Table 3. *Bond distances* (Å) *and angles* (°) *involving hydrogen bonds for* $C_{13}H_{23}NO_4.H_2O$

A—H···*B*	*A*—*B*	*A*—H	H—*B*	*A*—H—*B*
O(6)—HO(6)···O(8^{i})	2·707 (3)	0·90 (2)	1·82 (2)	169·9 (13)
N(8)—HN(8)···O*W*^{ii}	2·876 (4)	0·90 (2)	2·07 (1)	147·9 (12)
O*W*—HO*W*(1)···O(6)	2·817 (3)	1·14 (2)	1·69 (2)	171·5 (11)
O*W*—HO*W*(2)···O(8^{iii})	2·875 (4)	0·69 (1)	2·20 (1)	168·5 (15)

O(6)···O*W*···O(8^{i})	100·16 (11)	HO*W*(1)—O*W*—HO*W*(2)	103·0 (14)
O(6)···O*W*···N(8^{ii})	128·66 (12)	HO*W*(1)—O*W*···HN(8^{ii})	121·8 (8)
O(8^{i})···O*W*···N(8^{ii})	127·12 (12)	HO*W*(2)—O*W*···HN(8^{ii})	132·1 (12)
O*W*···O(6)—C(6)	127·5 (2)	HO*W*(1)···O(6)—C(6)	130·5 (5)
O*W*···O(8^{iii})—C(8^{iii})	128·4 (2)	HO*W*(2)···O(8^{iii})—C(8^{iii})	126·7 (4)
O*W*···N(8^{ii})—C(8^{ii})	134·0 (2)	HN(8)—N(8)—C(8)	113·2 (9)
O*W*···N(8^{ii})—C(9^{ii})	98·5 (2)	HN(8)—N(8)—C(9)	120·0 (9)

Symmetry code: (i) $-\frac{1}{2}+x, \frac{1}{2}+y, z$; (ii) $2-x, y, 2-z$; (iii) $2-x, 1+y, 2-z$.

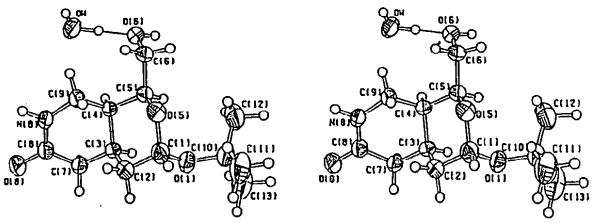

Fig. 1. Stereopair showing the molecular conformation and the atomic numbering of $C_{13}H_{23}NO_4.H_2O$. The ellipsoids correspond to 50% probability, except for the H atoms which are shown as spheres of arbitrary sizes.

sity fluctuation on the final difference Fourier synthesis +0·16 and −0·11 e Å^{-3}.

The scattering curves for the non-H atoms were taken from Cromer & Mann (1968) and those for the H atoms from Stewart, Davidson & Simpson (1965).

The final coordinates for the non-H atoms are given in Table 1, selected bond distances and angles in Table 2,* and bond distances and angles involving hydrogen bonds in Table 3. Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

Related literature. For the synthesis of the compound (3*a*), see Hanessian, Faucher & Léger (1990).

We would like to acknowledge NSERCC for financial support and a scholarship to AMF.

* Lists of structure factors, anisotropic thermal parameters, bond distances and angles involving H atoms, and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52891 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure Studies of Mitomycins. III. Structure of M-83

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Abstract. 7-*N*-(*p*-Hydroxyphenyl)mitomycin C* (I), $C_{21}H_{22}N_4O_6.H_2O$, $M_r = 444{\cdot}45$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}056$ (2), $b = 33{\cdot}832$ (9), $c = 7{\cdot}469$ (1) Å, $V = 2036$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}45$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 8{\cdot}8$ cm^{-1}, $F(000) = 936$, $T = 293$ K, $R = 0{\cdot}041$ for 1923 observed reflections with $F > 3\sigma(F)$. Although the overall structure, except the *p*-hydroxyphenyl group, is similar to mitomycin C, the bond lengths in the quinone ring are significantly influenced by the substituent. The phenyl and quinone rings are nearly planar and the two rings form a dihedral angle of

* Mitomycin C is [1*aR*]-6-amino-8-[[(aminocarbonyl)oxy]-methyl]-1,1a,2,8,8a,8b-hexahydro-8a-methoxy-5-methylazirino-[2′,3′:3,4]pyrrolo[1,2-*a*]indole-4,7-dione.

