We are indebted to J. C. Portheine for some of the tests of crystal quality.

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16-Demethoxycarbonyl-20-epiervatamine

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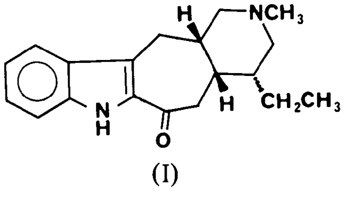
Abstract. $C_{19}H_{25}N_2OBr$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}212$ (4), $b = 13{\cdot}353$ (6), $c = 14{\cdot}217$ (7) Å, $Z = 4$, $M_r = 377{\cdot}1$, $d_x = 1{\cdot}43$ g cm^{-3}. Final R value: 0·078.

Introduction. The purpose of this investigation was to establish the molecular configuration of this alkaloid extracted from different species of *Hazunta* (Bui & Potier, unpublished). The X-ray analysis shows the compound to be 16-demethoxycarbonyl-20-epiervatamine (I) (Shafiee, Ahond, Bui, Langlois, Riche & Potier, 1976).



(I)

The stereochemistry of the related alkaloids ervatamine, epiervatamine and 19,20-dehydroervatamine has been recently determined (Husson, Langlois, Riche, Husson & Potier, 1973).

Preliminary photographs showed the crystals to be orthorhombic, space group $P2_12_12_1$. A crystal approximately 0·1 × 0·15 × 0·3 mm was used to measure the cell parameters and intensities on a Philips PW 1100 four-circle diffractometer. Data were collected to a maximum θ of 50° with graphite-monochromated Cu $K\alpha$ radiation. ($\lambda = 1{\cdot}5418$ Å, ω–2θ scan, scan width: 1·5°, scan speed: 0·12° s^{-1}; background was measured for 4 s on either side of the peak). Of the 2164 reflexions measured (hkl and $\bar{h}\bar{k}\bar{l}$), 2055 had intensities above $2\sigma(I)$ where $\sigma(I)$ was the standard deviation derived from counting statistics. No correction for absorption was made.

The structure was solved by the heavy-atom method and refined by full-matrix least squares (*ORFLS*: Busing, Martin & Levy, 1962) minimizing $\Sigma (F_o - |F_c|)/\sigma^2(F_o)$. The temperature factors were anisotropic for Br and isotropic for C, N and O. The H atoms were included in their calculated positions. The scattering factors used for C, N, O and Br^- were those of Doyle & Turner (1968) and for H, those of Stewart, Davidson & Simpson (1965). The component of the anomalous dispersion applied for Br was taken from *International Tables for X-ray Crystallography* (1968). All the observed reflexions (hkl and $\bar{h}\bar{k}\bar{l}$), which had been indexed according to a right-handed set of axes, were used. Two refinements were performed corresponding to the possible enantiomorphs. The R values were: $R(+) = 7{\cdot}8$, $R(-) = 8{\cdot}3$, $R_w(+) = 10{\cdot}3$, $R_w(-) = 10{\cdot}7\%$. Examination of several sensitive Bijvoet pairs indicated the correctness of the (+) enantiomorph.

Atomic coordinates and temperature factors are listed in Table 1, bond lengths and angles in Tables 2 and 3.*

Discussion. The molecule is shown in Fig. 1. H atoms on C(15) and C(16) are *cis*; the ethyl group on C(20) is α (absolute configuration 15*S*, 16*R* and 20*S*). The piperidine ring is in a chair conformation with substituents in equatorial positions. Torsion angles for the six- and seven-membered rings are in Table 3, and are compared with those for ervatamine (Riche, 1974). Fig. 2 shows Newman projections along C(15)—C(16). The α configuration of the ethyl group at C(20) (β in ervatamine) determines the conformation of ring *D* which adopts the sterically more favourable chair form. As a consequence, the configurations at N(4) in the two alkaloids are different. The methyl groups, both in equatorial positions, are β in ervatamine and α in the present compound. The indole nucleus is slightly bent along C(8)—C(13). The six- and five-membered rings are planar within $\pm 0{\cdot}04$ and $\pm 0{\cdot}09$ Å respectively. The angle between the normals to these planes is 1·4°.

