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Structure of 2′,3′-*O*-Isopropylideneguanosine Dimethylsulfoxide Solvate

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Abstract. $C_{13}H_{17}N_5O_5.C_2H_6OS$, $M_r = 401{\cdot}23$, orthorhombic, $P2_12_12_1$, grown from Me_2SO, $a = 10{\cdot}749$ (2), $b = 13{\cdot}219$ (2), $c = 14{\cdot}056$ (2) Å, $V = 1997{\cdot}23$ Å^3, $Z = 4$, $D_m = 1{\cdot}40$, $D_x = 1{\cdot}335$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 1{\cdot}694$ mm^{-1}, $F(000) = 848{\cdot}00$, $T = 293$ K, $R = 0{\cdot}0538$, $wR = 0{\cdot}0634$ for 2105 unique reflections with $F > 3\sigma(F)$. The asymmetric unit contains one nucleoside molecule with a disordered solvent Me_2SO molecule. The geometry about the C(4′)—C(5′) bond is *gauche–gauche*. The guanosine base is in the *anti* conformation with the furanose ring having C(3′)-*exo* (E_3) puckering. The bases do not show any stacking in contrast to other guanosine-containing structures. The crystal structure is stabilized by N—H···N and N—H···O hydrogen bonding.

Introduction. This investigation was undertaken as part of our studies on isopropylidene derivatives of nucleosides (Katti, Seshadri & Viswamitra, 1981; Gautham, Seshadri & Viswamitra, 1983; Gautham, Seshadri, Viswamitra & Salisbury, 1983). Such structures serve as models for studying the flexibility of nucleoside conformation under external cyclic constraints (Viswamitra & Salisbury, 1984). We now report the crystal structure of 2′,3′-*O*-isopropylideneguanosine.

Experimental. Plate-like crystals were obtained by slow evaporation of a solution of the compound (Sigma Chemicals) in Me_2SO. Crystals were unstable when exposed to air and had to be sealed in thin-walled glass capillary tubes. The density was measured by flotation in CCl_4/C_6H_6. Unit-cell parameters were refined using setting angles for 25 centred reflections ($7 < \theta < 45°$) on a Nonius CAD-4 diffractometer. Intensity data were collected using Cu $K\alpha$ radiation, ω–2θ scan, from a crystal of dimensions $0{\cdot}2 \times 0{\cdot}1 \times 0{\cdot}1$ mm up to $(\sin\theta)/\lambda = 0{\cdot}617$ Å^{-1}. The data were corrected for Lorentz and polarization factors. Absorption corrections were not applied. 2286 reflections were measured for $0 \le h \le 13$, $0 \le k \le 16$ and $0 \le l \le 17$, of which 2115 were considered observed $[F > 3\sigma(F)]$. The number of unique reflections was 2105. Two reflections $\bar{1}\bar{1}3$ and $\bar{3}\bar{2}3$ monitored during the data collection showed negligible variation indicating crystal and instrumental stability. The structure was solved by direct methods using *MULTAN*80 (Main *et al.*, 1980). Most non-H atoms were located from an *E* map computed with the best set of phases. Subsequent difference Fourier maps yielded peaks corresponding to the rest of the structure as well as five additional peaks which could be interpreted as belonging to a disordered solvent molecule. An analysis of the coordinates of the five additional peaks indicated that the S atom of the solvent molecule shows an umbrella inversion (Walden inversion) thus giving rise to two peaks S(1) and S(2) with half occupancies. The measured density of the crystal (1·40 Mg m^{-3}) also suggests the presence of one solvent molecule per asymmetric unit; the calculated density for two Me_2SO molecules is rather too high at 1·59 Mg m^{-3}. However, the observed density could also accommodate an additional water molecule, but we have not been able to see electron density in the difference Fourier maps suggesting this. It should also be mentioned that our crystallizations were done using only Me_2SO solvent. Full-matrix refinement on F [all calculations with *SHELX*76 (Sheldrick, 1976)] with anisotropic temperature factors reduced R to 0·070. The molecular H atoms except one were located either from difference Fourier maps or from geometric considerations. Two of the Me_2SO H atoms were also

