Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

An *ent*-abietane diterpenoid from *Doellingeria scaber*

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Key indicators

Single-crystal X-ray study
T = 286 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.088
Data-to-parameter ratio = 10.0

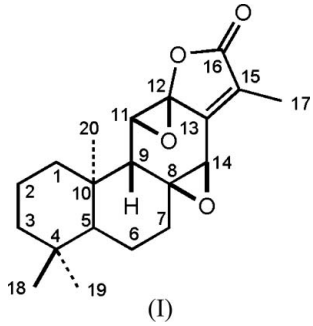
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 11 July 2005
Accepted 5 August 2005
Online 12 August 2005

The title compound, 4,4,8,11b-tetramethyl-2,3,4,4a,5,6,6a,7,-10a,11,11a,11b-dodecahydro-1*H*,9*H*-6a:7,10a:11-diepoxyphenanthro[3,2-*b*]furan-9-one, $C_{20}H_{26}O_4$, a natural *ent*-abietane diterpenoid, is composed of four fused rings with the expected *cis* and *trans* junctions. In the crystal structure, the molecules stack along the *b* axis and are linked by C—H···O hydrogen bonds.

Comment

The title compound, (I), a natural *ent*-abietane diterpenoid isolated from the medicinal plant *Doellingeria scaber Thunb*, is used for treatment of traumatic injury and snake bite. Compound (I) has been isolated previously from genus *Euphorbia* (Uemura & Hirata, 1972; Sutthivaiyakit *et al.*, 2000). In order to further confirm the structure and conformation of (I), we undertook an X-ray study.



(I)

The present analysis confirms the previously proposed molecular structure of (I) and shows its conformation (Fig. 1). There is a *trans* junction between rings *A* (atoms C1–C5/C10) and *B* (C5–C10), and a *cis* junction between rings *B* and *C* (C8–C14). In addition, the 11β,12β-epoxy and 8β,14β-epoxy substituents are observed and the α,β-unsaturated-γ-lactone adopts an α orientation at position C12.

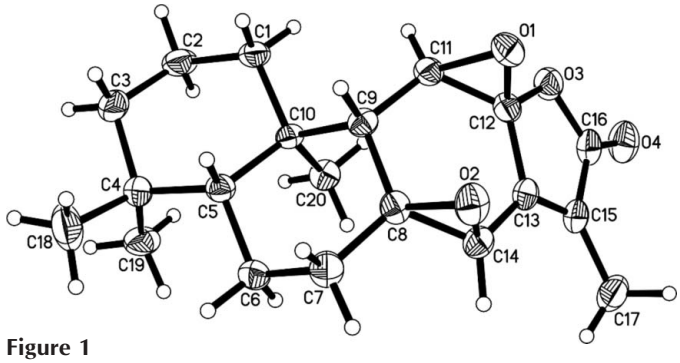


Figure 1
A view of the molecular structure of compound (I). Displacement ellipsoids are drawn at the 50% probability level.

