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3′-Amino-3′-deoxyinosine

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Abstract. $C_{10}H_{13}N_5O_4$, monoclinic, $P2_1$, $a = 4{\cdot}676$ (2), $b = 16{\cdot}003$ (8), $c = 7{\cdot}591$ (3) Å, $\beta = 106{\cdot}81$ (3)°, $M_r = 267{\cdot}3$, $Z = 2$, $D_x = 1{\cdot}63$ Mg m^{-3}. $R = 0{\cdot}038$ for 979 reflections. The conformation at the glycosidic bond N(9)–C(1′) is *anti* with $\chi_{CN} = 18{\cdot}1$ (3)°; that of the C(5′)–O(5′) bond relative to the ribose ring is *gauche–gauche*. The amino protons of N(3′) do not participate in hydrogen bonding. Inosine base stacking of the translation mode with no base overlap is observed. The distance between base planes is 3·35 Å.

Introduction. We have recently reported the structures of three 3′-*N*-substituted 3′-deoxyadenosines, namely 3′-amino-3′-deoxyadenosine (2), 3′-cyclobutylamino-3′-deoxyadenosine (3) and the oxazolidine (4) of 3′-(*N*-benzyl-*N*-methylamino)-3′-deoxyadenosine (Sheldrick & Morr, 1980). For comparison we have now determined the structure of 3′-amino-3′-deoxyinosine (1) (Fig. 1).

Cell dimensions were determined by a least-squares fit to settings for 15 reflections $\pm(hkl)$ on a Syntex $P2_1$ diffractometer (Cu $K\alpha$ radiation, $\lambda = 1{\cdot}54178$ Å). Data collection was carried out in the θ–2θ mode ($2\theta \leq 140°$) with graphite-monochromated Cu $K\alpha$ radiation. No absorption correction was applied ($\mu = 0{\cdot}98$ mm^{-1}). 979 independent reflections with $F \geq 3\sigma(F)$ were retained in the analysis. The structure was solved by direct methods and refined by full-matrix least squares with anisotropic temperature factors for the non-hydrogen atoms. The H atom positional parameters were refined with individual isotropic temperature factors under bond-length constraints. Extinction was particularly marked and an empirical isotropic extinction factor x (*SHELX*, G. M. Sheldrick) was refined to $0{\cdot}144\ (10) \times 10^{-4}$. The corrected calculated structure factors are then given by $F_c' = F_c(1 - xF_c^2/\sin\theta)$. Terminal values of R_w and R were respectively 0·040 and 0·038. The weights were $w = k[\sigma^2(F_o) + 0{\cdot}0001F_o^2]^{-1}$. Complex neutral-atom scattering factors were employed (Cromer & Waber, 1965; Cromer & Liberman, 1970). Table 1 lists the final non-hydrogen atom coordinates, Table 2 the bond distances and angles.†

Table 1. *Positional parameters* ($\times 10^4$)

	x	y	z	U_{eq}*
N(1)	4807 (7)	2399 (2)	−1074 (4)	39
C(2)	2664 (9)	1796 (2)	−1370 (4)	42
N(3)	2223 (7)	1288 (2)	−160 (3)	40
C(4)	4206 (7)	1412 (2)	1521 (4)	32
C(5)	6496 (8)	1993 (2)	1977 (4)	33
C(6)	6906 (8)	2539 (2)	603 (4)	36
O(6)	8843 (6)	3081 (2)	770 (3)	47
N(7)	8062 (7)	1921 (2)	3825 (3)	36
C(8)	6691 (7)	1316 (2)	4420 (4)	35
N(9)	4357 (6)	977 (2)	3074 (3)	31
O(1′)	2452 (5)	248 (2)	5145 (3)	35
C(1′)	2294 (7)	314 (2)	3276 (3)	29
C(2′)	3131 (7)	−533 (2)	2668 (4)	29
O(2′)	441 (5)	−1008 (2)	2082 (3)	39
C(3′)	5203 (7)	−871 (2)	4461 (4)	28
N(3′)	5552 (7)	−1779 (2)	4426 (3)	36
C(4′)	3732 (7)	−549 (2)	5878 (4)	31
C(5′)	5754 (8)	−432 (2)	7792 (4)	37
O(5′)	8242 (6)	53 (2)	7722 (3)	55

* The equivalent isotropic temperature factors are given by $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}\,a_i^*\,a_j^*\,\mathbf{a}_i\cdot\mathbf{a}_j$ (Willis & Pryor, 1975).

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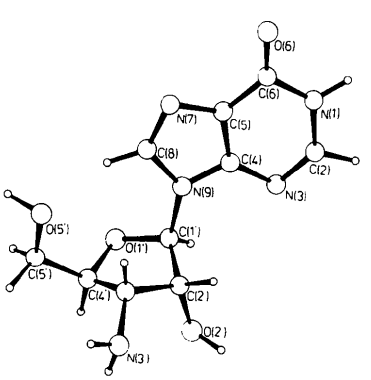

Fig. 1. The molecule of (1) in perspective.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35820 (9 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 2. *Bond lengths* (Å) *and angles* (°)

C(2)–N(1)	1·362 (5)	C(6)–N(1)	1·383 (4)
N(3)–C(2)	1·287 (5)	C(4)–N(3)	1·358 (3)
C(5)–C(4)	1·384 (5)	N(9)–C(4)	1·353 (4)
C(6)–C(5)	1·415 (5)	N(7)–C(5)	1·387 (4)
O(6)–C(6)	1·234 (5)	C(8)–N(7)	1·312 (5)
N(9)–C(8)	1·372 (4)	C(1′)–N(9)	1·471 (4)
C(1′)–O(1′)	1·403 (3)	C(4′)–O(1′)	1·451 (4)
C(2′)–C(1′)	1·521 (5)	O(2′)–C(2′)	1·426 (4)
C(3′)–C(2′)	1·523 (4)	N(3′)–C(3′)	1·462 (4)
C(4′)–C(3′)	1·525 (5)	C(5′)–C(4′)	1·499 (4)
O(5′)–C(5′)	1·413 (5)		

