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2-Hydroxyplatyphyllid

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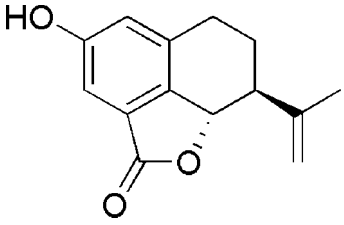
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.079; wR factor = 0.164; data-to-parameter ratio = 9.9.

The title compound [systematic name 4-hydroxy-3-(1-methylethylidene)-6,7,8,8a-tetrahydro-1-oxaacenaphthylen-2-one], $C_{14}H_{14}O_3$, is a tricyclic norsesquiterpene. The structure has a pseudo-centre of symmetry (possibility of space group *Pnma*). However, the unit cell contains only four molecules which have no internal symmetry. The molecules are linked into one dimensional chains along the *b* axis through O—H···O hydrogen bonding.

Related literature

For background on the biological activity of norsesquiterpenes, see: Gao & Jia (1998); Fu *et al.* (2002); Kaneko *et al.* (1982).



Experimental

Crystal data

$C_{14}H_{14}O_3$
M_r = 230.25
Orthorhombic, $P2_12_12_1$
a = 7.678 (4) Å
b = 8.063 (4) Å
c = 19.58 (1) Å
V = 1212.0 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.18 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.984, T_{max} = 0.991
5807 measured reflections
1549 independent reflections
1124 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.080
$wR(F^2)$ = 0.164
S = 1.20
1549 reflections
156 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1X···O2^{i}	0.83 (2)	1.90 (2)	2.719 (4)	170 (5)

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*.

This investigation was supported by a grant from the Fudan University Graduate Innovation Foundation (grant No. CQF301812) (to QW). We thank Professor Min-Qin Chen of the Research Centre of Analysis and Measurement of Fudan University for work on the X-ray crystal structure determination.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2135).

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supplementary materials

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Comment

Norsesquiterpenes isolated from many ligularia species show some biological activity, such as cytotoxicity (Gao *et al.*, 1998; Fu *et al.*, 2002). Knowledge of the three-dimensioned structure of these compounds is important in order to understand the biosynthetic pathway of terpenes and to establish structure–activity relationships (Kaneko *et al.*, 1982) in this class of compounds.

The molecular backbone of the title compound has a fused tricyclic ring system as shown in Fig. 1. In the structure, the hydrogen attached to O1 forms a strong intermolecular hydrogen bond with the O2, Table 1, forming a one dimensional molecular chain along the *b* axis (Fig.2).

Experimental

The dried and powdered roots and rhizomes of *Ligularia macrophylla* (5.0 kg) were extracted with 95% EtOH at reflux temperature three times and filtered. The filtrate was evaporated *in vacuo* to give a residue (360 g), a portion of which (350 g) was suspended in H_2O (2 *L*) and partitioned successively with petroleum ether, EtOAc, and *n*-BuOH. The EtOAc extract (50 g) was subjected to column chromatography on Si gel eluted successively with petroleum ether-acetone (30:1–1:1), the pure compound was obtained from petroleum ether:acetone (5:1).

Refinement

The hydrogen atoms (except the hydrogen in the OH group) were located geometrically and refined with a riding model with temperature factors of 1.2 or 1.5 times their covalently bonded atoms. The hydrogen in OH group was located by difference Fourier map and restained with 0.82 A to oxygen atoms during refinement. Since the anomalous scattering was not strong enough to determine the absolute structure of the molecule Friedel equivalents were merged before refinement.

Figures

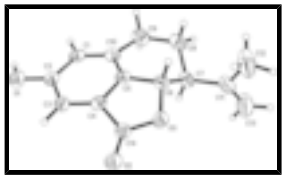

Fig. 1. The molecular strusture of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Fig. 2. A packing diagram showing the hydrogen bonds in the crystal structure of (I).

