Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

8α,9α-Epoxy-7-oxoroyleanon

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.006 Å
R factor = 0.060
wR factor = 0.152
Data-to-parameter ratio = 9.1

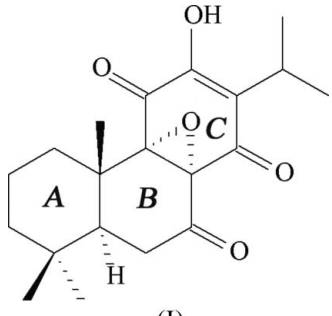
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 20 June 2006
Accepted 3 July 2006

The title compound, $C_{20}H_{26}O_5$, is a naturally occurring diterpenoid epoxyquinone. Its geometrical parameters are normal. The hydroxy group forms an intermolecular O—H···O hydrogen bond which links the molecules into infinite chains extended along the *b* axis.

Comment

The title compound, (I), was isolated from an Abyssinian Plectranthus species (Rüedi, 1984). Recently, we have obtained this compound from *Lycopodium japonicum* Thunb. Here we report its crystal structure.



(I)

Compound (I) crystallizes in the monoclinic space group $P2_1$ with one molecule in the asymmetric unit (Fig. 1). The geometrical parameters for (I) fall within their expected ranges (Allen *et al.*, 1987). The six-membered ring *A* adopts the usual chair conformation, while ring *B* adopts a twist-boat conformation. Ring *C* is essentially planar with an r.m.s. deviation of 0.042 (2) Å. The dihedral angle between the ring *C* and the three-membered ring C8/C9/O22 is 81.2 (3)°.

In the crystal structure, the hydroxy group forms an intermolecular O—H···O hydrogen bond (Table 21), which links the molecules into infinite chains extended along the *b* axis (Fig. 2).

Experimental

The air-dried and powdered whole plants (2.0 kg) were extracted with acetone (3 × 15 l) at room temperature and concentrated *in vacuo* to give a crude extract (50 g), which was directly subjected to column chromatography over MCI-gel CHP-20P, eluting with 95% ethanol. The elute from 95% ethanol (40 g) was concentrated *in vacuo* and then repeatedly subjected to column chromatography over silica gel. Further purification with silica gel column chromatography yielded 8α,9α-epoxy-7-oxoroyleanon (3 mg). A single crystal of the title compound was obtained by recrystallization from methanol by slow evaporation at 283 K.

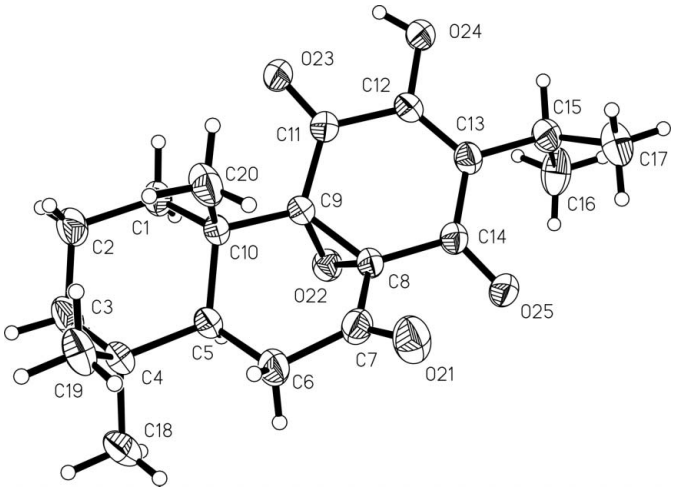


Figure 1
View of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by circles of arbitrary size.

Crystal data

$C_{20}H_{26}O_5$	$Z = 2$
$M_r = 346.41$	$D_x = 1.292$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 10.336$ (2) Å	$\mu = 0.09$ mm^{-1}
$b = 7.4770$ (15) Å	$T = 296$ (2) K
$c = 11.760$ (2) Å	Block, yellow
$\beta = 101.61$ (3)°	0.30 × 0.20 × 0.10 mm
$V = 890.2$ (3) Å^3	

Data collection

MAC DIP 2030K diffractometer	2066 independent reflections
ω scans	1845 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.038$
5289 measured reflections	$\theta_{max} = 27.1°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.071P)^2 + 0.3757P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.060$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.152$	$\Delta\rho_{max} = 0.25$ e Å^{-3}
$S = 1.09$	$\Delta\rho_{min} = -0.18$ e Å^{-3}
2066 reflections	
227 parameters	
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O24—H24A···O25^{i}	0.82	2.20	2.820 (4)	132

Symmetry code: (i) $x, y-1, z$.

In the absence of significant anomalous scatterers, Friedel pairs were merged. All H atoms were constrained to an ideal geometry, with C—H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH, C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH_2, C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH_3, and O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O).

Data collection: *DENZO* (Otwinowski & Minor, 1997); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*

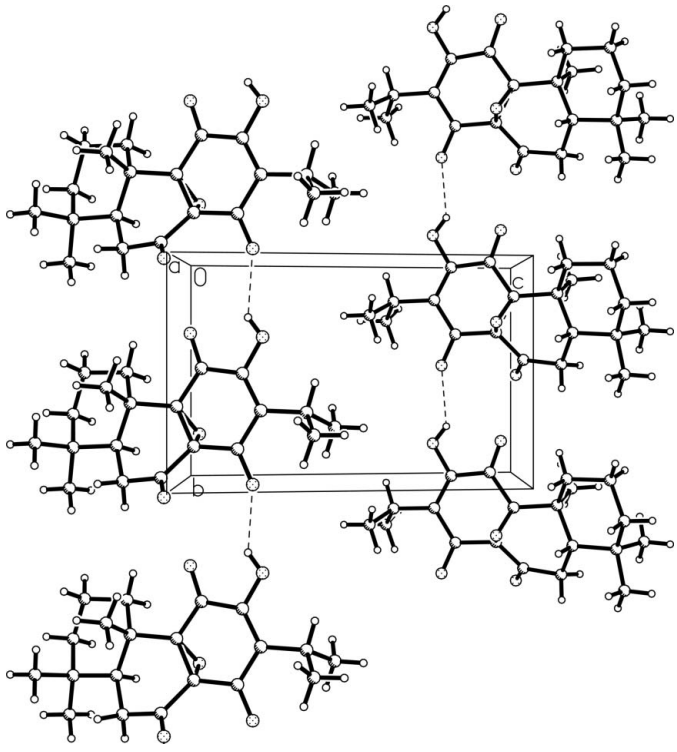


Figure 2
The molecular packing of (I), viewed along the b axis. Dashed lines indicate hydrogen-bonding interactions.

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