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24-Methylenecholest-4-ene-3β,6β-diol

Hui Lin, Shi-Hai Xu,* Xiang-Chao Zeng and Xiao-Jian Liao

Department of Chemistry, Jinan University, Guangzhou, Guangdong 510632, People's Republic of China

Correspondence e-mail: txush@jnu.edu.cn

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.006 Å
R factor = 0.061
wR factor = 0.215
Data-to-parameter ratio = 11.7

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

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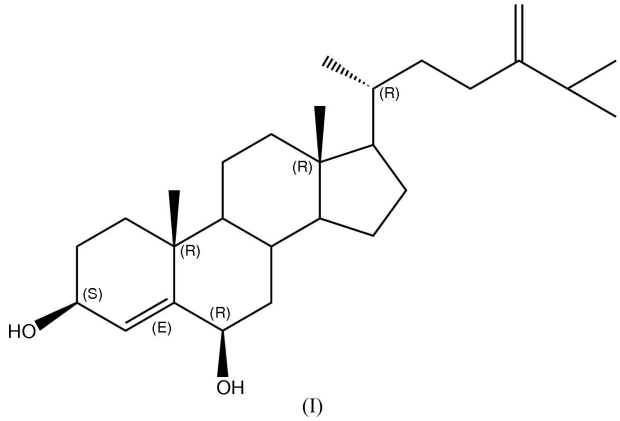
The title compound, $C_{28}H_{46}O_2$, was isolated from soft coral (*Nephthea* sp.) collected from Yongxing island of the Xisha archipelago in the South China Sea and its crystal structure has been determined. In the crystal structure, intermolecular O—H···O hydrogen-bond interactions link the molecules into extended ribbons parallel to the *a* axis.

Comment

24-Methylenecholest-4-ene-3β,6β-diol, (I), was first isolated by Zeng *et al.* (1995) from the soft coral *Alcyonium patagonicum* collected from the South China Sea. Its configuration had also been elucidated by the reduction of steroidal 4-ene-3,6-diones with KBH_4-C_2H_5OH (Cui *et al.*, 2002), but no three-dimensional coordinates were available. Antitumour studies have shown that (I) is cytotoxic against the P-388 cell line, with an IC_{50} value of 1 µg ml^{-1} (Zeng *et al.*, 1995). We report here the crystal structure of (I).



(I)

Bond lengths and angles are unexceptional and are in good agreement with the corresponding values in stigmast-4-ene-3β,6β-diol (Yang *et al.*, 2004).

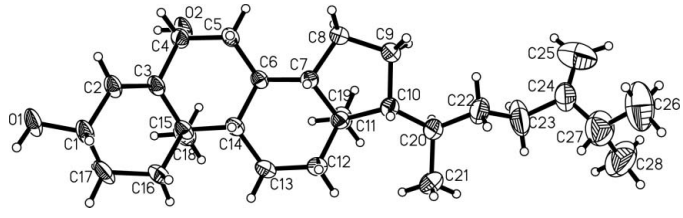


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

In the crystal structure of (I), there are two kinds of intermolecular hydrogen bonds. O1—H···O2 hydrogen bonds link neighbouring molecules together, generating one-dimensional chains (Fig. 2). Adjacent chains are further connected by O2—H···O1 hydrogen-bond interactions, generating extended ribbons running parallel to the *a* axis (Fig. 2).

