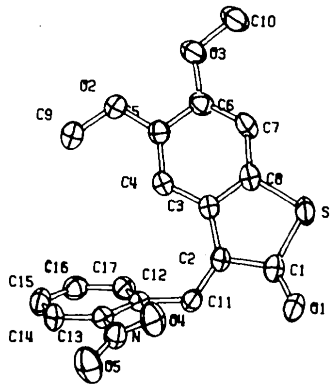

Fig. 1. An *ORTEP* diagram with 50% probability thermal ellipsoids, H atoms not included.

using *SHELX*76 (Sheldrick, 1976). H positions located from difference map but refined riding on C atoms at 0·98 Å. Least-squares refinement minimized $\sum w\Delta^2$, $1/w = \sigma^2(F_o) + 0{\cdot}00095|F_o|^2$. Non-H atoms anisotropic. $|\Delta/\sigma|_{\max} = 0{\cdot}003$. $\Delta\rho_{\max}/\Delta\rho_{\min}$, 0·14/−0·19 e Å^{-3}. *R*/*wR*, 0·0275/0·0345 for 1553 observed data; *R*/*wR*, 0·0311/0·0371 for all data. Atomic scattering factors from *SHELX*76. The final atomic parameters of the non-H atoms are given in Table 1.*

Bond distances and angles are given in Table 2. A view of the molecule is shown in Fig. 1 (Johnson, 1965).

Related literature. The structures of two other heterocyclic analogues of benzothiazinone have been published (Salem, Filippakis, Hountas & Terzis, 1986).

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* Lists of observed and calculated structure factors, anisotropic thermal parameters of the non-H atoms and positional and isotropic thermal parameters of the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44407 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1988). C**44**, 386–388

Structure of 9-Methoxy-11-demethylellipticine

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(*Received* 15 *May* 1987; *accepted* 9 *October* 1987)

Abstract. 9-Methoxy-5-methyl-6*H*-pyrido[4,3-*b*]-carbazole, $C_{17}H_{14}N_2O$, $M_r = 262{\cdot}31$, monoclinic, $P2_1/c$, $a = 6{\cdot}326$ (2), $b = 23{\cdot}535$ (6), $c = 9{\cdot}141$ (3) Å, $\beta = 99{\cdot}87$ (3)°, $V = 1340{\cdot}8$ (16) Å^3, $Z = 4$, $D_x = 1{\cdot}299$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}571$ mm^{-1}, $F(000) = 552$, $R = 0{\cdot}066$ for 1487 observed reflections at 298 K. The geometry and dimensions of the planar ring system are not significantly different from those of ellipticine and its derivatives. Crystal packing is also very similar: the molecules are stacked along the *a* axis and weakly hydrogen bonded, N(6)—H···N(2) 2·999 (4) Å. The presence or absence of a substituent at positions 9 or 11 does not significantly influence the structure.

Experimental. Two light-yellow crystals of the title compound (Gouyette, Reynaud, Sadet, Baaillargé, Gansser, Cros, Le Goffic, Le Pecq, Paoletti & Viel, 1980) were obtained with difficulty from methanol solution. Crystal dimensions: 0·25 × 0·12 × 0·10 mm. Enraf–Nonius CAD-4 diffractometer; graphite-monochromated Cu *K*α radiation, ω–2θ scan. $\theta_{\max} = 60°$ $(-7 \le h \le 7,\ 0 \le k \le 26,\ 0 \le l \le 10)$. Lattice parameters from least-squares refinement of 25

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *for non*-H *atoms and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	−313 (5)	7449 (2)	5140 (4)	45 (2)
N(2)	−692 (4)	7978 (1)	5406 (3)	48 (2)
C(3)	534 (5)	8375 (2)	4838 (4)	50 (2)
C(4)	2109 (5)	8235 (1)	4030 (4)	46 (2)
C(5)	4217 (5)	7487 (1)	2973 (3)	37 (2)
N(6)	5977 (4)	6627 (1)	2149 (3)	44 (2)
C(7)	6716 (6)	5598 (2)	1717 (4)	61 (3)
C(8)	6069 (6)	5060 (2)	1945 (5)	67 (3)
C(9)	4332 (6)	4949 (1)	2700 (4)	59 (3)
C(10)	3253 (5)	5394 (1)	3215 (4)	51 (2)
C(11)	1559 (5)	6670 (1)	4125 (3)	42 (2)
C(12)	1260 (5)	7243 (1)	4323 (3)	39 (2)
C(13)	2560 (4)	7664 (1)	3755 (3)	37 (2)
C(14)	4479 (4)	6912 (1)	2827 (3)	36 (2)
C(15)	3166 (4)	6496 (1)	3376 (3)	39 (2)
C(16)	3900 (5)	5948 (1)	2980 (3)	41 (2)
C(17)	5638 (5)	6050 (1)	2221 (4)	44 (2)
C(18)	5579 (6)	7909 (2)	2318 (4)	47 (2)
O(19)	3857 (5)	4387 (1)	2836 (4)	83 (2)
C(20)	2167 (8)	4254 (2)	3632 (7)	76 (4)