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Table 1. *Positional parameters and equivalent isotropic thermal parameters with their e.s.d.'s*

$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq} (Å^2)
O(5)	0·1921 (3)	0·53673 (5)	0·5564 (3)	3·52 (4)
O(8)	0·7870 (3)	0·58289 (5)	0·7955 (3)	3·30 (4)
O(9a)	0·2485 (3)	0·65552 (6)	0·9679 (3)	3·52 (4)
O(10)	0·6547 (4)	0·69932 (6)	0·8568 (3)	4·26 (5)
O(10a)	0·7535 (5)	0·74097 (7)	0·6490 (3)	6·26 (7)
O(14)	0·9437 (3)	0·35307 (5)	0·7971 (3)	3·64 (4)
N(1)	0·2844 (4)	0·67534 (7)	0·4754 (3)	3·40 (5)
N(4)	0·2369 (3)	0·61356 (6)	0·7067 (3)	2·73 (4)
N(7)	0·7645 (3)	0·51036 (6)	0·6942 (4)	3·17 (5)
N(10a)	0·7865 (6)	0·75283 (8)	0·9441 (4)	6·90 (9)
C(1)	0·2457 (4)	0·68187 (7)	0·6675 (4)	2·98 (5)
C(2)	0·1185 (4)	0·66535 (9)	0·5447 (5)	3·38 (6)
C(3)	0·1015 (4)	0·62171 (9)	0·5798 (5)	3·30 (6)
C(4a)	0·3598 (3)	0·58623 (7)	0·6853 (4)	2·34 (5)
C(5)	0·3310 (4)	0·54555 (7)	0·6139 (4)	2·43 (5)
C(6)	0·4685 (4)	0·51816 (7)	0·6178 (4)	2·48 (5)
C(6a)	0·4336 (4)	0·47842 (8)	0·5340 (4)	3·18 (6)
C(7)	0·6199 (4)	0·53061 (7)	0·6818 (4)	2·52 (5)
C(8)	0·6479 (4)	0·57317 (7)	0·7460 (4)	2·49 (5)
C(8a)	0·5084 (4)	0·59900 (7)	0·7421 (4)	2·43 (5)
C(9)	0·4956 (4)	0·64113 (7)	0·8101 (4)	2·45 (5)
C(9a)	0·3073 (4)	0·64956 (7)	0·7908 (4)	2·74 (5)
C(9b)	0·0709 (5)	0·6542 (1)	0·9929 (6)	5·57 (9)
C(10)	0·6157 (4)	0·67004 (7)	0·7236 (4)	2·97 (5)
C(10a)	0·7331 (5)	0·73205 (8)	0·8044 (5)	4·34 (7)
C(11)	0·8021 (4)	0·46978 (7)	0·7137 (4)	2·70 (5)
C(12)	0·6926 (4)	0·44286 (8)	0·7917 (4)	3·13 (5)
C(13)	0·7435 (4)	0·40409 (8)	0·8157 (4)	3·08 (5)
C(14)	0·9013 (4)	0·39220 (7)	0·7708 (4)	2·64 (5)
C(15)	1·0109 (4)	0·41898 (8)	0·6957 (4)	2·01 (5)
C(16)	0·9613 (4)	0·45772 (8)	0·6664 (4)	2·84 (5)
OW	0·7357 (5)	0·28542 (8)	0·7909 (4)	7·58 (8)

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°)

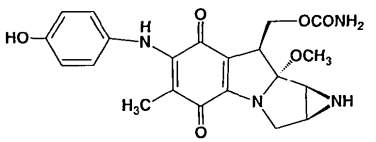
O(5)—C(5)	1·235 (4)	C(2)—C(3)	1·505 (4)
O(8)—C(8)	1·225 (4)	C(4a)—C(5)	1·493 (3)
O(9a)—C(9a)	1·420 (4)	C(4a)—C(8a)	1·342 (4)
O(9a)—C(9b)	1·445 (5)	C(5)—C(6)	1·445 (4)
O(10)—C(10)	1·439 (3)	C(6)—C(6a)	1·509 (4)
O(10)—C(10a)	1·333 (4)	C(6)—C(7)	1·377 (4)
O(10a)—C(10a)	1·211 (5)	C(7)—C(8)	1·534 (3)
O(14)—C(14)	1·381 (3)	C(8)—C(8a)	1·424 (4)
N(1)—C(1)	1·485 (4)	C(8a)—C(9)	1·517 (3)
N(1)—C(2)	1·472 (4)	C(9)—C(9a)	1·550 (4)
N(4)—C(3)	1·472 (5)	C(9)—C(10)	1·519 (4)
N(4)—C(4a)	1·364 (3)	C(11)—C(12)	1·395 (5)
N(4)—C(9a)	1·483 (3)	C(11)—C(16)	1·392 (4)
N(7)—C(7)	1·355 (4)	C(12)—C(13)	1·386 (4)
N(7)—C(11)	1·414 (3)	C(13)—C(14)	1·375 (4)
N(10a)—C(10a)	1·330 (5)	C(14)—C(15)	1·384 (5)
C(1)—C(2)	1·484 (4)	C(15)—C(16)	1·388 (4)
C(1)—C(9a)	1·513 (4)		