Experimental

Chopped soft coral (7.8kg) was extracted with EtOH (18l) at room temperature and then partitioned between EtOAc and H_2O (4l each). The organic phase was chromatographed on a silica-gel column, eluting with EtOAc–petroleum ether with gradually increasing amounts of EtOAc. The fraction eluted with EtOAc–petroleum ether in the ratio 65:35 gave the title compound. A sample was dissolved in pyridine at room temperature and normal pressure; colourless crystals of (I) suitable for X-ray analysis grew over a period of one month when the solution was exposed to the air. Spectroscopic analysis: ^{1}H NMR (THF, 500Hz, δ, p.p.m.): 5.39 (*s*, 1H, C2-H), 4.69 (*d*, 1H, C25-H), 4.64 (*s*, 1H, C25-H), 4.02 (*s*, 1H, C4-H), 3.99 (*m*, 1H, C1-H), 1.24 (*s*, 3H, C18-H), 1.02, 1.01 (*m*, 6H, C2-H, C28-H), 0.74 (*s*, 3H, C19-H); ^{13}C NMR (pyridine, δ, p.p.m.): 156.7 (1C, C24), 147.1 (1C, C3), 129.9 (1C, C2), 106.6 (1C, C25), 73.7 (1C, C4), 67.5 (1C, C1), 56.5 (1C, C10), 56.4 (1C, C7), 55.0 (1C, C14), 42.8 (1C, C11), 40.7 (1C, C12), 40.2 (1C, C5), 37.7(1C, C16), 37.3 (1C, C15), 36.0 (1C, C20), 35.1 (1C, C22), 34.1 (1C, C27), 31.3 (1C, C23), 31.0 (1C, C6), 30.2 (1C, C17), 28.4 (1C, C8), 24.5 (1C, C9), 22.1 (1C, C18), 21.9 (1C, C26), 21.6 (1C, C28), 21.3 (1C, C13), 18.9 (1C, C21), 12.2 (1C, C19).

Crystal data

$C_{28}H_{46}O_2$
M_r = 414.65
Orthorhombic, $P2_12_12_1$
a = 8.168 (3) Å
b = 10.933 (3) Å
c = 28.446 (8) Å
V = 2540.2 (13) Å^3
Z = 4
D_x = 1.084 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 965 reflections
θ = 2.84–21.54°
μ = 0.07 mm^{-1}
T = 273 (2) K
Block, colourless
0.50 × 0.40 × 0.20 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.97, T_{max} = 0.99
14474 measured reflections
3170 independent reflections
2044 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.1°
$h = -9 \rightarrow 10$
$k = -11 \rightarrow 13$
$l = -35 \rightarrow 36$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.215
S = 1.02
3170 reflections
272 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1393P)^2 + 0.1882P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

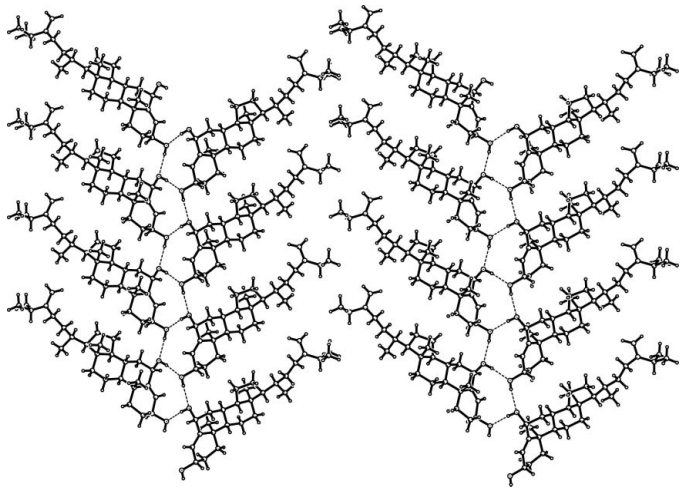


Figure 2
The extended ribbons formed by hydrogen bonds (dashed lines).

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	2.05	2.841 (4)	162
O2—H2···O1^{ii}	0.82	1.94	2.749 (4)	170

Symmetry codes: (i) $x+1, y, z$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+2$.

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. H atoms were positioned geometrically [C—H = 0.98 Å for CH, 0.97 Å for CH_2, 0.96 Å for CH_3 and 0.93 Å for CH(olefinic), and O—H = 0.82 Å], and refined using a riding model, with $U_{iso} = 1.2U_{eq}$(parent) ($1.5U_{eq}$ for hydroxyl groups).

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

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