scale factor and an isotropic extinction parameter $x = 0.005$ (1) $[F_c = F_c/(1.0 + 0.002xF_c^2/\sin 2\theta)^{0.25}]$. H atoms at idealized positions with fixed isotropic temperature factor $U = 0.06$ Å^2; in final cycle $R = 0.038$, $wR = 0.049$ for observed reflections only, $S = 1.39$, max. shift/e.s.d. 0.598, function minimized $\sum w(\Delta F)^2$, where $w = [\sigma(F_o)^2 + 0.0015\,(F_o)^2]^{-1}$, max. and min. heights in final $\Delta\rho$ synthesis: 0.16 and -0.19 e Å^{-3}, complex scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV); all calculations performed with *SHELXTL* (Sheldrick, 1983) on a NOVA 4S computer. Final atomic coordinates and equivalent isotropic U's are listed in Table 1,* bond distances and angles in Table 2. An *ORTEP*-like drawing of the molecule and numbering of the atoms are shown in Fig. 1. Fig. 2 shows a stereoview of the molecular packing.

Related literature. Biological activity (Andrews, Thomas, Polhke & Seubert, 1983). A similar *trans*-relation of carbonyl groups was observed in molecular mechanics studies of some hindered C(3)-substituted analogues (Rubio, Cetina & Pérez-Ibarra, 1991).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54232 (15 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 of 1-(Methoxymethyl)-16,17-didehydro-19-oxoalloyohimbane

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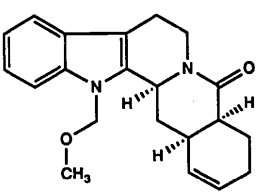
Abstract. $C_{21}H_{24}N_2O_2$, $M_r = 336.42$, orthorhombic, *Pcab*, $a = 9.882$ (4), $b = 17.357$ (5), $c = 19.794$ (6) Å, $V = 3395$ (2) Å^3, $Z = 8$, $D_x = 1.32$ g cm^{-3}, λ(Mo $K\alpha$) $= 0.7107$ Å, $\mu = 0.79$ cm^{-1}, $F(000) = 1440$, $T = 112$ K, $R = 0.063$ ($wR = 0.066$) for 2232 unique observed reflections. Three fused (six-membered) rings contain three bridgehead-bound H atoms; these three H atoms were *cis* to each other in the pair of enantiomers studied. This confirms the relative stereochemistry of the chiral centers as either C(10) (*S*), C(14) (*R*) and C(20) (*S*) or C(10) (*R*), C(14) (*S*) and C(20) (*R*).

Experimental. Crystals (colorless prisms) of $C_{21}H_{24}N_2O_2$ [hereafter (1)] obtained from a dichloromethane/hexane solution by Dr Paul Buonora and Professor A. I. Meyers (Colorado State University). Crystal size $0.15 \times 0.44 \times 0.53$ mm. Nicolet *R*3*m* diffractometer, unit-cell constants from least-squares fit of setting angles for 25 reflections ($2\theta_{av} = 23.56°$). Data collected ($\theta/2\theta$ scans) to $(\sin\theta)/\lambda = 0.5947$ Å^{-1}, $0 \le h \le 12$, $0 \le k \le 21$, $0 \le l \le 24$. Three standard reflections (200, 040, 002) measured every 97, no change in intensity; Lorentz and polarization corrections; no absorption correction applied due to low absorption coefficient; 2996 unique reflections, 2232 reflections with $F_o > 2.5\sigma(F_o)$ observed.