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Table 1. *Final positional parameters* ($\times 10^4$) *and equivalent isotropic temperature factors of non*-H *atoms, with e.s.d.'s in parentheses*

	x	y	z	B_{eq}* (Å^2)
N(1)	611 (2)	2216 (2)	4419 (2)	3·50 (5)
C(2)	336 (3)	1808 (2)	3534 (2)	3·71 (6)
N(2)	−879 (3)	1710 (3)	3332 (2)	5·28 (8)
N(3)	1181 (3)	1523 (2)	2913 (2)	3·93 (6)
C(4)	2341 (3)	1668 (2)	3234 (2)	3·51 (6)
C(5)	2718 (3)	2047 (2)	4104 (2)	3·46 (6)
C(6)	1816 (3)	2352 (2)	4771 (2)	3·43 (6)
O(6)	1967 (2)	2715 (2)	5568 (1)	4·71 (6)
N(7)	4002 (2)	2050 (2)	4175 (2)	3·85 (5)
C(8)	4388 (3)	1690 (2)	3360 (2)	4·07 (7)
N(9)	3424 (2)	1457 (2)	2751 (2)	3·78 (6)
C(1′)	3476 (3)	949 (2)	1840 (2)	4·25 (7)
C(2′)	4502 (4)	1310 (2)	1155 (2)	4·60 (8)
O(2′)	4020 (4)	1856 (2)	374 (2)	6·48 (9)
C(3′)	4996 (4)	319 (3)	739 (2)	5·11 (9)
O(3′)	4237 (4)	224 (2)	−84 (2)	6·72 (9)
C(4′)	4695 (4)	−479 (3)	1460 (3)	5·34 (9)
O(4′)	3655 (3)	−96 (2)	1991 (2)	5·99 (8)
C(5′)	5784 (6)	−716 (3)	2103 (3)	6·96 (13)
O(5′)	6308 (4)	169 (2)	2508 (2)	7·06 (9)
C(10)	4079 (6)	1205 (4)	−456 (3)	6·77 (13)
C(11)	5195 (7)	1535 (5)	−1045 (4)	9·60 (22)
C(12)	2866 (8)	1253 (6)	−969 (5)	10·47 (25)
S(1)	9563 (2)	766 (2)	844 (1)	6·01 (6)
S(2)	8836 (3)	−172 (2)	895 (2)	6·09 (7)
O(1)	8377 (4)	775 (4)	1521 (2)	9·45 (14)
C(13)	10381 (7)	−334 (5)	1192 (5)	9·36 (20)
C(14)	8834 (9)	338 (7)	−244 (4)	11·03 (28)

