

Fig. 1. Perspective view of a cation and anion of the title compound, showing the atomic numbering scheme. Radii are arbitrary and H atoms are omitted for clarity. The O...O hydrogen bond is indicated by a dashed line. Bond lengths and angles (see also *Abstract*): As—O 1.690 (7), As—C(11) 1.911 (12), As—C(21) 1.896 (12), As—C(31) 1.985 (12) Å, Cl(1)—Au—Cl(2) 90.8 (1), Cl(1)—Au—Cl(2) 89.2 (1), O—As—C(11) 110.4 (5), O—As—C(21) 106.9 (4), O—As—C(31) 111.3 (5), C(11)—As—C(21) 110.5 (5), C(11)—As—C(31) 108.1 (5), C(21)—As—C(31) 109.7 (6)°. Symmetry operator (i): $-x, 1-y, 1-z$.

Related literature. The preparation of the title compound has been described by Potts (1970) and more recently by Yuqiu & Shihua (1987). Structures of compounds with general formula $(RO)_2H^+ \cdot AuCl_4^-$ have been described by Jones & Sheldrick (1978) ($R = Ph_3P$; not isostructural with the title compound), Hussain & Schlemper (1982) ($R = pyridine$), Hussain & Al-

Hamoud (1984) ($R = \alpha$ -picoline), Hussain & Al-Hamoud (1985) ($R = 3$ -methylpyridine), Hussain & Al-Hamoud (1986) ($R = 4$ -methylpyridine), and Drew, Glaves & Hudson (1985) ($R = 2$ -nonylpyridine).

Calculations were performed with the program system *SHELX76* (Sheldrick, 1976) locally modified by its author. We thank the Fonds der Chemischen Industrie for financial support.

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Acta Cryst. (1988). **C44**, 2202–2204

The Structure of 3',5'-Di-*O*-acetyl-2'-deoxyadenosine

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(Received 28 April 1988; accepted 25 July 1988)

Abstract. $C_{14}H_{17}N_5O_5$, $M_r = 335.3$, orthorhombic, $P2_12_12_1$, $a = 7.75$ (1), $b = 12.89$ (2), $c = 15.47$ (1) Å, $U = 1546$ Å³, $Z = 4$, $D_x = 1.440$ Mg m⁻³, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 0.10$ mm⁻¹, $F(000) = 704$, $T = 293$ K, $R = 0.056$ for 919 unique observed [$F > 5\sigma(F)$] reflections. The *N*-glycosidic bond angle χ has a value of -86 (1)°, in the high *anti* range. The sugar pucker is 2T_1 with $P = 155$ (1)° and $\psi = 33$ (1)°. The C4'—C5' conformation is $-sc$ with $\gamma = -76$ (1)°. Symmetry-related molecules form self-base-paired hydrogen-bonded ribbons through N1...N6ⁱ and N6...N7ⁱⁱ contacts, both of 3.02 Å [(i) $-x, -\frac{1}{2}+y,$

$-\frac{1}{2}-z$; (ii) $-1-x, -\frac{1}{2}+y, -\frac{1}{2}-z$]. This type of base-pairing is similar to that found for 2',3',5'-tri-*O*-acetylated adenosine molecules. The base pair shows a propeller twist, defined as the angle between the base planes about a line joining them, of 31 (2)°.

Experimental. Crystals were obtained from aqueous solution. Space group and initial cell dimensions were obtained from Weissenberg photographs. Data were collected on a Nicolet P3 (four-circle) diffractometer in Aberdeen by RAH. The crystal had dimensions 0.9 × 0.26 × 0.08 mm. Cell parameters were measured

on the diffractometer using 14 reflections in the 2θ range $12\text{--}15^\circ$. Range of indices: $0 \leq h \leq 10$; $0 \leq k \leq 16$; $0 \leq l \leq 20$. Data measured using $\theta/2\theta$ scans in the range $0 < 2\theta < 50^\circ$. Standard reflections, 114 and 040, were measured every 50 reflections. 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. 1432 independent reflections measured, giving 919 observed [$F > 5\sigma(F)$] reflections used in the refinement. The structure was solved using the *SHELXS86* program (Sheldrick, 1986). The *E* map revealed positions of all non-hydrogen atoms. The positions of the hydrogens attached to atom N6 were obtained from a difference map. All other H atoms were included at calculated positions, C—H = 1.08 Å. The H

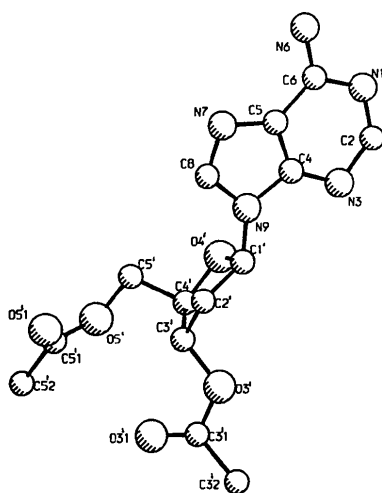


Fig. 1. Perspective view of molecule giving atomic numbering.

