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3-Hydroxy-3,6-dimethyl-2-(3-methylbut-2-enylidene)-3,3a,7,7a-tetrahydrobenzofuran-4(2*H*)-one

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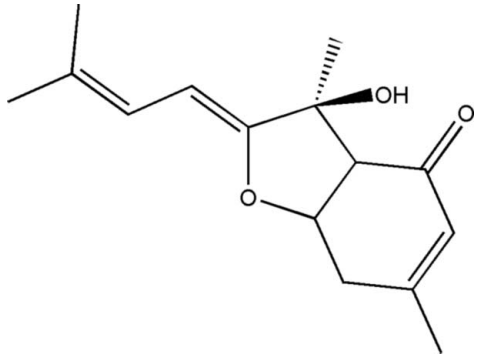
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.005 Å; R factor = 0.052; wR factor = 0.117; data-to-parameter ratio = 9.7.

The title compound, $C_{15}H_{20}O_3$, was obtained from *Angelica polymorpha* and shows antifeedant activity. The penta-1,3-diene fragment is essentially planar and the planarity extends to the adjacent C and O atoms of the benzofuran system. The six-membered ring of this system adopts a distorted chair conformation. The penta-1,3-diene fragment is essentially planar and the planarity extends to the adjacent C and O atoms of the benzofuran system. In the crystal structure, intermolecular O—H···O hydrogen bonds form zigzag rows along *b* and weak C—H···O interactions further stabilize the structure.

Related literature

Angelica polymorpha was described by Mi *et al.* (2003) and the bioactivity of the title compound was described by Mi *et al.* (1992) and Mi (1995). For related literature, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{15}H_{20}O_3$
M_r = 248.31
Orthorhombic, $P2_12_12_1$
a = 6.9797 (11) Å
b = 9.0174 (14) Å
c = 22.840 (4) Å
V = 1437.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 299 (2) K
0.20 × 0.20 × 0.06 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: none
12406 measured reflections
1648 independent reflections
1251 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.117
S = 1.07
1648 reflections
170 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.86 (4)	1.98 (5)	2.839 (3)	173 (4)
C10—H10*C*···O3^{ii}	0.96	2.69	3.528 (4)	146

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2284).

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supplementary materials

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3-Hydroxy-3,6-dimethyl-2-(3-methylbut-2-enylidene)-3,3a,7,7a-tetrahydrobenzofuran-4(2*H*)-one

J.-Z. Wang, K. Zou, F. Cheng and F.-J. Dan

Comment

Angelica polymorpha, belongs to parsley family, and is a species Chinese medicinal herb and has been widely used in folk medicine for the treatment of stomach ache, abdominal pain and rheumatism (Mi *et al.*, 1995). It contains coumarin, terpenoids and volatile oils (Mi *et al.*, 2003). Angelica polymorpha belongs to the same family as A. pubescens, A. silvesrtis and A. koreana. It was first obtained from Angelica polymorpha and shown to have strong antifeedant properties against insects (Mi *et al.*, 1992). We present here the crystal structure of the title compound, (I), a constituent of Angelica polymorpha (Fig. 1).

Within the molecule of (I), the bond lengths and angles present no unusual features. The penta-1,3,diene fragment is essentially planar and the planarity extends to the adjacent C and O atoms of the benzofuran system. The maximum deviation from the meanplane through atoms O3, C9, C11, C12, C13, C14 is -0.036 (2)Å for atom C12. The six-membered ring, C1/C2/C3/C4/C5/C6, has a total puckering amplitude of 0.327 (1) %A (Cremer & Pople, 1975) and a distorted chair conformation [θ = 51.9 (4)° and φ= 3.3 (4)°]. In the crystal structure, intermolecular O2—H2···O1 hydrogen bonds form zigzag rows along b and weak C10—H10c···O3 interactions further stabilise the structure, Fig 2. Table 1.

