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Incaniline (isoargentatin A) from *Parthenium argentatum*

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Key indicators

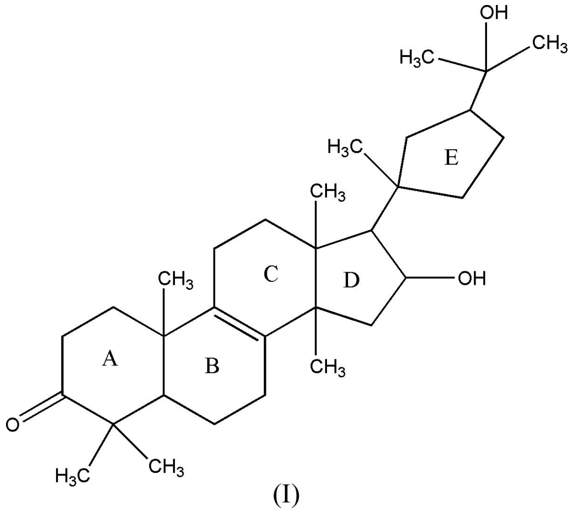
Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.117
Data-to-parameter ratio = 8.6

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

The title compound, (20*S*,24*R*)-incanilin, $C_{30}H_{48}O_4$, contains three six-membered rings and two five-membered rings. The six-membered ring *B* adopts a distorted envelope conformation and five-membered ring *D* adopts a twist conformation. In the crystal structure, the molecules exist as O—H···O hydrogen-bonded dimers.

Comment

Guayule (*Parthenium argentatum*) has been studied due to the high content of rubber in its roots and aerial parts. As a result of the industrial process used to obtain this rubber, high quantities of a resin by-product are also produced. Our research has shown that cycloartane-type triterpenes are the main constituents of this resin. We have also demonstrated that some of these triterpenes possess antimicrobial activity (Martínez-Vázquez *et al.*, 1994) and show a non-competitive inhibition of oestrogens in breast cancer (Calzada *et al.*, 1995).



(I)

Although there is a previous report on the stereochemistry of incaniline (isoargentatine A; Romo de Vivar & Matsubara, 1986), no report on the crystal structure determination of this compound has appeared. Therefore, due to this lack of data and as part of an effort to explore the structure–activity relationships of natural products isolated from guayule, the X-ray crystal structure determination of incaniline, (I), was undertaken.

Incaniline is a pentacyclic triterpene (Fig. 1), with three six-membered rings (*A*, *B* and *C*) and two five-membered rings (*D* and *E*). The *A*/*B* and *C*/*D* ring junctions are *trans* fused. Ring *A* adopts a slightly distorted chair conformation, as evidenced by the Cremer & Pople (1975) puckering parameters of q_2 =

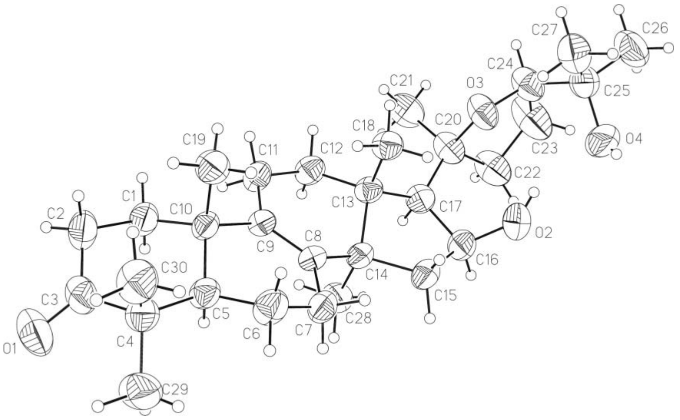


Figure 1
The molecular structure of (I), showing 40% probability displacment ellipsoids and the atomic numbering scheme.

0.083 (2) Å, q_3 = 0.512 (2) Å and φ_2 = −18 (2)°. This distortion is caused by the O1═C3 carbonyl bond [1.213 (4) Å]. The total puckering amplitude, Q_T [0.518 (2) Å], is slightly lower than the value for an ideal chair conformation [0.63 Å].