4-hydroxy-3-(1-methylethylidene)-6,7,8,8a-tetrahydro-1-oxaacenaphthylen-2-one

Crystal data

$C_{14}H_{14}O_3$	$F_{000} = 488$
$M_r = 230.25$	$D_x = 1.262$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 887 reflections
$a = 7.678$ (4) Å	$\theta = 2.7$–21.8º
$b = 8.063$ (4) Å	$\mu = 0.09$ mm^{-1}
$c = 19.58$ (1) Å	$T = 293$ (2) K
$V = 1212.0$ (11) Å^3	Prism, colourless
$Z = 4$	0.18 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1549 independent reflections
Radiation source: fine-focus sealed tube	1124 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.080$
$T = 293$(2) K	$\theta_{max} = 27.1$º
φ and ω scans	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{min} = 0.984$, $T_{max} = 0.991$	$k = -10 \rightarrow 9$
5807 measured reflections	$l = -24 \rightarrow 19$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.080$	$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.5001P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.164$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.20$	$\Delta\rho_{max} = 0.22$ e Å^{-3}
1549 reflections	$\Delta\rho_{min} = -0.18$ e Å^{-3}
156 parameters	Extinction correction: none
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.7457 (5)	0.0823 (4)	0.12179 (15)	0.0649 (10)
H1X	0.755 (9)	−0.014 (3)	0.107 (2)	0.09 (2)*
O2	0.7573 (5)	0.7554 (3)	0.08791 (15)	0.0643 (11)
O3	0.7270 (5)	0.7191 (3)	−0.02453 (15)	0.0587 (9)
C1	0.7226 (7)	0.1556 (5)	0.0023 (2)	0.0514 (11)
H1	0.7286	0.0443	−0.0099	0.062*
C2	0.7309 (6)	0.1984 (5)	0.0717 (2)	0.0504 (11)
C3	0.7310 (7)	0.3637 (5)	0.0914 (2)	0.0500 (11)
H3	0.7431	0.3943	0.1370	0.060*
C4	0.7123 (6)	0.4803 (5)	0.0405 (2)	0.0459 (10)
C5	0.6906 (5)	0.4361 (5)	−0.0268 (2)	0.0450 (10)
C6	0.6778 (6)	0.5828 (6)	−0.0707 (2)	0.0499 (11)
H6	0.5557	0.5975	−0.0839	0.060*
C7	0.7851 (6)	0.5607 (6)	−0.1347 (2)	0.0554 (12)
H7	0.9060	0.5393	−0.1213	0.066*
C8	0.7099 (8)	0.4001 (6)	−0.1669 (2)	0.0689 (14)
H8A	0.5875	0.4185	−0.1767	0.083*
H8B	0.7682	0.3815	−0.2102	0.083*
C9	0.7256 (9)	0.2425 (6)	−0.1243 (2)	0.0700 (15)
H9A	0.6370	0.1644	−0.1389	0.084*
H9B	0.8384	0.1924	−0.1328	0.084*
C10	0.7059 (6)	0.2734 (5)	−0.0480 (2)	0.0484 (11)
C11	0.7834 (8)	0.7051 (7)	−0.1837 (2)	0.0679 (14)
C12	0.6172 (10)	0.7755 (10)	−0.2038 (4)	0.128 (3)
H12A	0.5517	0.8041	−0.1638	0.192*
H12B	0.5534	0.6958	−0.2303	0.192*
H12C	0.6367	0.8733	−0.2308	0.192*
C13	0.9324 (6)	0.7596 (4)	−0.21134 (18)	0.130 (3)
H13A	0.9303	0.8436	−0.2439	0.157*
H13B	1.0380	0.7133	−0.1980	0.157*
C14	0.7347 (6)	0.6635 (4)	0.04079 (18)	0.0490 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.091 (3)	0.0379 (16)	0.0657 (19)	0.001 (2)	0.003 (2)	−0.0030 (15)
O2	0.095 (3)	0.0396 (16)	0.0580 (18)	−0.0083 (19)	−0.001 (2)	−0.0081 (14)
O3	0.079 (2)	0.0409 (15)	0.0566 (17)	−0.0037 (17)	−0.0022 (18)	0.0000 (14)
C1	0.052 (3)	0.033 (2)	0.069 (3)	−0.003 (2)	−0.003 (3)	−0.012 (2)
C2	0.049 (3)	0.040 (2)	0.063 (3)	−0.007 (2)	0.001 (2)	0.001 (2)
C3	0.056 (3)	0.046 (2)	0.048 (2)	−0.003 (2)	0.003 (3)	−0.0037 (19)
C4	0.044 (3)	0.041 (2)	0.053 (2)	0.000 (2)	0.004 (2)	−0.0050 (19)
C5	0.037 (2)	0.044 (2)	0.054 (2)	−0.002 (2)	−0.003 (2)	−0.003 (2)
C6	0.047 (3)	0.053 (3)	0.050 (2)	0.001 (2)	−0.009 (2)	−0.001 (2)
C7	0.045 (3)	0.074 (3)	0.046 (2)	0.003 (3)	−0.004 (2)	0.002 (2)
C8	0.074 (4)	0.086 (3)	0.047 (2)	0.004 (3)	−0.001 (3)	−0.016 (3)
C9	0.085 (4)	0.062 (3)	0.063 (3)	0.006 (3)	−0.005 (3)	−0.017 (2)
C10	0.044 (2)	0.048 (2)	0.053 (2)	0.000 (2)	−0.003 (2)	−0.010 (2)
C11	0.063 (3)	0.090 (4)	0.051 (3)	0.002 (3)	−0.007 (3)	0.007 (3)
C12	0.104 (6)	0.164 (8)	0.116 (6)	0.028 (5)	0.001 (4)	0.071 (6)
C13	0.092 (6)	0.169 (9)	0.130 (6)	−0.021 (5)	−0.017 (4)	0.082 (6)
C14	0.053 (3)	0.045 (2)	0.049 (2)	−0.003 (2)	0.001 (3)	−0.0041 (19)

