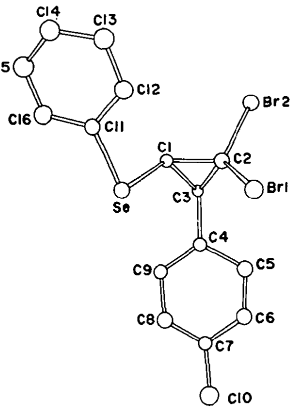

Fig. 1. Perspective view of one of the molecules showing the atom numbering.

molecules respectively. For an extensive review of the structural chemistry of organic compounds containing selenium, see Hargittai & Rozsondai (1986).

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Structure of a Chiral Intermediate in the Synthesis of (+)-19-Epiajmalicine

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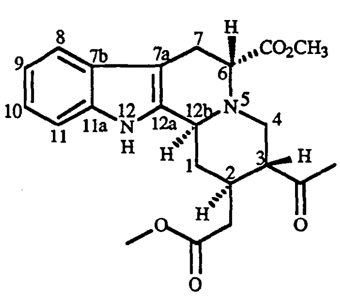
Abstract. [2*S**-(2β,3α,6α,12bβ)]-Methyl 3-acetyl-1,2,3,4,6,7,12,12b-octahydro-6-methoxycarbonyl-indolo[2,3-*a*]quinolizine-3-ethanoate, $C_{22}H_{26}N_2O_5$, $M_r = 398.46$, orthorhombic, $P2_12_12_1$, $a = 9.463\ (2)$, $b = 11.251\ (3)$, $c = 18.871\ (6)$ Å, $V = 2009.2\ (9)$ Å^3, $Z = 4$, $D_x = 1.32$ g cm^{-3} (178 K), λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.8762$ cm^{-1}, $F(000) = 848$, $T = 178$ K, $R = 0.0536$ for 1673 reflections [$F_o \geq 6\sigma(F_o)$]. Molecules are hydrogen bonded along the 2_1-screw axis parallel to **a**. The hydrogen-bond geometric parameters for N12—H12···O19 (related by $0.5 + x$, $1.5 - y$, $1 - z$) are N···O 2.986 (6), H···O 2.30 (5) Å, N—H···O 161 (5)°. The *C* and *D* rings are *trans* fused with ring-junction torsion angles of −39.6 (5) and 63.8 (5)° for C12a—C12b—N5—C6 and C1—C12b—N5—C4, respectively. The conformation of the *C* ring is half chair with N5 and C6 −0.168 (4) and 0.552 (5) Å, respectively, out of the plane defined by the remaining four atoms of the ring. The *D* ring is in the chair conformation.

Experimental. Compound (I) was synthesized *via* an intermolecular Michael addition (Hirai, Terada & Yamazaki, 1988) on an intermediate derived from D-tryptophan during studies directed toward the total synthesis of (+)-19-epiajmalicine (Melchio, Bouquet, Pais & Goutarel, 1977). The absolute configuration of (I) was deduced from the chiral center present in the optically pure starting material. Full details of the synthetic procedure will be published in due course (Martin & Corbett, 1991). Crystals were obtained by slow evaporation of a hexane–diethyl ether solution. The data crystal was a clear colorless needle of approximate dimensions 0.17 × 0.17 ×



(I)

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å²) *for the non-*H *atoms*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$, where $\mathbf{A}_j$ is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

	x	y	z	U_{eq}
C1	0.6608 (6)	0.7821 (5)	0.5662 (3)	0.028 (2)
C2	0.5551 (6)	0.8517 (4)	0.5198 (3)	0.027 (2)
C3	0.5073 (5)	0.9631 (5)	0.5593 (3)	0.027 (2)
C4	0.4484 (5)	0.9273 (4)	0.6326 (3)	0.026 (2)
N5	0.5555 (4)	0.8634 (3)	0.6728 (2)	0.0239 (13)
C6	0.5287 (5)	0.8563 (4)	0.7495 (3)	0.028 (2)
C7	0.6658 (6)	0.8184 (5)	0.7867 (3)	0.034 (2)
C7a	0.7243 (5)	0.7105 (4)	0.7523 (2)	0.0261 (15)
C7b	0.8173 (5)	0.6177 (4)	0.7775 (2)	0.0278 (15)
C8	0.8863 (6)	0.5953 (6)	0.8411 (3)	0.038 (2)
C9	0.9665 (6)	0.4928 (6)	0.8474 (3)	0.041 (2)
C10	0.9838 (6)	0.4144 (6)	0.7896 (3)	0.042 (2)
C11	0.9195 (6)	0.4360 (5)	0.7262 (3)	0.037 (2)
C11a	0.8350 (5)	0.5362 (5)	0.7201 (3)	0.0304 (15)
N12	0.7557 (5)	0.5795 (4)	0.6647 (2)	0.0290 (14)
C12a	0.6899 (5)	0.6815 (4)	0.6837 (2)	0.0235 (14)
C12b	0.5941 (5)	0.7509 (4)	0.6373 (2)	0.0215 (14)
C13	0.6127 (6)	0.8751 (5)	0.4442 (3)	0.033 (2)
C14	0.7219 (6)	0.9732 (5)	0.4438 (3)	0.034 (2)
O15	0.6949 (4)	1.0759 (3)	0.4342 (2)	0.0495 (13)
O16	0.8509 (4)	0.9312 (3)	0.4568 (2)	0.0377 (12)
C17	0.9621 (7)	1.0191 (6)	0.4598 (4)	0.045 (2)
C18	0.3979 (6)	1.0308 (5)	0.5193 (2)	0.028 (2)
O19	0.3037 (4)	0.9829 (3)	0.4872 (2)	0.0377 (12)
C20	0.4082 (7)	1.1644 (5)	0.5202 (3)	0.044 (2)
C21	0.4105 (6)	0.7762 (5)	0.7765 (3)	0.039 (2)
O22	0.3802 (4)	0.7684 (4)	0.8382 (2)	0.058 (2)
O23	0.3440 (4)	0.7170 (3)	0.7263 (2)	0.0416 (13)
C24	0.2352 (7)	0.6400 (5)	0.7538 (4)	0.056 (2)

