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#### Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.047  
 $wR$  factor = 0.117  
Data-to-parameter ratio = 13.3

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

## 6 $\alpha$ -Acetoxylvouacapan-7 $\beta$ ,17 $\beta$ -lactone

The structure of the title compound,  $\text{C}_{22}\text{H}_{28}\text{O}_5$ , which is a natural furan diterpene, has been determined. The crystal structure is stabilized by  $\text{C}-\text{H}\cdots\text{O}$  intermolecular interactions.

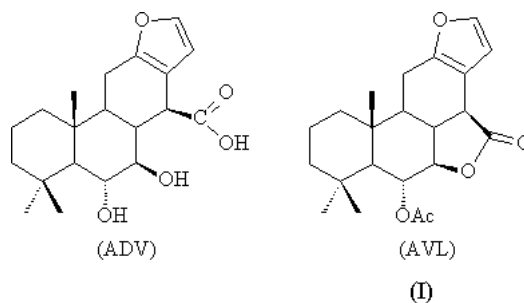
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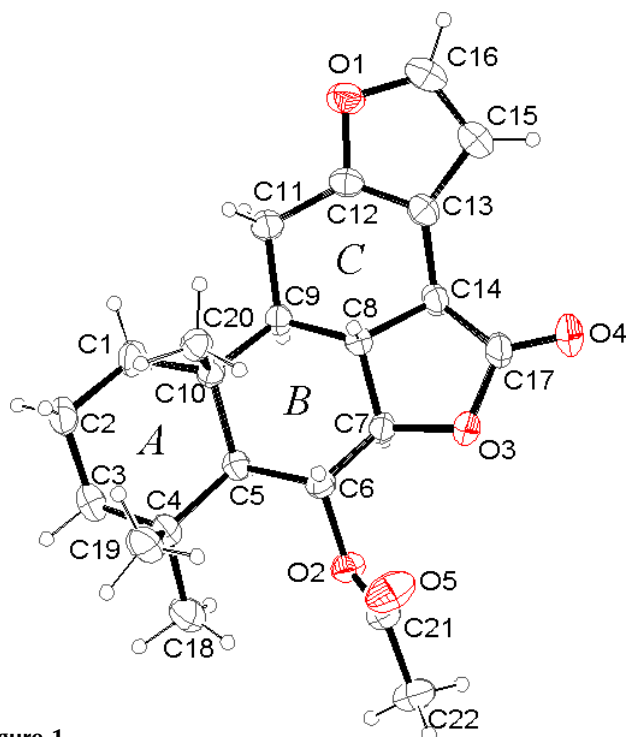
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#### Comment

For centuries, in many cultures, plants have been the base of folk medicine. Of the 20 best-selling non-protein drugs in 1999, nine were derived from or developed as the result of leads generated by natural products, with combined annual sales greater than US\$ 16 billion (Harvey, 2000). Phytochemical studies of the genus *Pterodon*, a native of Brazil, showed that the diterpenes 14,15-epoxygeranylgeraniol and 14,15-dihydroxy-14,15-dihydrogeranylgeraniol have prophylactic action against infection by *Schistosoma mansoni* (Mors *et al.*, 1967). Another 15 furan diterpenes were also isolated (Fascio *et al.*, 1976). Within this group, 6 $\alpha$ ,7 $\beta$ -dihydroxyvouacapan-17 $\beta$ -oic acid (ADV) possesses anti-inflammatory and analgesic properties (Nunan *et al.*, 1982; Duarte *et al.*, 1992). The crystallographic study of 6 $\alpha$ -acetoxylvouacapan-7 $\beta$ ,17 $\beta$ -lactone [AVL or (I)], another member of the above-mentioned group, is reported here. Both compounds showed the capacity to suppress the production of singlet molecular oxygen. This fact may be associated with damage reduction in tissues by anti-inflammatory processes (Di Mascio *et al.*, 1997). AVL can be isolated from *Pterodon* seed extracts or synthesized from ADV and used as an intermediate in the synthesis of nitrogenated derivatives of vouacapan (Rubinger *et al.*, 1991; Maltha *et al.*, 1995; Belinelo *et al.*, 2001; Di Mascio *et al.*, 1997).



