Acta Crystallographica Section E
Structure Reports Online

ISSN 1600-5368

3,4-Dihydro-8-hydroxy-3,5-dimethyl-7-methoxyisocoumarin (avicennin B)

Jun Wang,[a]* Hua-Rong Huang,[b] Xiao-Long Feng,[c] Yong-Cheng Lin[b] and Shi-Ning Zhou[d]

[a]School of Pharmaceutical Science, Sun Yat-Sen (Zhongshan) University, Guangzhou 510275, People's Republic of China, [b]School of Chemistry and Chemical Engineering, Sun Yat-Sen (Zhongshan) University, Guangzhou 510275, People's Republic of China, [c]Instrumentation Analysis and Research Center, Sun Yat-Sen (Zhongshan) University, Guangzhou 510275, People's Republic of China, and [d]School of Life Sciences, Sun Yat-Sen (Zhongshan) University, Guangzhou 510275, People's Republic of China

Correspondence e-mail: zwangjun@21cn.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.038
wR factor = 0.111
Data-to-parameter ratio = 8.1

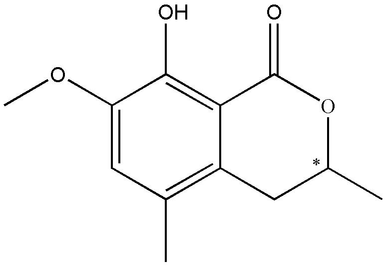
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{12}H_{14}O_4$, contains two molecules in the asymmetric unit, each with an intramolecular O—H···O hydrogen bond.

Received 4 January 2005
Accepted 25 January 2005

Comment

The title compound, (I), was isolated from the metabolites of the mangrove endophytic fungus (No. 2533). The structure of (I) was previously elucidated on the basis of spectroscopic analysis (Lin *et al.*, 2001). We report here its crystal structure.



(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 are unremarkable. A feature of the structure of (I) is the presence of intramolecular O—H···O hydrogen bonds between the hydroxyl groups and carbonyl O atoms (Table 1).

Experimental

A strain of the fungus (No.2533), an endophytic fungus, was isolated from mangrove young avicennia leaf from the South China Sea Coast and was stored in the Department of Applied Chemistry, Zhongshan University, Guangzhou, China. Culture conditions: GYT medium (glucose 5 g l^{-1}, peptone 1 g l^{-1}, yeast extract 0.5 g l^{-1}, beef extract 0.5 g l^{-1}, natural sea water 50 ml l^{-1}) and incubation at 298 K for 30 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 and the combined organic extracts were subjected to silica-gel column chromatography, eluting with petroleum ether/ethyl acetate to yield the title compound, (I). ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.67 (*qdd*, J = 6.5, 3 and 12 Hz), 2.99 (*dd*, J = 3 and 17 Hz), 2.68 (*dd*, J = 12 and 17 Hz), 6.89 (*s*), 1.54 (*d*, J = 6.5 Hz), 2.21 (*s*), 3.89 (*s*), 11.20 (*s*). Crystals of (I) were obtained by evaporation of a methanol solution.

doi:10.1107/S1600536805002771

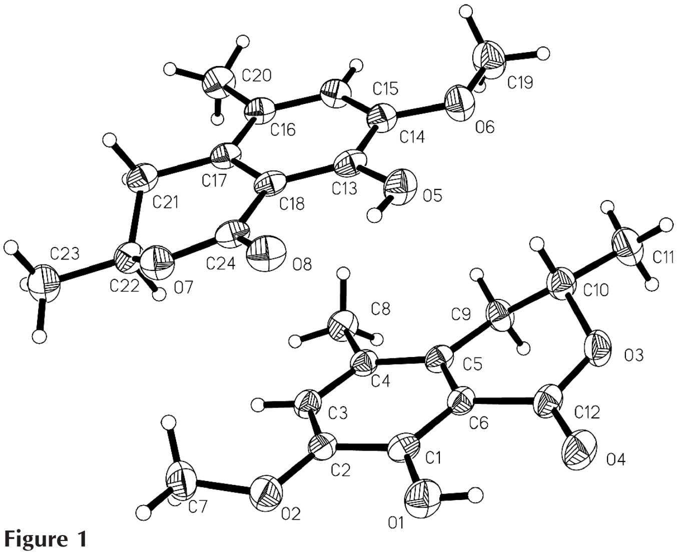


Figure 1
The structure of the asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Crystal data

$C_{12}H_{14}O_4$
$M_r = 222.23$
Tetragonal, $P4_3$
$a = 8.824$ (2) Å
$c = 27.733$ (10) Å
$V = 2159.4$ (11) Å^3
$Z = 8$
$D_x = 1.367$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1001 reflections
$\theta = 3.2$–26.6°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Block, colourless
$0.54 \times 0.48 \times 0.47$ mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.947$, $T_{\max} = 0.953$
12 988 measured reflections
2405 independent reflections
2027 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 27.0°$
$h = -11 \rightarrow 8$
$k = -10 \rightarrow 11$
$l = -35 \rightarrow 34$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.111$
$S = 1.07$
2405 reflections
297 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2 + 0.3661P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

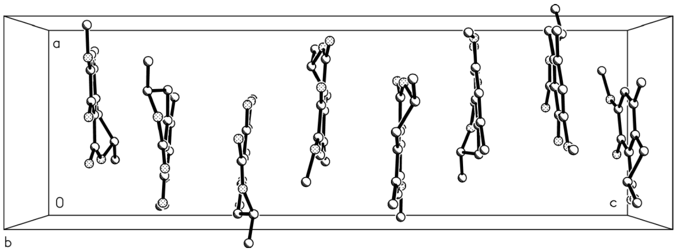


Figure 2
The packing of (I), viewed down the *b* axis. H atoms have been omitted.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4	0.82	1.88	2.598 (3)	145
O5—H5···O8	0.82	1.87	2.585 (3)	145

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 0.93–0.97 Å and an O—H distance of 0.82 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for H atoms on secondary and tertiary C atoms, and 1.5$U_{\rm eq}$(C) for methyl H atoms.. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

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*.

The authors thank the Guangdong Natural Science Foundation (grant No. 200308031527).

References

Bruker (1999). *SMART* (Version 5.054), *SAINT-Plus* (Version 6.45) and *SHELXTL* (Version 6.14). Bruker AXS Inc, Madison, Wisconsin, USA.

Lin, Y. C., Wang, J., Zhou, S. N. & Jones, E. B. G. (2001). *Chem. J. Internet*, **3**, 30–33.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.