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Methyl 3-hydroxy-4-(3-methylbut-2-enyloxy)benzoate

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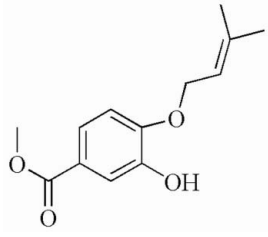
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.002 Å; R factor = 0.051; wR factor = 0.161; data-to-parameter ratio = 15.3.

The title compound, $C_{13}H_{16}O_4$, was isolated from culture extracts of the endophytic fungus *Cephalosporium* sp. The ester and ether substituents are twisted only slightly out of the benzene ring plane, making dihedral angles of 2.16 (2) and 3.63 (5)°, respectively. The non-H atoms of all three substituents are almost coplanar with the benzene ring, with an r.m.s. deviation of 0.0284 Å from the mean plane through all non-H atoms in the structure. A weak intramolecular O—H···O hydrogen bond contributes to this conformation. In the crystal structure, molecules are linked into a one-dimensional chain by intermolecular O—H···O hydrogen bonds. Weak non-classical C—H···π contacts are also observed in the structure.

Related literature

For structures with C—H···O and C—H···π contacts, see: Nangia (2002); Umezawa *et al.* (1999). For new bioactive secondary metabolites from the endophytic strain B60, see: Shao *et al.* (2007, 2008). For an investigation of the endophytic fungus, see: Shao *et al.* (2008). For a related structure, see: Huang *et al.* (2005).



Experimental

Crystal data

$C_{13}H_{16}O_4$
M_r = 236.26
Triclinic, $P\overline{1}$
a = 7.8401 (16) Å
b = 8.3899 (17) Å
c = 11.099 (2) Å
α = 100.655 (3)°
β = 98.771 (3)°
γ = 115.456 (3)°
V = 625.3 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 291 K
0.40 × 0.38 × 0.35 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.964, T_{max} = 0.968
4817 measured reflections
2420 independent reflections
2058 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.161
S = 1.05
2420 reflections
158 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O4	0.84 (3)	2.16 (2)	2.6519 (15)	117 (2)
O3—H3A···O2^{i}	0.84 (3)	2.20 (3)	2.9111 (16)	143 (2)
C9—H9B···$Cg1^{ii}$	0.97	2.90	3.7483 (2)	146
C13—H13B···$Cg1^{iii}$	0.96	2.96	3.688 (3)	134

Symmetry codes: (i) $x+1, y+1, z$; (ii) $-x, -y+1, -z$; (iii) $-x, -y+2, -z$. $Cg1$ is the centroid of the C3–C8 ring.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2576).

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supporting information

Acta Cryst. (2009). E**65**, o734–o735 [doi:10.1107/S160053680900806X]

Methyl 3-hydroxy-4-(3-methylbut-2-enyloxy)benzoate

Mei-yan Wei, Zhen Liu, Xiu-li Zhang, Chang-lun Shao and Chang-yun Wang

S1. Comment

Marine fungi have proven to be a rich source of novel structural compounds with interesting biological activities and a high level of biodiversity. As a continuation of our previous investigations aimed at finding new bioactive compounds, we found that an unidentified endophytic strain B60 isolated from the mangrove tree can produce new metabolites (Shao *et al.* 2007; Shao *et al.* 2008).

Although the structure of the title compound was previously elucidated on the basis of spectroscopic analysis (Shao *et al.* 2008), we have now determined its solid state structure, Fig. 1, which is reported here. All bond lengths and angles in the molecule are in good agreement with those reported in a related structure by Huang *et al.* (2005). In the title compound, the most striking feature is the interesting arrangement of the molecules, which linked to form a one-dimensional chain by intermolecular O—H···O hydrogen bonds, Table 1, Fig. 2. Further, weak non-classical C—H···π contacts, similar to those previously reported (Nangia, 2002; Umezawa *et al.* 1999) are also observed, in which C9—H9B and C13—H13B act as donors with the benzene ring as the acceptor.

S2. Experimental

An unidentified fungus (No. B60) was deposited in the School of Chemistry and Chemical Engineering, Sun Yat-sen University, Guangzhou, People's Republic of China. Strain No. B60 was cultivated without shaking in GYT medium (10 g of glucose, 2 g of peptone /*L*, 1 g of yeast extract /*L*, 2.5 g of NaCl, 1*L* of water) at 298 K for 4 weeks. The cultures (120 *L*) were filtered through cheesecloth. The filtrate was concentrated to 3 *L* below 323 K, then extracted five times by shaking with an equal volume of ethyl acetate. The extract was evaporated under reduced pressure below 323 K. The combined organic extracts were subjected to silica-gel column chromatography, eluting with petroleum ether/ethyl acetate (9:1, v:v), to yield the title compound, which was confirmed by spectral data including NMR and EI—MS. Crystals of the title compound were obtained by evaporation of an ethyl acetate solution.