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

C(1)–N(2)	1·300 (5)	C(8)–C(9)	1·418 (6)
C(1)–C(12)	1·427 (5)	C(9)–C(10)	1·377 (5)
N(2)–C(3)	1·372 (5)	C(9)–O(19)	1·366 (4)
C(3)–C(4)	1·378 (5)	C(10)–C(16)	1·394 (4)
C(4)–C(13)	1·406 (5)	C(11)–C(12)	1·379 (4)
C(5)–C(13)	1·429 (4)	C(11)–C(15)	1·381 (4)
C(5)–C(14)	1·371 (4)	C(12)–C(13)	1·440 (4)
C(5)–C(18)	1·505 (5)	C(14)–C(15)	1·431 (4)
N(6)–C(14)	1·390 (4)	C(15)–C(16)	1·437 (4)
N(6)–C(17)	1·377 (4)	C(16)–C(17)	1·418 (5)
C(7)–C(8)	1·359 (6)	O(19)–C(20)	1·427 (7)
C(7)–C(17)	1·385 (5)		
N(2)–C(1)–C(12)	126·2 (3)	C(11)–C(12)–C(13)	121·8 (3)
C(1)–N(2)–C(3)	116·6 (3)	C(4)–C(13)–C(5)	123·9 (3)
N(2)–C(3)–C(4)	123·2 (3)	C(4)–C(13)–C(12)	116·6 (3)
C(3)–C(4)–C(13)	120·8 (3)	C(5)–C(13)–C(12)	119·6 (3)
C(13)–C(5)–C(14)	116·6 (3)	C(5)–C(14)–N(6)	128·5 (3)
C(13)–C(5)–C(18)	121·7 (3)	C(5)–C(14)–C(15)	123·7 (3)
C(14)–C(5)–C(18)	121·7 (3)	N(6)–C(14)–C(15)	107·9 (2)
C(14)–N(6)–C(17)	109·3 (3)	C(11)–C(15)–C(14)	119·5 (3)
C(8)–C(7)–C(17)	119·2 (4)	C(11)–C(15)–C(16)	133·4 (3)
C(7)–C(8)–C(9)	121·7 (4)	C(14)–C(15)–C(16)	107·1 (3)
C(8)–C(9)–C(10)	119·8 (3)	C(10)–C(16)–C(15)	133·1 (3)
C(8)–C(9)–O(19)	115·0 (3)	C(10)–C(16)–C(17)	120·5 (3)
C(10)–C(9)–O(19)	125·2 (4)	C(15)–C(16)–C(17)	106·4 (3)
C(9)–C(10)–C(16)	118·9 (3)	N(6)–C(17)–C(7)	130·7 (3)
C(12)–C(11)–C(15)	118·9 (3)	N(6)–C(17)–C(16)	109·3 (3)
C(1)–C(12)–C(11)	121·6 (3)	C(7)–C(17)–C(16)	120·0 (3)
C(1)–C(12)–C(13)	116·6 (3)	C(9)–O(19)–C(20)	117·0 (3)

reflections with $15 < \theta < 43°$. 1989 unique reflections collected, 502 with $F < 4\sigma(F)$ considered unobserved [$\sigma(I)$ from counting statistics]. Three standard reflections showed no significant change in intensity. Lorentz and polarization corrections applied, no absorption correction. All non-H atoms located by direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Least-squares refinement followed by difference Fourier synthesis led to location of all H atoms, except on methyl C(18). Full-matrix least-squares refinement (on F's) with anisotropic thermal parameters on all non-H atoms (H atoms isotropic) gave $R = 0{\cdot}066$ and $wR = 0{\cdot}066$ with $w = 1/|\sigma^2(F) + 0{\cdot}001F^2|$, $S = 1{\cdot}60$, featureless analysis of variance as a function of $|F|$ and $\sin\theta$, $(\Delta/\sigma)_{max} = 0{\cdot}23$ for non-H atoms, $(\Delta/\sigma)_{max} = 1$ for an H atom on methyl C(18). Max. and min. values in final difference Fourier map 0·26, −0·40 e Å^{-3}. The peaks around C(18) and the poor quality of the crystal caused the rather high final R value. Computer programs: *SHELX*76 for refinement (Sheldrick, 1976), with scattering factors for C, N, O, H included in the program; *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) for geometry calculations; *ORTEP*II (Johnson, 1976) for drawings. Fig. 1 shows the molecule and numbering scheme. A projection of the structure down the a axis is given in Fig. 2. The atomic parameters and bond lengths and angles are listed in Tables 1 and 2 respectively.*

Related literature. The results show that in 9-methoxy-11-demethylellipticine the intermolecular distances and

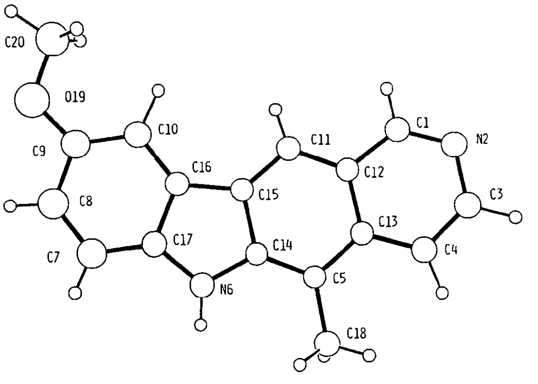

Fig. 1. Molecular structure of 9-methoxy-11-demethylellipticine showing the atom numbering.