Experimental

The roots of Angelica polymorpha Maxim (1.0 kg) were extracted four times, each for 1.5 h, with refluxing ethyl acetate. After the removal of solvent under reduced pressure, the extract was obtained. Chromatography on silica gel eluting with petroleum ether - EtOAc (4:1 v/v) yielded 46 50 ml fractions. The title compound, (I), was obtained from fractions 11-15 and was recrystallized from methanol.

Refinement

In the absence of significant anomalous scattering effects, 1164 Friedel pairs were merged. The OH proton H2 was located in a difference Fourier map and refined isotropically with U_{eq} = 1.5 U_{eq}(O). All other H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, U_{iso}=1.2U_{eq} (C) for aromatic 0.98 Å, U_{iso} = 1.2U_{eq} (C) for CH, 0.97 Å, U_{iso} = 1.2U_{eq} (C) for CH_2, 0.96 Å, U_{iso} = 1.5U_{eq} (C) for CH_3 atoms.

Figures

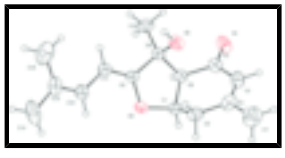

Fig. 1. A View of the molecule of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

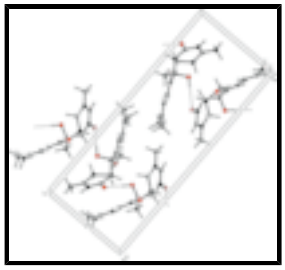

Fig. 2. The crystal packing, viewed along the X axis with hydrogen bonds drawn as dashed lines.

3-Hydroxy-3,6-dimethyl-2-(3-methylbut-2-enylidene)-3,3a,7,7a- tetrahydrobenzofuran-4(2*H*)-one

Crystal data

$C_{15}H_{20}O_3$	F_{000} = 536
M_r = 248.31	D_x = 1.147 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 1719 reflections
a = 6.9797 (11) Å	θ = 2.4–19.1º
b = 9.0174 (14) Å	μ = 0.08 mm^{-1}
c = 22.840 (4) Å	T = 299 (2) K
V = 1437.5 (4) Å^3	Plate, colorless
Z = 4	0.20 × 0.20 × 0.06 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer	1251 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.063
Monochromator: graphite	θ_{max} = 26.0º
T = 292(2) K	θ_{min} = 1.8º
φ and ω scans	h = −8→8
Absorption correction: none	k = −10→11
12406 measured reflections	l = −27→27
1648 independent reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.1864P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.052	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.117	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
1648 reflections	Extinction correction: none
170 parameters	

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
C1	0.9568 (4)	0.6484 (4)	0.22512 (13)	0.0481 (8)
H1	1.0231	0.5594	0.2112	0.058*
C2	1.0072 (5)	0.7777 (4)	0.18612 (16)	0.0633 (9)
H2A	0.9919	0.8686	0.2084	0.076*
H2B	1.1412	0.7698	0.1754	0.076*
C3	0.8915 (6)	0.7897 (4)	0.13172 (15)	0.0621 (9)
C4	0.7185 (5)	0.7263 (4)	0.12712 (14)	0.0610 (9)
H4	0.6499	0.7400	0.0926	0.073*
C5	0.6334 (5)	0.6381 (3)	0.17280 (13)	0.0504 (8)
C6	0.7421 (4)	0.6173 (3)	0.22880 (12)	0.0410 (7)
H6	0.7243	0.5148	0.2419	0.049*
C7	0.9765 (7)	0.8828 (5)	0.08408 (19)	0.1009 (16)
H7A	0.8968	0.8774	0.0499	0.151*
H7B	1.1023	0.8469	0.0748	0.151*
H7C	0.9848	0.9839	0.0970	0.151*
C8	0.6780 (4)	0.7213 (4)	0.27827 (13)	0.0453 (8)
C9	0.8597 (4)	0.7356 (3)	0.31434 (13)	0.0426 (7)
C10	0.5086 (4)	0.6627 (5)	0.31343 (17)	0.0761 (12)
H10A	0.4841	0.7276	0.3459	0.114*
H10B	0.5377	0.5651	0.3277	0.114*
H10C	0.3973	0.6583	0.2888	0.114*
C11	0.8786 (5)	0.8000 (4)	0.36580 (13)	0.0513 (8)
H11	0.7680	0.8346	0.3839	0.062*
C12	1.0591 (5)	0.8210 (4)	0.39641 (14)	0.0592 (9)
H12	1.1666	0.7778	0.3794	0.071*
C13	1.0875 (6)	0.8944 (5)	0.44550 (16)	0.0733 (12)
C14	1.2835 (7)	0.9062 (6)	0.47239 (19)	0.115 (2)
H14A	1.3730	0.8494	0.4497	0.173*

