

1-(2,6-Dihydroxyphenyl)ethanone

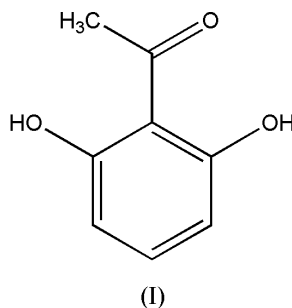
Hua-Rong Huang,^a Xue-Kui Xia,^a
Zhi-Gang She,^a Yong-Cheng
Lin,^{a*} L. L. P. Vrijmoed^b and
E. B. Gareth Jones^c^aSchool of Chemistry and Chemical Engineering,
Zhongshan University, Guangzhou 510275,
People's Republic of China, ^bDepartment of
Biology and Chemistry, City University of Hong
Kong, People's Republic of China, and
^cNational Center for Genetic Engineering and
Biotechnology, Bangkok, Thailand

Correspondence e-mail: ceslyc@zsu.edu.cn

Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.041
 wR factor = 0.122
Data-to-parameter ratio = 15.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_8\text{H}_8\text{O}_3$, has several intra- and intermolecular hydrogen bonds in its crystal structure. There are two molecules in the asymmetric unit, and they are extended into infinite chains along [011] by intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds.

Comment

The title compound, (I), was isolated from the extracts of cultures of the estuarine fungus (No. 3920). This substance was previously isolated from the extracts of cultures of *D. Concentrica* strain 26 A1 (Allport & Bu'Lock, 1960). The structure of (I) was previously elucidated on the basis of spectroscopic analysis. We report here the crystal structure of (I).The X-ray crystallographic study of (I) confirms the previously proposed molecular structure based on spectroscopic data. There are two crystallographically independent molecules in the asymmetric unit (Fig. 1). The C—O and C—C distances show no remarkable features (Table 1). A feature of the structure of (I) is the presence of both intra- and intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds between the hydroxy groups and the carbonyl O atom (Table 2), resulting in infinite chains along [011] (Fig. 2).

Experimental

A strain of fungus (No. 3920) was isolated from an endophyte NP 159/ Morphology Type10 from *Kandelia* Bark Mai Po, Hong Kong, and deposited in the Department of Applied Chemistry, ZhongShan University, Guangzhou, People's Republic of China. Culture conditions: GYT medium (glucose 10 g l^{-1} , peptone 2 g l^{-1} , yeast extract 1 g l^{-1} , NaCl 2 g l^{-1}) and incubation at 298 K for 28 d. Extraction and separation of metabolite: the cultures (100 l) were filtered through cheesecloth. The filtrate was concentrated to 5 l below 333 K, then extracted three times by shaking with an equal volume of ethyl acetate. The extract was evaporated under reduced pressure. The combined organic extracts were subjected to silica-gel column chromatography, eluting with petroleum ether/ethyl acetate, to yield theReceived 3 November 2004
Accepted 23 November 2004
Online 30 November 2004

title compound, (I). The compound's identity was confirmed by the NMR spectra. Crystals of (I) were obtained by evaporation of a methanol solution. ¹H NMR (300 MHz, actone-*d*₆): δ 2.69 (*s*, H), 6.40 (*d*, *J* = 8.1 Hz, H3, H5), 7.23 (*t*, *J* = 8.1, 16.2 Hz, H4), 11.44 (*s*, 2-OH, 6-OH).

Crystal data

C₈H₈O₃ Z = 4
M_r = 152.14 *D_x* = 1.400 Mg m⁻³
 Triclinic, *P*1̄ Mo *K*α radiation
a = 7.646 (3) Å Cell parameters from 816
b = 8.325 (3) Å reflections
c = 12.803 (5) Å θ = 2.8–26.0°
 α = 73.321 (6)° μ = 0.11 mm⁻¹
 β = 79.042 (7)° *T* = 293 (2) K
 γ = 68.230 (6)° Block, colorless
V = 721.8 (5) Å³ 0.48 × 0.42 × 0.35 mm