C(6)–N(1)–C(2)	124·0 (3)	N(3)–C(2)–N(1)	126·3 (3)
C(4)–N(3)–C(2)	111·9 (3)	C(5)–C(4)–N(3)	127·0 (3)
N(9)–C(4)–N(3)	126·4 (3)	N(9)–C(4)–C(5)	106·6 (2)
C(6)–C(5)–C(4)	119·4 (3)	N(7)–C(5)–C(4)	109·9 (3)
N(7)–C(5)–C(6)	130·7 (3)	C(5)–C(6)–N(1)	111·6 (3)
O(6)–C(6)–N(1)	120·8 (3)	O(6)–C(6)–C(5)	127·6 (3)
C(8)–N(7)–C(5)	104·0 (3)	N(9)–C(8)–N(7)	113·5 (3)
C(8)–N(9)–C(4)	106·0 (3)	C(1′)–N(9)–C(4)	126·2 (2)
C(1′)–N(9)–C(8)	127·7 (3)	C(4′)–O(1′)–C(1′)	110·5 (2)
O(1′)–C(1′)–N(9)	108·4 (2)	C(2′)–C(1′)–N(9)	112·2 (3)
C(2′)–C(1′)–O(1′)	107·2 (2)	O(2′)–C(2′)–C(1′)	106·8 (3)
C(3′)–C(2′)–C(1′)	101·5 (2)	C(3′)–C(2′)–O(2′)	111·4 (3)
N(3′)–C(3′)–C(2′)	112·5 (2)	C(4′)–C(3′)–C(2′)	102·1 (2)
C(4′)–C(3′)–N(3′)	115·1 (3)	C(3′)–C(4′)–O(1′)	104·3 (2)
C(5′)–C(4′)–O(1′)	109·6 (3)	C(5′)–C(4′)–C(3′)	116·1 (3)
O(5′)–C(5′)–C(4′)	109·1 (3)		

Discussion. The conformation at the glycosidic bond N(9)–C(1′) is *anti* with χ_{CN} = 18·1 (3)°. A similar conformation with χ_{CN} = 10·0 (2)° is observed for the analogous 3′-deoxyadenosine derivative (2) (Sheldrick & Morr, 1980). Steric barriers to rotation about N(9)–C(1′) computed by Haschemeyer & Rich (1967) have indicated that the mode of sugar puckering will determine the possible values of χ_{CN}. They calculated that for purine nucleosides and nucleotides with C(3′)-*endo* puckering, as observed for (1) and (2), only the *anti* conformation in the range −150 to +20° should be preferred. In (1) and (2), C(3′) is displaced by respectively 0·570 and 0·566 Å on the same side as C(5′) from the least-squares plane through the remaining four ribose ring atoms. With the nomenclature of Sundaralingam (1975), the conformation of the ribose ring in (1) and (2) may also be described as twist $^3T_2$. C(2′) and C(3′) are displaced by 0·199 and 0·411 Å in (1) and 0·260 and 0·358 Å in (2) on opposite sides of the plane through C(1′), O(1′) and C(4′).

Whereas the position of the C(5′)–O(5′) bond relative to the ribose ring is *gauche–trans* in (2), a *gauche–gauche* conformation is observed in (1). The torsion angles ψ_{OO} [O(5′)–C(5′)–C(4′)–O(1′)] and ψ_{OC} [O(5′)–C(5′)–C(4′)–C(3′)] are respectively 57·4 (2) and 175·7 (2)° in (2) and −66·4 (3) and 51·4 (3)° in (1). A *gauche–gauche* conformation similar to that in (1) also occurs for the 3′-deoxyadenosine derivatives (3) and (4). O(5′) participates in intermolecular O···H–N hydrogen bonding to N(6) in (2), but in intermolecular O–H···N hydrogen bonding to N(3) in (1), (3) and (4). As a result of the small value of χ_{CN} and the *gauche–gauche* position of C(5′)–O(5′), a very short O(5′)···C(8) intramolecular distance of 3·14 (1) Å with an O(5′)···H(8) distance of 2·13 (3) Å is observed in (1). This O(5′)···C(8) distance is even shorter than those of 3·16 (1) and 3·22 (1) Å in (3) and (4) and must be regarded as indicative of O···H–C hydrogen bonding.

N(3′) is involved in an N···H–N intermolecular hydrogen bond of length 2·82 (1) Å in (1). As in (2) and (3), the N(3′) amino protons in (1) do not take part in hydrogen bonds. The carbonyl O(6) in (1) is intermolecularly hydrogen bonded to O(2′) [d(O···H–O) = 2·71 (1) Å].

Parallel inosine bases in (1) are related to one another at a distance of 3·35 Å by translation along a. This distance is markedly shorter than that of 3·53 Å for the adenine bases in (2). There is no overlap of neighbouring inosine systems. This mode of stacking (pattern 1, Motherwell & Isaacs, 1972) is typical of purine nucleosides and nucleotides in the *anti* configuration with $\chi_{CN} \simeq 10 \pm 20°$ (Schomburg, 1978). Because of potential steric interaction between neighbouring ribose rings no base overlap is to be expected for this stacking mode.

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