H14B	1.2800	0.8686	0.5117	0.173*
H14C	1.3228	1.0082	0.4730	0.173*
C15	0.9326 (9)	0.9748 (6)	0.4781 (2)	0.121 (2)
H15A	0.8143	0.9670	0.4569	0.182*
H15B	0.9669	1.0774	0.4821	0.182*
H15C	0.9175	0.9316	0.5163	0.182*
O1	0.4750 (3)	0.5810 (3)	0.16660 (10)	0.0689 (7)
O2	0.6331 (4)	0.8598 (3)	0.25136 (10)	0.0624 (7)
H2	0.611 (7)	0.928 (5)	0.2772 (16)	0.094*
O3	1.0125 (3)	0.6779 (3)	0.28456 (9)	0.0586 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0400 (16)	0.0488 (19)	0.0555 (18)	0.0054 (15)	0.0016 (14)	−0.0070 (15)
C2	0.0491 (18)	0.065 (2)	0.076 (2)	−0.0119 (19)	0.0105 (18)	−0.0034 (18)
C3	0.077 (2)	0.045 (2)	0.065 (2)	−0.0107 (19)	0.012 (2)	−0.0007 (16)
C4	0.076 (2)	0.055 (2)	0.0519 (18)	−0.008 (2)	−0.0079 (18)	0.0064 (17)
C5	0.0547 (19)	0.0372 (17)	0.0594 (19)	−0.0026 (17)	−0.0058 (17)	−0.0043 (15)
C6	0.0439 (16)	0.0312 (15)	0.0480 (16)	0.0012 (14)	−0.0015 (14)	0.0017 (13)
C7	0.112 (4)	0.097 (4)	0.093 (3)	−0.026 (3)	0.018 (3)	0.025 (3)
C8	0.0384 (16)	0.0442 (18)	0.0535 (17)	0.0015 (14)	−0.0019 (13)	−0.0022 (14)
C9	0.0379 (15)	0.0402 (17)	0.0495 (17)	0.0023 (15)	0.0020 (14)	0.0016 (14)
C10	0.0425 (18)	0.106 (3)	0.079 (2)	−0.012 (2)	0.0075 (19)	−0.024 (2)
C11	0.0470 (17)	0.055 (2)	0.0514 (17)	0.0028 (17)	0.0028 (15)	−0.0028 (16)
C12	0.061 (2)	0.063 (2)	0.0534 (19)	−0.0045 (19)	−0.0076 (16)	−0.0009 (17)
C13	0.099 (3)	0.068 (3)	0.053 (2)	−0.016 (2)	−0.014 (2)	0.0022 (19)
C14	0.140 (5)	0.119 (4)	0.088 (3)	−0.039 (4)	−0.059 (3)	0.005 (3)
C15	0.173 (6)	0.102 (4)	0.088 (3)	−0.003 (4)	0.006 (4)	−0.034 (3)
O1	0.0621 (16)	0.0666 (17)	0.0780 (16)	−0.0244 (14)	−0.0229 (13)	0.0106 (13)
O2	0.0681 (15)	0.0475 (14)	0.0717 (15)	0.0215 (12)	−0.0140 (13)	−0.0070 (12)
O3	0.0374 (11)	0.0802 (17)	0.0583 (13)	0.0118 (12)	−0.0056 (10)	−0.0134 (12)

Geometric parameters (Å, °)

