Fig. 1. Atomic numbering scheme of molecule *A* of the title compound.

Bond lengths, bond angles and some selected torsion angles are given in Table 2.

Related literature. The method of preparation of this possible anti-AIDS compound has been described by Van Aerschot, Everaert, Balzarini, Augustyns, Jie, Janssen, Peeters, Blaton, De Ranter, De Clercq & Herdewijn (1990). There is a close conformational similarity between the title compound and 3′-azido-3′-deoxythymidine (Dyer, Low, Tollin, Wilson & Howie, 1988).

The authors wish to thank J. P. Van Cuyck for his help in preparing the figure.

References

Dyer, I., Low, N., Tollin, P., Wilson, H. R. & Howie, R. A. (1988). *Acta Cryst.* C44, 767–769.
Enraf–Nonius (1982). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.
Everaert, D. H., Peeters, O. M., Blaton, N. M., De Ranter, C. J., Van Aerschot, A. & Herdewijn, P. (1990). *Acta Cryst.* C46, 1936–1938.
IUPAC–IUB Joint Commission on Biochemical Nomenclature (1983). *Pure Appl. Chem.* 55, 1273–1280.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
Nardelli, M. (1983). *Comput. Chem.* 7, 95–98.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.
Van Aerschot, A., Everaert, D., Balzarini, J., Augustyns, K., Jie, L., Janssen, G., Peeters, O., Blaton, N., De Ranter, C., De Clercq, E. & Herdewijn, P. (1990). *J. Med. Chem.* 33, 1833–1839.

Structural Studies of Mitomycins. VI. Structure of Mitiromycin Monohydrate

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Abstract. 1,4b,5,5a,6,11b-Hexahydro-10-methoxy-5,9-dimethylazirino[2′,3′:3,4]pyrrolo[1,2-a][1,3]oxazino[4,5-b]indole-3,8,11(4H)-trione monohydrate, $C_{16}H_{17}N_3O_5.H_2O$, $M_r = 349.35$, orthorhombic, $P2_12_12_1$, $a = 11.770$ (1), $b = 14.276$ (2), $c = 9.488$ (1) Å, $V = 1594.3$ (5) Å^3, $Z = 4$, $D_x = 1.46$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.5405$ Å, $\mu = 8.1$ cm^{-1}, $F(000) = 736$, $T = 293$ K, $wR = 0.044$ for 1650 observed reflections with $I > 3\sigma(I)$. The quinone ring adopts a flat envelope conformation in which one carbonyl group deviates from the plane defined by the other five atoms in the ring, C by 0.065 (2) and O by 0.203 (2) Å. The dihedral angle between the quinone and the aziridine rings is 56.7 (1)°. The oxazinone ring has a boat conformation. The stereochemistries of the two junctions where the oxazinone ring is fused are determined as α and α. The absolute configuration of the molecule was suggested by referring to that of 7-*p*-bromoanilino-7-demethoxymitomycin B [Hirayama & Shirahata (1987). *Acta Cryst.* B43, 555–559].

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Table 1. *Positional parameters and equivalent isotropic temperature factors of the non-H atoms with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq} (Å^2)
O(5)	0·3786 (1)	0·9460 (1)	0·1764 (2)	4·14 (3)
O(7)	0·6134 (1)	0·6817 (1)	0·1718 (2)	4·24 (3)
O(8)	0·7345 (2)	0·7665 (1)	−0·0453 (2)	5·16 (4)
O(10)	0·8161 (1)	1·0496 (1)	−0·0312 (2)	3·34 (3)
O(11a)	0·7966 (1)	1·2000 (1)	0·0193 (2)	2·90 (3)
N(1)	0·4447 (1)	0·9944 (1)	−0·3133 (2)	2·80 (3)
N(4)	0·5124 (1)	1·0324 (1)	−0·0433 (2)	2·44 (3)
N(11a)	0·6668 (1)	1·1321 (1)	−0·1251 (2)	2·61 (3)
C(1)	0·5277 (1)	1·0689 (1)	−0·2850 (2)	2·52 (3)
C(1a)	0·4099 (2)	0·9895 (2)	−0·4616 (3)	4·04 (4)
C(2)	0·4075 (2)	1·0807 (1)	−0·2404 (3)	2·99 (4)
C(3)	0·4001 (2)	1·0697 (1)	−0·0834 (3)	3·19 (4)
C(4a)	0·5315 (2)	0·9464 (1)	0·0156 (2)	2·26 (3)
C(5)	0·4603 (2)	0·9044 (1)	0·1291 (2)	2·60 (3)
C(6a)	0·4244 (2)	0·7678 (2)	0·2947 (2)	3·62 (4)
C(6)	0·4953 (2)	0·8115 (1)	0·1823 (2)	2·72 (3)
C(7a)	0·7271 (2)	0·6574 (2)	0·2084 (3)	4·57 (5)
C(7)	0·5890 (2)	0·7701 (1)	0·1283 (2)	3·00 (4)
C(8)	0·6578 (2)	0·8114 (1)	0·0096 (2)	2·91 (3)
C(8a)	0·6251 (2)	0·9034 (1)	−0·0360 (2)	2·36 (3)
C(9)	0·6732 (1)	0·9588 (1)	−0·1572 (2)	2·23 (3)
C(9a)	0·5989 (1)	1·0490 (1)	−0·1557 (2)	2·28 (3)
C(10)	0·7977 (2)	0·9844 (1)	−0·1445 (3)	3·01 (4)
C(11a)	0·7598 (2)	1·1319 (1)	−0·0420 (2)	2·73 (3)
O(W)	0·4900 (1)	0·7894 (1)	0·6786 (2)	4·02 (3)