Geometric parameters (Å, °)

O1—C2	1.361 (5)	C7—C11	1.509 (6)
O1—H1X	0.83 (2)	C7—C8	1.552 (6)
O2—C14	1.196 (4)	C7—H7	0.9800
O3—C14	1.356 (4)	C8—C9	1.524 (6)
O3—C6	1.472 (5)	C8—H8A	0.9700
C1—C10	1.375 (6)	C8—H8B	0.9700
C1—C2	1.402 (6)	C9—C10	1.522 (6)
C1—H1	0.9300	C9—H9A	0.9700
C2—C3	1.387 (6)	C9—H9B	0.9700
C3—C4	1.378 (6)	C11—C13	1.339 (7)
C3—H3	0.9300	C11—C12	1.451 (9)
C4—C5	1.375 (6)	C12—H12A	0.9599
C4—C14	1.487 (5)	C12—H12B	0.9599
C5—C10	1.381 (6)	C12—H12C	0.9599
C5—C6	1.465 (6)	C13—H13A	0.9300
C6—C7	1.509 (6)	C13—H13B	0.9300
C6—H6	0.9800		
C2—O1—H1X	113 (4)	C9—C8—H8A	108.2
C14—O3—C6	110.1 (3)	C7—C8—H8A	108.2
C10—C1—C2	121.9 (4)	C9—C8—H8B	108.2
C10—C1—H1	119.1	C7—C8—H8B	108.2
C2—C1—H1	119.1	H8A—C8—H8B	107.4
O1—C2—C3	117.3 (4)	C10—C9—C8	113.1 (4)
O1—C2—C1	122.2 (4)	C10—C9—H9A	109.0

