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1-(2,6-Dihydroxyphenyl)butanone

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.049
wR factor = 0.143
Data-to-parameter ratio = 14.7

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

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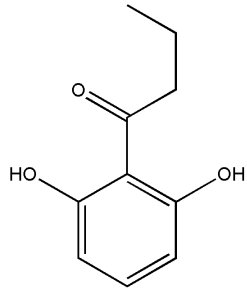
The title compound, $C_{10}H_{12}O_3$, has several intra- and intermolecular hydrogen bonds in the crystal structure. The molecules are linked into infinite chains along [011] *via* intermolecular O—H···O hydrogen bonds. There are two molecules in the asymmetric unit.

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).



(I)

The X-ray 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. A structural feature of (I) is the presence of both intra- and intermolecular O—H···O hydrogen bonds; one is between the hydroxy groups and the carbonyl O atom, and the other between the hydroxy groups (Table 1), resulting in infinite chains along [011] (Fig. 2). Positional disorder of the propyl chain was resolved for one of the molecules in the asymmetric unit.

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. For the extraction and separation of the metabolite, the cultures (100 l) of (I) were filtered through cheesecloth. The filtrate was concentrated to 5 l below 323 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 (I). The compound's identity was confirmed by NMR spectroscopy. Crystals of (I) were obtained by evaporation of a methanol solution. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.00 (*t*, *J* = 7.2 and 14.7 Hz, 3H), 1.75 (*m*, 2H), 3.13 (*t*, *J* = 7.2 and 14.7 Hz, 2H), 6.40 (*d*, *J* = 8.1 Hz, 2H, H3, H5), 7.20 (*t*, *J* = 8.1 and 16.5 Hz, 1H, H4), 9.94 (*s*, 2-OH, 6-OH).

Crystal data

$C_{10}H_{12}O_3$
M_r = 180.20
Triclinic, $P\overline{1}$
a = 7.805 (3) Å
b = 10.705 (5) Å
c = 11.805 (5) Å
α = 107.729 (7)°
β = 98.068 (7)°
γ = 91.090 (8)°
V = 928.2 (7) Å^3
Z = 4
D_x = 1.289 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 897 reflections
θ = 3.4–26.5°
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colorless
0.40 × 0.38 × 0.35 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.963, T_{max} = 0.968
7939 measured reflections
3993 independent reflections
2322 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.1°
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.143
S = 1.03
3993 reflections
271 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 0.0999P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.0001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O6	0.90 (2)	1.66 (2)	2.498 (2)	153 (2)
O5—H5···O1	0.85 (2)	1.90 (2)	2.744 (2)	169 (2)
O1—H1···O3	0.96 (3)	1.58 (3)	2.460 (2)	151 (3)
O2—H2···O6^{i}	0.90 (3)	1.83 (3)	2.712 (2)	166 (2)

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

C-bound H atoms were positioned geometrically and were included in the refinement in the riding-model approximation, with distances of 0.96 (CH_3), 0.97 (CH_2) and 0.93 Å (CH); U_{iso}(H) = 1.2U_{eq}(C) for H atoms on secondary and tertiary C atoms, and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms. Hydroxy H atoms were located in difference Fourier maps and refined isotropically.

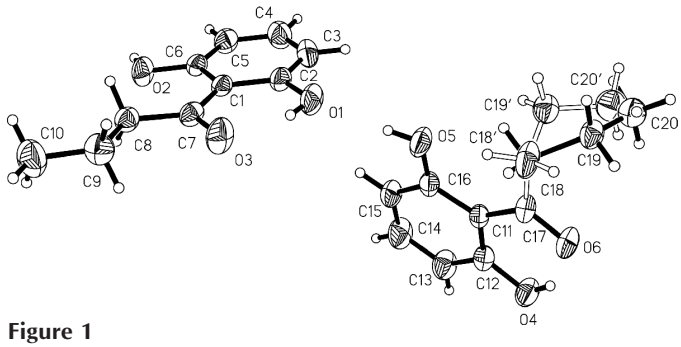


Figure 1
The structure of the asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme

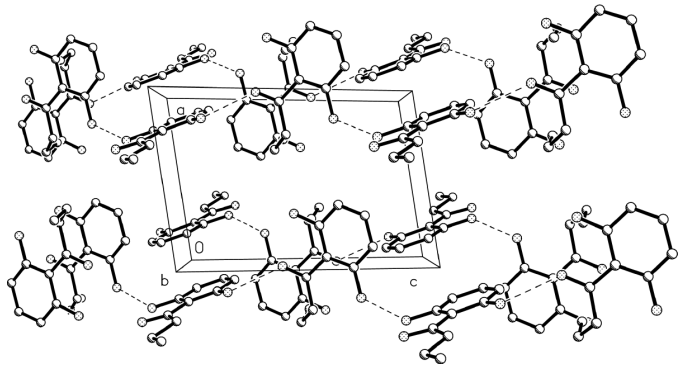


Figure 2
A packing diagram of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms have been omitted.

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

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