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In the molecule of the title compound (systematic name: 1α-hydroxy-6β,7β-diacetoxy-8,13-epoxylabd-14-en-11-one), $C_{24}H_{36}O_7$, rings *A* and *B* adopt chair conformations, while ring *C* has a twist–boat conformation. The crystal structure is stabilized by intermolecular O—H$\cdots$O and C—H$\cdots$O hydrogen bonds, linking the molecules into networks approximately parallel to the (110) plane.

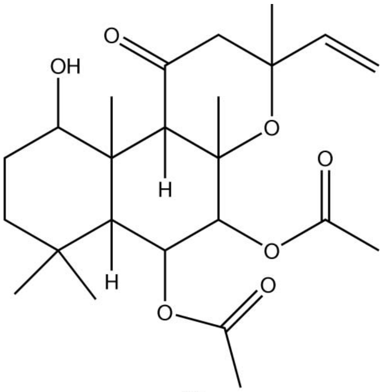
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.035
wR factor = 0.093
Data-to-parameter ratio = 7.6

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

Comment

Coleus forskohlii (Willd.) Briq. (Labiatae), which grows mostly in the tropical and subtropical regions of India, Pakistan and Sri Lanka, has been used for medical treatment in Hindu and Ayurvedic medicine (Shah *et al.*, 1980; Gabetta, *et al.*, 1989). It is a rich source of 8,13-epoxylabd-14-en-11-one diterpenes, many of which have positive inotropic, anti-hypertensive and adenylate cyclase-stimulating activities (Bhat *et al.*, 1977; Seamon *et al.*, 1983; Jin & He, 1998; Lindner *et al.*, 1978). The title compound, (I), was isolated from an ethanol extract of the plant and its structure was identified by NMR spectroscopy. Here, the crystal structure of (I) is reported.



(I)

The molecule of compound (I) consists of three fused six-membered ring systems, in addition to carbonyl, methyl, hydroxyl, allyl and acetoxy groups (Fig. 1). The bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and are comparable with the corresponding values in analogous compounds (Bick *et al.*, 1993).

Rings *A* (C1–C5/C10) and *B* (C5–C10) adopt chair conformations, with puckering parameters (Cremer & Pople, 1975) of Q_T = 0.559 (1) Å, θ = 2.57 (1)° and φ = 69.86 (18)°, and Q_T = 0.542 (1) Å, θ = 5.14 (1)° and φ = −61.06 (14)°, respectively, while ring *C* (O6/C8/C9/C11–C13) adopts a twist–boat conformation, with puckering parameters of Q_T =

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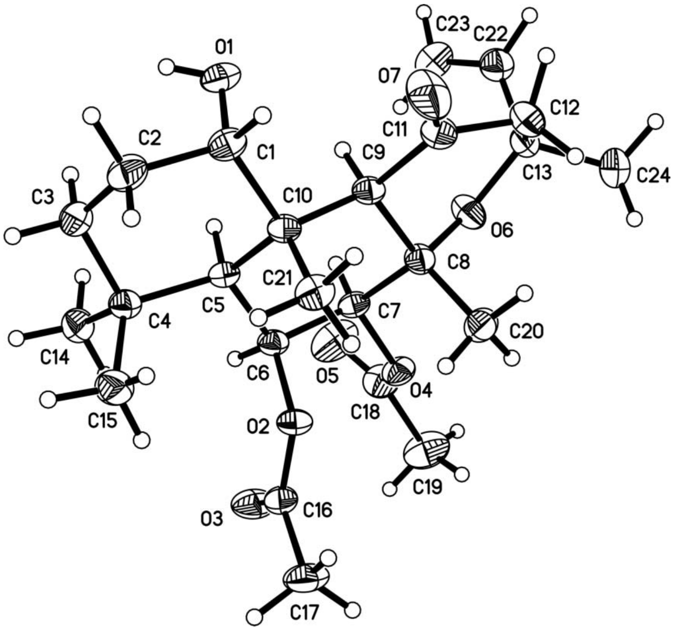

Figure 1
The molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

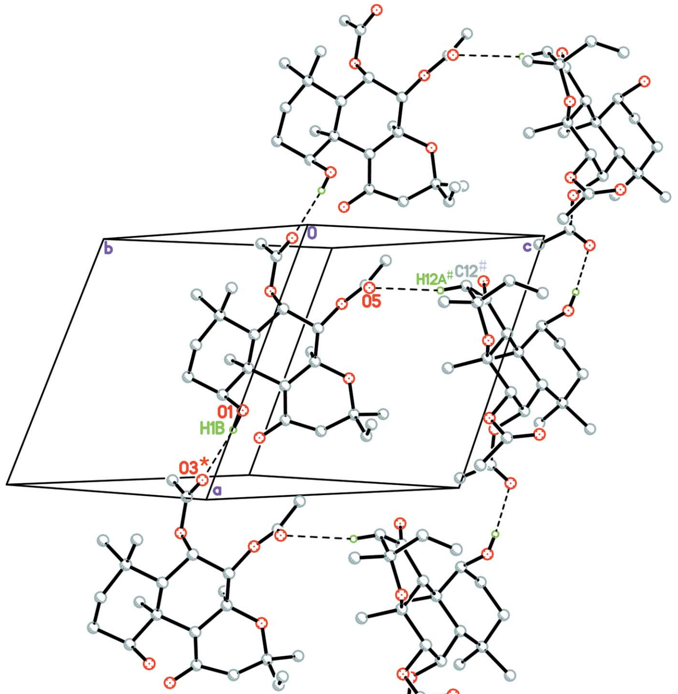

Figure 2
A packing diagram for (I). Hydrogen bonds are shown as dashed lines. Atoms marked with an asterisk (*) or hash sign (#) are at the symmetry positions $(1 + x, y, z)$ and $(1 - x, y - \frac{1}{2}, 2 - z)$, respectively. For the sake of clarity, some H atoms have been omitted.

