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## Structure of 3 $\beta$ -(D-Galactopyranosyl)pregna-5,20-diene–Dimethyl Sulfoxide Solvate

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**Abstract.** C<sub>27</sub>H<sub>42</sub>O<sub>6</sub>·C<sub>2</sub>H<sub>6</sub>OS,  $M_r = 540.8$ , orthorhombic,  $P2_12_12_1$ ,  $a = 9.011(3)$ ,  $b = 38.68(8)$ ,  $c = 8.338(7)$  Å,  $V = 2906.2$  Å<sup>3</sup>,  $D_x = 1.236$  g cm<sup>-3</sup> at 138 K,  $Z = 4$ ,  $F(000) = 1176$ ,  $\mu(\text{Cu K}\alpha) = 12.5$  cm<sup>-1</sup>,  $\lambda(\text{Cu K}\alpha) = 1.5418$  Å. Structure was determined from 3436 reflections at 138±2K and refined to a final  $R = 0.074$  for 2330 observed reflections [ $I > 2\sigma(I)$ ]. The conformation of the steroid nucleus is quite typical for a  $\Delta^5$ -steroid. Rings *A* and *C* are in a chair conformation, whereas the unsaturated ring *B* assumes an 8 $\beta$ ,9 $\alpha$  half-chair conformation. The five-membered ring *D* has a half-chair conformation and the orientation of the 17 $\beta$  side chain is consistent with other related pregnene structures. The galactopyranoside ring has the normal <sup>4</sup>C<sub>1</sub> chair conformation. There is a  $\beta$ -linkage between the galactose and the steroid nucleus.

**Introduction.** Marine invertebrates have been shown to be rich sources of many natural products including terpenes and steroids. Although a great variety of steroid derivatives have been extracted from various marine organisms (Tursch, Hootele, Kaisin, Losman & Karlsson, 1976; Moldowan, Tan & Djerassi, 1975; Ballantine, Williams & Burke, 1977; Fenical, 1979; Weinheimer, Chang & Matson, 1979), the steroidal glycosides from the marine organisms are relatively rare (Bandurraga & Fenical, 1985). The title compound was isolated from the gorgonian *Pseudoplexaura wagenari* (collected from Key Biscayne, Florida) and its isolation and identification have been

reported in an earlier communication (Wasylyk, Alam, Martin, Weinheimer, Hossain & van der Helm, 1987). The crystal structure determination of the compound was carried out to confirm the chemical structure and to investigate the stereochemical features of a steroidal glycoside, in particular to ascertain the nature of the linkage ( $\alpha$  or  $\beta$ ) of the galactose to the steroid nucleus.

**Experimental.** Crystals grown from Me<sub>2</sub>SO showed large mosaic spread ( $\sim 1.8^\circ$ ), a rectangular pillar shaped crystal, 0.60 × 0.20 × 0.18 mm, used for X-ray work; all measurements on an Enraf–Nonius CAD-4 diffractometer fitted with a liquid-N<sub>2</sub> low-temperature set up; cell parameters by least-squares fit to  $\pm 2\theta$  of 22 reflections,  $15 < \theta < 23^\circ$  measured at 138 K using Cu K $\alpha_1$  radiation, space group uniquely determined from systematic extinctions,  $h00$ ,  $h = 2n+1$ ,  $0k0$ ,  $k = 2n+1$ ,  $00l$ ,  $l = 2n+1$ ; intensities of 3436 unique reflections with  $2\theta \leq 150^\circ$  at 138±2K using Cu K $\alpha$  radiation,  $h = 0 \rightarrow 11$ ,  $k = 0 \rightarrow 48$ ,  $l = 0 \rightarrow 10$ ;  $\theta$ - $2\theta$  scan technique, scan width  $(0.90 + 0.20 \tan \theta)^\circ$ , extended 25% on each side for background measurement, horizontal aperture  $(5.0 + 0.86 \tan \theta)$  mm, three standard reflections measured every 7200 s of X-ray exposure, maximum variation, 5%; 2330 observed reflections on the basis  $I \geq 2\sigma(I)$ ; Lorentz and polarization corrections, no absorption correction; partial structure obtained from *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1976), and the full structure by difference Fourier syntheses; solvent

