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17β-Hydroxy-28-norolean-12-ene-3,16-dione

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Key indicators

Single-crystal X-ray study
T = 286 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.089
Data-to-parameter ratio = 10.5

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

Molecules of the title compound, $C_{29}H_{44}O_3$, are linked into chains by intermolecular O—H···O hydrogen bonds involving the carbonyl and hydroxyl groups.

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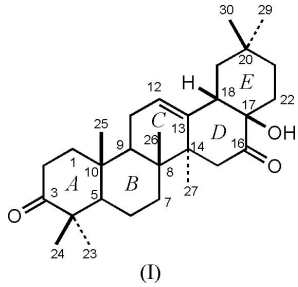
Comment

The title compound, (I), is a 28-noroleanane-type triterpene isolated from the medicinal plant *Doellingeria scaber* Thunb used for treatment of traumatic injury and snake bite. Compound (I) has been reported previously (Itokawa *et al.*, 1981), but its structure, determined by spectroscopic methods, seems questionable. The crystal structure analysis of (I) was undertaken to establish the structure unambiguously.



(I)

An *ORTEP*-3 drawing (Farrugia, 1997) of the molecule is shown in Fig. 1. The X-ray analysis of (I) shows that the hydroxyl group with a β-orientation is located on C17, not on C18 as reported previously based on spectroscopic methods (Itokawa *et al.*, 1981). The *A*/*B* and *B*/*C* ring junctions show *trans* fusion and the geometry of the rings is *cis* at the *D*/*E* ring junction. The bond lengths and angles in (I) have normal values (Allen *et al.*, 1987), with the following average values (Å): Csp^3—Csp^3 = 1.539 (3), Csp^3—Csp^2 = 1.515 (3), Csp^2=Csp^2 = 1.313 (3), C=O = 1.211 (4) and C—O = 1.432 (3). Rings *B* and *E* have slightly flattened chair conformations, with average torsion angles of 53.8 (3) and 52.6 (3)°, respectively. Rings *A* and *D* also adopt chair conformations, with average torsion angles of 47.9 (3) and 48.0 (3)°, respectively. Ring *C* adopts an envelope conformation. The crystal packing is stabilized by intermolecular O—H···O hydrogen bonds involving the hydroxyl group and the C16-carbonyl group (Table 1). The hydrogen bonds link the molecules into chains along the *b* axis (Fig. 2).

Experimental

The dried and powdered roots of *Doellingeria scaber* (6.5 kg) were extracted three times with petroleum–methanol–diethyl ether (1:1:1) at room temperature. After evaporation under reduced pressure, the residue was separated by repeated silica gel (200–300 mesh) column

chromatography and recrystallization, giving compound (I) (yield: 80 mg; m.p. 505–506 K; optical rotation: $[\alpha]_D^{25}$ +42°). Crystals suitable for X-ray diffraction measurements were obtained by slow evaporation of a solution of (I) in acetone at room temperature.

Crystal data

$C_{29}H_{44}O_3$
M_r = 440.64
Orthorhombic, $P2_12_12_1$
a = 7.273 (1) Å
b = 11.298 (2) Å
c = 30.344 (5) Å
V = 2493.4 (7) Å^3
Z = 4
D_x = 1.174 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 28 reflections
θ = 2.2–11.7°
μ = 0.07 mm^{-1}
T = 286 (2) K
Block, colourless
0.56 × 0.38 × 0.32 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
3381 measured reflections
3124 independent reflections
1830 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 27.0°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 14$
$l = -1 \rightarrow 38$
3 standard reflections
every 97 reflections
intensity decay: 2.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.089
S = 0.86
3124 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0099 (7)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···O2^{i}	0.82	2.24	3.054 (3)	173

Symmetry code: (i) $2-x, y-\frac{1}{2}, \frac{3}{2}-z$.

All H atoms were placed in calculated positions (O—H = 0.82 Å and C—H = 0.93–0.97 Å) and allowed to ride on the carrier atom, with U_{iso}(H) values constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for remaining H atoms. Friedel reflections were merged before the final refinement because of the absence of significant anomalous scattering effects.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997*b*); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

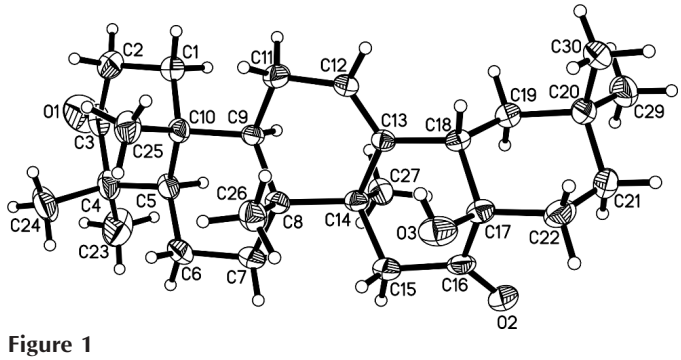


Figure 1
An *ORTEP-3* drawing (Farrugia, 1997) of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

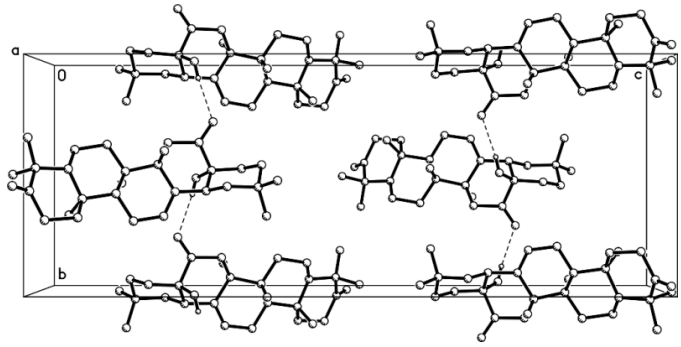


Figure 2
The molecular packing of (I), viewed along the *a* axis. Dashed lines represent O—H···O hydrogen bonds. Only the H atoms involved in hydrogen bonding are shown.

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