0.635 (1) Å, θ = 75.22 (1)° and φ = 20.42 (1)°. In analogous compounds, the conformation of ring *C* was reported as a boat in solution and a chair in the solid state (Roy *et al.*, 1993; Kogler & Fehlhaber, 1991).

Ring systems *A*/*B* and *B*/*C* are *trans*-fused about the C5—C10 and C8—C9 bonds, as evidenced by the torsion angles C21—C10—C5—H5 (179.4°) and C20—C8—C8—H9 (170.8°), and this is very similar to what was observed in the analogous compound 1,9-dideoxy-coleonol-B (Roy *et al.*, 1993). The hydroxyl and allyl groups are axially attached to rings *A* and *C*, respectively. Atoms H5 and H9 are both axially linked to rings *A* and *B*. The two acetoxy groups are on the same side, as supported by the O2—C6—C7—O4 torsion angle of 49.69 (3)°.

The crystal structure of (I) is stabilized by intermolecular O—H···O and C—H···O hydrogen bonds (Table 1), which link the molecules into networks approximately parallel to the (110) plane (Fig. 2).

Experimental

Material from the whole plant of *Coleus forskohlii* (Willd.) Briq. (Labiatae) (4 kg), collected from Yunnan Province in China, was air-dried, powdered and extracted with 95% EtOH (3 × 24 l) for 2.5 h at 353 K. The concentrated EtOH extract (0.5 kg) was dissolved in H_2O and successively extracted with petroleum ether (330–360 K), EtOAc and *n*-BuOH. The petroleum ether-soluble fraction (108 g) was loaded onto a silica-gel column (100–200 mesh, 800 g) eluted with a gradient from 100% petroleum ether to petroleum ether–EtOAc (100:50), pooled by common thin-layer chromatography characteristics. The petroleum ether–EtOAc (100:8) fraction, subjected to repeated chromatography over silica-gel and Sephadex LH-20 columns, gave compound (I) (yield 30 mg, m.p. 506 K). Prismatic crystals suitable for X-ray studies were grown from petroleum ether–acetone (1:1 *v*/*v*) by slow evaporation at room temperature. Spectroscopic analysis: ESI-MS: *m*/*z* 459 $[M+\text{Na}]^+$; ^{1}H NMR ($CDCl_3$, 300 MHz, δ, p.p.m.): 0.93 (3H, *s*, H—CH_3), 0.98 (3H, *s*, H—CH_3), 1.24 (3H, *s*, H—CH_3), 1.40 (3H, *s*, H—CH_3), 1.51 (3H, *s*, H—CH_3), 2.07 [3H, *s*, H—(C═O)—CH_3], 2.08 [3H, s, H—(C═O)—CH_3], 1.62 (1H, *d*, *J* = 2.5 Hz, H5α), 2.59 (1H, *d*, *J* = 18.2 Hz, H12*a*), 2.71 (1H, *d*, *J* = 18.2 Hz, H12*b*), 3.60 (1H, *s*, H9α), 4.39 (1H, *bt*, *J* = 2.7 Hz, H1β), 5.10 (1H, *d*, *J* = 3.4 Hz, H7α), 5.75 (1H, *dd*, *J* = 2.5 and 3.4 Hz, H6α), 5.08 (1H, *d*, *J* = 10.8 Hz, H15*a*), 5.21 (1H, *d*, *J* = 17.2 Hz, H15*b*), 5.97 (1H, *dd*, *J* = 10.8, 17.2 Hz, H14); ^{13}C NMR ($CDCl_3$, 75 MHz, δ, p.p.m.): 71.0 (C1), 25.5 (C2), 36.3 (C3), 33.9 (C4), 46.2 (C5), 69.9 (C6), 78.6 (C7), 77.9 (C8), 57.8 (C9), 41.9 (C10), 207.1 (C11), 49.6 (C12), 74.8 (C13), 145.8 (C14), 112.8 (C15), 31.5 (C16), 24.0 (C17), 32.6 (C18), 22.8 (C19), 17.8 (C20) 170.0 (C═O6 or 7-Ac), 170.2 (C═O6 or 7-Ac), 20.9 (CH_3-6 or 7-Ac), 21.3 (CH_3-6 or 7-Ac). The MS, IR and NMR data are consistent with the reference data (Shen *et al.*, 2002).

Crystal data

$C_{24}H_{36}O_7$
M_r = 436.53
Monoclinic, $P2_1$
a = 9.839 (3) Å
b = 11.981 (4) Å
c = 10.908 (4) Å
β = 114.982 (4)°
V = 1165.5 (7) Å^3

Z = 2
D_x = 1.244 Mg m^{-3}
Mo *K*α radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
Prism, colourless
0.48 × 0.45 × 0.40 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.958$, $T_{\max} = 0.965$
6139 measured reflections
2168 independent reflections
1884 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.093$
$S = 1.00$
2168 reflections
287 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.222P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.016$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1B···O3$^{\rm i}$	0.82	2.16	2.972 (3)	172
C12—H12A···O5$^{\rm ii}$	0.97	2.61	3.564 (5)	167

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

H atoms were positioned geometrically, with O—H = 0.82 and C—H = 0.93 and 0.98 (for methine H), 0.93 and 0.97 (for methylene H) or 0.96 Å (for methyl H), and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = xU_{\rm eq}$(C,O), where $x = 1.2$ for methine and methylene H and $x = 1.5$ for all other H. In the absence of significant anomalous scattering effects, Friedel pairs were merged, and the absolute configuration is assigned arbitrarily.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; 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, 2000); software used to prepare material for publication: *SHELXTL*.

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