Table 1. Atomic positions ( $\times 10^4$ ) and  $U_{eq}$  values for non-hydrogen atoms with e.s.d.'s for last digit in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
C(1)	6400 (9)	7942 (2)	9270 (9)	0.022 (2)
C(2)	6206 (9)	8318 (2)	9858 (10)	0.026 (2)
C(3)	6426 (8)	8561 (2)	8463 (9)	0.022 (2)
C(4)	5245 (10)	8488 (2)	7205 (11)	0.023 (3)
C(5)	5300 (8)	8122 (2)	6619 (10)	0.021 (2)
C(6)	5333 (8)	8054 (2)	5070 (10)	0.023 (2)
C(7)	5381 (9)	7702 (2)	4326 (9)	0.019 (2)
C(8)	5113 (8)	7407 (2)	5557 (9)	0.021 (2)
C(9)	5870 (8)	7489 (2)	7152 (9)	0.018 (2)
C(10)	5320 (8)	7839 (2)	7943 (9)	0.020 (2)
C(11)	5748 (10)	7180 (2)	8311 (10)	0.024 (2)
C(12)	6377 (10)	6844 (2)	7577 (10)	0.025 (2)
C(13)	5572 (8)	6759 (2)	6020 (10)	0.025 (2)
C(14)	5734 (9)	7069 (2)	4881 (10)	0.023 (2)
C(15)	5167 (10)	6933 (2)	3314 (12)	0.032 (3)
C(16)	5768 (10)	6552 (2)	3284 (11)	0.032 (3)
C(17)	6337 (9)	6479 (2)	4979 (11)	0.025 (2)
C(18)	3962 (9)	6657 (2)	6353 (11)	0.029 (3)
C(19)	3772 (8)	7803 (2)	8644 (10)	0.023 (2)
C(20)	6127 (11)	6108 (2)	5486 (12)	0.039 (3)
C(21)	7095 (13)	5927 (3)	6245 (14)	0.062 (4)
O(1)	6270 (6)	8919 (1)	8880 (6)	0.026 (2)
O(2)	8951 (7)	9288 (1)	7930 (7)	0.039 (2)
O(3)	9282 (6)	9914 (1)	9698 (7)	0.031 (2)
O(4)	6790 (5)	9868 (1)	11600 (6)	0.030 (2)
O(5)	6593 (6)	9114 (1)	11388 (6)	0.025 (1)
O(6)	7316 (8)	9467 (1)	15302 (7)	0.051 (2)
C(1')	7356 (9)	9052 (2)	9911 (10)	0.027 (2)
C(2')	7980 (9)	9384 (2)	9169 (10)	0.027 (3)
C(3')	8867 (8)	9591 (2)	10416 (10)	0.026 (2)
C(4')	7989 (9)	9642 (2)	11913 (10)	0.025 (2)
C(5')	7509 (10)	9289 (2)	12547 (11)	0.029 (3)
C(6')	6528 (12)	9306 (2)	14047 (11)	0.036 (3)
S(1)	8486 (2)	5209.4 (5)	11255 (3)	0.0359 (7)
O(D)	7081 (6)	4995 (1)	11196 (8)	0.044 (2)
C(D)1	8431 (13)	5492 (3)	9565 (14)	0.045 (4)
C(D)2	8152 (17)	5525 (3)	12792 (14)	0.053 (5)

identified as  $\text{Me}_2\text{SO}$ ; refinement by full-matrix least squares, *SHELX76* (Sheldrick, 1976), with minimization of  $\sum w(|F_o| - |F_c|)^2$ ,  $w = 1/\sigma(F)^2$ ; all H atoms, except one, from difference Fourier map, refined isotropically (a few H atoms kept fixed in the final cycles), final  $R = 0.074$ ,  $wR = 0.071$ ,  $S = 1.8$  for 2330 observations and 457 variables,  $(\Delta/\sigma)_{\max} = 0.24$ , maximum/minimum peak height in the final difference map,  $\pm 0.5 e \text{\AA}^{-3}$ . Atomic scattering factors those of *SHELX76*.

**Discussion.** The final parameters for the non-hydrogen atoms are listed in Table 1.\* Bond distances and the atom-numbering scheme are shown in Fig. 1(a), while the endocyclic torsion angles are given in Fig. 1(b). A stereoview of a single molecule is shown in Fig. 2. Bond angles are listed in Table 2. The linkage of galactose to the steroid skeleton is  $\beta$  in nature.

