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Structure of 2',3',5'-Tri-*O*-acetyl-8-bromoguanosine

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Abstract. $C_{16}H_{18}BrN_5O_8$, $M_r = 488.2$, triclinic, $P1$, $a = 8.201$ (5), $b = 8.761$ (5), $c = 15.392$ (13) Å, $\alpha = 95.38$ (6), $\beta = 103.11$ (6), $\gamma = 110.25$ (6)°, $U = 992$ Å³, $Z = 2$, $D_x = 1.63$ g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 20.5$ cm⁻¹, $F(000) = 496$, $T = 293$ K, $R = 0.071$ for 4066 unique observed [$F > 3\sigma(F)$] reflections. The *N*-glycosidic torsion angles χ have values 98.1 (1) and 85.6 (1)°, in the high *anti* range, which are unusual for ribose nucleosides. (Molecule I values are given first throughout.) The sugar pucker are ${}_2E$ (C2'-*exo*), with $P = -19$ (1)° and $\psi_m = 22$ (1)°, which is unusual, and ${}_3T$ (C2'-*exo*/C3'-*endo*), with $P = 5$ (1)° and $\psi_m = 8$ (1)°. The C4–

C5 conformations, with $\gamma = 41.7$ (3) and 51.3 (3)°, are +*sc* (*gauche-gauche*).

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 0.5 × 0.46 × 0.08 mm. Cell parameters were measured on the diffractometer using 14 reflections in the 2θ

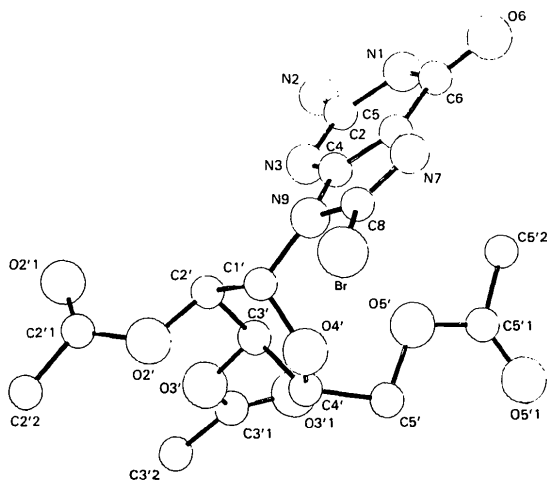


Fig. 1. Atomic numbering of molecule I viewed perpendicular to N7, C1' and O4'.

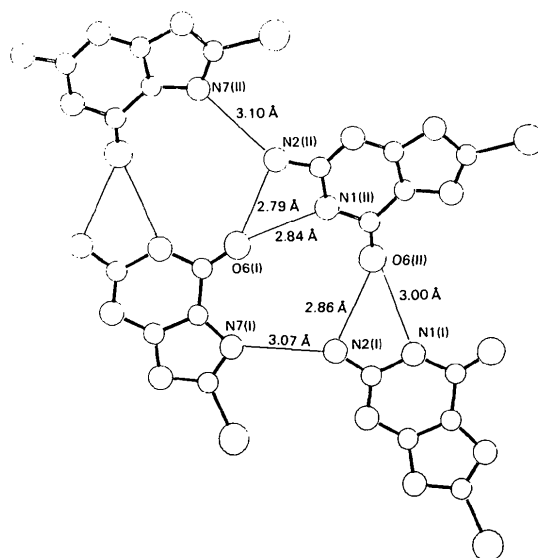


Fig. 2. Packing of bases viewed down *c*. Ribose rings omitted for clarity.

range 20–22°. Range of indices: $0 \leq h \leq 10$; $-12 \leq k \leq 11$; $-21 \leq l \leq 20$. Data measured using $\theta/2\theta$ scans in the range $0 < 2\theta < 60^\circ$. Standard reflections, 144 and 242, were measured every 50 reflections. No significant change in the intensities of these reflections was found throughout data collection. No absorption correction. 5262 independent reflections measured, giving 4066 observed [$F > 3\sigma(F)$] reflections used in the refinement. The structure was solved by locating the Br atom positions from a Patterson

synthesis. Fourier recycling, using the program package *MITHRIL* (Gilmore, 1983) completed the definition of the structure. Blocked full-matrix refinement (on F) was carried out using the program *SHELX76* (Sheldrick, 1976). The H atoms were included at calculated positions. All H atoms were given fixed isotropic temperature factors of approximately 1.5 times that of the parent atom. Anisotropic temperature factors were used for all non-H atoms, and the refinement converged at $R = 0.071$, $wR = 0.069$, $w = 1.3161[\sigma^2(F) + 0.002077F^2]$. 549 refined parameters; max. shift/e.s.d. < 0.1 ; max. difference peak, $1.17 \text{ e } \text{\AA}^{-3}$, min. difference peak, $-1.36 \text{ e } \text{\AA}^{-3}$. The largest of the difference map peaks were associated with the Br atoms.