Data collection

Bruker SMART CCD area-detector 3115 independent reflections
 diffractometer 2065 reflections with *I* > 2σ(*I*)
 φ and ω scans *R_{int}* = 0.015
 Absorption correction: multi-scan θ_{max} = 27.1°
 (SADABS; Sheldrick, 1996) *h* = -9 → 9
T_{min} = 0.950, *T_{max}* = 0.963 *k* = -10 → 10
 6117 measured reflections *l* = -15 → 16

Refinement

Refinement on *F*² *w* = 1/[σ²(*F_o*²) + (0.0579*P*)²
R[*F*² > 2σ(*F*²)] = 0.041 + 0.1147*P*]
wR(*F*²) = 0.122 where *P* = (*F_o*² + 2*F_c*²)/3
S = 1.03 (Δ/σ)_{max} < 0.001
 3115 reflections Δρ_{max} = 0.24 e Å⁻³
 201 parameters Δρ_{min} = -0.13 e Å⁻³
 H-atom parameters constrained

Table 1

Selected bond lengths (Å).

C1–C2	1.414 (2)	C9–C14	1.413 (2)
C1–C6	1.415 (2)	C9–C10	1.420 (2)
C1–C7	1.472 (2)	C9–C15	1.461 (2)
C2–O1	1.3519 (19)	C10–O4	1.3469 (19)
C2–C3	1.382 (2)	C10–C11	1.379 (2)
C3–C4	1.369 (2)	C11–C12	1.365 (3)
C4–C5	1.379 (2)	C12–C13	1.372 (3)
C5–C6	1.379 (2)	C13–C14	1.381 (2)
C6–O2	1.3539 (18)	C14–O5	1.352 (2)
C7–O3	1.2378 (19)	C15–O6	1.2425 (19)
C7–C8	1.489 (2)	C15–C16	1.486 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1–H1A...O6 ⁱ	0.82	1.95	2.767 (2)	176
O2–H2A...O3	0.82	1.74	2.472 (2)	148
O4–H4A...O6	0.82	1.79	2.513 (2)	146
O5–H5A...O2 ⁱⁱ	0.82	1.97	2.7878 (19)	180

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

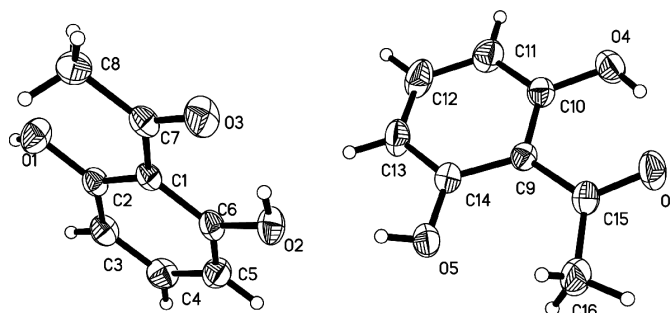


Figure 1

The asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

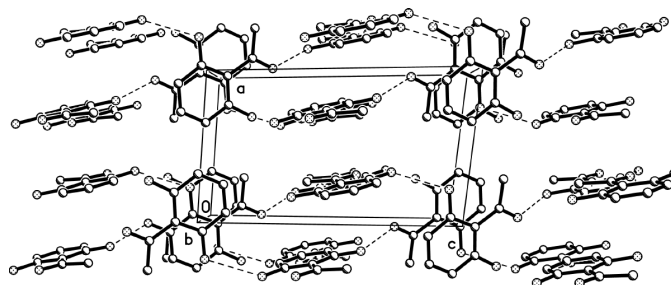


Figure 2

The packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

The H atoms were positioned geometrically and were treated as riding on their parent C and O atoms, with C–H distances in the range 0.93–0.96 Å and O–H distances of 0.82 Å.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT-Plus (Bruker, 1999); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1999); software used to prepare material for publication: SHELXTL.

The authors thank the 863 Foundation of China (No. 2003AA624010) and the Natural Science Foundation of Guangdong Province (No. 2003A2050401).

References

Allport, D. C. & Bu'Lock, J. D. (1960). *J. Chem. Soc.* pp. 654–662.
 Bruker (1999). SMART (Version 5.054), SAINT-Plus (Version 6.45) and SHELXTL (Version 6.14). Bruker AXS Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.