Table 1. *Atomic parameters* ($\times 10^4$) *and standard deviations* (*in parentheses*)

	x	*y*	*z*	*B*
N(1)	1658 (10)	8026 (7)	8380 (6)	2·52 (20)
C(2)	1959 (12)	7755 (8)	9300 (7)	2·00 (21)
C(3)	2503 (14)	6758 (8)	9532 (8)	2·37 (24)
N(4)	1481 (11)	6866 (7)	13088 (7)	2·74 (21)
C(5)	2010 (13)	7695 (9)	12450 (8)	2·52 (23)
C(6)	1769 (14)	8581 (8)	10922 (7)	2·54 (24)
C(7)	1567 (12)	8529 (8)	9890 (7)	1·83 (21)
C(8)	1049 (13)	9317 (8)	9301 (8)	2·18 (23)
C(9)	509 (14)	10279 (9)	9499 (8)	2·82 (25)
C(10)	45 (14)	10852 (9)	8747 (10)	3·67 (26)
C(11)	134 (16)	10486 (10)	7817 (9)	3·92 (31)
C(12)	668 (14)	9561 (9)	7586 (9)	3·08 (26)
C(13)	1143 (12)	8972 (8)	8361 (8)	2·10 (24)
C(14)	2543 (12)	6465 (9)	10525 (8)	2·09 (22)
C(15)	1097 (12)	6696 (8)	11032 (7)	1·82 (22)
C(16)	1163 (11)	7722 (8)	11527 (7)	1·82 (21)
C(17)	2368 (17)	6824 (11)	14026 (10)	4·69 (35)
C(18)	−194 (16)	4714 (11)	10368 (10)	4·38 (32)
C(19)	593 (14)	4828 (9)	11301 (9)	3·58 (26)
C(20)	615 (13)	5893 (9)	11712 (8)	2·31 (24)
C(21)	1520 (14)	5908 (9)	12639 (9)	3·25 (27)
O	2835 (8)	6183 (6)	8900 (6)	3·21 (17)
Br	3204 (1)	7833 (1)	6257 (1)	*

* Anisotropic temperature factors: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$. $\beta_{11} = 0{\cdot}0098$ (2), $\beta_{21} = 0{\cdot}0060$ (1), $\beta_{33} = 0{\cdot}0032$ (1), $\beta_{12} = -0{\cdot}0009$ (1), $\beta_{13} = -0{\cdot}0002$ (1), $\beta_{23} = -0{\cdot}0001$ (1).

The Br^- ion is involved in two hydrogen bonds linking two different molecules: N(1)—H···Br^- (3·35 Å), N^+(4)—H···Br^- (3·18 Å) (Fig. 3).

The authors are grateful to Dr A. Ahond for helpful discussions.

Table 2. *Interatomic distances* (Å) *and angles* (°)

The estimated standard deviations are 0·015 Å and 1° respectively.

N(1)—C(2)	1·39	C(8)—C(9)	1·41
N(1)—C(13)	1·35	C(8)—C(13)	1·42
C(2)—C(3)	1·46	C(9)—C(10)	1·38
C(2)—C(7)	1·38	C(10)—C(11)	1·41
C(3)—C(14)	1·46	C(11)—C(12)	1·37
C(3)—O	1·22	C(12)—C(13)	1·42
N(4)—C(5)	1·51	C(14)—C(15)	1·55
N(4)—C(17)	1·56	C(15)—C(16)	1·54
N(4)—C(21)	1·43	C(15)—C(20)	1·51
C(5)—C(16)	1·53	C(18)—C(19)	1·52
C(6)—C(7)	1·48	C(19)—C(20)	1·54
C(6)—C(16)	1·54	C(20)—C(21)	1·56
C(7)—C(8)	1·43		
C(2)—N(1)—C(13)	110	C(9)—C(10)—C(11)	121
N(1)—C(2)—C(3)	121	C(10)—C(11)—C(12)	124
N(1)—C(2)—C(7)	109	C(11)—C(12)—C(13)	115
C(3)—C(2)—C(7)	129	N(1)—C(13)—C(8)	108
C(2)—C(3)—C(14)	118	N(1)—C(13)—C(12)	130
C(2)—C(3)—O	119	C(8)—C(13)—C(12)	122
C(14)—C(3)—O	122	C(3)—C(14)—C(15)	112
C(5)—N(4)—C(17)	112	C(14)—C(15)—C(16)	111
C(5)—N(4)—C(21)	112	C(14)—C(15)—C(20)	114
C(17)—N(4)—C(21)	110	C(16)—C(15)—C(20)	111
N(4)—C(5)—C(16)	112	C(5)—C(16)—C(6)	108
C(7)—C(6)—C(16)	118	C(5)—C(16)—C(15)	113
C(2)—C(7)—C(6)	127	C(6)—C(16)—C(15)	115
C(2)—C(7)—C(8)	106	C(18)—C(19)—C(20)	116
C(6)—C(7)—C(8)	126	C(15)—C(20)—C(19)	115
C(7)—C(8)—C(9)	132	C(15)—C(20)—C(21)	112
C(7)—C(8)—C(13)	107	C(19)—C(20)—C(21)	110
C(9)—C(8)—C(13)	121	N(4)—C(21)—C(20)	112
C(8)—C(9)—C(10)	117		