The geometric parameters of the steroid nucleus are quite normal and agree with the average values of bond

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44280 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

distances and angles for other  $\Delta^5$ -steroids listed by Duax & Norton (1975). The overall steroid conformation is also consistent with the reported structures. The A and C rings are in a chair conformation with  $\langle \tau \rangle$  values of  $53.8$  and  $56.6^\circ$  respectively. The unsaturated ring B has an  $8\beta,9\alpha$ -half-chair conformation. The asymmetry parameters defined by Duax & Norton (1975) for ring B are  $\Delta C_2(5-6) = 7.4$  and  $\Delta C_5(6) = 15.6^\circ$ . These values indicate that the deviation from the perfect half-chair ( $\Delta C_2 = 0^\circ$ ) is towards a  $9\alpha$ -sofa conformation. The five-membered ring (D) is in a half-chair conformation and has a pseudo-rotation parameter (Altona, Geise & Romers, 1968)  $\Delta = 8.4^\circ$ . This observation is consistent with the conclusion of Duax, Weeks & Rohrer (1976) that the D rings in steroids containing all- $sp^3$  hybrid C atoms usually have a conformation intermediate between  $13\beta,14\alpha$  half-chair

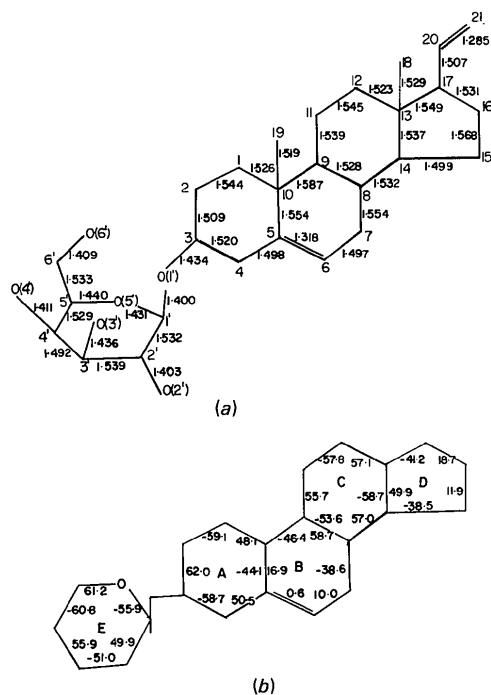


Fig. 1. (a) Atom numbering with bond lengths in Å. Estimated standard deviations range between 0.009 and 0.013 Å. (b) Endocyclic torsion angles in degrees. Estimated standard deviations range between 0.7 and 1.1°.

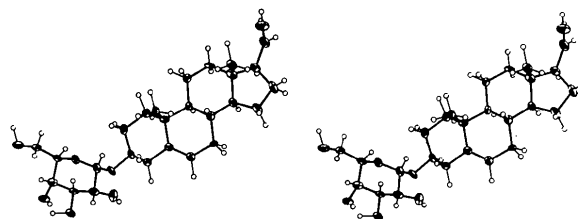


Fig. 2. Stereoview of a single molecule.

Table 2. Bond angles (°) (e.s.d.'s range between 0.6 and 0.8°)

C(2)–C(1)–C(10)	113.7	C(9)–C(11)–C(12)	112.2
C(1)–C(2)–C(3)	109.1	C(11)–C(12)–C(13)	110.2
C(2)–C(3)–C(4)	108.9	C(12)–C(13)–C(14)	108.2
C(2)–C(3)–O(1')	113.7	C(12)–C(13)–C(17)	114.7
C(4)–C(3)–O(1')	106.1	C(12)–C(13)–C(18)	110.7
C(3)–C(4)–C(5)	112.2	C(14)–C(13)–C(17)	99.0
C(4)–C(5)–C(10)	115.7	C(14)–C(13)–C(18)	113.7
C(4)–C(5)–C(6)	120.6	C(17)–C(13)–C(18)	110.1
C(6)–C(5)–C(10)	123.8	C(8)–C(14)–C(13)	113.9
C(5)–C(6)–C(7)	126.0	C(8)–C(14)–C(15)	119.7
C(6)–C(7)–C(8)	113.0	C(13)–C(14)–C(15)	103.5
C(7)–C(8)–C(9)	110.7	C(14)–C(15)–C(16)	103.0
C(7)–C(8)–C(14)	109.2	C(15)–C(16)–C(17)	106.0
C(9)–C(8)–C(14)	109.5	C(16)–C(17)–C(13)	103.8
C(8)–C(9)–C(10)	113.5	C(16)–C(17)–C(20)	113.2
C(8)–C(9)–C(11)	110.8	C(13)–C(17)–C(20)	116.9
C(10)–C(9)–C(11)	112.3	C(17)–C(20)–C(21)	124.8
C(9)–C(10)–C(1)	108.9	C(3)–O(1')–C(1')	115.8
C(1)–C(10)–C(5)	109.8	O(1')–C(1')–O(5')	104.7
C(1)–C(10)–C(19)	109.3	O(1')–C(1')–C(2')	108.4
C(5)–C(10)–C(9)	108.1	C(2')–C(1')–O(5')	112.5
C(5)–C(10)–C(19)	109.2	C(1')–C(2')–C(3')	110.8
C(9)–C(10)–C(19)	111.5	C(1')–C(2')–O(2')	107.8
C(1')–O(5')–C(5')	112.4	C(3')–C(4')–O(4')	109.5
C(3')–C(2')–O(2')	108.1	C(5')–C(4')–O(4')	113.5
C(2')–C(3')–C(4')	111.1	C(4')–C(5')–O(5')	110.5
C(2')–C(3')–O(3')	108.0	C(4')–C(5')–C(6')	114.0
C(4')–C(3')–O(3')	111.8	C(6')–C(5')–O(5')	103.7
C(3')–C(4')–C(5')	108.7	C(5')–C(6')–O(6')	109.5

