$C_{22}H_{20}O_5$

Fig. 1. Molecular configuration and atom-numbering scheme, thermal ellipsoids at the 50% probability level. H atoms are shown as isotropic spheres with B's of $1{\cdot}0$ Å^2 and are unlabeled to improve clarity.

Related literature. The title compound was prepared as part of the studies of the synthesis of antibiotic gilvocarcins (Takahashi, Yoshida, Tomita & Shirahata, 1981). Despite many efforts with various kinds of NMR techniques including 2D NOESY (Homo-nuclear Nuclear Overhauser Effect correlated two dimensionally) experiments, we were unable to
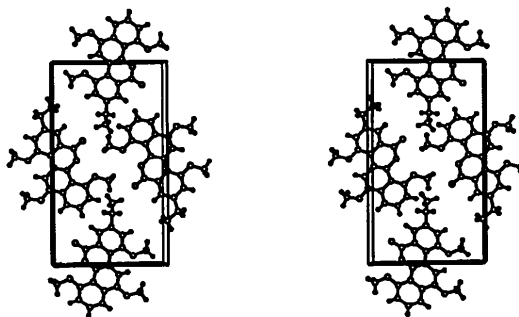

Fig. 2. Stereoscopic illustration of the molecular packing. b is vertical, a is horizontal and c is into the plane of the paper.

differentiate the title compound from its—C-1 methoxy isomer, 8-ethyl-1,10,12-trimethoxy-6H--benzo[d]naphtho[1,2-b]pyran-6-one. This study allows us to characterize each isomer unequivocally. The structure of gilvocarcin M (possessing a C-1 hydroxy group) has been reported earlier (Hirayama, Takahashi, Shirahata, Ohashi & Sasada, 1981).

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Structure of a Praziquantel Analogue*

By R. A. Toscano,† M. Rubio, R. Cetina and B. M. Pérez-Ibarra

Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Univesitaria, Coyoacán, 04510 México DF, Mexico

Abstract. 2-(m-Nitrobenzoyl)-1,2,3,6,7,11b-hexa-hydro-4H-pyrazino[2,1-a]isoquinolin-4-one, $C_{19}H_{17}N_3O_4$, $M_r = 351{\cdot}4$, monoclinic, $P2_1/n$, $a = 14{\cdot}803$ (3), $b = 7{\cdot}991$ (1), $c = 15{\cdot}253$ (3) Å, $\beta = 115{\cdot}24$ (1)°, $V = 1632{\cdot}1$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}43$ Mg m^{-3}, λ(Cu $K\alpha$) $= 1{\cdot}54178$ Å, $\mu = 8{\cdot}04$ cm^{-1}, $F(000) = 736$, $T = 293$ K, final $R = 0{\cdot}038$ for 1821 unique observed reflections $[F_o > 3\sigma(F_o)]$. The hydropyrazine ring adopts a half-chair conformation, while the tetrahydropyridine moiety has a sofa conformation with C(6) $0{\cdot}66$ Å out of the plane formed by C(7)—C(7a)—C(11a)—C(11b)—N(5). The nitrophenyl group is essentially planar [maximum deviation O(4)

Table 1. *Atom coordinates* ($\times 10^4$) *and temperature factors* ($\text{Å}^2 \times 10^3$)