An ORTEP-3 (Farrugia, 1997) drawing of AVL is shown in Fig. 1. The bond distances and angles are within normal ranges (Allen *et al.*, 1987). According to the puckering parameters (Cremer & Pople, 1975; Iulek & Zuckerman-Schpector, 1997), cyclohexane rings *A* [ $q_2 = 0.042$  (6) Å,  $q_3 = 0.556$  (6) Å,  $Q = 0.558$  (6) Å,  $\theta = 4.3$  (6)° and  $\varphi = 49$  (8)°] and *B* [ $q_2 = 0.031$  (9) Å,  $q_3 = -0.612$  (8) Å,  $Q = 0.612$  (8) Å,  $\theta = 177.1$  (8)° and  $\varphi = 85$  (13)°] adopt chair conformations. Ring *C* has a half-



**Figure 1**  
An ORTEP-3 (Farrugia, 1997) view of AVL, with displacement ellipsoids drawn at the 30% probability level.

boat conformation [ $q_2 = 0.504(8) \text{ \AA}$ ,  $q_3 = -0.330(7) \text{ \AA}$ ,  $Q = 0.602(7) \text{ \AA}$ ,  $\theta = 123.2(7)^\circ$  and  $\varphi = 239(7)^\circ$ ]. Similar behaviour is observed in ADV (Ruggiero *et al.*, 1997), and its amide (Branco *et al.*, 1999) and lactone derivatives (Abrahão Júnior *et al.*, 1997, 2004). As in the  $6\alpha$ -hydroxyvouacapan- $7\beta,17\beta$ -lactone compound (Abrahão-Junior *et al.*, 1997), the lactone ring is in an envelope conformation, while the furan ring is planar, with an r.m.s. deviation of fitted atoms of  $0.002 \text{ \AA}$ .

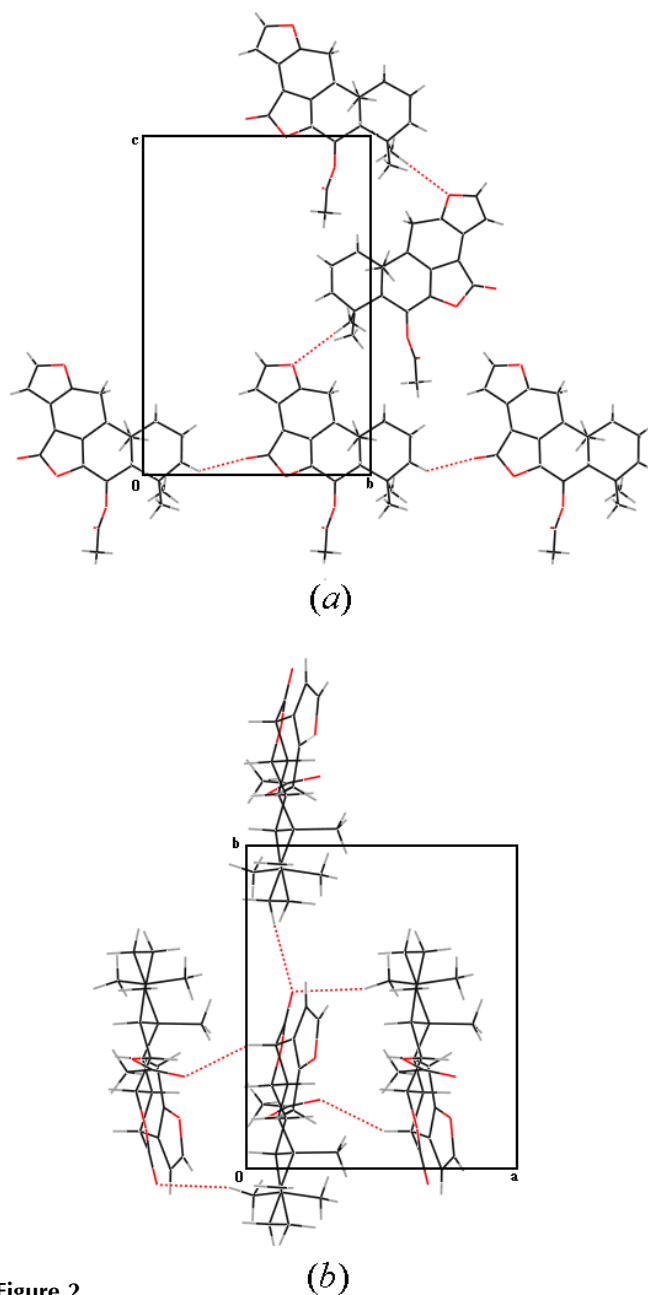
The crystal packing is supported by C—H...O interactions (Table 1) in the [100], [010] and [001] directions (Fig. 2). Both  $6\alpha$ -hydroxy- $17\beta$ -azavouacapan- $17,7\beta$ -carbolactone and  $6\alpha$ -hydroxyvouacapan- $7\beta,17\beta$ -lactone show intermolecular hydrogen bonds only in the [100] direction (Abrahão-Júnior *et al.*, 1997, 2004), while ADV exhibits intermolecular hydrogen bonds in the [100] and [010] directions (Ruggiero *et al.*, 1997).