S3. Refinement

All H atoms were positioned geometrically and treated as riding, with C—H bonding lengths constrained to 0.93 (aromatic CH), 0.96 Å (methyl CH_3), 0.97 Å (methylene CH_2), and O—H = 0.84 Å, and with *U*ĩso~(H) = 1.2Ueq(CH) or *U*ĩso~(H) = 1.5Ueq(CH_3, methylene C or OH).

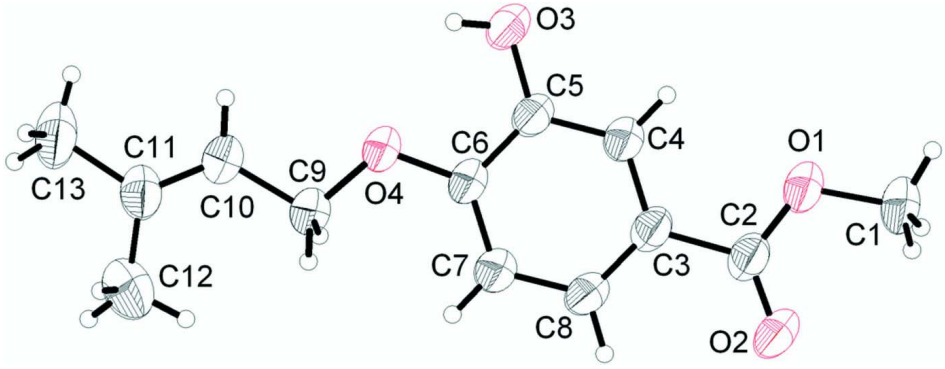

Figure 1

The structure of (I) with displacement ellipsoids for the non-hydrogen atoms drawn at the 30% probability level.

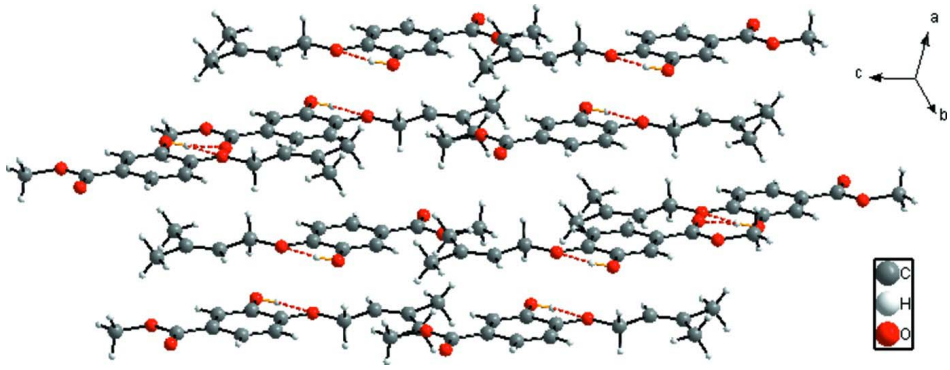

Figure 2

Crystal packing of (I) viewed down the *b* axis with hydrogen bonds drawn as dashed lines.

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Crystal data

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$M_r = 236.26$
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$\beta = 98.771\ (3)°$
$\gamma = 115.456\ (3)°$
$V = 625.3\ (2)$ Å^3
$Z = 2$
$F(000) = 252$
$D_x = 1.255$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4817 reflections
$\theta = 1.9$–$26.0°$
$\mu = 0.09$ mm^{-1}
$T = 291$ K
Block, colorless
0.40 × 0.38 × 0.35 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.964$, $T_{max} = 0.968$
4817 measured reflections
2420 independent reflections
2058 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.9°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.161$
$S = 1.05$
2420 reflections
158 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1024P)^2 + 0.0667P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	−0.4408 (3)	0.2951 (3)	0.50642 (16)	0.0716 (5)
H1A	−0.3697	0.3458	0.5944	0.107*
H1B	−0.4681	0.1692	0.4784	0.107*
H1C	−0.5616	0.3009	0.4960	0.107*
C2	−0.4029 (2)	0.34671 (19)	0.30738 (14)	0.0490 (4)
C3	−0.2748 (2)	0.46837 (17)	0.24198 (13)	0.0453 (3)
C4	−0.0924 (2)	0.61796 (19)	0.30835 (13)	0.0481 (4)
H4A	−0.0474	0.6410	0.3955	0.058*
C5	0.02077 (19)	0.73115 (19)	0.24510 (13)	0.0457 (3)
C6	−0.04479 (19)	0.69490 (18)	0.11391 (13)	0.0452 (3)
C7	−0.2253 (2)	0.5465 (2)	0.04830 (14)	0.0558 (4)
H7A	−0.2702	0.5224	−0.0389	0.067*
C8	−0.3388 (2)	0.4343 (2)	0.11268 (14)	0.0544 (4)
H8A	−0.4597	0.3346	0.0682	0.065*
C9	0.0269 (2)	0.7902 (2)	−0.07071 (14)	0.0557 (4)
H9A	−0.0895	0.8036	−0.0942	0.067*
H9B	0.0003	0.6684	−0.1169	0.067*
C10	0.1952 (2)	0.9336 (2)	−0.10072 (15)	0.0578 (4)
H10A	0.3061	1.0114	−0.0342	0.069*
C11	0.1993 (2)	0.9589 (2)	−0.21421 (14)	0.0546 (4)
C12	0.0315 (3)	0.8410 (3)	−0.32730 (17)	0.0836 (6)
H12A	−0.0725	0.7516	−0.3026	0.125*
H12B	0.0724	0.7793	−0.3892	0.125*
H12C	−0.0138	0.9160	−0.3632	0.125*