Table 1. Fractional coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$) for non-H atoms with e.s.d.'s in parentheses

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

Molecule I	x	y	z	U_{eq}
Br	9986	0	9997	48 (1)
N1	7038 (8)	5245 (8)	11199 (5)	31 (1)
C2	5370 (11)	3943 (10)	10828 (5)	31 (2)
N2	3952 (10)	4286 (9)	10913 (7)	53 (2)
N3	5190 (9)	2491 (8)	10425 (5)	32 (2)
C4	6810 (10)	2376 (9)	10452 (5)	27 (2)
C5	8536 (10)	3574 (9)	10807 (6)	28 (2)
C6	8713 (10)	5179 (10)	11215 (6)	36 (2)
O6	10112 (8)	6420 (7)	11586 (5)	46 (2)
N7	9872 (9)	3004 (9)	10705 (5)	36 (2)
C8	8941 (11)	1488 (10)	10287 (6)	30 (2)
N9	7074 (8)	1002 (8)	10108 (4)	27 (1)
C1'	5867 (10)	-538 (9)	9469 (6)	29 (2)
C2'	3850 (10)	-1024 (8)	9349 (6)	24 (2)
O2'	3127 (8)	-2783 (7)	9010 (4)	33 (1)
C2'1	1584 (11)	-3690 (11)	9225 (7)	39 (2)
C2'2	947 (15)	-5477 (12)	8753 (8)	57 (3)
O2'1	962 (10)	-3129 (9)	9713 (6)	64 (2)
C3'	3282 (11)	-170 (9)	8578 (5)	28 (2)
O3'	1421 (9)	-1133 (8)	8069 (5)	40 (3)
C3'1	522 (15)	-335 (14)	7582 (8)	50 (1)
C3'2	-1361 (17)	-1427 (18)	7107 (9)	70 (4)
O3'1	1190 (13)	1086 (12)	7540 (7)	82 (3)
C4'	4605 (12)	-173 (10)	8003 (6)	34 (2)
C5'	5316 (16)	1378 (12)	7610 (7)	47 (3)
O5'	5739 (10)	2781 (8)	8314 (5)	48 (2)
C5'1	6311 (13)	4257 (13)	8072 (7)	42 (3)
C5'2	6477 (16)	5624 (11)	8778 (8)	54 (3)
O5'1	6646 (12)	4429 (9)	7367 (5)	60 (2)
O4'	6092 (8)	-408 (9)	8590 (5)	45 (2)
Molecule II				
Br	1605 (1)	3906 (1)	3158 (1)	46 (1)
N1	-6380 (8)	-1121 (8)	1872 (5)	28 (1)
C2	-6651 (10)	117 (9)	2364 (6)	27 (2)
N2	-8360 (9)	-224 (9)	2383 (6)	42 (2)
N3	-5362 (8)	1546 (8)	2800 (5)	27 (1)
C4	-3742 (9)	1653 (8)	2695 (5)	22 (1)
C5	-3315 (10)	511 (9)	2222 (5)	25 (2)
C6	-4710 (10)	-1025 (10)	1758 (6)	28 (2)
O6	-4587 (8)	-2175 (7)	1290 (4)	39 (1)
N7	-1451 (8)	1074 (8)	2298 (5)	28 (1)
C8	-847 (9)	2540 (9)	2820 (5)	26 (2)
N9	-2114 (8)	2993 (8)	3104 (4)	27 (1)
C1'	-1626 (11)	4335 (10)	3869 (6)	33 (2)
C2'	-3102 (11)	4987 (9)	3960 (6)	29 (2)
O2'	-2138 (9)	6698 (7)	4426 (4)	36 (1)
C2'1	-1612 (14)	7836 (10)	3891 (7)	43 (3)
C2'2	-531 (20)	9501 (13)	4429 (9)	67 (4)
O2'1	-1957 (15)	7422 (9)	3099 (6)	74 (3)
C3'	-3859 (10)	4031 (10)	4647 (6)	28 (2)
O3'	-4635 (9)	4909 (8)	5158 (5)	40 (2)
C3'1	-5983 (12)	3986 (13)	5483 (7)	45 (3)
C3'2	-6660 (16)	5062 (17)	5982 (8)	57 (4)
O3'1	-6500 (11)	2509 (10)	5359 (6)	63 (2)
C4'	-2246 (11)	3806 (11)	5263 (6)	34 (2)
C5'	-2657 (16)	2223 (12)	5657 (6)	46 (3)
O5'	-3712 (11)	851 (8)	4903 (5)	52 (2)
C5'1	-4850 (15)	-479 (11)	5108 (8)	48 (3)
C5'2	-5936 (23)	-1793 (17)	4244 (9)	81 (5)
O5'1	-4943 (15)	-636 (11)	5870 (7)	78 (3)
O4'	-1086 (9)	3791 (10)	4689 (5)	48 (2)

Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Also used were the program packages *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). All calculations were carried out on the Dundee University DEC-10 computer. The atomic numbering is shown in the perspective drawing (Fig. 1), and tables of atomic parameters, bond lengths and angles are given

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

	Mol. I	Mol. II	Mol. I	Mol. II
C8-Br	1.864 (10)	1.868 (6)	C6-N1-C2	125.6 (8) 124.5 (6)
C2-N1	1.394 (8)	1.374 (12)	N2-C2-N1	114.8 (8) 114.9 (6)
C6-N1	1.389 (12)	1.394 (12)	N3-C2-N1	123.3 (9) 124.4 (8)
N2-C2	1.327 (14)	1.335 (11)	N3-C2-N2	121.8 (7) 120.7 (8)
N3-C2	1.306 (12)	1.319 (8)	C4-N3-C2	112.3 (6) 111.6 (7)
C4-N3	1.359 (12)	1.347 (11)	C5-C4-N3	129.2 (8) 129.0 (6)
C5-C4	1.384 (9)	1.369 (12)	N9-C4-N3	126.3 (6) 124.7 (7)
N9-C4	1.375 (11)	1.397 (7)	N9-C4-C5	104.6 (7) 106.3 (7)
C6-C5	1.431 (12)	1.419 (9)	C6-C5-C4	118.0 (8) 119.0 (7)
N7-C5	1.384 (12)	1.407 (10)	N7-C5-C4	112.8 (7) 111.8 (6)
O6-C6	1.244 (8)	1.229 (11)	N7-C5-C6	129.2 (6) 129.2 (8)
C8-N7	1.294 (10)	1.313 (10)	C5-C6-N1	111.5 (6) 111.4 (8)
N9-C8	1.392 (10)	1.374 (12)	O6-C6-N1	119.5 (8) 120.6 (6)
C1'-N9	1.467 (8)	1.459 (10)	O6-C6-C5	129.0 (9) 128.0 (8)
O2'-C1'	1.520 (11)	1.534 (14)	C8-N7-C5	102.3 (6) 101.8 (7)
C4'-C1'	1.415 (12)	1.423 (12)	N7-C8-Br	123.3 (7) 121.3 (7)
O2'-C2'	1.441 (8)	1.451 (8)	N9-C8-Br	121.4 (5) 122.5 (5)
C3'-C2'	1.531 (12)	1.519 (13)	N9-C8-N7	115.2 (9) 116.3 (6)
C2'1-O2'	1.373 (11)	1.370 (12)	C8-N9-C4	105.2 (6) 103.9 (6)
C2'2-C2'1	1.517 (13)	1.465 (13)	C1'-N9-C4	132.9 (7) 131.0 (7)
O2'1-C2'1	1.158 (15)	1.178 (14)	C1'-N9-C8	120.1 (7) 122.6 (6)
O3'-C3'	1.448 (9)	1.438 (13)	C2'-C1'-N9	116.7 (7) 116.5 (6)
C4'-C3'	1.549 (14)	1.527 (13)	O4'-C1'-N9	109.6 (6) 108.8 (7)
C3'1-O3'	1.341 (15)	1.354 (12)	O4'-C1'-C2'	105.2 (6) 107.2 (6)
C3'2-C3'1	1.473 (14)	1.485 (20)	O2'-C2'-C1'	103.1 (7) 105.2 (8)
O3'1-C3'1	1.189 (15)	1.195 (14)	C3'-C2'-C1'	103.2 (7) 101.3 (7)
C5'-C4'	1.523 (14)	1.527 (14)	C3'-C2'-O2'	108.5 (6) 106.3 (7)
O4'-C4'	1.433 (12)	1.440 (13)	C2'1-O2'-C2'	116.0 (7) 116.1 (7)
C5'-C5'	1.445 (12)	1.450 (10)	C2'2-C2'1-O2'	108.4 (9) 111.9 (9)
O5'1-O5'	1.334 (13)	1.333 (13)	O2'1-C2'1-O2'	123.3 (8) 120.8 (8)
C5'2-C5'1	1.486 (16)	1.534 (15)	O2'1-C2'1-C2'2	128.3 (9) 127.2 (11)
O5'1-C5'1	1.190 (14)	1.209 (17)	O3'-C3'-C2'	109.4 (6) 111.8 (7)
			C4'-C3'-C2'	102.7 (8) 104.9 (7)
			C4'-C3'-O3'	111.7 (6) 111.3 (7)
			C3'1-O3'-C3'	116.9 (7) 117.0 (7)
			C3'2-C3'1-O3'	112.1 (10) 110.7 (9)
			O3'1-C3'1-O3'	123.2 (9) 121.8 (11)
			O3'1-C3'1-C3'2	124.6 (13) 127.5 (11)
			C5'-C4'-C3'	116.0 (9) 116.5 (7)
			O4'-C4'-C3'	105.9 (7) 104.2 (7)
			O4'-C4'-C5'	109.5 (7) 108.4 (9)
			O5'-C5'-C4'	107.0 (8) 107.0 (7)
			C5'1-O5'-C5'	115.2 (8) 115.4 (8)
			C5'2-C5'1-O5'	112.5 (9) 109.8 (11)
			O5'1-C5'1-O5'	122.6 (10) 124.5 (9)
			O5'1-C5'1-C5'2	124.9 (10) 125.7 (10)
			C4'-O4'-C1'	111.9 (7) 111.5 (8)