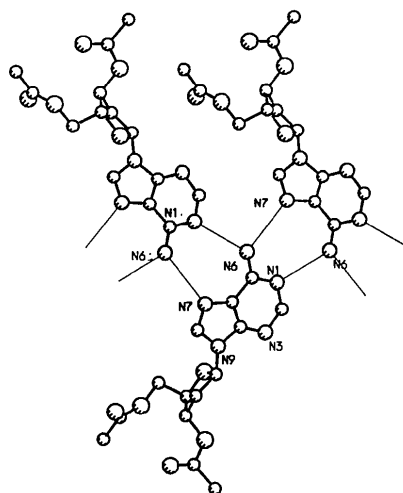


Fig. 2. View of the self-base-paired molecules.

Table 1. Coordinates ($\times 10^4$) for non-hydrogen atoms with *e.s.d.*'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq} (\text{\AA}^2 \times 10^3)$
N1	2688 (8)	-3606 (5)	-5681 (4)	36 (2)
C2	2983 (12)	-4553 (6)	-6023 (5)	42 (3)
N3	1965 (9)	-5386 (5)	-6048 (4)	37 (2)
C4	469 (10)	-5170 (6)	-5650 (5)	31 (3)
C5	-23 (9)	-4264 (6)	-5260 (5)	27 (2)
C6	1178 (11)	-3425 (6)	-5278 (5)	35 (2)
N6	876 (8)	-2504 (5)	-4942 (5)	43 (2)
N7	-1688 (8)	-4322 (5)	-4935 (4)	40 (2)
C8	-2161 (11)	-5259 (6)	-5134 (5)	39 (3)
N9	-910 (8)	-5843 (4)	-5555 (4)	32 (2)
C1'	-1048 (10)	-6851 (5)	-5946 (4)	30 (2)
C2'	-2280 (10)	-7586 (5)	-5479 (4)	32 (2)
C3'	-2921 (10)	-8295 (5)	-6182 (5)	33 (2)
O3'	-1760 (7)	-9165 (4)	-6256 (3)	41 (1)
C3'1	-2455 (14)	-10034 (7)	-6603 (6)	44 (3)
C3'2	-1203 (12)	-10913 (6)	-6619 (5)	58 (3)
O3'1	-3894 (9)	-10061 (5)	-6844 (4)	70 (2)
C4'	-2851 (10)	-7609 (6)	-6990 (5)	35 (2)
C5'	-4558 (11)	-7116 (6)	-7248 (6)	47 (3)
O5'	-5616 (8)	-7914 (5)	-7650 (3)	50 (2)
C5'1	-6904 (12)	-8326 (7)	-7180 (6)	45 (3)
C5'2	-7883 (12)	-9100 (8)	-7683 (6)	69 (4)
O5'1	-7160 (8)	-8121 (5)	-6439 (4)	61 (2)
O4'	-1754 (7)	-6741 (4)	-6784 (3)	45 (2)

Table 2. Interatomic distances (Å) and angles ($^\circ$)

C2—N1	1.350 (9)	C6—N1	1.347 (9)
N3—C2	1.333 (9)	C4—N3	1.343 (9)
C5—C4	1.368 (9)	N9—C4	1.385 (9)
C6—C5	1.428 (10)	N7—C5	1.388 (9)
N6—C6	1.317 (9)	C8—N7	1.299 (9)
N9—C8	1.390 (9)	C1'—N9	1.437 (8)
C2'—C1'	1.526 (9)	O4'—C1'	1.415 (7)
C3'—C2'	1.506 (9)	O3'—C3'	1.442 (8)
C4'—C3'	1.531 (9)	C3'1—O3'	1.355 (10)
C3'2—C3'1	1.493 (12)	O3'1—C3'1	1.177 (11)
C5'—C4'	1.522 (10)	O4'—C4'	1.441 (8)
O5'—C5'	1.455 (9)	C5'1—O5'	1.346 (10)
C5'2—C5'1	1.476 (12)	O5'1—C5'1	1.193 (9)
C6—N1—C2	119.1 (7)	N3—C2—N1	129.7 (8)
C4—N3—C2	109.3 (6)	C5—C4—N3	128.4 (7)
N9—C4—N3	125.8 (7)	N9—C4—C5	105.8 (7)
C6—C5—C4	117.1 (7)	N7—C5—C4	111.9 (7)
N7—C5—C6	130.9 (8)	C5—C6—N1	116.4 (7)
N6—C6—N1	119.6 (7)	N6—C6—C5	124.0 (8)
C8—N7—C5	103.1 (7)	N9—C8—N7	114.7 (7)
C8—N9—C4	104.4 (6)	C1'—N9—C4	125.4 (7)
C1'—N9—C8	129.4 (7)	C2'—C1'—N9	114.1 (6)
O4'—C1'—N9	108.9 (6)	O4'—C1'—C2'	104.7 (6)
C3'—C2'—C1'	104.0 (5)	O3'—C3'—C2'	108.8 (6)
C4'—C3'—C2'	103.1 (6)	C4'—C3'—O3'	111.2 (6)
C3'1—O3'—C3'	115.2 (7)	C3'2—C3'1—O3'	112.1 (9)
O3'1—C3'1—O3'	121.8 (10)	O3'1—C3'1—C3'2	126.1 (9)
C5'—C4'—C3'	115.1 (7)	O4'—C4'—C3'	106.8 (6)
O4'—C4'—C5'	104.3 (6)	O5'—C5'—C4'	107.9 (6)
C5'1—O5'—C5'	117.8 (7)	C5'2—C5'1—O5'	111.3 (8)
O5'1—C5'1—O5'	123.8 (9)	O5'1—C5'1—C5'2	124.9 (10)
C4'—O4'—C1'	110.6 (5)		