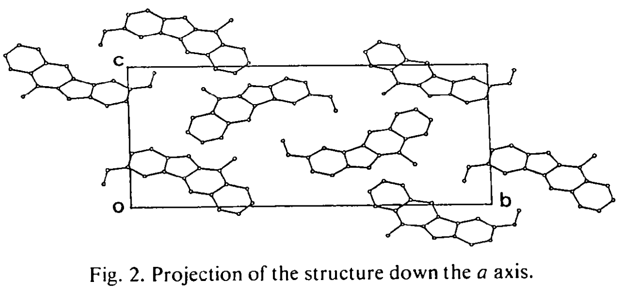

Fig. 2. Projection of the structure down the a axis.

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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 44434 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the values of the angles are comparable with those of ellipticine (Courseille, Busetta & Hospital, 1974) and its derivatives (Aggarwal, Neidle & Sainsbury, 1983; Courseille, Busetta & Hospital, 1982; Kuroda & Sainsbury, 1984; Garlich, Kaiser & Schlemper, 1984, and references therein). It follows that a substituent in position 9 or 11 only exerts a minor influence on the molecular conformation, which is not surprising considering the rigidity of such types of aromatic molecule.

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Acta Cryst. (1988). C**44**, 388–389

Structure of Lumi-arteannuin B

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(*Received* 27 *July* 1987; *accepted* 25 *September* 1987)

Abstract. $C_{15}H_{20}O_3$, $M_r = 248{\cdot}25$, monoclinic, $P2_1$, $a = 6{\cdot}970\,(1)$, $b = 14{\cdot}004\,(2)$, $c = 14{\cdot}124\,(2)$ Å, $\beta = 104{\cdot}94\,(1)°$, $V = 1332{\cdot}0\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}238$ Mg m^{-3}, $\lambda(\text{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}65$ mm^{-1}, $F(000) = 536$, $T = 295$ K, final $R = 0{\cdot}044$, $wR = 0{\cdot}042$ for 1473 independent observed reflections. Formed by rearrangement upon irradiation of arteannuin B. Both molecules in the asymmetric unit have the same conformation. All three O atoms lie on the same side of the best plane through the nine-membered ring and consequently there are close O···O intramolecular approaches (2·82–3·24 Å). The nine-membered ring is flattened on one end with C1—C2···C8—C9 forming a plane. The epoxy ring is at an angle of 105° to this plane.

Experimental. Colorless, 0·06 × 0·32 × 0·18 mm crystal, from benzene/cyclohexane. Isolated by N. Roth of the Division of Experimental Therapeutics of the Walter Reed Army Institute of Research, m.p. = 437–439 K. Automated Nicolet $R3m$ diffractometer with incident-beam graphite monochromator; 25 centered reflections within $26 < 2\theta < 60°$ used for determining cell parameters. Data corrected for Lorentz and polarization effects, absorption ignored. $2\theta_{\max} = 110°$; range of hkl: $0 \le h \le 7$, $0 \le k \le 14$, $-15 \le l < 13$. Standards, $1\bar{1}0$, 040, 004, monitored every 60 reflections with random variation 2·4% over data collection, θ–2θ mode, scan width $(2{\cdot}0 + \Delta_{\alpha1,\alpha2})$, scan rate a function of count rate (6° min^{-1} minimum, 60° min^{-1} maximum); 2041 reflections measured, 1763 unique, $R_{\text{int}} = 0{\cdot}019$, 1473 observed $[F_o > 3\sigma(F_o)]$.

Structure solved by direct methods. The least-squares

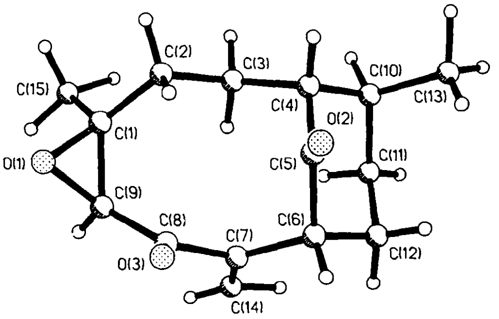

Fig. 1. Perspective drawing of the results of the X-ray study on lumi-arteannuin B. Only one of the two molecules in the asymmetric unit is shown. Numbering of the second molecule was set by adding 20 to the number of the corresponding atom in the illustrated molecule.

0108-2701/88/020388-02$03.00