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5α,8α-Epidioxyergosta-6,22-dien-3β-ol (ergosterol peroxide) methanol solvate

Jian-Feng Wang,[a]* Yao-Jian Huang,[b] Mei-Juan Fang,[a] Wen-Ling Xie,[b] Wen-Jin Su[b] and Yu-Fen Zhao[a]

[a]The Key Laboratory for Chemical Biology of Fujian Province, Xiamen University, Xiamen 361005, China, and [b]Department of Biology, Xiamen University, Xiamen 361005, China

Correspondence e-mail: jfwang@yanan.xmu.edu.cn

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.009 Å
R factor = 0.068
wR factor = 0.170
Data-to-parameter ratio = 8.6

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

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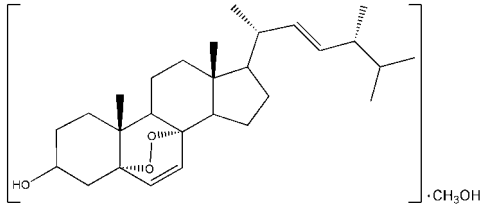
The title compound was isolated from Tubercularia *sp*., an endophytic fungus of *Taxus mairei*, and crystallizes as a methanol solvate, $C_{28}H_{44}O_3\cdot CH_3OH$. The crystal structure shows that the OH group complexes with the methanol solvent molecule *via* intermolecular hydrogen bonds, and that the peroxy unit has an O—O bond length of 1.482 (5) Å and a C—O—O—C torsion angle of −6.0 (6)°.

Comment

5α,8α-Epidioxyergosta-6,22-dien-3β-ol (ergosterol peroxide), was isolated from Tubercularia *sp*., an endophytic fungus with antitumor activity (Wang *et al.*, 2000). It is found that ergosterol peroxide has various important biological activities, including immunosuppressive (Fujimoto *et al.*, 1994), anti-inflammatory (Yasukawa *et al.*, 1996), antiviral (Lindequist *et al.*, 1989; Nakanishi *et al.*, 1998), antiplasmodial (Kuria *et al.*, 2002) and antitumor (Bok *et al.*, 1999; Nam *et al.*, 2001) activities.



(I)

The title compound crystallizes as a methanol solvate, (I) (Fig. 1), and the OH group complexes with the methanol solvent molecule *via* intermolecular hydrogen bonds, with an O1*S*···O1($-x+1$, $y-\frac{1}{2}$, $-z+2$) distance of 2.666 (9) Å and an O1···O1*S*(x, $y+1$, z) distance of 2.695 (12) Å. The structure of (I) is similar to that of 5α,8α-epidioxyergosta-6,22-

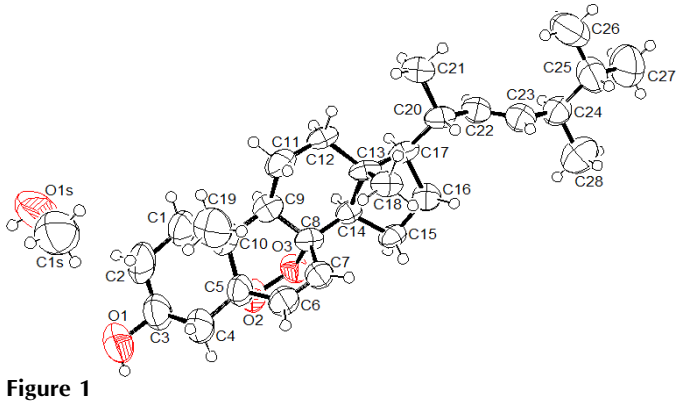


Figure 1
ORTEP-3 (Farrugia, 1997) plot of (I) at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

dien-3β-acetate, (II) (Kutshabsky *et al.*, 1990). The two structures differ in that (I) contains a hydroxy group instead of an acetate group. The peroxy distance [O—O = 1.482 (5) Å] in (I) is close to that in (II) [O—O = 1.48 (1) Å]. The torsion angle [C—O—O—C = −6.0 (6)°] of the peroxy unit in (I) is very similar to that in (II) [C—O—O—C = −5.6 (7)°].

Experimental

The title compound was isolated from an endophytic fungus, Tubercularia *sp*., which was found in the inner bark of *Taxus mairei* of Fujian Province, China. Crystals were grown from methanol. The molecular formula of the title compound was deduced from the high resolution EI–MS spectrum as $C_{28}H_{44}O_3$, showing an accurate mass of *m*/*z* 428.3304 (M^+, Δ − 1.4 mmu).

Crystal data

$C_{28}H_{44}O_3\cdot CH_4O$
M_r = 460.67
Monoclinic, $P2_1$
a = 10.022 (5) Å
b = 7.373 (5) Å
c = 19.156 (5) Å
β = 91.556 (5)°
V = 1415.0 (12) Å^3
Z = 2
D_x = 1.081 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 684 reflections
θ = 2.9–24.4°
μ = 0.07 mm^{-1}
T = 298 (2) K
Needle, colorless
0.43 × 0.18 × 0.11 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.909, T_{max} = 0.992
7222 measured reflections
2692 independent reflections
1468 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 25.0°
$h = -11 \rightarrow 10$
$k = -8 \rightarrow 8$
$l = -14 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.170
S = 1.09
2692 reflections
312 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.013
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1*S*—H1*S*···O1^{i}	0.82	1.89	2.666 (9)	158
O1—H1···O1*S*^{ii}	0.79 (8)	1.94 (8)	2.695 (12)	160 (9)

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

The H atom on atom O1 was located in a difference Fourier synthesis and refined freely. All other H atoms were positioned geometrically (C—H = 0.93, 0.96, 0.97 and 0.98 Å, and O—H = 0.82 Å) and were included in the refinement in the riding-model approximation. The displacement parameters of H atoms were set to $1.2U_{eq}$ of their parent atoms. The absolute configuration was taken as that of 5α,8α-epidioxyergosta-6,22-dien-3β-acetate (Kutshabsky *et al.*, 1990). In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

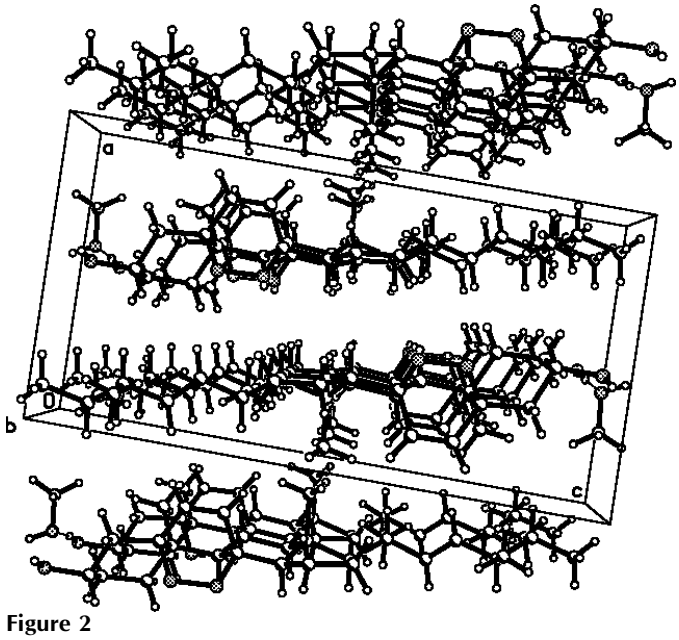


Figure 2
Packing diagram (*ORTEP*II; Johnson, 1976) of (I).

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