(1)

Structure solved by direct methods (*SOLV*) in *Pcab* (variant of *Pbca*, No. 61); block-diagonal (maximum 103 parameters/block, 229 parameters total, data/parameters = 9.7) weighted $\{w = [\sigma^2(F)$

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Table 1. *Atomic coordinates and isotropic thermal parameters* ($Å^2 \times 10^3$)

Equivalent isotropic U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	−0·1674 (3)	0·1813 (2)	0·4423 (1)	21 (1)
C(2)	−0·2344 (3)	0·1782 (2)	0·3800 (1)	24 (1)
C(3)	−0·3001 (3)	0·1105 (2)	0·3641 (1)	28 (1)
C(4)	−0·3010 (3)	0·0477 (2)	0·4084 (2)	31 (1)
C(5)	−0·2342 (3)	0·0508 (2)	0·4696 (2)	28 (1)
C(6)	−0·1643 (3)	0·1180 (2)	0·4868 (1)	25 (1)
C(7)	−0·0812 (3)	0·1400 (2)	0·5431 (1)	24 (1)
C(8)	−0·0262 (3)	0·0915 (2)	0·5996 (1)	28 (1)
C(9)	0·1056 (3)	0·1279 (2)	0·6226 (2)	28 (1)
C(10)	0·0577 (3)	0·2581 (2)	0·5757 (1)	24 (1)
C(11)	−0·0398 (3)	0·2139 (2)	0·5319 (1)	21 (1)
C(12)	−0·0705 (3)	0·3152 (2)	0·4397 (1)	25 (1)
C(13)	−0·2889 (3)	0·3665 (2)	0·4202 (2)	36 (1)
C(14)	0·0114 (3)	0·3383 (2)	0·5964 (1)	26 (1)
C(15)	0·1074 (3)	0·3694 (2)	0·6504 (1)	27 (1)
C(16)	0·0917 (3)	0·4540 (2)	0·6645 (1)	31 (1)
C(17)	0·0412 (3)	0·4816 (2)	0·7221 (2)	33 (1)
C(18)	−0·0141 (4)	0·4321 (2)	0·7770 (2)	37 (1)
C(19)	−0·0291 (3)	0·3477 (2)	0·7560 (2)	36 (1)
C(20)	0·0926 (3)	0·3216 (2)	0·7140 (1)	26 (1)
C(21)	0·0904 (3)	0·2357 (2)	0·7010 (1)	28 (1)
N(1)	−0·0909 (2)	0·2407 (1)	0·4703 (1)	23 (1)
N(2)	0·0867 (2)	0·2105 (1)	0·6357 (1)	25 (1)
O(1)	−0·1671 (2)	0·3708 (1)	0·4586 (1)	31 (1)
O(2)	0·0977 (2)	0·1906 (1)	0·7489 (1)	43 (1)

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

C(1)—C(2)	1·401 (4)	C(1)—C(6)	1·409 (4)
C(1)—N(1)	1·394 (3)	C(2)—C(3)	1·379 (4)
C(3)—C(4)	1·399 (4)	C(4)—C(5)	1·379 (4)
C(5)—C(6)	1·399 (4)	C(6)—C(7)	1·435 (4)
C(7)—C(8)	1·502 (4)	C(7)—C(11)	1·363 (4)
C(8)—C(9)	1·517 (4)	C(9)—N(2)	1·468 (4)
C(10)—C(11)	1·506 (4)	C(10)—C(14)	1·521 (4)
C(10)—N(2)	1·475 (3)	C(11)—N(1)	1·400 (3)
C(12)—N(1)	1·441 (3)	C(12)—O(1)	1·408 (3)
C(13)—O(1)	1·425 (4)	C(14)—C(15)	1·527 (4)
C(15)—C(16)	1·503 (4)	C(15)—C(20)	1·515 (4)
C(16)—C(17)	1·333 (4)	C(17)—C(18)	1·489 (4)
C(18)—C(19)	1·530 (4)	C(19)—C(20)	1·531 (4)
C(20)—C(21)	1·513 (4)	C(21)—N(2)	1·365 (4)
C(21)—O(2)	1·231 (4)		