atoms were given fixed isotropic temperature factors approximately 1.5 times that of the parent atom and allowed to ride on their parent atoms. All other atoms were refined anisotropically. Blocked full-matrix refinement (on *F*) was carried out using the program

SHELX76 (Sheldrick, 1976). The refinement converged at $R = 0.056$, $wR = 0.048$, $w = 1.4632[\sigma^2(F) + 0.000086F^2]^{-1}$. 217 refined parameters; average shift/e.s.d. = 0.003; max. shift/e.s.d. < 0.01; max. difference peak, 0.23, min., -0.22 e Å⁻³.

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. The atomic numbering is shown in the perspective drawing (Fig. 1). Tables 1 and 2 give the atomic parameters, bond lengths and angles.* Fig. 2 shows the hydrogen bonding between symmetry-related molecules. The chirality of the molecule was not determined.

Related literature. The conformational parameters are in accordance with the IUPAC-IUB Joint Commission on Biochemical Nomenclature (1983) guidelines. The

* 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 51266 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

related compounds 2',3',5'-tri-*O*-acetyladenosine and its 8-bromo derivative have been studied by Wilson, Tollin & Howie (1986) and Boyd, Low & Tollin (1987). Propeller twists between self-base-paired adenine bases are discussed by Wilson & Tollin (1987).

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Acta Cryst. (1988). **C44**, 2204-2206

The Absolute Structure of 3-[*N*-Methyl-*N*-(*S*)- α -methylbenzyl]carbamoyl-2,4-dimethylpyridinium Chloride Monohydrate

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(Received 11 May 1988; accepted 20 July 1988)

Abstract. C₁₇H₂₁ClN₂O.H₂O, $M_r = 322.83$, monoclinic, $P2_1$, $a = 13.667$ (1), $b = 8.585$ (1), $c = 16.106$ (4) Å, $\beta = 106.09$ (1)°, $V = 1815.7$ (5) Å³, $Z = 4$, $D_x = 1.181$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 19.4$ cm⁻¹, $F(000) = 688$, $T = 295$ K, $R = 0.0659$ for 6021 observed reflections with $I > 2.5\sigma(I)$. The molecules contain three planar parts, the pyridinium and phenyl rings and the carbamoyl fragment in which the carbonyl and *N*-methyl groups are in *anti* position. The water molecules are disordered over two positions with site occupation factors of 0.52 (1) and 0.48 (1) respectively. The Cl⁻ ions serve as hydrogen-bond

acceptors for the N-H groups of the pyridinium cations and for the disordered water molecules.

Experimental. A colourless plate-shaped crystal, 1.00 × 0.40 × 0.06 mm, was glued on top of a glass fibre and transferred to an Enraf-Nonius CAD-4F diffractometer. Unit-cell parameters and their e.s.d.'s were derived from a least-squares treatment of 12 reflections ($10.0 < \theta < 14.4^\circ$). 7232 intensity data were collected in 'FLAT' mode within two Friedel-related quadrants of the reflection sphere (hkl and $\bar{h}\bar{k}\bar{l}$ with $0 < h < 16$; $0 < k < 10$; $-19 < l < 19$; $2.86 < \theta < 70.0^\circ$) using Ni-filtered Cu $K\alpha$ radiation in $\omega/2\theta$ scan mode with $\Delta\omega = (0.60 + 0.15\tan\theta)^\circ$. Three reference

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