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2-Hexylidene-3-methylsuccinic acid

Xiao-Hong Liu,[a,b] Fang Xu,[b] Yong-Cheng Lin,[a,b]* Wing-Lai Chan[c,d] and Zhong-Yuan Zhou[c,d]

[a]School of Chemistry and Chemical Engineering, Sun Yat-sen University, Guangzhou 510275, People's Republic of China, [b]School of Pharmaceutical Sciences, Sun Yat-sen University, Guangzhou 510080, People's Republic of China, [c]Department of Biology and Chemistry, The Hong Kong Polytechnic University, Hong Kong, People's Republic of China, and [d]Open Laboratory of Chirotechnology of the Institute of, Molecular Technology for Drug Discovery and Synthesis, The Hong Kong Polytechnic University, Hong Kong, People's Republic of China

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

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The title compound, $C_{11}H_{18}O_4$, also known as piliformic acid, was isolated from the endophytic fungus *Xylariasp sp.* No. 2508 from mangrove trees from the coast of the South China Sea. The two carboxyl groups form intermolecular O—H···O hydrogen bonds, which link the molecules into infinite chains extending along the *c* axis.

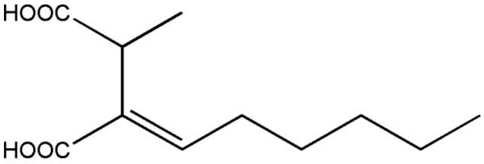
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.066
wR factor = 0.224
Data-to-parameter ratio = 14.3

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

Comment

The title compound, (I), also known as piliformic acid, was isolated from the endophytic fungus *Xylariasp sp.* No. 2508 from mangrove trees from the coast of the South China Sea. The mangrove fungus strain No. 2508 has earlier been found to produce rich secondary metabolites (Lin, Wu, Feng, Jiang, Zhou *et al.*, 2001; Lin, Wu, Feng, Jiang, Luo *et al.*, 2001; Wu *et al.*, 2005). This substance was previously isolated from extracts of cultures of xylariaceous fungi *H. deustum* (Anderson *et al.*, 1985), and its molecular structure was elucidated on the basis of spectroscopic analysis. We report here the crystal structure of (I).



(I)

The X-ray crystallographic study of (I) confirms the previously proposed molecular structure (Fig. 1). All bond lengths and angles show normal values. In the crystal structure, the two carboxyl groups form intermolecular O—H···O hydrogen bonds (Table 1), which link the molecules into infinite chains extending along the *c* axis (Fig. 2).

Experimental

A strain of the fungus *Xylaria sp.* (No. 2508) was isolated from seeds of an angiosperm tree in Mai Po, Hong Kong, and was stored at the Department of Applied Chemistry, Zhongshan University, Guangzhou, China. Culture conditions: GYT medium (glucose 12 g l^{-1}, peptone 2 g l^{-1}, yeast extract 1 g l^{-1}, NaCl 30 g l^{-1}), incubation at 303 K for 5–7 d. Extraction and separation of metabolite: the cultures (170 l) were filtered through cheesecloth. The filtrate was concentrated to 3.5 l below 333 K, then extracted five times by shaking with an equal volume of EtOAc. The combined extracts were concentrated under reduced pressure to give a brown oil and chromatographed on silica gel using a gradient elution from petroleum ether to ethyl acetate, to obtain the title compound, (I), from the 30% ethyl acetate/petroleum ether fraction. Crystals of (I) were obtained

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by solvent evaporation of a petroleum ether–ethyl acetate (60%) solution.

Crystal data

$C_{11}H_{18}O_4$
$M_r = 214.25$
Triclinic, $P\overline{1}$
$a = 6.7208$ (14) Å
$b = 9.488$ (2) Å
$c = 10.139$ (2) Å
$\alpha = 85.994$ (4)°
$\beta = 89.221$ (4)°
$\gamma = 84.457$ (4)°
$V = 641.9$ (2) Å^3
$Z = 2$
$D_x = 1.109$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 294$ (2) K
Block, colourless
0.32 × 0.24 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.793$, $T_{\max} = 0.988$
5098 measured reflections
2497 independent reflections
1402 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 26.0$°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.224$
$S = 1.03$
2497 reflections
175 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.114P)^2 + 0.1365P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D$$\cdots$$A$	D—H$\cdots$$A$
O2—H2$\cdots$O1$^{\rm i}$	0.82	1.83	2.652 (3)	176
O4—H4$\cdots$O3$^{\rm ii}$	0.82	1.82	2.617 (3)	164

Symmetry codes: (i) $-x+3, -y+1, -z$; (ii) $-x+3, -y+1, -z+1$.

The C-bound H atoms were positioned geometrically, with C—H = 0.93–0.97 Å, and refined as riding, with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for H atoms on secondary and tertiary C atoms, and $1.5U_{\rm eq}$(C) for methyl H atoms. The O-bound H atoms were located in a difference Fourier map and refined as riding, with O—H = 0.82 Å and $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(O). The hexyl chain was treated as disordered over two sets of positions, with refined occupancies of 0.520 (8) and 0.480 (8).

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*.

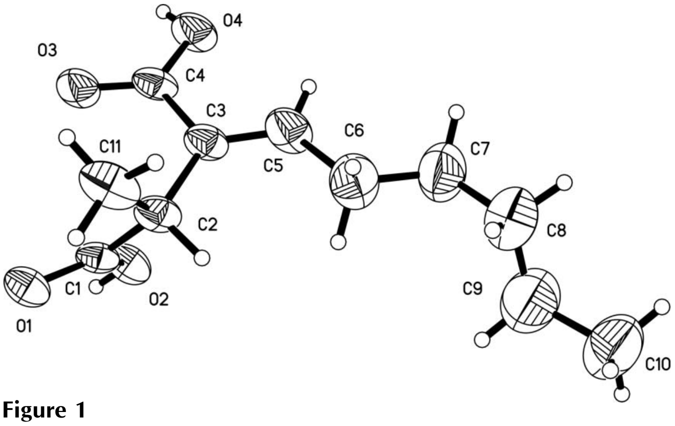

Figure 1
View of (I), showing the atomic numbering and displacement ellipsoids drawn at the 30% probability level. Only the major component of the disordered hexyl group is shown.

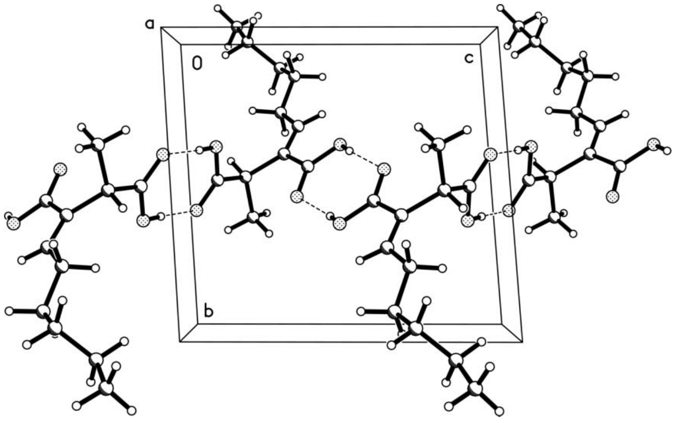

Figure 2
A packing diagram of (I), viewed down the a axis. Hydrogen bonds are shown as dashed lines. Only the major component of the disordered hexyl group is shown.

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