(Tables 1 and 2).^{*} Fig. 2 shows a view of the molecular packing of the bases; the ribose and acetyl atoms are omitted for clarity.

Related literature. The conformational parameters used follow the guidelines of the IUPAC–IUB Joint Commission on Biochemical Nomenclature (1983). The related compound 2',3',5'-tri-*O*-acetylguanosine has been studied recently (Wilson, Low & Tollin, 1985; Low, Tollin, Wilson & Scrimgeour, 1986). Conformational parameters for nucleosides are discussed by Saenger (1984).

^{*} 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 44344 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the ABC Ring Synthone of Dendrobine

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Abstract. (1*R*,4*S*,8*S*,11*R*)-11-Methyl-3-[(*R*)- α -phenylethyl]-3-azatricyclo[6.2.1.0^{4,11}]undec-5-en-2-one, C₁₉H₂₃NO, $M_r = 281.40$, orthorhombic, $P2_12_12_1$, $a = 9.534$ (2), $b = 11.055$ (4), $c = 14.372$ (4) Å, $V = 1514.8$ (8) Å³, $Z = 4$, $D_x = 1.23$ (163 K), $D_m = 1.15$ g cm⁻³ (295 K), $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.702$ cm⁻¹, $F(000) = 608$, $T = 163$ K, $R = 0.0387$ for 2310 reflections. The absolute configuration is assigned on the basis of internal comparison with the α -methylbenzylamine moiety. The tricyclic ring system is concave. The N appears to be sp^2 hybridized [N is 0.0661 (1) Å from the plane through C atoms bound to it] and conjugated through the carbonyl group {short C–N bond [1.351 (2) Å]}. Ring strain appears to cause a slight increase in the C–C bond lengths of the center atom of the tricyclic ring system. The average bond length from this atom to the three other ring C atoms is 1.557 (1) Å while the average bond length for all other sp^3 -C to sp^3 -C bonds is 1.529 (1) Å.

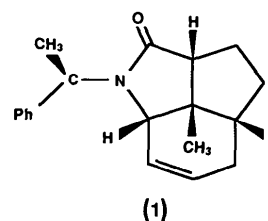
Experimental. The title compound (1) was synthesized by an intramolecular Diels–Alder reaction of the

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corresponding trienamide which was heated in a sealed tube in xylene. Details of the experimental procedure will be reported elsewhere (Martin & Li, 1987).



(*R*)-(+)- α -Methylbenzylamine, upon which the assignment of absolute configuration was made, was purchased from the Aldrich Chemical Company. Colorless block, cut from a larger crystal, 0.49 × 0.61 × 0.87 mm, from 1:3 ethyl acetate–hexane. Crystal density by flotation method in aqueous ZnCl₂. Syntex $P2_1$ diffractometer, graphite monochromator with Syntex LT-1 low-temperature delivery system (163 K). Lattice parameters from least-squares refinement of 30 reflections with $25.5 < 2\theta < 31.5^\circ$. ω -scan technique (5018 reflections, 2516 unique, $R_{int} = 0.0294$ from merging hkl and $\bar{h}\bar{k}l$ reflections; $h - 13 \rightarrow 15$;