Table 2. *Selected bond lengths (Å) and angles (°)*

O(5)—C(5)	1·217 (2)	C(2)—C(3)	1·501 (3)
O(8)—C(8)	1·223 (3)	C(4a)—C(5)	1·491 (3)
O(10)—C(10)	1·438 (3)	C(4a)—C(8a)	1·353 (2)
O(10)—C(11a)	1·353 (2)	C(5)—C(6)	1·478 (3)
O(11a)—C(11a)	1·213 (3)	C(6)—C(7)	1·354 (3)
N(4)—C(3)	1·475 (2)	C(7)—C(8)	1·507 (3)
N(4)—C(4a)	1·367 (2)	C(8)—C(8a)	1·435 (2)
N(4)—C(9a)	1·495 (2)	C(8a)—C(9)	1·506 (3)
N(11a)—C(9a)	1·459 (2)	C(9)—C(9a)	1·557 (2)
N(11a)—C(11a)	1·349 (3)	C(9)—C(10)	1·517 (2)
C(1)—C(9a)	1·513 (3)		

C(10)—O(10)—C(11a)	115·6 (2)	C(7)—C(8)—C(8a)	116·1 (2)
C(3)—N(4)—C(4a)	125·3 (1)	C(4a)—C(8a)—C(8)	121·6 (2)
C(3)—N(4)—C(9a)	111·7 (2)	C(4a)—C(8a)—C(9)	110·1 (1)
C(4a)—N(4)—C(9a)	108·9 (1)	C(8)—C(8a)—C(9)	127·6 (2)
C(9a)—N(11a)—C(11a)	124·0 (2)	C(8a)—C(9)—C(9a)	102·5 (1)
C(2)—C(1)—C(9a)	108·6 (2)	C(8a)—C(9)—C(10)	115·4 (2)
C(1)—C(2)—C(3)	109·1 (2)	C(9a)—C(9)—C(10)	110·0 (1)
N(4)—C(3)—C(2)	104·0 (2)	N(4)—C(9a)—N(11a)	111·1 (1)
N(4)—C(4a)—C(5)	124·3 (2)	N(4)—C(9a)—C(1)	103·4 (1)
N(4)—C(4a)—C(8a)	113·2 (2)	N(4)—C(9a)—C(9)	104·9 (1)
C(5)—C(4a)—C(8a)	122·4 (2)	N(11a)—C(9a)—C(1)	108·2 (1)
O(5)—C(5)—C(4a)	121·0 (2)	N(11a)—C(9a)—C(9)	111·6 (1)
O(5)—C(5)—C(6)	122·2 (2)	C(1)—C(9a)—C(9)	117·3 (1)
C(4a)—C(5)—C(6)	116·9 (2)	O(10)—C(10)—C(9)	111·2 (2)
C(5)—C(6)—C(7)	119·3 (2)	O(10)—C(11a)—O(11a)	119·0 (2)
C(6)—C(7)—C(8)	123·4 (2)	O(10)—C(11a)—N(11a)	116·3 (2)
O(8)—C(8)—C(7)	120·6 (2)	O(11a)—C(11a)—N(11a)	124·7 (2)
O(8)—C(8)—C(8a)	123·3 (2)		

Experimental. Crystal dimensions 0·40 × 0·30 × 0·20 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions from setting angles of 25 independent reflections with $35 \leq \theta \leq 49°$. ω–2θ scans. 1952 reflections surveyed in the range $1 \leq 2\theta \leq 150°$; $0 \leq h \leq 13$, $0 \leq k \leq 16$, $0 \leq l \leq 12$; 1831 reflections were unique, 1650 observed with $I > 3\sigma(I)$. Three reference reflections monitored periodically showed no significant variation in intensity. Absorption correction was not applied. Secondary-extinction correction (Zachariasen, 1963) was made (final refined extinction coefficient $5·1 \times 10^{-6}$). Structure solved using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier-map recycling. Refinement using the *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 refined isotropically. $w = 4F_o^2/[\sigma(I_o)^2 + (0·04I_o)^2]^{1/2}$/Lp, final $R = 0·030$, $wR = 0·044$, $S = 1·56$, maximum shift/e.s.d in the final least-squares cycle 0·08, maximum peak in
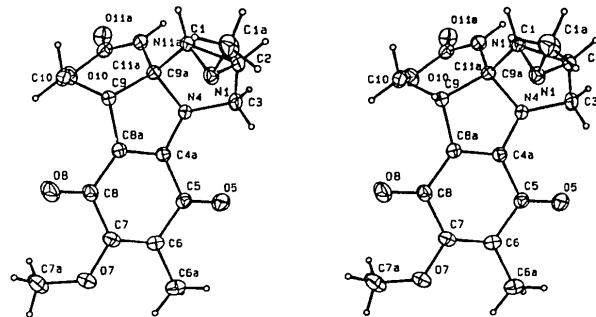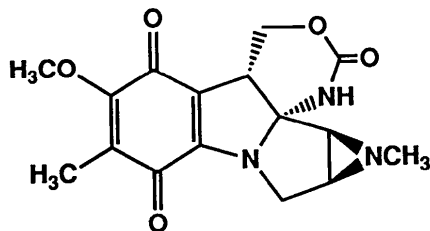

