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Key indicators

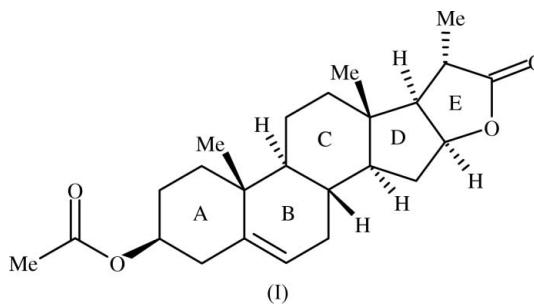
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
Disorder in main residue
 R factor = 0.047
 wR factor = 0.127
Data-to-parameter ratio = 10.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**3 β -Acetoxy-16 β -hydroxypregn-5-ene-20-carboxylic acid 16-lactone (vespertilin acetate)**

In the title compound, $\text{C}_{24}\text{H}_{34}\text{O}_4$, the six-membered rings *A*, *B* and *C* of the steroid structure have chair, half-chair and slightly flattened chair conformations, respectively, while the five-membered rings *D* and *E* have twisted and envelope conformations, respectively. The *B/C* and *C/D* ring junctions are both *trans*, whereas the tetrahydrofuran ring *E* is *cis*-fused to the cyclopentane ring *D*.

Received 1 August 2006
Accepted 5 August 2006

Comment

20-Carboxylic acid-16-lactones or bisnorcholanolic lactones have been isolated from different vegetal sources. Several naturally occurring (González *et al.*, 1983) and synthetic (Mola-Gárate *et al.*, 2003) members of this family of compound have attracted some interest because of their biological activity. We have recently reported a convenient procedure that allows the production of such compounds by $\text{BF}_3 \cdot \text{Et}_2\text{O}$ -induced abnormal Beckmann rearrangement of 23-hydroxyiminosapogenins (Iglesias-Arteaga & Alvarado-Nuño, 2006). In the present paper, we report the structure of the title compound, (I), which has been derived from 23-hydroxyiminosapogenin acetate, (II), by abnormal Beckmann rearrangement. The structure of (II) was reported previously (Iglesias-Arteaga *et al.*, 2004).



The *B/C* and *C/D* rings junctions are *trans*, while the tetrahydrofuran ring *E* is *cis*-fused to the cyclopentane ring *D* (Fig. 1). The presence of the acetoxy group bonded to C3 does not disturb the chair conformation of ring *A* [puckering parameters (Cremer & Pople, 1975) ($\text{C}1-\text{C}5/\text{C}10$) $Q = 0.553(3)\text{ \AA}$, $\theta = 3.3(3)^\circ$ and $\varphi = 79(5)^\circ$; all asymmetry parameters less than $4.4(3)^\circ$ (Duax *et al.*, 1976)]. Rotational symmetry is dominant; a pseudo- C_2 axis bisects the $\text{C}3-\text{C}4$ bond with asymmetry parameters $\Delta\text{C}_2(\text{C}3-\text{C}4) = 3.9(4)^\circ$ and $\Delta\text{C}_s(\text{C}3) = 1.3(3)^\circ$, while ring *B* assumes an almost perfect half-chair, which contains a double bond between atoms $\text{C}5$ and $\text{C}6$ [puckering parameters ($\text{C}5-\text{C}10$) $Q = 0.520(3)\text{ \AA}$, $\theta = 52.2(3)^\circ$ and $\varphi = 207.4(4)^\circ$; asymmetry parameter $\Delta\text{C}_2(\text{C}5-$

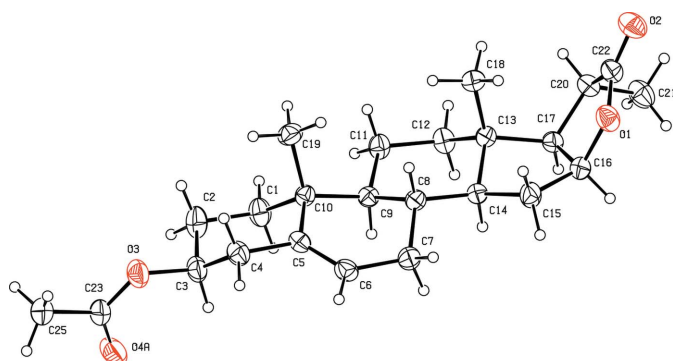


Figure 1

The structure of (I) with displacement ellipsoids at the 30% probability level. One component of the disordered O4 site has been omitted.