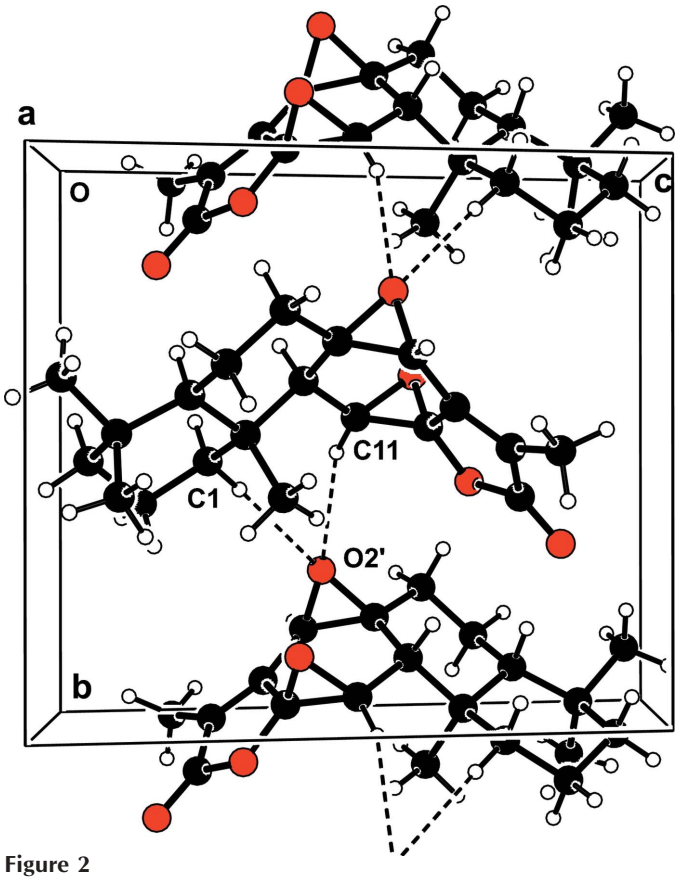


Figure 2
The crystal packing of (I), viewed along the *a* axis, showing the C—H···O hydrogen bonds as dashed lines [symmetry code: (′) $1-x, \frac{1}{2}+y, 1-z$].

The bonds lengths and angles in (I) are within expected ranges (Allen *et al.*, 1987), with average values (Å) Csp^3—Csp^3 (except for C11—C12 and C8—C14) = 1.532 (3), Csp^3—Csp^3 (in oxirane) = 1.473 (3), Csp^3—Csp^2 = 1.474 (3), C═C = 1.330 (3), Csp^2—Csp^2 (in C═C—C═O) = 1.477 (4), C═O = 1.200 (3), Csp^3—O = 1.436 (3) and Csp^2—O = 1.386 (3). Rings *A* and *B* have chair conformations, with average torsion angles of 53.2 (3) and 53.8 (3)°, respectively. Ring *C* adopts a highly flattened boat conformation, while ring *D* (O3/C12/C13/C15/C16) is planar, with an average torsion angle of 0.9 (3)°.

It was not possible to determined the absolute configuration of compound (I) by anomalous dispersion effects, but the positive optical rotation showed this compound to be in the *ent*-abietane series as depicted in genus Euphorbia (Uemura & Hirata, 1972; Sutthivaiyakit *et al.*, 2000; Lal *et al.*, 1990), rather than in the abietane series.

In the crystal structure, the molecules stack along the *b* axis and are linked by C—H···O hydrogen bonds (see Table 1 and Fig. 2 for details).

Experimental

The dried and powdered roots of *Doellingeria scaber* (6.5 kg) were extracted three times with petroleum ether–MeOH–Et_2O (1:1:1) at room temperature. After evaporation under reduced pressure, the residue was separated by repeated silica gel (200–300 mesh) column chromatography and recrystallization, giving 20 mg of compound (I) (m.p. 486–488 K). Optical rotation: $[\alpha]_D^{25}$ +164° (*c* 0.56, $CHCl_3$). Crystals suitable for X-ray analysis were obtained by slow evaporation of a solution of compound (I) in acetone at room temperature.

Crystal data

$C_{20}H_{26}O_4$
M_r = 330.41
Monoclinic, $P2_1$
a = 6.912 (1) Å
b = 10.751 (1) Å
c = 12.080 (2) Å
β = 103.18 (1)°
V = 874.1 (2) Å^3
Z = 2
D_x = 1.255 Mg m^{-3}
D_m = ? Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 27 reflections
θ = 3.1–14.4°
μ = 0.09 mm^{-1}
T = 286 (2) K
Plate, colourless
0.54 × 0.46 × 0.24 mm

Data collection

Siemens P4 diffractometer
ω scans
Absorption correction: none
2505 measured reflections
2219 independent reflections
1496 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 28.0°
h = 0 → 9
k = 0 → 14
l = −15 → 15
3 standard reflections
every 97 reflections
intensity decay: 2.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.088
S = 0.89
2219 reflections
222 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.068 (5)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···O2^{i}	0.97	2.54	3.499 (3)	173
C11—H11···O2^{i}	0.98	2.53	3.444 (3)	156

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

All H atoms were included in calculated positions and refined as riding atoms, with C—H = 0.96–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C). As mentioned above, the absolute configuration could not be determined crystallographically and Friedel pairs were merged. The choice of enantiomer was based on comparison of the optical rotation with that of related compounds with known stereochemistry.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997b); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

This work was supported by the National Natural Science Foundation of China (No. 20172025).

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