Rings *B* and *C* are fused through the C═C double bond [C8═C9 1.335 (4) Å]. The puckering parameters of ring *B* show that this ring adopts a half-boat conformation, with q_2 = 0.401 (2) Å, q_3 = 0.331 (2) Å, φ_2 = 11.0 (3)° and Q_T = 0.520 (2) Å. The C10—C9—C8—C7—C6 fragment in ring *B* is planar (r.m.s deviation 0.038 Å), with atom C5 deviating from it by 0.698 (5) Å. Ring *C* also adopts a half-boat conformation, with q_2 = 0.418 (2) Å, q_3 = 0.324 (2) Å, φ_2 = −107.0 (3)° and Q_T = 0.528 (2) Å. Atom C13 deviates from the C14/C8/C9/C11/C12 plane (r.m.s deviation 0.045 Å) by 0.720 (4) Å.

Ring *D* is twisted around the C13—C14 bond, with puckering parameters q_2 = 0.464 (2) Å and φ_2 = −163.6 (3)°. Ring *E* adopts a slightly distorted envelope conformation, with q_2 = 0.350 (2) Å and φ_2 = −132.0 (4)°. The relative orientation of rings *D* and *E* in (I) is different from that observed in Beesioside J (Ju *et al.*, 2002) and 3,15,16,18-tetra-acetoxy-20,24-epoxy-9,19-cyclolanostane-3,15,25-triol-*D*-pyranoside hemihydrate (Sakurai *et al.*, 1986).

In the crystal structure of (I), the molecules exist as O—H⋯O hydrogen-bonded dimers (Table 2).

Experimental

The title compound was isolated from the roots of *Parthenium argentatum*, as previously reported by Romo de Vivar *et al.* (1990). Crystals of (I) suitable for X-ray diffraction were grown by recrystallization from an ethanol solution.

Crystal data

$C_{30}H_{48}O_4$
M_r = 472.68
Orthorhombic, $C222_1$
a = 13.9158 (7) Å
b = 14.8078 (8) Å
c = 26.3807 (14) Å
V = 5436.1 (5) Å^3
Z = 8
D_x = 1.155 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 8001 reflections
θ = 2.2–31.0°
μ = 0.07 mm^{-1}
T = 291 (2) K
Prism, colourless
0.40 × 0.30 × 0.30 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
22368 measured reflections
2652 independent reflections
2096 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.047
$\theta_{\rm max}$ = 25.0°
$h = -16 \rightarrow 16$
$k = -17 \rightarrow 17$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.117
S = 1.02
2652 reflections
309 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.006
$\Delta\rho_{\rm max}$ = 0.17 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.16 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.210 (4)	C1—C10	1.530 (4)
O2—C16	1.433 (4)	C2—C3	1.481 (5)
O3—C24	1.435 (4)	C3—C4	1.517 (5)
O3—C20	1.439 (4)	C4—C5	1.564 (5)
O4—C25	1.404 (5)	C5—C10	1.549 (4)
C1—C2	1.525 (5)		
C24—O3—C20	109.7 (2)	O3—C20—C17	110.8 (3)
O1—C3—C2	120.6 (4)	O3—C24—C25	107.8 (3)
O1—C3—C4	122.7 (4)	O3—C24—C23	106.5 (3)
O2—C16—C15	109.6 (3)	O4—C25—C27	109.8 (4)
O2—C16—C17	114.6 (3)	O4—C25—C24	106.3 (3)
O3—C20—C21	108.5 (3)	O4—C25—C26	108.2 (3)
O3—C20—C22	103.1 (3)		
C10—C1—C2—C3	−56.7 (4)	C10—C9—C11—C12	176.0 (3)
C1—C2—C3—C4	51.3 (5)	C11—C12—C13—C14	54.1 (3)
C7—C8—C9—C11	177.5 (3)	C15—C16—C17—C13	15.8 (3)
C7—C8—C9—C10	0.8 (5)	O3—C20—C22—C23	−36.6 (4)
C8—C9—C11—C12	−0.9 (5)		

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O2—H2⋯O4$^{\rm i}$	0.82	2.00	2.689 (4)	141

Symmetry code: (i) $x, -y+1, -z+1$.

H atoms were included in calculated positions (O—H = 0.82 Å and C—H = 0.96–0.98 Å) and refined using a riding model, with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(carrier) for hydroxy and methyl H atoms and 1.2$U_{\rm eq}$(carrier) for other H atoms. A rotating-group refinement was used for the OH groups. In the absence of significant anomalous scattering effects, Freidel pairs were merged during refinement and the absolute configuration was not determined; the enantiomer used in the refined model was assigned arbitrarily.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PARST95* (Nardelli, 1995).

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