## Experimental

The title compound was synthesized from ADV according to the procedure described in the literature (Rubinger *et al.*, 1991). Suitable single crystals of the compound were obtained by slow evaporation of a benzene–ethanol (1:2 *v/v*) solution.

### Crystal data

$C_{22}H_{28}O_5$	Mo $K\alpha$ radiation
$M_r = 372.44$	Cell parameters from 4243 reflections
Orthorhombic, $P2_12_12_1$	$\theta = 1.0\text{--}27.5^\circ$
$a = 9.6398(3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 11.5164(4) \text{ \AA}$	$T = 293(2) \text{ K}$
$c = 17.1539(5) \text{ \AA}$	Prism, colourless
$V = 1904.35(10) \text{ \AA}^3$	$0.16 \times 0.11 \times 0.06 \text{ mm}$
$Z = 4$	
$D_x = 1.299 \text{ Mg m}^{-3}$	



**Figure 2**  
Short contacts (dotted lines) in the packing of the title compound, viewed (a) down the  $a$  axis and (b) down the  $c$  axis.

### Data collection

Nonius KappaCCD diffractometer	$R_{\text{int}} = 0.037$
$\omega$ and $\varphi$ scans with $\kappa$ offsets	$\theta_{\text{max}} = 25^\circ$
Absorption correction: none	$h = -11 \rightarrow 11$
5957 measured reflections	$k = -13 \rightarrow 12$
3320 independent reflections	$l = -20 \rightarrow 20$
2683 reflections with $I > 2\sigma(I)$	

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.3858P]$
$R[F^2 > 2\sigma(F^2)] = 0.047$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.117$	$(\Delta/\sigma)_{\text{max}} = 0.004$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
3320 reflections	$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$
249 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: $0.014(4)$

**Table 1**

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C18—H18A $\cdots$ O4 <sup>i</sup>	0.96	2.59	3.517 (5)	162
C14—H14A $\cdots$ O5 <sup>i</sup>	0.98	2.54	3.450 (4)	154
C3—H3B $\cdots$ O4 <sup>ii</sup>	0.97	2.55	3.342 (3)	138
C19—H19A $\cdots$ O1 <sup>iii</sup>	0.96	2.69	3.641 (4)	173

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

In the absence of significant anomalous scattering effects, the Flack (1983) parameter is essentially meaningless. Friedel pairs were merged before refinement. H atoms were positioned geometrically ( $C-H = 0.93-0.98$  Å) and a riding model was used during the refinement process with  $U_{iso}$  set at 1.5 (for methyl H atoms) or 1.2 (for the remaining H atoms) times the value of  $U_{eq}$  of the atom to which they are attached. The absolute configuration was assigned arbitrarily.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *MERCURY* (CCDC, 2003); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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