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Eudesma-5,12-dien-13-oic acid from *Laggera pterodonta*

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Key indicators

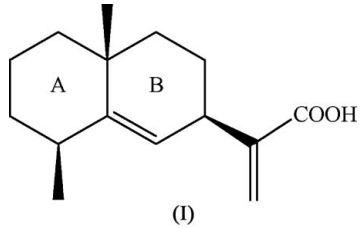
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.105
Data-to-parameter ratio = 10.1

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

The title compound, $C_{15}H_{22}O_2$, is an eudesmane sesquiterpenoid which was isolated from *Laggera pterodonta* (DC) Benth. The molecule contains two six-membered rings. An intermolecular hydrogen bond is formed between the hydroxy and carbonyl groups.

Comment

Laggera pterodonta (DC) Benth is a perennial herbaceous plant growing in southwestern China, mainly in Yunnan province. The aerial part of *L. pterodonta* has been used as a folk medicine in traditional Chinese medicine. Pharmacological research indicates that the extract of *L. pterodonta* has antileukaemic, antibacterial, anti-inflammatory and antimalarial activities (Dou, 1998; Zhao *et al.*, 1997; Li & Ding, 1996). To investigate the bioactive natural products from *L. pterodonta*, chemical studies of *L. pterodonta* have been undertaken, and we obtained the title compound, (I). The structure of (I) was elucidated by spectroscopic analysis, including two-dimensional NMR spectroscopy, and confirmed by single-crystal X-ray diffraction analysis.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The molecule contains two six-membered rings. Atoms C5 and C10 bridge rings *A* (atoms C1–C5/C10) and *B* (C5–C10). Ring *A* adopts a chair conformation, while ring *B* adopts a slightly distorted half-chair conformation, as a result of the double bond between atoms C5 and C6. The packing of the molecules in the solid state is stabilized by intermolecular O—H···O interactions between the hydroxy and carbonyl groups (Table 1).

This investigation was performed independently of another study which reports the same structure in the preceding paper (Mei *et al.*, 2006).

Experimental

Laggera pterodonta (DC) Benth was collected from Kunming, Yunnan Province, China, in August 2002 and identified by Professor Wen-Yuan Gao. A voucher specimen (D20020818) was deposited in the laboratory of the School of Pharmacy, Tianjin Medical University, China. The dried aerial parts (850 g) of *Laggera pterodonta* were crushed and extracted three times with EtOH (95%, 10 l each) at

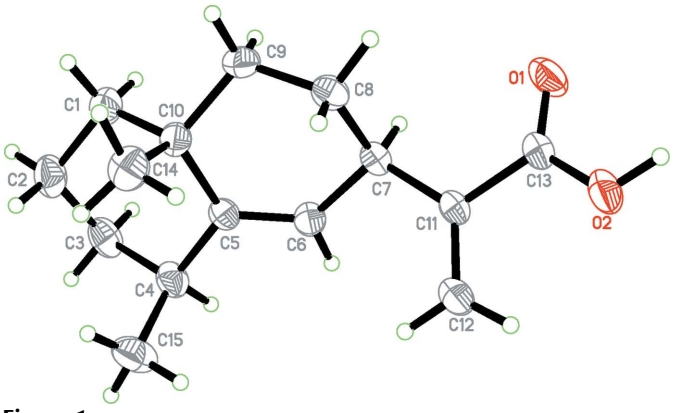


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

333 K for 6 h. The EtOH extract was concentrated under reduced pressure to give a residue (110 g), which was suspended in H_2O and then partitioned with petroleum ether, EtOAc and *n*-BuOH. The EtOAc layer (20 g) was chromatographed on a silica-gel column eluted with solvents of increasing polarity ($CHCl_3$–MeOH: 95:5, 9:1, 8:2) to yield eight fractions. Fraction 1 (0.439 g) was further separated on Toyopear HW-40 ($CHCl_3$–MeOH 2:1) to give four fractions (fraction 1.1–1.4). Fraction 1.3 (0.098 g) was purified by high-performance liquid chromatography (ODS, MeOH–H_2O 9:1) to give 0.038 g of the pure title compound, (I) (m.p. 369.5–370.5 K). ^{13}C NMR (125 MHz, C_5D_5N): δ 42.8 (C1), 18.5 (C2), 30.1 (C3), 39.0 (C4), 148.7 (C5), 124.9 (C6), 39.6 (C7), 27.6 (C8), 42.6 (C9), 35.2 (C10), 148.0 (C11), 122.8 (C12), 169.9 (C13), 28.0 (C14), 23.9 (C15). Crystals suitable for X-ray structure analysis were obtained by slow evaporation of an acetone solution at room temperature.

Crystal data

$C_{15}H_{22}O_2$
M_r = 234.33
Orthorhombic, $P2_12_12_1$
a = 6.3452 (12) Å
b = 14.032 (3) Å
c = 15.132 (3) Å
V = 1347.2 (5) Å^3
Z = 4
D_x = 1.155 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 294 (2) K
Prism, colourless
0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.981, T_{max} = 0.985
7658 measured reflections
1619 independent reflections
1203 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.02
1619 reflections
160 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.0486P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.90 (4)	1.83 (4)	2.708 (2)	165 (3)

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

Hydroxy atom H2 was located in a difference density map and the atomic coordinates allowed to refine freely. All other H atoms were positioned geometrically and refined as riding (C—H = 0.93–0.98 Å). For the CH and CH_2 groups, U_{iso}(H) values were set equal to 1.2U_{eq}(C) and for the methyl groups they were set equal to 1.5U_{eq}(C). The absolute configuration could not be established because of the absence of significant anomalous effects. Friedel pairs were merged for the final cycles of refinement.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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