$*B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

N(1)–C(2)	1·386 (4)	C(2′)–C(3′)	1·529 (5)
N(1)–C(6)	1·399 (4)	O(2′)–C(10)	1·452 (5)
C(2)–N(2)	1·343 (5)	C(3′)–C(3′)	1·421 (5)
C(2)–N(3)	1·316 (4)	C(3′)–C(4′)	1·499 (5)
N(3)–C(4)	1·340 (4)	O(3′)–C(10)	1·408 (6)
C(4)–C(5)	1·381 (4)	C(4′)–C(4′)	1·437 (5)
C(4)–N(9)	1·377 (4)	C(4′)–C(5′)	1·511 (7)
C(5)–C(6)	1·408 (4)	C(5′)–C(5′)	1·417 (5)
C(5)–N(7)	1·383 (4)	C(10)–C(11)	1·521 (9)
C(6)–O(6)	1·229 (3)	C(10)–C(12)	1·491 (10)
N(7)–C(8)	1·308 (4)	S(1)–O(1)	1·592 (4)
C(8)–N(9)	1·378 (4)	S(1)–C(13)	1·769 (7)
N(9)–C(1′)	1·448 (4)	S(1)–C(14)	1·809 (7)
C(1′)–C(2′)	1·540 (5)	S(2)–O(1)	1·608 (5)
C(1′)–O(4′)	1·410 (4)	S(2)–C(13)	1·727 (8)
C(2′)–O(2′)	1·412 (4)	S(2)–C(14)	1·737 (7)
C(2)–N(1)–C(6)	124·4 (3)	O(2′)–C(2′)–C(3′)	105·6 (3)
N(1)–C(2)–N(2)	115·8 (3)	C(2′)–C(2′)–C(10)	107·7 (3)
N(1)–C(2)–N(3)	124·0 (3)	C(2′)–C(3′)–O(3′)	100·8 (3)
N(2)–C(2)–N(3)	120·2 (3)	C(2′)–C(3′)–C(4′)	105·7 (3)
C(2)–N(3)–C(4)	112·2 (3)	O(3′)–C(3′)–C(4′)	111·4 (3)
N(3)–C(4)–C(5)	128·6 (3)	C(3′)–C(3′)–C(10)	106·9 (3)
N(3)–C(4)–N(9)	126·2 (3)	C(3′)–C(4′)–O(4′)	105·7 (3)
C(5)–C(4)–N(9)	105·2 (2)	C(3′)–C(4′)–C(5′)	112·5 (4)
C(4)–C(5)–C(6)	119·4 (3)	O(4′)–C(4′)–C(5′)	111·4 (3)
C(4)–C(5)–N(7)	111·0 (3)	C(1′)–O(4′)–C(4′)	111·9 (3)
C(6)–C(5)–N(7)	129·6 (3)	C(4′)–C(5′)–O(5′)	112·2 (4)
N(1)–C(6)–C(5)	111·4 (3)	O(2′)–C(10)–O(3′)	104·6 (4)
N(1)–C(6)–C(6)	119·7 (3)	O(2′)–C(10)–C(11)	107·6 (4)
C(5)–C(6)–C(6)	128·9 (3)	O(2′)–C(10)–C(12)	109·0 (5)
C(5)–N(7)–C(8)	104·6 (2)	O(3′)–C(10)–C(11)	111·8 (5)
N(7)–C(8)–N(9)	112·7 (3)	O(3′)–C(10)–C(12)	108·9 (5)
C(4)–N(9)–C(8)	106·5 (2)	C(11)–C(10)–C(12)	114·5 (5)
C(4)–N(9)–C(1′)	124·3 (2)	C(1)–S(1)–C(13)	103·8 (3)
C(8)–N(9)–C(1′)	128·6 (3)	O(1)–S(1)–C(14)	99·2 (3)
N(9)–C(1′)–C(2′)	115·9 (2)	C(13)–S(1)–C(14)	101·0 (4)
N(9)–C(1′)–O(4′)	109·0 (2)	O(1)–S(2)–C(13)	105·1 (3)
C(2′)–C(1′)–O(4′)	107·5 (3)	O(1)–S(2)–C(14)	101·6 (4)
C(1′)–C(2′)–O(2′)	112·4 (3)	C(13)–S(2)–C(14)	105·8 (4)
C(1′)–C(2′)–C(3′)	102·8 (3)		

located from difference Fourier maps. H atoms were refined isotropically. After the final cycles of refinement, $R = 0\cdot0538$, $wR = 0\cdot0634$ and $S = 0\cdot6$. The function minimized in the final stages of refinement was $\sum w(|F_o| - |F_c|)^2$ where $w^{-1} = \sigma^2(F) + 0\cdot012325F^2$. Scattering factors for non-H atoms taken from Cromer & Waber (1965), for H atoms computed from Stewart, Davidson & Simpson (1965). Max. and min. electron densities in the final difference Fourier map were 0·23 and −0·22 e Å^{-3} respectively. Max. $\Delta/\sigma = 0\cdot996$.

Discussion.

Molecular geometry. The positional parameters of the non-H atoms and the bond lengths and bond angles are given in Tables 1 and 2.* Fig. 1 shows the geometry of the molecule; the crystal structure is illustrated in Fig. 2.

Guanosine base. The nucleoside has the common *anti* conformation with the glycosidic torsion angle χ [O(4′)–C(1′)–N(9)–C(4)] (IUPAC–IUB Joint Commission on Biochemical Nomenclature, 1983) = −94·1 (3)°. The guanosine base is essentially planar.

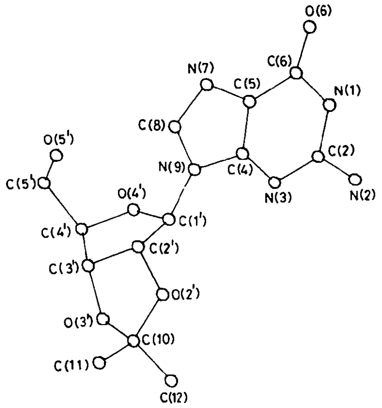

Fig. 1. Molecular geometry showing atomic labelling.

