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Pterodontic acid

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.126
Data-to-parameter ratio = 11.3

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

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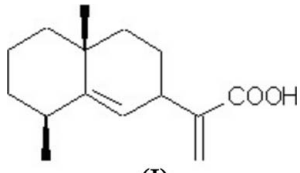
In the title compound, $C_{15}H_{22}O_2$, the C=C bond lengths in the cyclohexene ring and the allylic acid group [1.325 (3) and 1.324 (3) Å] are almost equal and are shorter than those observed in ethylene. The C=O and C—O bond lengths in the allylic acid group [1.201 (3) and 1.321 (3) Å] are almost equal to those of formic acid. There is an intramolecular hydrogen bond between the allylic acid H atom and the hydroxy O atom [C···O = 2.673 (2) Å and C—H···O = 101°].

Comment

Laggera pterodonta (DC) Benth (Compositae) is widely distributed in southwestern China, especially in Yunnan province. It has been used as a traditional herbal medicine for its anti-inflammatory and antibacterial activities (Jiangsu New Medicial College, 1977). Previous investigations of this plant led to the isolation of 55 eudesmane sesquiterpenes and nine flavonoid compounds (Mei *et al.*, 2005). Some eudesmane sesquiterpenes isolated from this plant showed cytotoxicity towards tumour cells (Xiao *et al.*, 2003) and antibacterial activities (Wei *et al.*, 1995). These interesting activities of eudesmane sesquiterpenes have prompted us to isolate more sesquiterpenes to evaluate their biological activities. As a result, one eudesmane sesquiterpene, pterodontic acid, (I) (Li *et al.*, 1996), was isolated from an EtOAc fraction of this plant.



(I)

The molecular structure of (I) is shown in Fig. 1, the molecular packing is shown in Fig. 2 and selected bond lengths are listed in Table 1. This X-ray study confirms the previously proposed molecular structure. The C=C bond lengths in the cyclohexene ring and the allylic acid group are almost equal, and shorter than those observed in ethylene (1.34 Å; Jerry, 1985). The C=O and C—O bond lengths in the allylic acid group are almost the same as those in formic acid (1.20 and 1.34 Å; Jerry, 1985). There is an intramolecular hydrogen bond between the allylic acid H atom and the hydroxy O atom (Table 2). Molecules are further linked by intermolecular hydrogen bonds between the carbonyl and hydroxy groups.

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

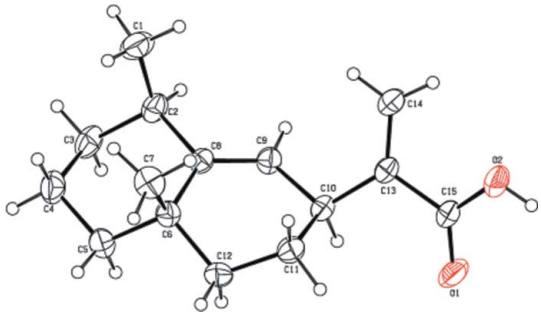


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

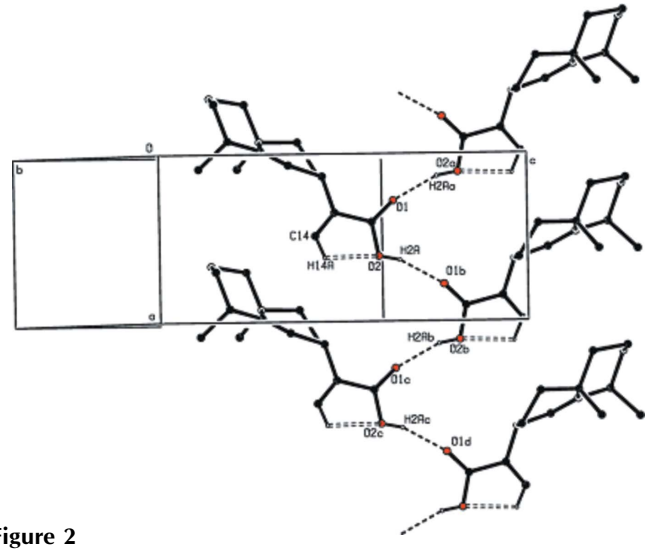


Figure 2
Plot of the crystal packing, showing the linkage of the molecules by hydrogen bonds (dashed lines), H atoms not involved in hydrogen bonding have been omitted for clarity.

Experimental

The air-dried aerial parts of the whole plant (8 kg, dry weight) were powdered and extracted twice with 70% ethanol. The residue (845 g) obtained by removal of the solvent *in vacuo* was suspended in water and extracted successively with petroleum ether, ethyl acetate and *n*-butanol. The ethyl acetate layer was evaporated to give a residue (250 g) which was subjected to column chromatography over 1500 g silica gel and eluted with a petroleum ether/ethyl acetate gradient (9:1, 8:2, 7:3, 1:1, 3:7, 0:10) and then crystallized slowly from acetone to yield compound (I) (1.2 g). ^{1}H NMR (300 MHz, $CDCl_3$, coupling constants in Hz in parentheses): δ 6.31 (*br s*, H14*a*), 5.68 (*br s*, H14*b*), 5.18 (*br s*, H9), 1.25 (*s*, H7), 1.16 (*d*, 7.5, H1). ^{13}C NMR (125 MHz, $CDCl_3$): δ 23.44 (C1), 34.61 (C2), 29.94 (C3), 17.74 (C4), 42.07 (C5), 33.40 (C6), 27.45 (C7), 149.33 (C8), 126.09 (C9), 38.34 (C10), 26.83 (C11), 41.72 (C12), 145.18 (C13), 123.01 (C14), 172.76 (C15).

Crystal data

$C_{15}H_{22}O_2$
M_r = 234.33
Orthorhombic, $P2_12_12_1$
a = 6.3425 (7) Å
b = 14.0242 (15) Å
c = 15.1156 (16) Å
V = 1344.5 (3) Å^3
Z = 4
D_x = 1.158 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 292 (2) K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.978, T_{max} = 0.985
10414 measured reflections
1865 independent reflections
1676 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.126
S = 1.08
1865 reflections
165 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0813P)^2 + 0.0392P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C15	1.201 (3)	C6—C5	1.543 (3)
O2—C15	1.321 (3)	C9—C8	1.325 (3)
C15—C13	1.490 (2)	C8—C2	1.530 (2)
C10—C13	1.510 (3)	C11—C12	1.520 (3)
C10—C9	1.510 (2)	C13—C14	1.324 (3)
C10—C11	1.531 (3)	C2—C1	1.532 (4)
C6—C8	1.521 (3)	C2—C3	1.538 (3)
C6—C12	1.540 (3)	C5—C4	1.520 (3)
C6—C7	1.542 (3)	C4—C3	1.507 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14*A*···O2	0.93	2.33	2.673 (2)	101
O2—H2*A*···O1^{i}	0.82 (1)	1.91 (2)	2.706 (2)	162 (4)

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

The hydroxy atom H2*A* and atoms H10 and H2 were located in difference density maps and their atomic coordinates allowed to refine freely. 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 and the hydroxy group they were set equal to 1.5U_{eq}(C,O). 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, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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