U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}*
C(1)	4242 (1)	3745 (3)	3256 (1)	42 (1)
N(2)	5204 (1)	3908 (2)	3223 (1)	40 (1)
C(3)	5214 (1)	3201 (2)	2347 (1)	43 (1)
C(4)	4278 (1)	3540 (2)	1444 (1)	44 (1)
O(1)	4260 (1)	3068 (2)	673 (1)	63 (1)
N(5)	3529 (1)	4372 (2)	1520 (1)	43 (1)
C(6)	2675 (1)	4904 (3)	646 (1)	53 (1)
C(7)	1773 (1)	3913 (3)	541 (1)	55 (1)
C(7a)	1614 (1)	4038 (2)	1449 (1)	42 (1)
C(8)	669 (2)	3783 (2)	1419 (2)	53 (1)
C(9)	501 (2)	3915 (3)	2237 (2)	56 (1)
C(10)	1286 (1)	4309 (3)	3116 (2)	52 (1)
C(11)	2227 (1)	4556 (2)	3162 (1)	45 (1)
C(11a)	2413 (1)	4419 (2)	2339 (1)	39 (1)
C(11b)	3455 (1)	4745 (2)	2429 (1)	39 (1)
C(12)	5943 (1)	4847 (2)	3879 (1)	39 (1)
O(2)	5830 (1)	5643 (2)	4514 (1)	55 (1)
C(13)	6939 (1)	4874 (2)	3843 (1)	38 (1)
C(14)	7392 (1)	3419 (2)	3726 (1)	40 (1)
C(15)	8334 (1)	3520 (2)	3758 (1)	42 (1)
N(1)	8794 (1)	1971 (2)	3624 (1)	54 (1)
O(3)	8318 (1)	665 (2)	3490 (1)	64 (1)
O(4)	9618 (1)	2055 (2)	3637 (2)	85 (1)
C(16)	8854 (1)	4997 (3)	3913 (1)	49 (1)
C(17)	8415 (2)	6433 (3)	4042 (1)	52 (1)
C(18)	7458 (1)	6380 (3)	4002 (1)	48 (1)

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

C(1)—N(2)	1·453 (3)	C(1)—C(11b)	1·528 (2)
N(2)—C(3)	1·455 (3)	N(2)—C(12)	1·352 (2)
C(3)—C(4)	1·504 (2)	C(4)—O(1)	1·224 (3)
C(4)—N(5)	1·340 (3)	N(5)—C(6)	1·455 (2)
N(5)—C(11b)	1·468 (3)	C(6)—C(7)	1·501 (3)
C(7)—C(7a)	1·503 (3)	C(7a)—C(8)	1·395 (3)
C(7a)—C(11a)	1·402 (2)	C(8)—C(9)	1·377 (4)
C(9)—C(10)	1·385 (3)	C(10)—C(11)	1·379 (3)
C(11)—C(11a)	1·399 (3)	C(11a)—C(11b)	1·511 (3)
C(12)—O(2)	1·229 (3)	C(12)—C(13)	1·500 (3)
C(13)—C(14)	1·391 (3)	C(13)—C(18)	1·392 (3)
C(14)—C(15)	1·376 (3)	C(15)—N(1)	1·469 (3)
N(1)—O(4)	1·214 (3)	N(1)—O(3)	1·226 (2)
C(17)—C(18)	1·392 (3)	C(16)—C(17)	1·374 (3)