C(9a)—O(9a)—C(9b)	116·6 (3)	C(7)—C(8)—C(8a)	116·9 (2)
C(10)—O(10)—C(10a)	118·1 (2)	C(4a)—C(8a)—C(8)	120·9 (2)
C(1)—N(1) C(2)	60·3 (3)	C(4a)—C(8a)—C(9)	110·4 (2)
C(3)—N(4)—C(4a)	126·1 (2)	C(8)—C(8a)—C(9)	128·5 (3)
C(3)—N(4)—C(9a)	113·8 (3)	C(8a)—C(9)—C(9a)	102·0 (2)
C(4a)—N(4)—C(9a)	109·2 (2)	C(8a)—C(9)—C(10)	114·9 (2)
C(7)—N(7)—C(11)	133·0 (2)	C(9a)—C(9)—C(10)	117·7 (2)
N(1)—C(1)—C(2)	59·5 (3)	O(9a)—C(9a)—N(4)	112·5 (2)
N(1)—C(1)—C(9a)	114·3 (2)	O(9a)—C(9a)—C(1)	110·8 (2)
C(2)—C(1)—C(9a)	109·3 (2)	O(9a)—C(9a)—C(9)	105·4 (2)
N(1)—C(2)—C(1)	60·3 (3)	N(4)—C(9a)—C(1)	102·1 (2)
N(1)—C(2)—C(3)	111·7 (3)	N(4)—C(9a)—C(9)	105·2 (3)
C(1)—C(2)—C(3)	108·9 (3)	C(1)—C(9a)—C(9)	120·8 (2)
N(4)—C(3)—C(2)	103·2 (2)	O(10)—C(10)—C(9)	106·7 (2)
N(4)—C(4a)—C(5)	123·6 (2)	O(10)—C(10a)—O(10a)	123·6 (4)
N(4)—C(4a)—C(8a)	113·2 (2)	O(10)—C(10a)—N(10a)	111·2 (3)
C(5)—C(4a)—C(8a)	123·2 (2)	O(10a)—C(10a)—N(10a)	125·2 (3)
O(5)—C(5)—C(6)	123·1 (2)	N(7)—C(11)—C(12)	122·8 (3)
C(4a)—C(5)—C(6)	117·7 (2)	N(7)—C(11)—C(16)	117·1 (2)
C(5)—C(6)—(6a)	114·8 (2)	C(12)—C(11)—C(16)	119·9 (2)
C(5)—C(6)—C(7)	119·4 (2)	C(11)—C(12)—C(13)	119·0 (3)
C(6a)—C(6)—C(7)	125·6 (3)	C(12)—C(13)—C(14)	121·2 (3)
N(7)—C(7)—C(6)	129·2 (2)	O(14)—C(14)—C(13)	118·4 (2)
N(7)—C(7)—C(8)	109·0 (2)	O(14)—C(14)—C(15)	121·8 (3)
C(6)—C(7)—C(8)	121·8 (2)	C(13)—C(14)—C(15)	119·9 (2)
O(8)—C(8)—C(7)	118·7 (2)	C(14)—C(15)—C(16)	119·9 (3)
O(8)—C(8)—C(8a)	124·3 (2)	C(11)—C(16)—C(15)	120·1 (3)

C(9b)—O(9a)—C(9a)—C(9)	−165·1 (3)	C(10a)—O(10)—C(10)—C(9)	−167·6 (3)
C(10)—O(10)—C(10a)—O(10a)	10·2 (6)	C(11)—N(7)—C(7)—C(6)	−27·6 (5)
C(7)—N(7)—C(11)—C(12)	−25·5 (5)	C(9a)—C(9)—C(10)—O(10)	91·2 (3)

46·36 (7)°. Two quinone O atoms deviate from the least-squares planes of the quinone ring on the same side of the plane.