Table 3. *Torsion angles in the seven-membered ring* (*C*) *and the piperidine ring* (*D*)

	Ervatamine	This work
C(6)—C(7)—C(2)—C(3)	−10°	8°
C(7)—C(2)—C(3)—C(14)	−13	6
C(2)—C(3)—C(14)—C(15)	63	48
C(3)—C(14)—C(15)—C(16)	−35	−94
C(14)—C(15)—C(16)—C(6)	−46	47
C(15)—C(16)—C(6)—C(7)	83	29
C(16)—C(6)—C(7)—C(2)	−36	−54
C(20)—C(15)—C(16)—C(5)	−46	50
C(15)—C(16)—C(5)—N(4)	56	−51
C(16)—C(5)—N(4)—C(21)	−64	55
C(5)—N(4)—C(21)—C(20)	61	−57
N(4)—C(21)—C(20)—C(15)	−53	56
C(21)—C(20)—C(15)—C(16)	45	−51

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* A list of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31976 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

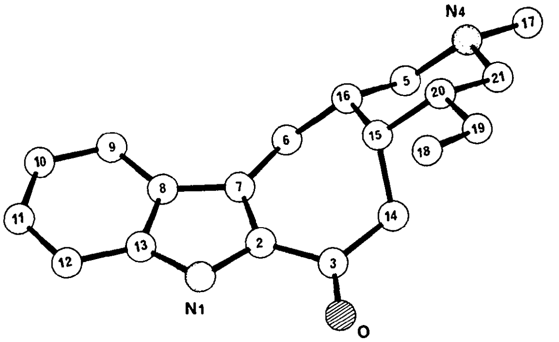

Fig. 1. Molecular structure and atom numbering. *ORTEP* (Johnson, 1965) was used for this drawing and for Fig. 3.

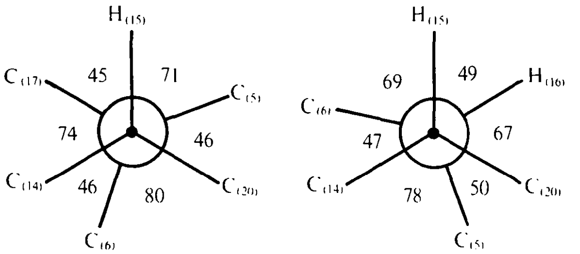

Fig. 2. Newman projections along C(15)—C(16). (*a*) Ervatamine. (*b*) This work.

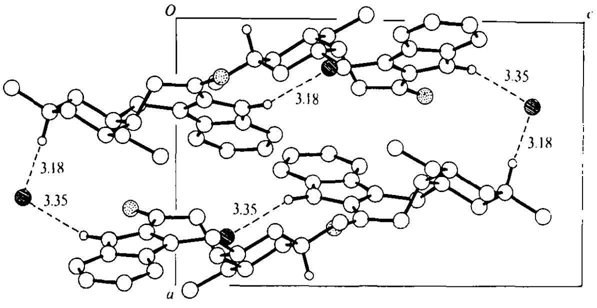

Fig. 3. Crystal structure viewed along **b**.

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Trimethyltin Methylsulphinate

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Abstract. $C_4H_{12}O_2SSn$, orthorhombic, $C222_1$; $a = 17{\cdot}569$ (20), $b = 12{\cdot}293$ (20), $c = 8{\cdot}240$ (12) Å, $U = 1778$ Å^3, $Z = 8$, $D_x = 1{\cdot}81$ g cm^{-3}, μ(Mo $K\alpha$) = 28·18 cm^{-1}. The structure was solved by the heavy-atom method and refined to an R of 0·043 for 719 unique two-circle diffractometer data. Planar trimethyltin groups are linked by disordered —O—S(Me)—O— units to form infinite helical chains along **c**, with trigonal bipyramidal geometry about Sn.

Introduction. Several tetraorganotin derivatives undergo insertion of SO_2 into the Sn—C bond to give stable, crystalline mono-inserted products which have been shown by IR and NMR studies to be aggregated in solution (Fong & Kitching, 1970). In view of the facility with which Sn increases its coordination number beyond four when a suitable bridging ligand is available, it seemed likely that these insertion compounds are polymeric in the solid state, and this has been confirmed by the structure of $Me_3SnOS(O)CH_2CCH$ (Ginderow & Huber, 1973) and by the determination reported here.

The sample was prepared by dissolving Me_4Sn in dry liquid SO_2 under vacuum. Evaporation of excess SO_2 left a white solid which was recrystallized from CCl_4 to afford a few plate-like crystals which were mounted in