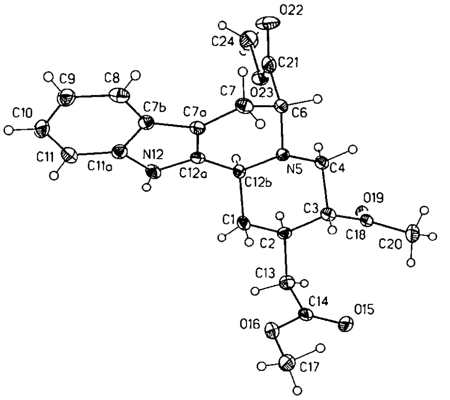

Fig. 1. View of (I) showing the atom-labelling scheme for molecule 1. The non-H atoms are scaled to the 30% probability level while the H atoms are drawn at an arbitrary size.

0.85 mm that was cut from a larger crystal. The data were collected on a Nicolet *R*3 diffractometer using a graphite monochromator and a Nicolet LT-2 low-temperature delivery system. Lattice parameters were obtained from the least-squares refinement of 40 reflections with $16.3 < 2\theta < 20.8°$. The data were collected using the ω-scan technique with a 2θ range of 4.0–55.0°, with a 1.2° ω scan at 5–10° min^{-1} (h $-12 \rightarrow 12$, k $0 \rightarrow 15$, l $0 \rightarrow 25$). A total of 5036 reflections were collected of which 2670 were unique (ignoring the small anomalous-dispersion effects; $R_{\rm int} = 0.0294$). Four reflections ($\bar{1}\bar{2}\bar{3}$, $21\bar{2}$, 116 and $12\bar{3}$) were remeasured every 96 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these check reflections was used to scale the data. The scaling factor ranged from 0.972 to 1.00. The data were also corrected for Lp effects but not absorption. The data reduction and decay correction were applied using the *SHELXTL-Plus* software package (Sheldrick, 1991). Reflections having $F_o < 6\sigma(F_o)$ were considered unobserved (997 reflections). The structure was solved by direct methods and refined by full-matrix least squares (Sheldrick, 1991). In all, 345 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. Most H-atom positions were obtained from a ΔF map and refined with isotropic thermal parameters. The methyl H atoms on C22 and C24 were idealized (C—H 0.96 Å) while the $U_{\rm iso}$ for the H atoms on C24 was set to $1.2 \times U_{eq}$. The $U_{\rm iso}$ for H atoms on C22 was refined. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0.5kI^{-1/2}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$. The intensity, I, is given by $(I_{\rm peak} - I_{\rm background}) \times$