Experimental. The title compound (I) was synthesized from mitomycin C (Imai, Ashizawa, Urakawa, Morimoto & Nakamura, 1980). Crystal dimensions



(I)

0·40 × 0·30 × 0·20 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions from setting angles of 24 independent reflections with $35·0 \le \theta \le 43·0°$. 2577 reflections surveyed in the range $1 \le 2\theta \le 150°$ (Cu $K\alpha$); $0 \le h \le 10$, $0 \le k \le 42$, $0 \le l \le 9$; 2474 reflections were unique, 1923 observed with $F > 3\sigma(F)$. Three reference reflections monitored periodically showed no significant variation in intensity. Absorption correction was not applied. Structure solved using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier-map recycling. Refinement using *SDP* package (Frenz, 1985), full-matrix least-squares refinement on F, with non-H atoms having anisotropic temperature factors. Most of the H atoms were located from difference Fourier syntheses and were refined with isotropic temperature parameters. $w = 4F_o^2/[\sigma^2(I_o) + (0·04I_o)^2]^{1/2}$/Lp, final $R = 0·041$, $wR = 0·053$, $S = 1·75$, maximum shift/e.s.d. in the final least-squares cycle of 0·30, maximum peak in the final difference map 0·30 (6) e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Final fractional coordinates and equivalent B values are listed in Table 1. Distances, angles and selected torsion angles are listed in Table 2.*

Fig. 1 shows a stereoview of the molecule with the atomic numbering.

* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53006 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

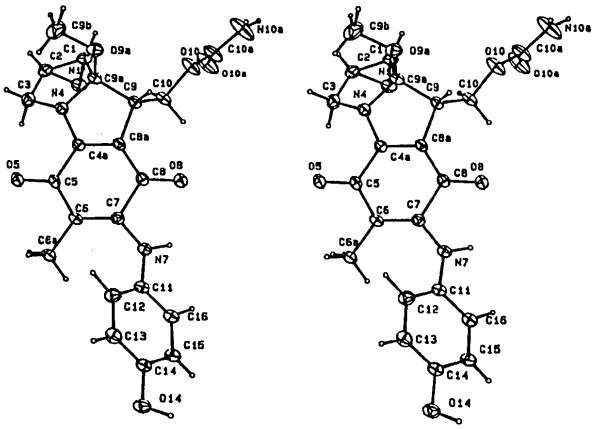


Fig. 1. *ORTEP*II (Johnson, 1976) drawing showing 30% ellipsoids.

Related literature. Mitomycin C is one of the successful potent anticancer antibiotics in clinical use today. In order to screen for derivatives more active than mitomycin C, several 7-*N*-alkylated derivatives of mitomycin C were synthesized (Imai, Ashizawa, Urakawa, Morimoto & Nakamura, 1980). M83, a 7-*N*-(*p*-hydroxylphenyl) derivative, showed more potent antitumor activity against the ascitic form of P-388 leukemia than mitomycin C (Kobayashi, Inaba, Tsukagoshi, Sakurai, Imai & Morimoto, 1981). The crystal structure of mitomycin C itself has been determined for the two different crystal forms (Arora, 1979; Ogawa, Nomura, Fujiwara & Tomita, 1979). The absolute configuration was determined by the Bijvoet method (Shirahata & Hirayama, 1983).

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Structure of 10,10-Dihydro-10-deoxo-10a-methyl-10a-aza-10a-homoerythronolide A

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Abstract. $C_{22}H_{43}NO_7$, $M_r = 433{\cdot}58$, monoclinic, $P2_1$, $a = 20{\cdot}427\ (5)$, $b = 6{\cdot}995\ (2)$, $c = 8{\cdot}372\ (2)$ Å, $\beta = 97{\cdot}60\ (1)°$, $V = 1185{\cdot}7$ Å^3, $Z = 2$, $D_m = 1{\cdot}22$ (by flotation), $D_x = 1{\cdot}218$ g cm^{-3}, λ(Mo $K\alpha$) = 1·5418 Å, $\mu = 6{\cdot}45$ cm^{-1}, $F(000) = 476$, $T = 293$ K, $R = 0{\cdot}039$ for 1709 reflections. The geometry and dimensions of the 15-membered aglycone ring are not significantly different from those found in the analogous azaerythronolide A derivatives. The presence of the methyl group linked to the N atom does not significantly influence the structure. The distances between O(61)···O(131^{i}) [(i) x, y, $1+z$] of 2·821 (5) and O(61)···O(141^{ii}) [(ii) x, $1+y$, $1+z$] of 2·861 (5) Å suggest the existence of intermolecular hydrogen bonding while the O(71)···N(11) distance of 2·816 (5) Å suggests intramolecular hydrogen bonding.

Experimental. As part of a wider investigation of a novel 15-membered macrolide antibiotic the title

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