C3—C2—C1	120.5 (4)	C8—C9—H9A	109.0
C4—C3—C2	116.9 (4)	C10—C9—H9B	109.0
C4—C3—H3	121.5	C8—C9—H9B	109.0
C2—C3—H3	121.5	H9A—C9—H9B	107.8
C5—C4—C3	122.0 (4)	C1—C10—C5	116.6 (4)
C5—C4—C14	106.0 (4)	C1—C10—C9	125.6 (4)
C3—C4—C14	131.5 (4)	C5—C10—C9	117.3 (4)
C4—C5—C10	121.6 (4)	C13—C11—C12	120.9 (5)
C4—C5—C6	111.2 (4)	C13—C11—C7	120.1 (5)
C10—C5—C6	126.6 (4)	C12—C11—C7	118.8 (5)
C5—C6—O3	103.0 (3)	C11—C12—H12A	109.5
C5—C6—C7	110.8 (4)	C11—C12—H12B	109.5
O3—C6—C7	117.2 (4)	H12A—C12—H12B	109.5
C5—C6—H6	108.5	C11—C12—H12C	109.5
O3—C6—H6	108.5	H12A—C12—H12C	109.5
C7—C6—H6	108.5	H12B—C12—H12C	109.5
C11—C7—C6	115.6 (4)	C11—C13—H13A	120.0
C11—C7—C8	112.5 (4)	C11—C13—H13B	120.0
C6—C7—C8	103.4 (4)	H13A—C13—H13B	120.0
C11—C7—H7	108.3	O2—C14—O3	121.9 (3)
C6—C7—H7	108.3	O2—C14—C4	129.5 (4)
C8—C7—H7	108.3	O3—C14—C4	108.6 (3)
C9—C8—C7	116.4 (4)		
C10—C1—C2—O1	179.3 (4)	C6—C7—C8—C9	−61.5 (6)
C10—C1—C2—C3	−3.2 (8)	C7—C8—C9—C10	35.3 (7)
O1—C2—C3—C4	−178.7 (4)	C2—C1—C10—C5	−2.5 (7)
C1—C2—C3—C4	3.7 (7)	C2—C1—C10—C9	169.3 (5)
C2—C3—C4—C5	1.4 (7)	C4—C5—C10—C1	7.7 (7)
C2—C3—C4—C14	−168.7 (5)	C6—C5—C10—C1	177.6 (4)
C3—C4—C5—C10	−7.4 (7)	C4—C5—C10—C9	−164.8 (5)
C14—C4—C5—C10	165.0 (4)	C6—C5—C10—C9	5.1 (7)
C3—C4—C5—C6	−178.7 (4)	C8—C9—C10—C1	−176.7 (5)
C14—C4—C5—C6	−6.4 (5)	C8—C9—C10—C5	−4.9 (7)
C4—C5—C6—O3	10.2 (5)	C6—C7—C11—C13	134.4 (5)
C10—C5—C6—O3	−160.6 (5)	C8—C7—C11—C13	−107.1 (5)
C4—C5—C6—C7	136.4 (4)	C6—C7—C11—C12	−49.9 (7)
C10—C5—C6—C7	−34.4 (6)	C8—C7—C11—C12	68.6 (7)
C14—O3—C6—C5	−10.4 (4)	C6—O3—C14—O2	−173.4 (5)
C14—O3—C6—C7	−132.3 (4)	C6—O3—C14—C4	7.0 (5)
C5—C6—C7—C11	−179.4 (4)	C5—C4—C14—O2	−180.0 (5)
O3—C6—C7—C11	−61.6 (5)	C3—C4—C14—O2	−8.7 (10)
C5—C6—C7—C8	57.2 (4)	C5—C4—C14—O3	−0.5 (6)
O3—C6—C7—C8	175.0 (4)	C3—C4—C14—O3	170.9 (5)
C11—C7—C8—C9	173.0 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1X···O2^{i}	0.83 (2)	1.90 (2)	2.719 (4)	170 (5)

Symmetry codes: (i) *x*, *y*−1, *z*.

Fig. 1

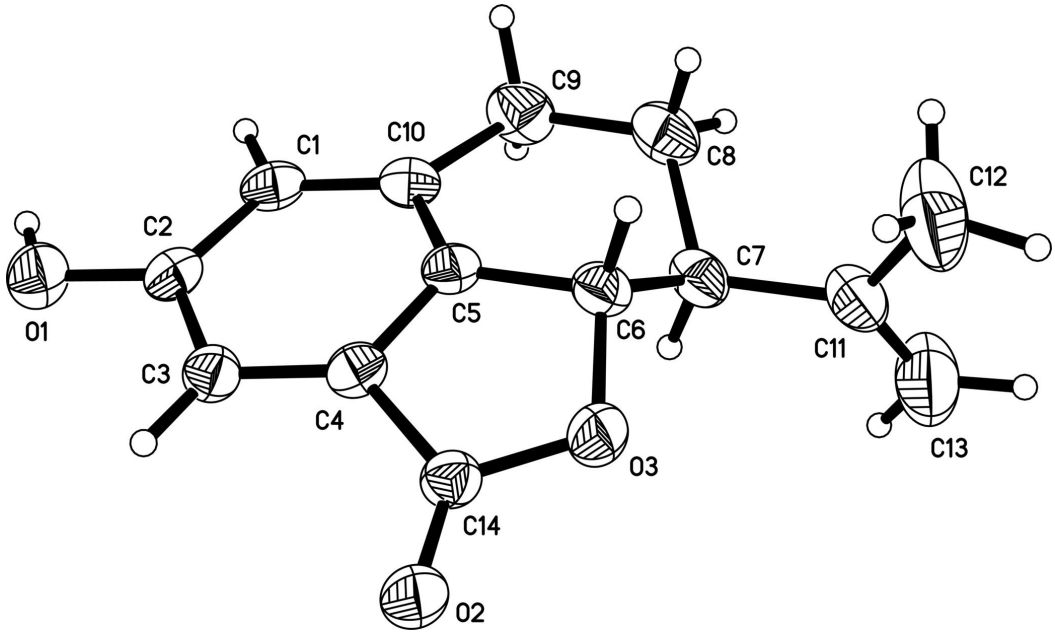

Fig. 2