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6α-Hydroxy-12-oleanen-3-one: a triterpenoid from the stalks of *Celastrus hypoleucus*

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.107
Data-to-parameter ratio = 9.7

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

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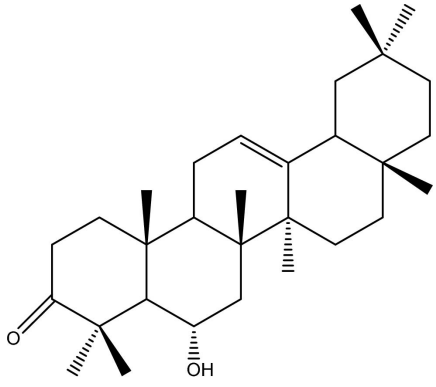
The title compound, $C_{30}H_{48}O_2$, is a triterpenoid which was isolated from the stalks of *Celastrus hypoleucus* (Oliv.) *Warb*. The molecule contains five six-membered rings adopting distorted boat, half-chair and chair conformations. The hydroxy groups serve as hydrogen-bond donors and the carbonyl groups as acceptors, forming molecular chains parallel to the *b* axis.

Comment

Celastraceae plants have been the subject of continued and growing interest, due to the range of biological activities shown by many members of this family (Bruning & Wagner, 1978; Tu, 1990, 1991; Jiang *et al.*, 1996). Pharmaceutical studies and clinical practice have demonstrated that sesquiterpenes and triterpenes possess notable antibacterial, antitumour, insect antifeedant and cytoxic activities (Chen & Liang, 1999). Our investigation of the bioactive constituents of *Celastrus hypoleucus* (Oliv.) Warb., a perennial plant belonging to the Celastraceae family, led to the isolation of 6α-hydroxyl-12-oleanene-3-one, (I). The structure of (I) was elucidated by spectroscopic analysis, including two-dimensional NMR spectroscopy, and was confirmed by single-crystal X-ray diffraction analysis, the results of which are presented here.



(I)

The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. The molecule contains five six-membered rings (*A* atoms C1–C5/C10, *B* C5–C10, *C* C8/C9/C11–C14, *D* C13–C18 and *E* C17–C22). Rings *B*, *D* and *E* adopt chair conformations, while ring *A* adopts a slightly distorted boat conformation and ring *C* a slightly distorted half-chair conformation, as a result of the C3 carbonyl group and the C12═C13 double bond, respectively. All rings are *trans* fused, except for the *D*/*E* junction, which is *cis*. The orientation of the hydroxy group is axial.

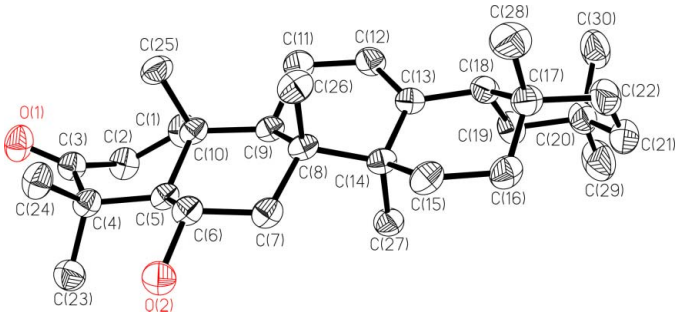


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

In the crystal structure of (I), the hydroxy groups serve as hydrogen-bond donors and the carbonyl groups as acceptors, forming molecular chains running parallel to the *b* axis.

Experimental

Stalks of *Celastrus hypoleucus* (Oliv.) Warb. were collected in Jiujiang city, Jiangxi Province, China, in September 2002. The shade-dried powder of the stalks (10 kg) was extracted at room temerature three times with methanol (3 × 20 l). The extracts were evaporated *in vacuo* affording a gummy residue (514 g). This residue was partitioned in H_2O and extracted at room temerature with petroleum ether (4 × 3000 ml). The petroleum ether extract (103 g) was adsorbed on to silica gel (100 g) and then subjected to column chromatography (silica gel, 1 kg, 200–300 mesh), eluted with petroleum ether–AcOEt (gradients 10:0–0:10). The eluted fractions were evaluated by thin-layer chromatography and combined to give 16 main fractions. Fraction 8 (4 g) was rechromatographed on a silica-gel (80 g) column with petroleum ether–acetone (5:1) to afford the pure title compound, (I) (m. p. 492–494 K). ^{13}C NMR (125 MHz, $CDCl_3$, δ, p.p.m.): 219.6 (C3), 144.8 (C13), 121.9 (C12), 68.0 (C6), 59.0 (C5), 47.5 (C9), 47.4 (C4), 47.0 (C19), 45.9 (C18), 43.4 (C7), 42.3 (C8), 41.0 (C14), 39.2 (C1), 38.3 (C10), 37.3 (C22), 34.9 (C21), 33.5 (C29), 33.3 (C2), 32.8 (C17), 32.0 (C23), 31.3 (C20), 28.7 (C28), 27.2 (C16), 26.3 (C15), 26.0 (C27), 23.9 (C11), 23.9 (C30), 20.4 (C24), 17.5 (C25), 17.5 (C26). Crystals of (I) suitable for X-ray structure analysis were obtained by slow evaporation of a methanol solution at room temperature.

Crystal data

$C_{30}H_{48}O_2$
M_r = 440.68
Orthorhombic, $P2_12_12_1$
a = 11.5254 (14) Å
b = 14.5357 (18) Å
c = 15.769 (2) Å
V = 2641.7 (6) Å^3
Z = 4
D_x = 1.108 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3643 reflections
θ = 2.2–21.5°
μ = 0.07 mm^{-1}
T = 293 (2) K
Prism, colourless
0.46 × 0.40 × 0.33 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
14520 measured reflections
2915 independent reflections
2407 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 26.0°
$h = -14 \rightarrow 14$
$k = -8 \rightarrow 17$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.107
S = 1.07
2915 reflections
299 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.1992P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0025 (6)

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.215 (3)	C3—C4	1.524 (3)
O2—C6	1.432 (3)	C12—C13	1.330 (3)
C2—C3	1.491 (3)		
C3—C2—C1	112.2 (2)	C3—C4—C23	103.8 (2)
O1—C3—C2	122.3 (2)	O2—C6—C7	109.01 (17)
O1—C3—C4	122.3 (2)	O2—C6—C5	108.53 (18)
C2—C3—C4	115.3 (2)	C7—C6—C5	110.97 (17)
C3—C4—C24	109.9 (2)		
C1—C2—C3—O1	121.6 (3)	C2—C3—C4—C5	30.4 (3)
C1—C2—C3—C4	−60.7 (3)	C3—C4—C5—C6	159.00 (19)
O1—C3—C4—C24	−24.4 (3)	C10—C5—C6—O2	−178.24 (17)
C2—C3—C4—C24	157.9 (2)	C4—C5—C6—O2	52.2 (2)
O1—C3—C4—C23	90.4 (3)	C10—C5—C6—C7	−58.5 (2)
C2—C3—C4—C23	−87.3 (2)	C4—C5—C6—C7	171.98 (18)
O1—C3—C4—C5	−152.0 (2)	O2—C6—C7—C8	178.33 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O1^{i}	0.82	2.08	2.901 (2)	175

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

H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å and O—H = 0.82 Å, and refined as riding atoms, with U_{iso}(H) set equal to 1.2U_{eq}(C) or 1.5U_{eq}(O). The absolute configuration could not be determined from the X-ray analysis, as no strong anomalous scatterers are present; 2242 Friedel pairs were therefore merged before refinement.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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