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8β-Hydroxy-2,6,6-trimethyl-5βH,10βH-eudesma-1,7(11)-dien-12,8-olide

Guang-Wen Zhang,[a] Cheng-Zhu Liao,[b] Xin-Sheng Yao,[a] Hiroshi Kurihara,[a] Jing-Yu Su[c] and Long-Mei Zeng[c]*

[a]Institute of Traditional Chinese Medicine and Natural Products, Jinan University, Guangzhou 510632, People's Republic of China, [b]Instrumentation Analysis and Research Center, Sun Yat-Sen (Zhongshan) University, Guangzhou 510275, People's Republic of China, and [c]The School of Chemistry and Chemical Engineering, Sun Yat-Sen (Zhongshan) University, Guangzhou 510275, People's Republic of China

Correspondence e-mail: lcz613@163.com

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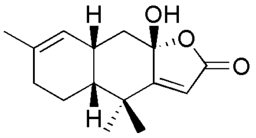
In the title compound, $C_{15}H_{20}O_3$, molecules are linked in an infinite chain along the *a* axis, with graph-set motifs $C_2^2(12)$ or $C_2^2(14)$, through O—H···O hydrogen bonds.

Comment

8β-Hydroxy-2,6,6-trimethyl-5βH,10βH-eudesma-1,7(11)-dien-12,8-olide, (I), was isolated from the marine sponge *Dysidea avara*; previously it has been isolated from the marine sponge *Dysidea fragilis* (Zhong *et al.*, 1995). A pharmacological experiment shows that, when the concentration of compound (I) is 5.58×10^{-4} M, it significantly inhibited spontaneous movement of rabbit ileum, controlled autonomy activity and muscular activity.



(I)

The X-ray study of (I) further confirms the previously proposed structure based on spectroscopic data. The asymmetric unit of (I) consists of one independent molecule (Fig. 1). In the crystal structure, molecules are linked in an infinite chain along the *a* axis, described as $C_2^2(12)$ or $C_2^2(14)$ according to graph-set notation (Etter, 1990), through O3—H1···O1 hydrogen bonds (Fig. 2 and Table 1). The most characteristic feature is that (I) can be described as a hydrophobic–hydrophilic layered structure along the *c* axis, with the two six-membered rings forming the hydrophobic portion, while the five-membered lactone rings form the hydrophilic portion.

In the crystal structure, the middle six-membered ring adopts a chair conformation and one of the attached methyl groups and the hydroxy group occupy axial positions.

Experimental

Freshly collected sponge *Dysidea avara* was cut into pieces and extracted with 95% EtOH three times. The combined extracts were concentrated *in vacuo* and the crude extract was partitioned between water and EtOAc. The EtOAc-soluble portion was subjected to vacuum liquid chromatography (VLC), eluting with EtOAc–hexane, to yield (I). The compound's identity was confirmed by NMR spectroscopy. Crystals of (I) were obtained from hexane–EtOAc by solvent diffusion. ^{1}H NMR (500 MHz, $CDCl_3$): 5.66 (1H, *s*, H11), 5.37 (1H, *m*, H1), 2.82 (1H, *dd*, J = 13.0 and 4.0 Hz, H10), 2.32 (1H, *dd*, J = 14.0 and 4.0 Hz, H9*A*), 1.95 (2H, *m*, H4), 1.71 (1H, *m*, H5), 1.67 (1H,

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.095
Data-to-parameter ratio = 10.3

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

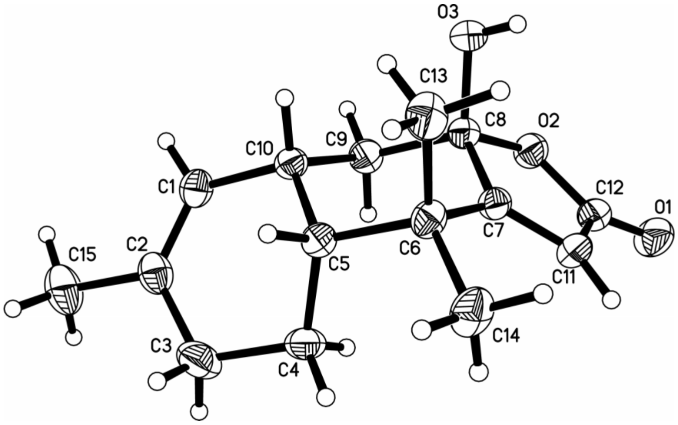


Figure 1
View of the title compound, showing 30% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