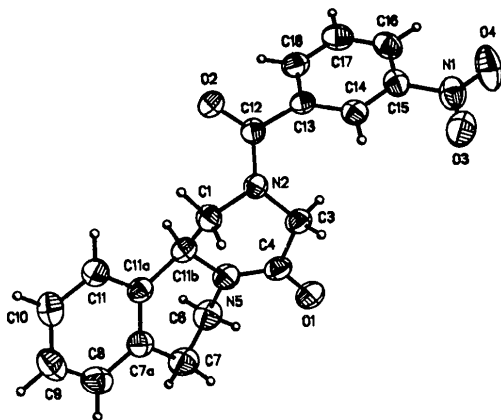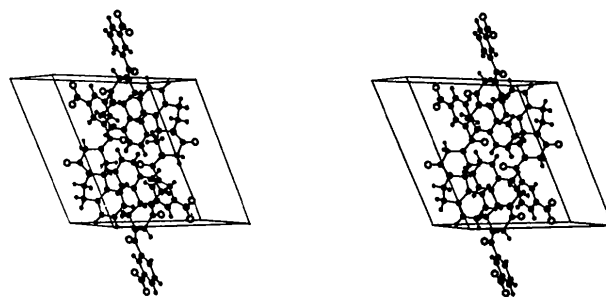|				
N(2)—C(1)—C(11b)	109·8 (2)	C(1)—N(2)—C(3)	113·0 (1)
C(1)—N(2)—C(12)	121·3 (2)	C(3)—N(2)—C(12)	125·0 (2)
N(2)—C(3)—C(4)	113·9 (2)	C(3)—C(4)—O(1)	117·3 (2)
C(3)—C(4)—N(5)	118·9 (2)	O(1)—C(4)—N(5)	123·7 (1)
C(4)—N(5)—C(6)	119·6 (2)	C(4)—N(5)—C(11b)	125·5 (1)
C(6)—N(5)—C(11b)	114·8 (2)	N(5)—C(6)—C(7)	109·2 (2)
C(6)—C(7)—C(7a)	110·4 (2)	C(7)—C(7a)—C(8)	120·6 (2)
C(7)—C(7a)—C(11a)	120·5 (2)	C(8)—C(7a)—C(11a)	118·9 (2)
C(7a)—C(8)—C(9)	121·7 (2)	C(8)—C(9)—C(10)	119·6 (2)
C(9)—C(10)—C(11)	119·6 (2)	C(10)—C(11)—C(11a)	121·6 (2)
C(7a)—C(11a)—C(11)	118·6 (2)	C(7a)—C(11a)—C(11b)	122·1 (2)
C(11)—C(11a)—C(11b)	119·2 (1)	C(1)—C(11b)—N(5)	109·6 (2)
C(1)—C(11b)—C(11a)	112·0 (2)	N(5)—C(11b)—C(11a)	111·4 (1)
N(2)—C(12)—O(2)	122·2 (2)	N(2)—C(12)—C(13)	118·6 (2)
O(2)—C(12)—C(13)	119·2 (2)	C(12)—C(13)—C(14)	121·9 (2)
C(12)—C(13)—C(18)	118·9 (2)	C(14)—C(13)—C(18)	119·0 (2)
C(13)—C(14)—C(15)	118·9 (2)	C(14)—C(15)—N(1)	117·9 (2)
C(14)—C(15)—C(16)	122·8 (2)	N(1)—C(15)—C(16)	119·3 (2)
C(15)—N(1)—O(3)	118·3 (2)	C(15)—N(1)—O(4)	118·3 (2)
O(3)—N(1)—O(4)	123·4 (2)	C(15)—C(16)—C(17)	118·5 (2)
C(16)—C(17)—C(18)	120·2 (2)	C(13)—C(18)—C(17)	120·6 (2)

Fig. 1. The molecular conformation of the title compound showing the atom numbering. The thermal ellipsoids are drawn at the 50% probability level.

Fig. 2. Stereopacking of the molecule viewed along the *b* axis.

0·029 Å] and makes a dihedral angle of 42·9 (4)° with the amide group N(2)—C(12)—O(2)—C(13). The phenyl group in the isoquinoline moiety is also planar and the two carbonyls are *trans* oriented to each other [angle between planes 135·2 (4)°]. Crystal packing is entirely due to van der Waals forces.

Experimental. The title compound was synthesized by the method described by Yuste, Pallas, Barrios, Ortiz & Sánchez-Obregón (1986). A yellow prismatic crystal of dimensions 0·15 × 0·32 × 0·42 mm showed

systematic abscences *h*0*l* for *h* + *l* odd and 0*k*0 for *k* odd. Nicolet *P3F* diffractometer, Ni-filtered Cu *Kα* radiation; unit-cell parameters from least-squares fitting of 25 reflections (11·0 < 2*θ* < 30·78°), intensity data (*h* = 0 to 15, *k* = 0 to 8, *l* = − 16 to 16) by 2*θ*/*θ* scans, 2*θ* < 110°; variable scan speed; two standards (10̄1, 3̄23) measured every 50 reflections, no significant fluctuation; 2341 reflections measured, 2060 unique, 239 unobserved [$F_o < 3\sigma(F_o)$], Lp corrections, absorption ignored. Structure was solved by direct methods and by block-diagonal least-squares refinement of 236 parameters, including positional and anisotropic thermal parameters for non-H atoms, a

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

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* 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.

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).

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Structure of 1-(Methoxymethyl)-16,17-didehydro-19-oxoalloyohimbane

BY MARK A. THOMSON AND OREN P. ANDERSON*

*Department of Chemistry, Colorado State University, Fort Collins, Colorado 80523, USA*

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

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* Author to whom correspondence should be addressed.

($2\theta_{av} = 23.56°$). 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.
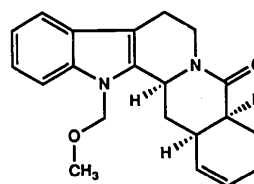
(1)

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 {$w = [\sigma^2(F)$