Furanose sugar. C(3′) of the ribose moiety deviates by 0·390 (3) Å on the opposite side to C(5′) from the least-squares plane defined by the remaining four atoms of the furanose ring. Thus, the sugar pucker is C(3′)-*exo* (E_3). P, the phase angle of pseudorotation, and $\tau_{m,\max}$, the maximum amplitude of pucker, are equal to

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44649 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

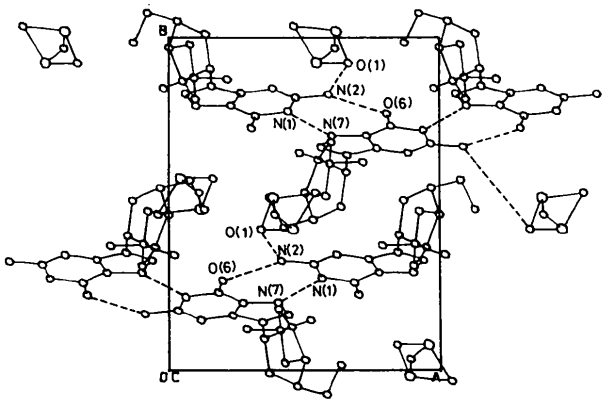

Fig. 2. Packing diagram viewed down the z axis showing hydrogen bonding between molecules in the unit cell.

196·42 and 25·72° respectively. The conformation about the C(4')—C(5') bond is *gauche–gauche* with torsion angles φ_{OO}[O(5')—C(5')—C(4')—O(4')] and φ_{OC}[O(5')—C(5')—C(4')—C(3')] —67·5 (5) and 51·0 (5)° respectively.

Dioxolane ring. The dioxolane ring assumes O(3')-*endo* geometry with torsional angle C(10)—O(2')—C(2')—C(3') = 6·0 (4)° and O(3') displaced by −0·530 (4) Å. The pseudorotation parameters for the dioxolane ring are $P = -45{\cdot}49$ and $\tau_{m,\max} = 38{\cdot}43°$.

Molecular packing. The crystal structure shows no base stacking in contrast to other guanosine-containing crystals where stacking of bases is normally observed. The structure is stabilized by three hydrogen bonds as shown in Fig. 2. Molecules related by ($x-\frac{1}{2}$, $-y+\frac{1}{2}$, $-z+1$) form two hydrogen bonds [N(1)—H···N(7) = 2·801 (4) Å, 166·0°; N(2)—H···O(6) = 2·887 (4) Å, 167·9°]. The third bond [N(2)—H···O(1) = 2·941 (5) Å, 170·5°] involves the O(1) atom of the solvent Me_2SO molecule related by $x-1, y, z$.

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Structure of the 1/1 Complex of [2.2]Metacyclophane* with Tetracyanoethylene

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Abstract. $C_{16}H_{16}.C_6N_4$, $M_r = 336{\cdot}4$, triclinic, $P\bar{1}$, $a = 9{\cdot}380\,(4)$, $b = 8{\cdot}700\,(5)$, $c = 6{\cdot}888\,(4)$ Å, $\alpha = 102{\cdot}20\,(5)$, $\beta = 99{\cdot}60\,(5)$, $\gamma = 119{\cdot}70\,(5)°$, $V = 452\,(1)$ Å^3, $Z = 1$, $D_x = 1{\cdot}23$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71093 Å, $\mu = 0{\cdot}081$ mm^{-1}, $F(000) = 176$, $T = 243$ K. The structure was refined to a wR value of 0·062 for 589 observed intensities. The structure is isomorphous to those of the related compounds, charge-transfer complexes of [2.2]paracyclophane, [3.3]paracyclophane and [2.2]metaparacyclophane with tetracyanoethylene (TCNE). The molecules of [2.2]metacyclophane and TCNE are on centers of symmetry and are alternately stacked along the a axis, as in the three compounds mentioned above.

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* [2.2]Metabenzenophane.

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