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1-(2,4-Dihydroxy-3,5-dimethylphenyl)-ethanone (Clavatol)

Sheng-Ying Li,[a] Jian-Feng Wang,[b]* Zhong-Hui Zheng,[a] Qing-Yan Xu,[a] Yao-Jian Huang,[a] Yu-Fen Zhao[b] and Wen-Jin Su[a]

[a]Department of Biology, Xiamen University, Xiamen 361005, People's Republic of China, and [b]Department of Chemistry, Xiamen University, Xiamen 361005, People's Republic of China

Correspondence e-mail: jfwang@yanan.xmu.edu.cn

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.052
wR factor = 0.155
Data-to-parameter ratio = 16.6

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

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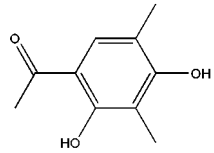
The structure of the title molecule, $C_{10}H_{12}O_3$, is stabilized by an intramolecular O—H···O hydrogen bond. In the crystal structure, the glide-related molecules are linked by O—H···O hydrogen bonds to form molecular chains along [201]. The structure is further stabilized by $\pi\cdots\pi$ interactions.

Comment

The title compound, (I), was isolated from *Aspergillus clavatus*, as a phenolic substance (Bergel *et al.*, 1944) lacking antibacterial activity (Gatenbeck & Brunsberg, 1966; Hasall & Todd, 1947). However, our studies show that it exhibits relative antifungal activity toward *Candida albicans* and *Aspergillus niger* with MIC (Minimum Inhibitory Concentration) of 20 and 40 μg ml^{-1}, respectively, as well as moderate antitumor activity against the Raji, KB and A549 cancer cell lines with IC_{50} values of 9.1, 6.1, and 14.5 μg ml^{-1}, respectively (Li, 2003). Here we report the structure of (I).



(I)

The title molecule, as a whole, excluding H atoms, is planar within 0.062 (2) Å (Fig. 1 and Table 1). The planarity is stabilized by an O—H···O intramolecular hydrogen bond involving atoms O1 and O2 (Table 2). The crystal structure shows that the glide-related molecules are linked by O—H···O hydrogen bonds involving a hydroxyl group and the

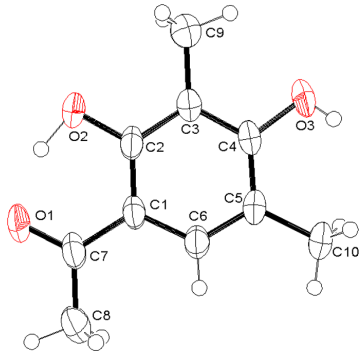


Figure 1
*ORTEP*III (Farrugia, 1997) view of the title compound, with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

carbonyl O atom to form molecular chains running approximately along [201] (Fig. 2). Furthermore, the glide-related molecules are stacked along the *c* axis, with the centroids of the aromatic rings separated by 3.618 (1) Å, indicating significant $\pi\cdots\pi$ interactions.

Experimental

The title compound was isolated from *Aspergillus sp*. strain BYY-1, an endophytic fungus of *Avicennia marina* that was collected from the mangrove forest in Fujian Province, People's Republic of China. Crystals were grown using methanol as solvent.

Crystal data

$C_{10}H_{12}O_3$
M_r = 180.20
Monoclinic, $P2_1/c$
a = 8.318 (1) Å
b = 15.200 (3) Å
c = 7.204 (1) Å
β = 96.896 (3)°
V = 904.2 (2) Å^3
Z = 4
D_x = 1.324 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3017 reflections
θ = 2.4–28.0°
μ = 0.10 mm^{-1}
T = 298 (2) K
Block, colorless
0.35 × 0.23 × 0.18 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.947, T_{max} = 0.983
5370 measured reflections
2078 independent reflections
1725 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 28.3°
$h = -10 \rightarrow 10$
$k = -17 \rightarrow 20$
$l = -8 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.155
S = 1.04
2078 reflections
125 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0945P)^2 + 0.142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.034 (8)

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.243 (2)	C3—C4	1.387 (2)
O2—C2	1.350 (2)	C3—C9	1.496 (2)
O3—C4	1.352 (2)	C4—C5	1.406 (2)
C1—C6	1.402 (2)	C5—C6	1.369 (2)
C1—C2	1.405 (2)	C5—C10	1.499 (2)
C1—C7	1.448 (2)	C7—C8	1.489 (2)
C2—C3	1.390 (2)		
C2—O2—H2	109.5	O3—C4—C3	115.4 (1)
C4—O3—H3	109.5	O3—C4—C5	121.9 (1)
C6—C1—C2	117.6 (1)	C3—C4—C5	122.7 (1)
C6—C1—C7	121.8 (1)	C6—C5—C4	117.1 (1)
C2—C1—C7	120.6 (1)	C6—C5—C10	121.7 (1)
O2—C2—C3	117.2 (1)	C4—C5—C10	121.2 (1)
O2—C2—C1	121.3 (1)	C5—C6—C1	123.1 (1)
C3—C2—C1	121.4 (1)	O1—C7—C1	120.8 (1)
C4—C3—C2	118.1 (1)	O1—C7—C8	118.5 (1)
C4—C3—C9	121.0 (1)	C1—C7—C8	120.7 (1)
C2—C3—C9	121.0 (1)		
C2—C1—C7—O1	1.7 (2)	C6—C1—C7—C8	2.5 (2)

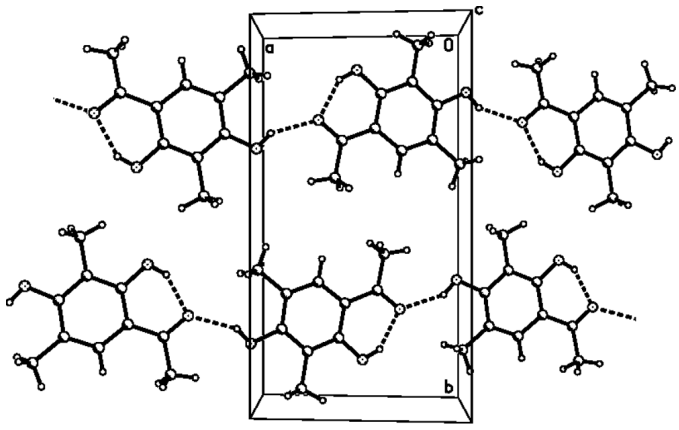


Figure 2
O—H···O hydrogen-bonded chains in the unit cell, viewed down the *c* axis.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.82	1.82	2.542 (2)	147
O3—H3···O1^{i}	0.82	2.01	2.749 (1)	149

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

All H atoms were positioned geometrically and were included in the refinement in the riding-model approximation [C—H = 0.93–0.96 Å, O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(parent atom)]. A rotating-group model was used for the methyl and hydroxyl groups.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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