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(3*S**,16*S**,20*R**,22*R**,24*S**)-3,22,25-Trihydroxy-16,24:20,24-diepoxycholest-5-en-3-yl acetate methanol solvate

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The title compound, $C_{29}H_{44}O_6{\cdot}CH_4O$, namely suberoretisteroid A methanol solvate, is a fused hexacyclic steroid which was isolated from the South China Sea gorgonian *Suberogorgia reticulata*. There are two independent molecules in the asymmetric unit. Suberoretisteroid A molecules and methanol solvent molecules are linked *via* intermolecular O—H···O hydrogen bonds to form two independent two-dimensional frameworks.

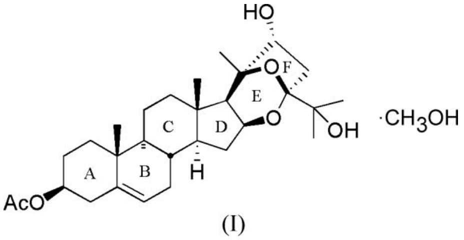
Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.013 Å
R factor = 0.071
wR factor = 0.173
Data-to-parameter ratio = 8.2

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

Comment

In the course of searching for bioactive substances from marine organisms, we have chemically investigated the gorgonian *Suberogorgia reticulata*, collected from the South China Sea. The discovery of suberoretisteroid A methanol solvate, (I), from the diethyl ether extract of *S. reticulata* (Zhang *et al.*, 2005) leads to the structural revision of some steroids previously found in the Indian gorgonian *Gorgonella umbraculum* (Subrahmanyam & Kuamr, 2000; Anjaneyulu *et al.*, 2003). The crystal structure of (I) further confirms the structure established by spectroscopic methods, *i.e.* NMR, IR and MS.



(I)

Figs. 1 and 2 show the two independent suberoretisteroid A molecules. In both, the molecular skeleton contains a fused hexacyclic system, involving the rare 24-ketal function. Rings *A*, *C* and *E* are in chair conformations, while ring *B* adopts a half-chair conformation. The five-membered rings *D* and *F* both have envelope conformations. The *trans* linkage between

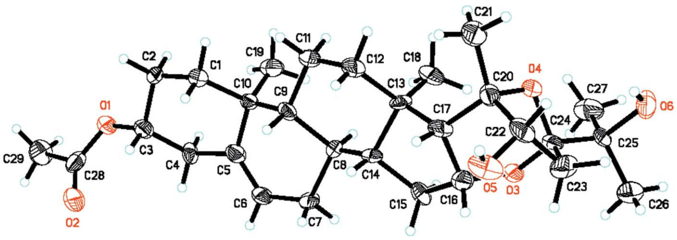


Figure 1
View of the first independent molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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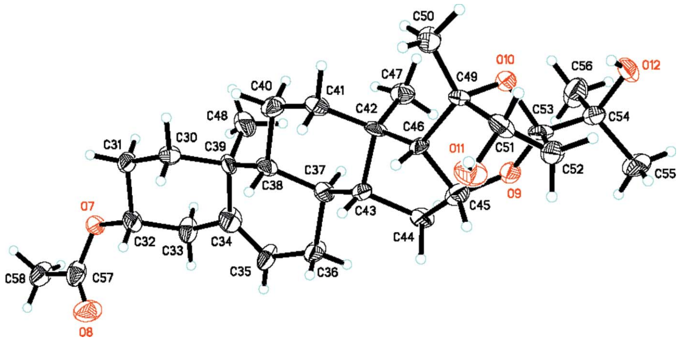

Figure 2
View of the second independent molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

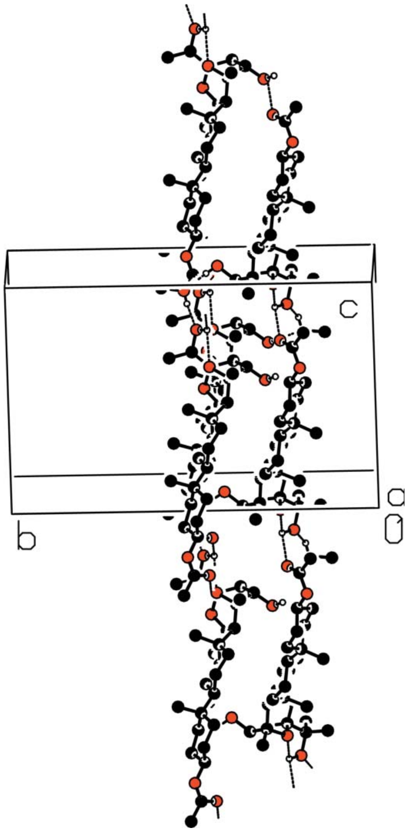

Figure 3
Partial packing diagram (Spek, 2003) of (I). Dahed lines indicate hydrogen bonds.