C6) = 3.3 (4)°]. Ring C shows a slightly flattened chair conformation [puckering parameters (C8/C9/C11–C14) $Q = 0.565$ (3) Å, $\theta = 13.6$ (3)° and $\varphi = 250.1$ (11)°]. The five-membered D ring has a C13–C14 twisted conformation with puckering parameters (C13–C17) $q_2 = 0.448$ (3) Å and $\varphi_2 = 194.1$ (4)° [pseudorotation (Altona *et al.*, 1968) and asymmetry parameters: $\Delta = 2.1$ (2), $\tau_m = 45.5$ (2), $\Delta C_s(14) = 20.4$ (4) and $\Delta C_2(13,14) = 4.7$ (3)°]. Finally, ring E has a 17-envelope conformation with puckering parameters (O1/C16/C17/C20/C22) $q_2 = 0.251$ (3) Å and $\varphi_2 = 76.2$ (6)°. In (I), the molecules are linked by van der Waals interactions, forming a ribbon structure along the [100] direction.

Comparing the structures of (I) and (II), there are differences in the values of the torsion angles in the A–D rings. As a consequence, there are variations in the asymmetry parameters. However, the conformations of the rings of both compounds are the same except for ring D, *i.e.* twisted and envelope forms in (I) and (II), respectively.

Experimental

The title compound was obtained in 84% yield by BF₃·Et₂O-induced abnormal Beckmann rearrangement of 23-hydroxyiminodiosgenin acetate (Iglesias-Arteaga & Alvarado-Nuño, 2006). Crystals suitable for X-ray analysis were obtained from a solution of (I) (100 mg, 0.26 mmol) in ethyl acetate (2 ml) by slow evaporation overnight.

Crystal data

C ₂₄ H ₃₄ O ₄	Z = 2
$M_r = 386.51$	$D_x = 1.202$ Mg m ⁻³
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 9.8290$ (12) Å	$\mu = 0.08$ mm ⁻¹
$b = 6.6580$ (18) Å	$T = 298$ (2) K
$c = 16.566$ (2) Å	Thick plate, colorless
$\beta = 99.910$ (9)°	0.5 × 0.4 × 0.2 mm
$V = 1067.9$ (3) Å ³	

Data collection

Siemens P4 diffractometer	$R_{int} = 0.034$
2 θ/ω scans	$\theta_{max} = 28^\circ$
Absorption correction: none	3 standard reflections
3706 measured reflections	every 97 reflections
2756 independent reflections	intensity decay: 7.2%
2167 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.127$
 $S = 1.02$
 2756 reflections
 267 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0744P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.004$
 $\Delta\rho_{max} = 0.18$ e Å⁻³
 $\Delta\rho_{min} = -0.18$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C1–C2	1.542 (4)	C22–O2	1.200 (4)
C3–O3	1.472 (3)	C22–O1	1.347 (4)
C5–C6	1.339 (3)	C23–O3	1.324 (4)
C16–O1	1.467 (4)		
C10–C1–C2–C3	–56.5 (4)	C6–C7–C8–C9	–47.7 (3)
C1–C2–C3–C4	56.6 (4)	C7–C8–C9–C10	64.4 (3)
C2–C3–C4–C5	–55.3 (3)	C4–C5–C10–C1	–51.4 (3)
C3–C4–C5–C10	54.6 (3)	C6–C5–C10–C9	11.7 (4)
C10–C5–C6–C7	3.6 (5)	C2–C1–C10–C5	51.7 (3)
C5–C6–C7–C8	15.2 (4)	C8–C9–C10–C5	–44.7 (3)

The carboxyl O atom is disordered over two sites (O4A and O4B) with occupation factors 0.56 (9) and 0.44 (9), respectively. H atoms attached to C atoms were placed in geometrically idealized positions and refined as riding on their parent atoms, with C–H = 0.93–0.98 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$, or $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups. In the absence of significant anomalous scattering, Friedel pairs were merged. The absolute configurations of each chiral center in the title compound were assumed to be the same as those of the starting material (Iglesias-Arteaga & Alvarado-Nuño, 2006).

Data collection: XSCANS (Siemens, 1993); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SIR2004 (Burla *et al.*, 2005); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

Thanks are due to the Dirección General de Asuntos del Personal Académico (DGAPA–UNAM) for support *via* project IN200105. MFA is indebted to Dr R Moreno-Esparza for useful comments.

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