Table 2. *Bond lengths* (Å) *and angles* (°) *for non-*H *atoms*

A	*B*	*C*	*A*—*B*	*A*—*B*—*C*
C2	C1	C12b	1.543 (7)	110.4 (4)
C12b	C1		1.524 (7)	
C3	C2	C13	1.527 (7)	114.5 (4)
C3	C2	C1		109.4 (4)
C13	C2	C1	1.549 (7)	112.4 (4)
C4	C3	C18	1.545 (7)	109.7 (4)
C4	C3	C2		109.3 (4)
C18	C3	C2	1.490 (7)	112.2 (4)
N5	C4	C3	1.456 (6)	110.2 (4)
C6	N5	C12b	1.472 (6)	116.2 (3)
C6	N5	C4		114.8 (4)
C12b	N5	C4	1.477 (6)	111.0 (4)
C7	C6	C21	1.536 (8)	107.7 (4)
C7	C6	N5		108.6 (4)
C21	C6	N5	1.523 (7)	119.2 (4)
C7a	C7	C6	1.484 (7)	110.0 (4)
C7b	C7a	C12a	1.445 (7)	106.4 (4)
C7b	C7a	C7		132.4 (4)
C12a	C7a	C7	1.374 (6)	121.2 (4)
C8	C7b	C11a	1.388 (7)	118.9 (5)
C8	C7b	C7a		134.6 (5)
C11a	C7b	C7a	1.430 (7)	106.6 (4)
C9	C8	C7b	1.386 (9)	118.9 (5)
C10	C9	C8	1.412 (8)	121.1 (5)
C11	C10	C9	1.364 (8)	120.9 (5)
C11a	C11	C10	1.386 (8)	118.4 (5)
N12	C11a	C7b	1.377 (7)	106.5 (4)
N12	C11a	C11		131.7 (5)
C7b	C11a	C11		121.8 (5)
C12a	N12	C11a	1.354 (6)	110.4 (4)
C12b	C12a	C7a	1.483 (6)	125.2 (4)
C12b	C12a	N12		124.8 (4)
C7a	C12a	N12		110.0 (4)
C1	C12b	N5		107.7 (4)
C1	C12b	C12a		112.8 (4)
N5	C12b	C12a		109.5 (4)
C14	C13	C2	1.511 (8)	111.7 (4)
O15	C14	O16	1.197 (7)	124.4 (5)
O15	C14	C13		124.1 (5)
O16	C14	C13	1.332 (6)	111.5 (5)
C17	O16	C14	1.445 (7)	115.7 (4)
O19	C18	C20	1.205 (6)	120.0 (5)
O19	C18	C3		122.7 (5)
C20	C18	C3	1.507 (7)	117.3 (5)
O22	C21	O23	1.202 (7)	123.0 (5)
O22	C21	C6		122.9 (5)
O23	C21	C6	1.319 (7)	114.1 (5)
C24	O23	C21	1.443 (7)	112.7 (4)

(scan rate); where 0.02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction for Lp effects and decay. $\sigma(I)$ was estimated from counting statistics: $\sigma(I) = [(I_{\rm peak} + I_{\rm background})^{1/2} \times \text{(scan rate)}]$. Final $R = 0.0536$ for 1673 reflections, with $wR = 0.0582$ ($R_{\rm all} = 0.0894$, $wR_{\rm all} = 0.0687$) and goodness of fit = 1.636. Maximum $|\Delta/\sigma| < 0.1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were -0.26 and $0.50\ {\rm e\ Å^{-3}}$, respectively. Scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). Scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear-absorption coefficient were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55).* Fig. 1, showing the atom-labelling scheme, was generated using *SHELXTL-Plus* (Sheldrick, 1991). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. The structure of (I) was determined as part of a generalized approach to the syntheses of alkaloids of the indole family (Martin, Rüeger, Williamson & Grzejszczak, 1987; Martin, Benage & Hunter, 1988).

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* Lists of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles involving H atoms, torsion angles, and structure-factor amplitudes, as well as a unit-cell packing diagram, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55205 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0565]

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2-Menthyl (2-Hydroxyphenyl)glycolate

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Abstract. 2-Menthyl hydroxy(2-hydroxyphenyl)ethanoate, $C_{18}H_{26}O_4$, $M_r = 306.40$, monoclinic, $P2_1$, $a = 13.686\,(2)$, $b = 5.874\,(2)$, $c = 11.030\,(3)$ Å, $\beta = 96.62\,(3)^\circ$, $V = 880.8$ Å^3, $Z = 2$, $D_x = 1.16\ {\rm g\ cm^{-3}}$, $\lambda({\rm Cu}\ K\alpha) = 1.5418$ Å, $\mu = 6.14\ {\rm cm^{-1}}$, $F(000) = 332$, room temperature, $R = 0.044$ for 1715 unique observed reflections with $I \geq 2\sigma(I)$. The cyclohexane ring adopts the chair conformation. The absolute configuration at C7 was inferred to be R on the basis of those of the compounds used in the synthesis. A short intramolecular bond [O2···H1O1 = 1.84 (6) Å] is observed.

Experimental. A prismatic crystal $0.11 \times 0.19 \times 0.27$ mm was used for data collection on a Siemens AED single-crystal diffractometer equipped with an IBM PS2/30 personal computer (Belletti, Cantoni & Pasquinelli, 1988), and Cu $K\alpha$ radiation. Cell parameters were determined by least-squares fit of the setting angles of 27 reflections with $11.02 \leq \theta \leq 31.32^\circ$. 1933 reflections were measured ($3 \leq \theta \leq 70^\circ$) using a modified version (Belletti *et al.*, 1988) of the Lehmann & Larsen (1974) procedure; 1847 were unique ($R_{\rm int} = 0.017$) and 1715 with $I \geq 2\sigma(I)$ observed ($-16 \leq h \leq 16$, $0 \leq k \leq 7$, $0 \leq l \leq 13$). One

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