C(2)—C(1)—C(6)	122·1 (3)	C(2)—C(1)—N(1)	129·4 (2)
C(6)—C(1)—N(1)	108·4 (2)	C(1)—C(2)—C(3)	117·1 (3)
C(2)—C(3)—C(4)	121·7 (3)	C(3)—C(4)—C(5)	121·1 (3)
C(4)—C(5)—C(6)	118·9 (3)	C(1)—C(6)—C(5)	119·1 (3)
C(1)—C(6)—C(7)	106·9 (2)	C(5)—C(6)—C(7)	134·0 (3)
C(6)—C(7)—C(8)	129·5 (2)	C(6)—C(7)—C(11)	107·2 (2)
C(8)—C(7)—C(11)	122·6 (2)	C(7)—C(8)—C(9)	107·5 (2)
C(8)—C(9)—N(2)	110·5 (2)	C(11)—C(10)—C(14)	115·3 (2)
C(11)—C(10)—N(2)	107·6 (2)	C(14)—C(10)—N(2)	110·7 (2)
C(7)—C(11)—C(10)	125·3 (2)	C(7)—C(11)—N(1)	110·2 (2)
C(10)—C(11)—N(1)	124·2 (2)	N(1)—C(12)—O(1)	114·1 (2)
C(10)—C(14)—C(15)	109·0 (2)	C(14)—C(15)—C(16)	114·3 (2)
C(14)—C(15)—C(20)	109·2 (2)	C(16)—C(15)—C(20)	111·7 (2)
C(15)—C(16)—C(17)	123·3 (3)	C(16)—C(17)—C(18)	123·6 (3)
C(17)—C(18)—C(19)	112·9 (2)	C(18)—C(19)—C(20)	110·8 (3)
C(15)—C(20)—C(19)	111·4 (2)	C(15)—C(20)—C(21)	113·5 (2)
C(19)—C(20)—C(21)	111·9 (2)	C(20)—C(21)—N(2)	118·5 (2)
C(20)—C(21)—O(2)	119·7 (2)	N(2)—C(21)—O(2)	121·7 (3)
C(1)—N(1)—C(11)	107·2 (2)	C(1)—N(1)—C(12)	125·0 (2)
C(11)—N(1)—C(12)	127·8 (2)	C(9)—N(2)—C(10)	115·4 (2)
C(9)—N(2)—C(21)	118·6 (2)	C(10)—N(2)—C(21)	126·0 (2)
C(12)—O(1)—C(13)	113·3 (2)		

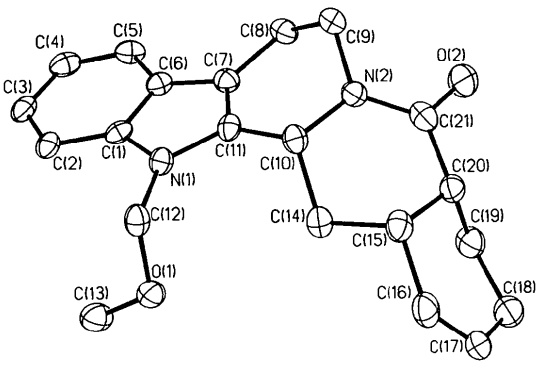

Fig. 1. The structure of (1) (50% probability thermal ellipsoids). H atoms have been omitted for clarity.

$+ gF^2]^{-1}$, $g = 8·5 \times 10^{-4}$} least-squares refinement on F. H atoms in idealized positions [C—H = 0·96 Å, $U(H) = 1·2 \times U_{iso}(C)$]. Non-H atoms refined with anisotropic thermal parameters. At convergence [$(\Delta/\sigma)_{max} = 0·087$, $(\Delta/\sigma)_{mean} = 0·013$ for last three cycles], $R = 0·063$, $wR = 0·066$, $S = 1·302$, slope of normal probability plot = 1·16, $(\Delta\rho)_{max} = 0·26$, $(\Delta\rho)_{min} = -0·30$ e $Å^{-3}$. Neutral-atom scattering factors and anomalous-dispersion corrections used (*International Tables for X-ray Crystallography*, 1974, Vol. IV); all calculations performed using *SHELXTL* program library (Sheldrick, 1983). Table 1 gives atomic coordinates and Table 2 gives bond lengths and angles.* Fig. 1 shows the structure of (1), as well as the numbering scheme used.

Related literature. Nauclefiline (Lin, Liu, Yu, Dou, Zhang & Li, 1985) also contains the oxoalloyohimbane framework. Nauclefiline differs from (1) both in the location of unsaturation and the presence of an oxygen heteroatom within the oxoalloyohimbane framework.

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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 54265 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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