A/*B*/*C*/*D* is in agreement with that in natural steroids. In addition, there are *cis* linkages between rings *D*/*E* and *E*/F.

Suberoretisteroid molecules and methanol solvent molecules are linked *via* intermolecular O—H···O hydrogen bonds to form two independent two-dimensional frameworks (Table 1 and Fig. 3).

Experimental

The gorgonian *S. reticulata* was collected along the coast of Xiaodong Hai, Hainan Province, China, in December 2001, at a depth of 20 m and identified as *S. reticulata*. A voucher specimen is available for inspection at the Shanghai Institute of Materia Medica, Institutes for Biological Sciences, Chinese Academy of Sciences. The frozen animals (dry weight 460.7 g) were cut into small pieces and then extracted with acetone at room temperature. The crude extract of *S. reticulata* was partitioned between Et_2O and H_2O after filtration. The Et_2O extract was evaporated yielding the dark-green residue (4.7 g) which was fractionated by silica-gel column chromatography (light petroleum ether/acetone gradient) followed by repeated column chromatography on Sephadex-LH-20, and normal- and reversed-phase silica gel to afford suberoretisteroid A, (I) (11.9 mg). The title compound was recrystallized from MeOH.

Crystal data

$C_{29}H_{44}O_6 \cdot CH_4O$
$M_r = 520.68$
Monoclinic, $P2_1$
$a = 8.6596$ (17) Å
$b = 22.247$ (4) Å
$c = 15.891$ (3) Å
$\beta = 105.314$ (3)°
$V = 2952.7$ (10) Å^3
$Z = 4$
$D_x = 1.171$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 946 reflections
$\theta = 4.9–33.2°$
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.42 × 0.21 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.738$, $T_{\max} = 0.980$
15224 measured reflections
5608 independent reflections
2037 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.150$
$\theta_{\max} = 25.5°$
$h = -10 \rightarrow 10$
$k = -26 \rightarrow 26$
$l = -19 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.173$
$S = 0.77$
5608 reflections
688 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.090$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O13	0.82	1.92	2.742 (11)	178
O6—H6···O2^{i}	0.82	2.25	2.752 (10)	120
O12—H12···O8^{ii}	0.82	2.42	2.780 (10)	108
O11—H11···O14^{ii}	0.82	2.00	2.755 (10)	153
O12—H12···O10	0.82	2.47	2.776 (8)	104
O13—H13···O12^{iii}	0.82	2.19	2.708 (9)	121
O14—H14*A*···O6^{iv}	0.82	2.03	2.673 (9)	135

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

All H atoms were located in a difference Fourier map, but were introduced in calculated positions and treated as riding on their parent atoms [C—H = 0.93–0.98 Å and O—H = 0.82 Å; $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C), $U_{\rm iso}$(H) = $1.5U_{\rm eq}$($C_{\rm methyl}$) and $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(O). In the absence of significant anomalous dispersion effects, Friedel pairs were merged. The absolute configuration is unknown and was randomly chosen for this determination. The higher than normal $R_{\rm int}$ value of 0.15 can be attributed to the small amount of observed data (*ca* 36%). This can lower the precision of the structure determination.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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References

Anjaneyulu, A. S. R., Rao, V. L. & Sastry, V. G. (2003). *Nat. Prod. Res.* **17**, 149–152.

Bruker (2000). *SMART* (Version 5.6), *SAINT* (Version 6.0) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.

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

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Subrahmanyam, C. & Kuamr, S. R. (2000). *J. Chem. Res. (S)*, pp. 182–183.

Zhang, W., Guo, Y.-W., Gavagnin, M., Mollo, E. & Cimino, G. (2005). *Helv. Chim. Acta*, **88**, 87–94.