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The pseudoguaianolide peruvin

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The title compound, (3a*R*,4a*S*,7a*R*,8*S*,9a*R*)-decahydro-7a-hydroxy-4a,8-dimethyl-3-methyleneazuleno[6,5-*b*]furan-2,5-dione, $C_{15}H_{20}O_4$, from *Ambrosia artemisiifolia*, has its seven-membered ring in a twist conformation. Molecules form intermolecular O···O hydrogen bonds of length 2.920 (2) Å.

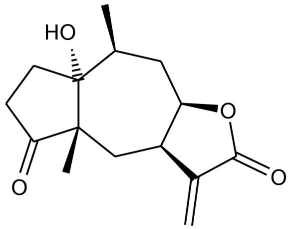
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.109
Data-to-parameter ratio = 13.6

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

Comment

The title ambrosanolide-class sesquiterpene lactone, (I), has been isolated from several species of *Ambrosia* (Compositae), including *A. peruviana* (Joseph-Nathan & Romo, 1966), *A. confertiflora* (Yoshioka *et al.*, 1970), *A. tenuifolia* (Oberti *et al.*, 1986; Schmeda Hirshmann *et al.*, 1986), *A. cumanensis* (Del Amo & Anaya, 1978), and *A. artemisiifolia* (hog-weed; Porter & Mabry, 1969; Rybalko *et al.*, 1979; Watanabe *et al.*, 1981). It has been shown to have allelopathic activity, stimulating the germination of witchweed (Fischer *et al.*, 1990), and inhibiting germination of lettuce and growth of rice seedlings (Watanabe *et al.*, 1981). It has also been shown to have insect antifeedant activity (Bloszyk, 1988).



(I)

The cyclopentanone ring is *trans*-fused to the seven-membered ring, and the lactone ring is *cis*-fused at C7—C8 (Fig. 1). The conformation of the seven-membered ring is nearest the twist chair, with C10 on the local C_2 axis. Parthenin (Fronczek *et al.*, 1989), which differs from peruvin only by having a C2═C3 double bond and having the lactone *cis*-fused at C6—C7 rather than C7—C8, has the seven-membered ring in the chair conformation, also with C10 on the local symmetry axis (Table1). The cyclopentanone ring of peruvin is near the envelope conformation, with C1 at the flap position, and the lactone ring is in a flattened envelope, with C7 at the flap. Molecules are linked by intermolecular hydrogen bonds into chains in the [001] direction (Table2).

The cell dimensions of peruvin at 298 K are a = 7.1425 (8), b = 12.0672 (8) and c = 8.2319 (6) Å, and β = 103.802 (7)°.

Experimental

Crystals of (I) were kindly provided by Amber Hale, Marwa Donia and Flor Mora, who isolated the compound from *Ambrosia artemisiifolia*.

Crystal data

$C_{15}H_{20}O_4$
$M_r = 264.31$
Monoclinic, $P2_1$
$a = 7.0647\ (8)$ Å
$b = 12.0073\ (13)$ Å
$c = 8.1788\ (11)$ Å
$\beta = 103.247\ (8)^\circ$
$V = 675.33\ (14)$ Å^3
$Z = 2$
$D_x = 1.300$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 21.1$–43.5°
$\mu = 0.76$ mm^{-1}
$T = 120$ K
Prism, colorless
0.47 × 0.35 × 0.32 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.757$, $T_{\max} = 0.791$
2686 measured reflections
2400 independent reflections
2395 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$
$\theta_{\max} = 74.9^\circ$
$h = -8 \rightarrow 8$
$k = -13 \rightarrow 15$
$l = -10 \rightarrow 0$
3 standard reflections frequency: 120 min intensity decay: 2.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.109$
$S = 1.10$
2400 reflections
176 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 0.3022P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.033 (3)
Absolute structure: Flack (1983); 944 Friedel pairs
Flack parameter = 0.1 (2)

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.211 (3)	O4—C1	1.450 (2)
O3—C12	1.210 (3)	C11—C13	1.328 (3)
C5—C1—C2—C3	−41.16 (18)	C11—C7—C8—O2	−10.39 (18)
C1—C2—C3—C4	21.2 (2)	C6—C7—C8—C9	−8.2 (2)
C2—C3—C4—C5	6.5 (2)	C7—C8—C9—C10	−72.4 (2)
C3—C4—C5—C1	−30.98 (19)	C5—C1—C10—C9	38.0 (2)
C2—C1—C5—C4	43.62 (17)	C8—C9—C10—C1	50.5 (2)
C10—C1—C5—C6	−63.3 (2)	C8—C7—C11—C12	9.80 (19)
C1—C5—C6—C7	−12.6 (3)	C8—O2—C12—C11	−1.5 (2)
C5—C6—C7—C8	61.2 (2)	C7—C11—C12—O2	−5.6 (2)
C12—O2—C8—C7	7.79 (19)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O3$^{\rm i}$	0.84	2.09	2.920 (2)	169

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

H atoms were placed in calculated positions, with C—H bond distances of 0.95–1.00 Å, O—H distances of 0.84 Å and $U_{\rm iso} = 1.2U_{\rm eq}$ of the attached atom ($1.5U_{\rm eq}$ for OH and methyl groups), and thereafter treated as riding. A torsional parameter was refined for each methyl and OH group. The absolute configuration was determined by refinement of the Flack (1983) parameter. The reported enantiomer, which agrees with the accepted configuration of sesquiterpene lactones from higher plants (Fischer *et al.*, 1979), yielded $x = 0.1\ (2)$, while the inverse configuration yielded $x = 1.1\ (2)$.

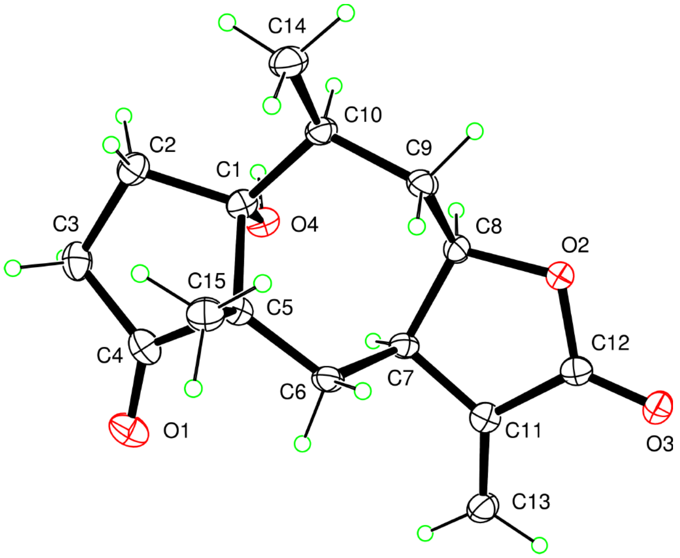


Figure 1
The atom-numbering scheme and ellipsoids at the 50% probability level for (I).

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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