C13	0.3751 (3)	1.1067 (3)	−0.2368 (2)	0.0752 (5)
H13A	0.4748	1.1741	−0.1582	0.113*
H13B	0.3387	1.1889	−0.2699	0.113*
H13C	0.4242	1.0518	−0.2968	0.113*
O1	−0.32509 (16)	0.39913 (15)	0.43211 (10)	0.0640 (4)
O2	−0.56125 (16)	0.21602 (16)	0.25485 (12)	0.0702 (4)
O3	0.19557 (16)	0.87793 (16)	0.31218 (11)	0.0663 (4)
H3A	0.231 (4)	0.943 (4)	0.263 (2)	0.102 (8)*
O4	0.08089 (14)	0.81567 (13)	0.06303 (9)	0.0537 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0731 (11)	0.0727 (11)	0.0615 (10)	0.0165 (9)	0.0292 (9)	0.0378 (9)
C2	0.0446 (7)	0.0438 (7)	0.0559 (8)	0.0141 (6)	0.0170 (6)	0.0222 (6)
C3	0.0414 (7)	0.0383 (7)	0.0526 (8)	0.0122 (6)	0.0151 (6)	0.0191 (6)
C4	0.0452 (7)	0.0459 (7)	0.0446 (7)	0.0117 (6)	0.0118 (6)	0.0186 (6)
C5	0.0377 (7)	0.0407 (7)	0.0492 (7)	0.0089 (5)	0.0108 (5)	0.0166 (6)
C6	0.0434 (7)	0.0397 (7)	0.0494 (8)	0.0127 (6)	0.0169 (6)	0.0192 (6)
C7	0.0535 (8)	0.0494 (8)	0.0451 (7)	0.0067 (7)	0.0093 (6)	0.0177 (6)
C8	0.0456 (7)	0.0441 (7)	0.0523 (8)	0.0029 (6)	0.0078 (6)	0.0166 (6)
C9	0.0579 (9)	0.0501 (8)	0.0481 (8)	0.0119 (7)	0.0171 (6)	0.0208 (6)
C10	0.0588 (9)	0.0500 (8)	0.0534 (8)	0.0114 (7)	0.0210 (7)	0.0205 (7)
C11	0.0688 (10)	0.0527 (8)	0.0556 (8)	0.0316 (8)	0.0311 (7)	0.0252 (7)
C12	0.0918 (14)	0.0981 (15)	0.0565 (10)	0.0370 (12)	0.0193 (10)	0.0314 (10)
C13	0.0933 (14)	0.0714 (11)	0.0806 (12)	0.0382 (11)	0.0533 (11)	0.0417 (10)
O1	0.0574 (6)	0.0626 (7)	0.0561 (6)	0.0075 (5)	0.0189 (5)	0.0305 (5)
O2	0.0530 (7)	0.0590 (7)	0.0689 (7)	−0.0030 (5)	0.0148 (5)	0.0275 (6)
O3	0.0486 (6)	0.0604 (7)	0.0555 (7)	−0.0055 (5)	0.0056 (5)	0.0255 (5)
O4	0.0496 (6)	0.0489 (6)	0.0470 (6)	0.0055 (5)	0.0153 (4)	0.0212 (5)

Geometric parameters (Å, º)