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of mitiromycin with thermal ellipsoids at 30% probability.

the final difference map 0·18 (3) e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final fractional coordinates and equivalent B values are listed in Table 1.* Selected bond lengths and angles are shown in Table 2. An *ORTEP*II (Johnson, 1976) drawing of the molecule with the atomic numbering is shown in Fig.1.

Related literature. Mitiromycin, which was isolated from the fermentation broth of *Streptomyces* (Lefemine, Barbatschi, Hausemann, Zbinovsky,

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* 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 53529 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Monnikendam, Adam & Bohomos, 1962), is a member of the mitomycin family. The structure possesses the quite unique skeleton of an oxazinone ring fused at the 9 and 9a positions (Morton, Van Lear & Fulmer, 1970). The compound was synthesized recently (Kono, Kasai, Shirahata & Hirayama, 1990). The absolute configuration was determined by referring to that of 7-p-bromoanilino-7-demethoxy-mitomycin B (Hirayama & Shirahata, 1987).

References

Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package.* Version 1.1. Enraf–Nonius, Delft, The Netherlands.

Hirayama, N. & Shirahata, K. (1987). *Acta Cryst.* **B43**, 555–559.
Johnson, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kono, M., Kasai, M., Shirahata, K. & Hirayama, N. (1990). *J. Antibiot.* In the press.
Lefemine, D. V., Barbatschi, F., Hausemann, W. K., Zbinovsky, V., Monnikendam, P., Adam, J. & Bohomos, N. (1962). *J. Am. Chem. Soc.* **84**, 3184–3185.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Morton, G. O., Van Lear, G. E. & Fulmer, W. (1970). *J. Am. Chem. Soc.* **92**, 2588–2590.
Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Structure of Cholest-5-en-3-one

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Abstract. $C_{27}H_{44}O$, $M_r = 384.7$, orthorhombic, $P2_12_12_1$, $a = 11.109 (1)$, $b = 11.213 (2)$, $c = 19.104 (2)$ Å, $V = 2379.4 (5)$ Å^3, $Z = 4$, $D_x = 1.07$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.44$ mm^{-1}, $F(000) = 856$, $R = 0.069$, $wR = 0.068$ for 2600 unique observed reflections with $F_o > 4\sigma(F_o)$. The A and C rings have chair conformations and the B and D rings assume half-chair conformations. The cholesterol side chain is fully extended with a *gauche, trans* conformation of the terminal C26 and C27 methyl groups.

Experimental. Irregularly shaped crystal with dimensions $0.7 \times 0.7 \times 0.5$ mm. Nonius CAD-4 diffractometer, space group determined from Weissenberg photographs, cell dimensions from 25 centered reflections $(25 < 2\theta < 32°)$, Cu $K\alpha$ radiation, Ni filtered, scan width $(1.2 + 0.14 \tan\theta)°$, $\theta_{max} = 75°$, $0 < h < 13$, $0 < k < 13$, $0 < l < 23$, 2704 unique reflections measured using θ–2θ scan mode. Three standard reflections $(\overline{1}48, 0,\overline{4},11, 6\overline{35})$ were measured every 1 h and varied in intensity by less than 3% during the data collection.

Positions of all non-H atoms were found using *SHELXS*86 (Sheldrick, 1986) and refined anisotropically by full-matrix least squares on F_o values, using

2600 reflections for which $F_o > 4\sigma(F_o)$ by means of *SHELX*76 (Sheldrick, 1976). All H atoms were placed geometrically and refined as 'riding groups' (Sheldrick, 1976). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.069$, $wR = 0.068$, $S = 2.988$, $w = 1/\sigma^2$, $(\Delta/\sigma)_{max} = 0.1$. Final difference map showed maximum negative and positive peaks of -0.25 and 0.30 e Å^{-3}.

Atomic fractional coordinates and equivalent thermal parameters for all non-H atoms are given in Table 1. Bond lengths and valency angles are listed in Table 2.* A stereoplot (Johnson, 1970) of the molecule with the atomic numbering and the packing diagram (Motherwell, 1976) are given in Figs. 1 and 2, respectively.

Related literature. Observed bond lengths and bond angles are in good agreement (within three e.s.d.'s) with corresponding values of 'the average' 5-ene

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* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53478 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.