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6α-Hydroxy-17β-azavouacapane-17,7β-carbolactone

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The title compound, $C_{20}H_{27}NO_4$, is an intermediate of nitrogenated vouacapan derivatives. The crystal structure has intermolecular hydrogen bonds that establish a regular arrangement in the [100] direction.

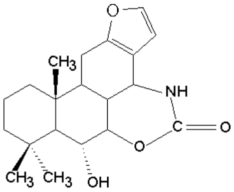
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.117
Data-to-parameter ratio = 8.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The cyclic urethane 6α-hydroxy-17β-azavouacapane-17,7β-carbolactone (HVC), was prepared from 6α,7β-dihydroxy-vouacapan-17β-oic acid (ADV) according to the procedure described in the literature (Maltha *et al.*, 1995). ADV was isolated from the hexane extract of Pterodon polygalaeflorus Benth fruits, of the leguminous family, widely used in Brazilian folk medicine (Mahajan & Monteiro, 1973). This class of compounds exhibits anti-inflammatory and analgesic activities (Duarte *et al.*, 1992). An inhibitory effect on the radicle growth of *S. bicolor L.* and a stimulatory growth effect on the radicle of *C. Sativus L.* has also been reported (Demuner *et al.*, 1998). The crystal and molecular structure of HVC, (I), is now reported.



(I)

Fig. 1 is an *ORTEP*-3 (Farrugia, 1997) view of the molecule. According to Cremer and Pople parameters (Cremer & Pople, 1975; Iulek & Zuckerman-Schpector, 1997) the ring fused to furan [θ = 50.4 (9)° and φ = 5 (1)°] is in a half-boat conformation, while rings C1–C5/C10 [θ = 9.9 (8)° and φ = 38 (5)°] and

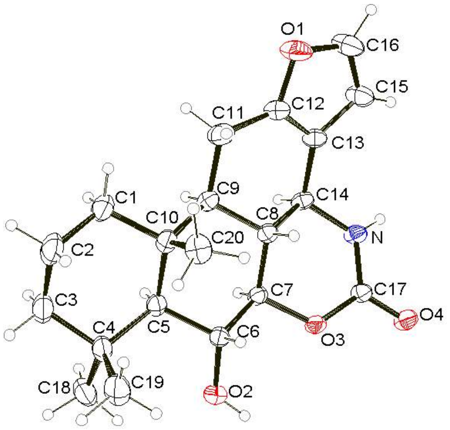


Figure 1
An *ORTEP*-3 view (Farrugia, 1997) of HVC, showing 30% probability displacement ellipsoids.

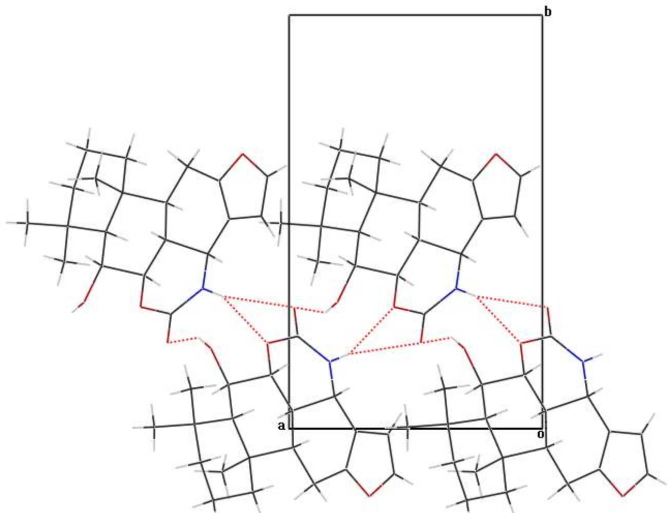


Figure 2
A *MERCURY* view (CCDC, 2003) of the hydrogen bonds in the crystal packing of HVC.

C5–C10 [θ = 10.6 (8)° and φ = 356 (3)°] adopt chair conformations, as in ADV (Ruggiero *et al.*, 1997). The carbolactone ring, O3/C7/C8/C14/N/C17 [θ = 51.1 (8)° and φ = 111 (1)°] is in a half-boat conformation.

Intermolecular hydrogen bonds (Table 2) establish a regular arrangement in the [100] direction (Fig. 2), as seen in 6α-hydroxyvouacapan-7β,17β-lactone (Abrahão-Júnior *et al.*, 1997).

Experimental

Recrystallization from dichloromethane/petroleum ether (1:2) gave colorless crystals (m.p. 339.8–341.7 K).

Crystal data

$C_{20}H_{27}NO_4$
M_r = 345.43
Orthorhombic, $P2_12_12_1$
a = 8.354 (1) Å
b = 13.631 (1) Å
c = 15.493 (1) Å
V = 1764.3 (3) Å^3
Z = 4
D_x = 1.3 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 3.0–26.3°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colorless
0.8 × 0.4 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: none
2065 measured reflections
2044 independent reflections
1719 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.023
$\theta_{\rm max}$ = 26.3°
$h = -10 \rightarrow 3$
$k = 0 \rightarrow 16$
$l = -3 \rightarrow 19$
3 standard reflections
frequency: 120 min
intensity decay: 7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.117
S = 1.06
2044 reflections
236 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0775P)^2 + 0.1708P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.28 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.012 (3)

Table 1
Selected geometric parameters (Å, °).

N—C17	1.343 (3)	O3—C17	1.349 (3)
N—C14	1.452 (3)	O3—C7	1.458 (3)
O1—C16	1.365 (4)	O4—C17	1.217 (3)
O1—C12	1.369 (3)	C7—C8	1.499 (3)
O2—C6	1.418 (3)	C8—C14	1.522 (3)
C17—N—C14	125.2 (2)	N—C14—C8	107.38 (18)
C16—O1—C12	105.7 (2)	O4—C17—N	124.2 (2)
C17—O3—C7	120.45 (17)	O4—C17—O3	117.2 (2)
N—C14—C13	113.7 (2)	N—C17—O3	118.66 (19)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N—HN···O3$^{\rm i}$	0.81 (4)	2.30 (4)	3.057 (3)	155 (3)
N—HN···O4$^{\rm i}$	0.81 (4)	2.52 (4)	3.096 (3)	129 (3)
O2—HO2···O4$^{\rm ii}$	0.83 (4)	2.17 (4)	2.929 (3)	151 (3)

Symmetry codes: (i) $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$; (ii) $x-\frac{1}{2}, \frac{3}{2}-y, 1-z$.

In the absence of significant anomalous scattering effects, the Flack (1983) parameter is essentially meaningless. Friedel pairs were merged before refinement. Atoms HO2 and HN were located by difference Fourier synthesis and positional parameters were refined. The remaining H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined as riding. For methyl H atoms, the $U_{\rm iso}$ values were set at 1.5$U_{\rm eq}$ of the parent atoms and for the others, 1.2$U_{\rm eq}$.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *MERCURY* (CCDC, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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