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(25*R*)-Spirost-5-en-3-ol dimethyl sulfoxide solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.063
wR factor = 0.199
Data-to-parameter ratio = 15.9

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

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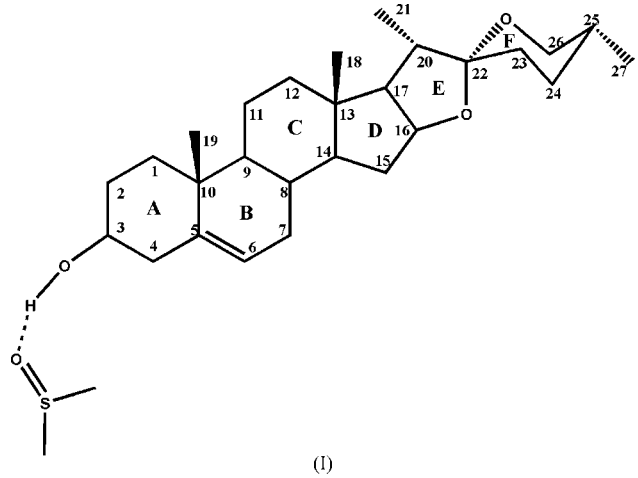
In the crystal structure of the title compound, $C_{27}H_{42}O_3\cdot C_2H_6OS$, intermolecular O—H···O hydrogen bonds link the hydroxyl groups with the dimethyl sulfoxide solvent molecules.

Comment

Diosgenin is a very important class of steroidal saponin with a C_{27} skeleton. The compound has attracted scientific attention because of its significant biological activities, such as iimmunostimulant (Moreira *et al.*, 2001), protein binding (Lee *et al.*, 2003), gastroprotective (Matsuda *et al.*, 2003), antibacterial (Quan *et al.*, 2002), *etc*. X-ray powder diffraction data for (25*R*)-spirost-5-en-3-ol have been reported (Poveda & Henao, 2005), but its crystal structure has, to our knowledge, not been determined so far. An unambiguous crystal structure of (25*R*)-spirost-5-en-3-ol dimethyl sulfoxide solvate, (I), is reported here for the first time.



(I)

Compound (I) possesses *B*/*C trans*, *C*/*D trans* and *D*/*E cis* ring-fusion geometry and an *R* configuration at spiro atom C22. The 21-methyl group is α-oriented (20*S*) and the 19-methyl group is β-oriented. Ring *F* is in a chair conformation, as shown in Fig. 1. The molecule has a 25*R* configuration with an equatorial 27α-methyl group. The crucial structural feature is an O—H···O hydrogen bond between the hydroxyl group and the dimethyl sulfoxide solvent molecule (Table 1). The hydroxyl group at C3 acts as a donor for the hydrogen bond and the sulfone group in dimethyl sulfoxide as an acceptor; each compound is thus bonded to one dimethyl sulfoxide molecule *via* a hydrogen bond, yielding a stable crystal structure. There is a close similarity in crystal structure between (I) and that of diosgenyl 3,4,6-tri-*O*-acetyl-2-dexoy-2-

tetrachlorophthalimido-β-D-gulcopyranoside (Myszka *et al.*, 2002), the difference being that related to the hydroxyl group at C3. Structurally, it is the hydroxyl group at C3 that plays an important role in the formation of diverse compounds of steroidal saponins.

Experimental

Paris Polyphylla Smith was collected from HuBei and Hunan provinces of China in May 2004. The rhizomes of the plant (3.5 kg) were extracted with 95% ethanol to yield a dark-brown extract (720 g). Fractionation of the CH_3Cl fraction (32 g) using chromatograghy (silica gel) eluting with *n*-hexane–EtOAc of increasing polarity afforded the title compound (2.3 g) as a white powder. Crystals were grown from mixed solvents of dimethyl sulfoxide and ethanol at 277 K. The clear solutions were slowly evaporated to give needle-like colourless crystals of good quality (m.p. 480–482 K). IR (KBr, cm^{-1}): 3452, 2952, 1668, 1377, 1242, 1173, 1053, 981, 920, 899, 866, 810; ^{1}C NMR ($CDCl_3$): δ 37.19, 31.58, 71.67, 42.23, 140.77, 121.39, 31.81, 31.40, 50.01, 36.61, 20.84, 39.75, 40.23, 56.48, 32.02, 80.79, 62.04, 16.27, 19.40, 41.57, 14.51, 109.27, 31.34, 28.76, 30.26, 66.81, 17.11.

Crystal data

$C_{27}H_{42}O_3{\cdot}C_2H_6OS$
M_r = 492.73
Orthorhombic, $P2_12_12_1$
a = 7.4688 (15) Å
b = 9.6157 (19) Å
c = 39.192 (8) Å
V = 2814.7 (10) Å^3
Z = 4
D_x = 1.163 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15087 reflections
θ = 2.1–25.1°
μ = 0.15 mm^{-1}
T = 293 (2) K
Needle, colourless
0.55 × 0.12 × 0.08 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.923, T_{max} = 0.988
15087 measured reflections
4891 independent reflections
2574 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 25.1°
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -45 \rightarrow 45$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.199
S = 1.05
4891 reflections
308 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.094P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Absolute structure: Flack (1983) 1998 Friedel pairs
Flack parameter: 0.1 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—HO3···O4	0.82	1.94	2.741 (7)	164

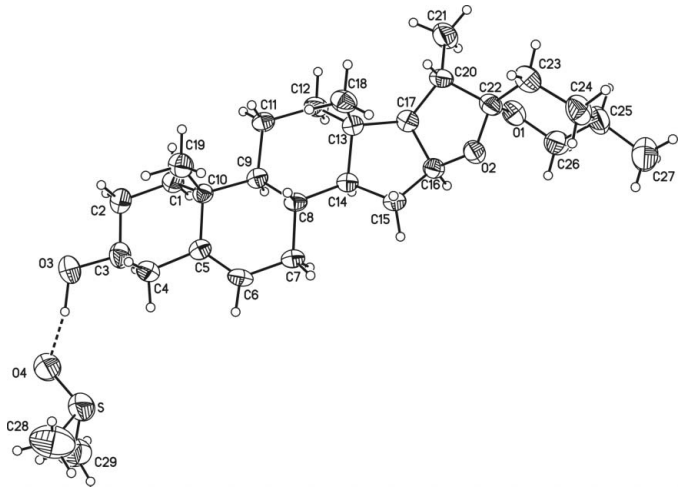


Figure 1
View of the asymmetric unit (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

All H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined as riding. For the CH and CH_2 groups, U_{iso}(H) values were set equal to $1.2U_{eq}$(carrier atom) and for the methyl groups they were set equal to $1.5U_{eq}$(carrier atom).

Data collection: *RAPID-AUTO* (Rigaku, 2000); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1990); software used to prepare material for publication: *SHELXL97*.

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