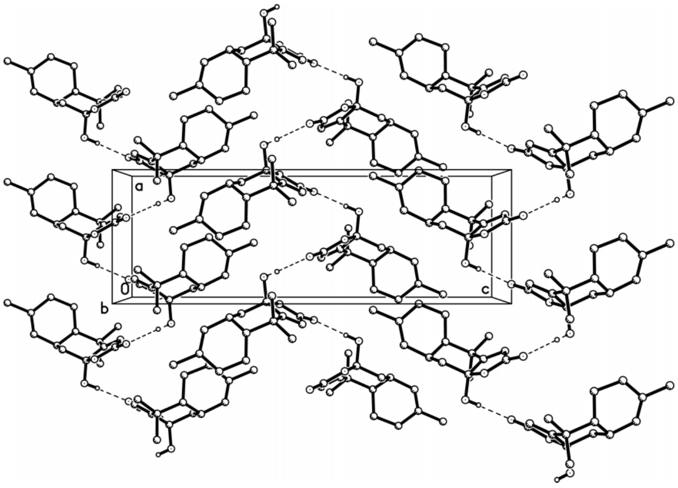


Figure 2
Packing arrangement of the title compound, projected on the *ac* plane. All H atoms attached to C atoms have been omitted for clarity. Hydrogen bonds are shown as dashed lines.

m, H3*B*), 1.62 (1H, *s*, H15), 1.55 (1H, *t*, $J = 13.5$ Hz, H9*B*), 1.41 (1H, *s*, H13), 1.22 (1H, *s*, H14), 1.13 (1H, *m*, H3*A*) ^{13}C NMR: 123.6 (*d*, C1), 134.3 (*s*, C2), 18.7 (*t*, C3), 31.0 (*t*, C4), 47.3 (*d*, C5), 38.6 (*s*, C6), 175.3 (*s*, C7), 105.5 (*s*, C8), 40.9 (*t*, C9), 30.4 (*d*, C10), 114.9 (*d*, C11), 170.8 (*s*, C2), 26.8 (*q*, C13), 25.3 (*q*, C14), 23.11 (*q*, C15).

Crystal data

$C_{15}H_{20}O_3$
$M_r = 248.31$
Orthorhombic, $P2_12_12_1$
$a = 7.1843$ (9) Å
$b = 8.7554$ (11) Å
$c = 21.310$ (3) Å
$V = 1340.4$ (3) Å^3
$Z = 4$

$D_x = 1.230$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 929 reflections
$\theta = 2.5$–$27.0°$
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.50 × 0.40 × 0.35 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.959$, $T_{\max} = 0.971$
7880 measured reflections

1714 independent reflections
1619 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$
$\theta_{\max} = 27.0°$
$h = -9 \rightarrow 4$
$k = -10 \rightarrow 11$
$l = -27 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.095$
$S = 1.05$
1714 reflections
167 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 0.1304P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1$^{\rm i}$	0.82	2.00	2.8128 (19)	172

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

H atoms were positioned geometrically and were treated as riding on the parent C and O atoms, with C—H distances of 0.93–0.98 Å and an O—H distance of 0.82 Å [$U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) or $1.5U_{\rm eq}$(C,O)]. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was assigned arbitrarily.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (1998). *SMART* (Version 5.0) and *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT-Plus*. Version 6. Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zhong, Y.-L., Su, J.-Y. & Zeng, L.-M. (1995). *Bopu Zaizhi*, **12**, 47–54. (In Chinese.)

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