C1—O3	1.437 (4)	C8—C10	1.524 (4)
C1—C2	1.509 (5)	C9—C11	1.318 (4)
C1—C6	1.527 (4)	C9—O3	1.368 (3)
C1—H1	0.9800	C10—H10A	0.9600
C2—C3	1.485 (5)	C10—H10B	0.9600
C2—H2A	0.9700	C10—H10C	0.9600
C2—H2B	0.9700	C11—C12	1.454 (4)
C3—C4	1.340 (5)	C11—H11	0.9300
C3—C7	1.497 (5)	C12—C13	1.317 (5)
C4—C5	1.441 (4)	C12—H12	0.9300
C4—H4	0.9300	C13—C15	1.500 (7)
C5—O1	1.228 (4)	C13—C14	1.503 (6)
C5—C6	1.499 (4)	C14—H14A	0.9600
C6—C8	1.535 (4)	C14—H14B	0.9600

C6—H6	0.9800	C14—H14C	0.9600
C7—H7A	0.9600	C15—H15A	0.9600
C7—H7B	0.9600	C15—H15B	0.9600
C7—H7C	0.9600	C15—H15C	0.9600
C8—O2	1.426 (4)	O2—H2	0.86 (4)
C8—C9	1.517 (4)		
O3—C1—C2	110.6 (3)	O2—C8—C6	106.4 (2)
O3—C1—C6	104.4 (2)	C9—C8—C6	102.0 (2)
C2—C1—C6	113.8 (3)	C10—C8—C6	113.7 (3)
O3—C1—H1	109.3	C11—C9—O3	122.2 (3)
C2—C1—H1	109.3	C11—C9—C8	127.2 (3)
C6—C1—H1	109.3	O3—C9—C8	110.5 (2)
C3—C2—C1	115.0 (3)	C8—C10—H10A	109.5
C3—C2—H2A	108.5	C8—C10—H10B	109.5
C1—C2—H2A	108.5	H10A—C10—H10B	109.5
C3—C2—H2B	108.5	C8—C10—H10C	109.5
C1—C2—H2B	108.5	H10A—C10—H10C	109.5
H2A—C2—H2B	107.5	H10B—C10—H10C	109.5
C4—C3—C2	121.6 (3)	C9—C11—C12	125.0 (3)
C4—C3—C7	122.6 (4)	C9—C11—H11	117.5
C2—C3—C7	115.7 (3)	C12—C11—H11	117.5
C3—C4—C5	123.4 (3)	C13—C12—C11	127.3 (4)
C3—C4—H4	118.3	C13—C12—H12	116.4
C5—C4—H4	118.3	C11—C12—H12	116.4
O1—C5—C4	121.3 (3)	C12—C13—C15	123.9 (4)
O1—C5—C6	120.1 (3)	C12—C13—C14	121.4 (4)
C4—C5—C6	118.6 (3)	C15—C13—C14	114.7 (4)
C5—C6—C1	115.3 (3)	C13—C14—H14A	109.5
C5—C6—C8	113.8 (2)	C13—C14—H14B	109.5
C1—C6—C8	102.4 (2)	H14A—C14—H14B	109.5
C5—C6—H6	108.3	C13—C14—H14C	109.5
C1—C6—H6	108.3	H14A—C14—H14C	109.5
C8—C6—H6	108.3	H14B—C14—H14C	109.5
C3—C7—H7A	109.5	C13—C15—H15A	109.5
C3—C7—H7B	109.5	C13—C15—H15B	109.5
H7A—C7—H7B	109.5	H15A—C15—H15B	109.5
C3—C7—H7C	109.5	C13—C15—H15C	109.5
H7A—C7—H7C	109.5	H15A—C15—H15C	109.5
H7B—C7—H7C	109.5	H15B—C15—H15C	109.5
O2—C8—C9	110.1 (2)	C8—O2—H2	111 (3)
O2—C8—C10	111.1 (3)	C9—O3—C1	109.2 (2)
C9—C8—C10	113.0 (3)		
O3—C1—C2—C3	−156.0 (3)	C1—C6—C8—C9	−27.2 (3)
C6—C1—C2—C3	−39.0 (4)	C5—C6—C8—C10	85.7 (3)
C1—C2—C3—C4	22.0 (5)	C1—C6—C8—C10	−149.2 (3)
C1—C2—C3—C7	−160.9 (3)	O2—C8—C9—C11	75.5 (4)
C2—C3—C4—C5	−2.3 (5)	C10—C8—C9—C11	−49.4 (5)
C7—C3—C4—C5	−179.1 (4)	C6—C8—C9—C11	−171.9 (3)