C1—O1	1.4418 (17)	C8—H8A	0.9300
C1—H1A	0.9600	C9—O4	1.4289 (18)
C1—H1B	0.9600	C9—C10	1.489 (2)
C1—H1C	0.9600	C9—H9A	0.9700
C2—O2	1.2066 (18)	C9—H9B	0.9700
C2—O1	1.3300 (18)	C10—C11	1.319 (2)
C2—C3	1.4808 (18)	C10—H10A	0.9300
C3—C8	1.378 (2)	C11—C12	1.484 (3)
C3—C4	1.399 (2)	C11—C13	1.500 (2)
C4—C5	1.3763 (18)	C12—H12A	0.9600
C4—H4A	0.9300	C12—H12B	0.9600
C5—O3	1.3594 (17)	C12—H12C	0.9600
C5—C6	1.397 (2)	C13—H13A	0.9600
C6—O4	1.3563 (16)	C13—H13B	0.9600
C6—C7	1.386 (2)	C13—H13C	0.9600

C7—C8	1.382 (2)	O3—H3A	0.84 (3)
C7—H7A	0.9300		
O1—C1—H1A	109.5	O4—C9—C10	106.75 (12)
O1—C1—H1B	109.5	O4—C9—H9A	110.4
H1A—C1—H1B	109.5	C10—C9—H9A	110.4
O1—C1—H1C	109.5	O4—C9—H9B	110.4
H1A—C1—H1C	109.5	C10—C9—H9B	110.4
H1B—C1—H1C	109.5	H9A—C9—H9B	108.6
O2—C2—O1	123.26 (13)	C11—C10—C9	125.11 (15)
O2—C2—C3	124.42 (14)	C11—C10—H10A	117.4
O1—C2—C3	112.31 (12)	C9—C10—H10A	117.4
C8—C3—C4	119.39 (13)	C10—C11—C12	121.92 (15)
C8—C3—C2	118.91 (13)	C10—C11—C13	121.91 (16)
C4—C3—C2	121.69 (13)	C12—C11—C13	116.16 (15)
C5—C4—C3	120.19 (13)	C11—C12—H12A	109.5
C5—C4—H4A	119.9	C11—C12—H12B	109.5
C3—C4—H4A	119.9	H12A—C12—H12B	109.5
O3—C5—C4	119.06 (13)	C11—C12—H12C	109.5
O3—C5—C6	120.93 (12)	H12A—C12—H12C	109.5
C4—C5—C6	120.01 (12)	H12B—C12—H12C	109.5
O4—C6—C7	126.11 (13)	C11—C13—H13A	109.5
O4—C6—C5	114.18 (12)	C11—C13—H13B	109.5
C7—C6—C5	119.71 (13)	H13A—C13—H13B	109.5
C8—C7—C6	119.92 (14)	C11—C13—H13C	109.5
C8—C7—H7A	120.0	H13A—C13—H13C	109.5
C6—C7—H7A	120.0	H13B—C13—H13C	109.5
C3—C8—C7	120.78 (13)	C2—O1—C1	117.18 (12)
C3—C8—H8A	119.6	C5—O3—H3A	105.7 (18)
C7—C8—H8A	119.6	C6—O4—C9	118.40 (11)
O2—C2—C3—C8	0.9 (2)	C5—C6—C7—C8	−0.6 (2)
O1—C2—C3—C8	−178.13 (12)	C4—C3—C8—C7	−0.3 (2)
O2—C2—C3—C4	−179.95 (14)	C2—C3—C8—C7	178.79 (13)
O1—C2—C3—C4	1.0 (2)	C6—C7—C8—C3	0.2 (3)
C8—C3—C4—C5	0.8 (2)	O4—C9—C10—C11	−177.76 (15)
C2—C3—C4—C5	−178.28 (12)	C9—C10—C11—C12	−0.9 (3)
C3—C4—C5—O3	178.57 (13)	C9—C10—C11—C13	−179.89 (15)
C3—C4—C5—C6	−1.2 (2)	O2—C2—O1—C1	−1.6 (2)
O3—C5—C6—O4	0.8 (2)	C3—C2—O1—C1	177.47 (13)
C4—C5—C6—O4	−179.48 (12)	C7—C6—O4—C9	−0.6 (2)
O3—C5—C6—C7	−178.70 (14)	C5—C6—O4—C9	179.99 (12)
C4—C5—C6—C7	1.0 (2)	C10—C9—O4—C6	−176.59 (12)
O4—C6—C7—C8	−179.96 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O4	0.84 (3)	2.16 (2)	2.6519 (15)	117 (2)
O3—H3*A*···O2^{i}	0.84 (3)	2.20 (3)	2.9111 (16)	143 (2)
C9—H9*B*···*Cg*1^{ii}	0.97	2.90	3.7483 (2)	146
C13—H13*B*···*Cg*1^{iii}	0.96	2.96	3.688 (3)	134

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