacement of C(1′) from the imidazole ring is also appreciable. The length of the glycosidic bond is 0·016 Å shorter than that of 2′-deoxyadenosine (Sato, 1984). This shortening seems to be correlated with an elongation (0·023 Å) of the O(4′)—C(1′) bond.

The packing scheme in the crystal is shown in Fig. 2. In the present structure, no appreciable base stacking is observed. All available hydrogens participate in hydrogen bonding, *i.e.* N(6)—H···O(3′) [2·941 (3) Å], O(3′)—H···O(5′) [2·722 (3) Å] and O(5′)—H···N(1) [2·755 (3) Å]. There is an intermolecular contact between C(8)—H and N(3) [C···N 3·208 (3) Å; H···N 2·29 (3) Å]. The hydrogen attached to C(8) has been frequently observed to be in close contact with hydrogen-accepting atoms. A hydrogen-bonding-like interaction has been suggested for this (Seeman, Sussman, Berman & Kim, 1971).

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