C3—C4—C5—O1	−179.4 (3)	O2—C8—C9—O3	−101.1 (3)
C3—C4—C5—C6	0.8 (5)	C10—C8—C9—O3	133.9 (3)
O1—C5—C6—C1	161.2 (3)	C6—C8—C9—O3	11.5 (3)
C4—C5—C6—C1	−19.1 (4)	O3—C9—C11—C12	1.5 (5)
O1—C5—C6—C8	−81.0 (4)	C8—C9—C11—C12	−174.8 (3)
C4—C5—C6—C8	98.8 (3)	C9—C11—C12—C13	174.3 (4)
O3—C1—C6—C5	158.4 (2)	C11—C12—C13—C15	−1.8 (6)
C2—C1—C6—C5	37.7 (4)	C11—C12—C13—C14	179.7 (4)
O3—C1—C6—C8	34.2 (3)	C11—C9—O3—C1	−166.2 (3)
C2—C1—C6—C8	−86.4 (3)	C8—C9—O3—C1	10.6 (3)
C5—C6—C8—O2	−36.9 (3)	C2—C1—O3—C9	94.3 (3)
C1—C6—C8—O2	88.2 (3)	C6—C1—O3—C9	−28.5 (3)
C5—C6—C8—C9	−152.2 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.86 (4)	1.98 (5)	2.839 (3)	173 (4)
C10—H10C···O3^{ii}	0.96	2.69	3.528 (4)	146

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*−1, *y*, *z*.

Fig. 1

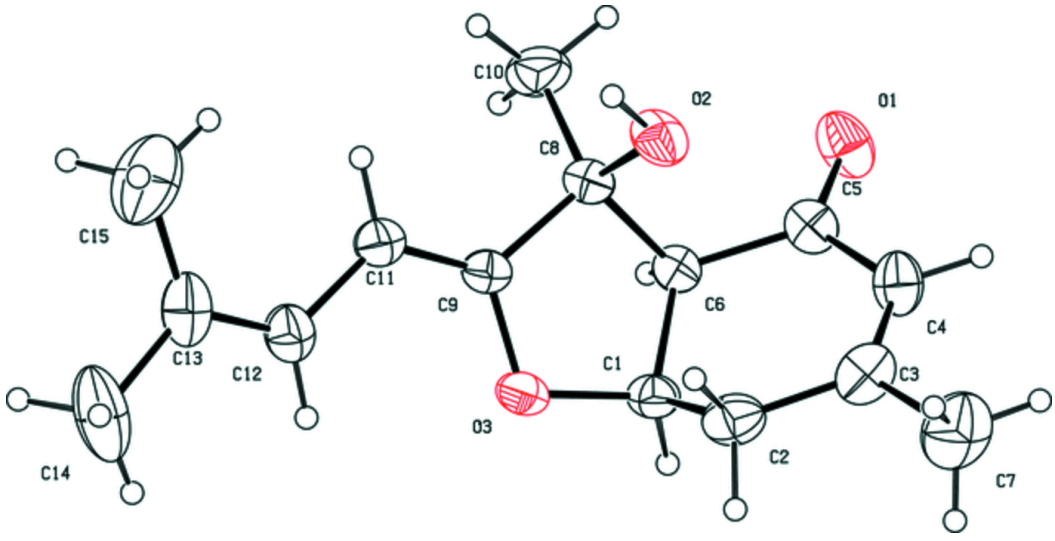

Fig. 2