( $\Delta = 0^\circ$ ) and  $13\beta$  envelope ( $\Delta = 35^\circ$ ). The present structure has one of the most uncommon  $17\beta$  side chains, an ethenyl group. Most pregnane steroids have a 20-keto group in their side chain. However, the conformation of the two-carbon chain has some common features of other pregnanes. The torsion angle C(18)–C(13)–C(17)–C(20) of  $-47.1$  ( $10^\circ$ ) falls in the range,  $-44.7$  to  $-51.0^\circ$ , observed in 15 other pregnane structures with  $17\alpha$ -hydrogen substitution (Duax & Norton, 1975). It is interesting to note that the C(20) hydrogen takes up the position [C(16)–C(17)–C(20)–H(20) =  $-25(5)^\circ$ ] analogous to that occupied by the C(20) carbonyl in nearly 100 pregnane derivatives (Duax, Griffin & Rohrer, 1981), where the torsion angle C(16)–C(17)–C(20)–O(20) ranges between 0 and  $-46^\circ$ .

The galactopyranose ring is in the normal chair conformation. The conformation about C(5')–C(6') is *trans-gauche* with torsion angle O(5')–C(5')–C(6')–O(6') of  $178.7$  ( $6^\circ$ ). A similar conformation has been observed in some of the substituted glycosides, like 2,3,4,6-tetra-*O*-acetyl-1-cyano- $\beta$ -D-galactopyranose (torsion angle  $178.4^\circ$ ) (Foces-Foces, Cano & Garcia-Blanco, 1976), methyl 6-*O*-acetyl- $\beta$ -D-galactopyranoside (torsion angle  $172.3^\circ$ ) (Lindberg, 1976) and methyl 7-deoxy-D-glycero- $\beta$ -D-galacto-heptopyranoside (torsion angle  $-175.2^\circ$ ) (Avenel, Ohanessian, Gillier-Pandraud & Boullanger, 1982), while in the unsubstituted sugars the conformation is *gauche-trans*, as in methyl  $\beta$ -D-galactopyranoside (torsion angle  $63.9^\circ$ ) (Takagi & Jeffrey, 1978). The torsion angle of the glycosidic bond C(1')–O(1') [C(3)–O(1')–C(1')–O(5')] of  $-110.7$  ( $6^\circ$ ) is significantly different from those observed in other galactose structures,  $-74.1^\circ$  in 7-

deoxy-D-glycero- $\beta$ -D-galacto-heptopyranoside,  $-77.6^\circ$  in methyl  $\beta$ -D-galactopyranoside and  $-82.3^\circ$  in methyl 6-*O*-acetyl-D-galactopyranoside. Such a conformation is possibly needed to accommodate the bulky steroid. The galactose structure gives four equatorial and only one [at C(4')] axial substituents. The angle O(5')–C(1')–O(1') of  $104.7$  ( $6^\circ$ ) deviates significantly from the tetrahedral value and is much smaller than the average ( $107.4^\circ$ ) observed in the three methyl galactopyranoside structures referred to above. Beside this, the overall effect on the geometry of the galactose moiety of the steroidal substitution appears to be negligible. The present structure determination shows that the combination of a steroid skeleton and a glycoside has little or no influence on each other's overall stereochemical features.

The Me<sub>2</sub>SO molecule has a trigonal pyramidal geometry with S(1)–O(D) =  $1.514$  (6), S(1)–C(D)1 =  $1.784$  (12), S(1)–C(D)2 =  $1.796$  (12) Å, and angles C(D)1–S(1)–C(D)2 =  $98.2$  (5), C(D)1–S(1)–O(D) =  $106.6$  (5) and C(D)2–S(1)–O(D) =  $104.8$  (5)°. The two S–C distances are nearly equal, in agreement with the Me<sub>2</sub>SO structure at 213 K (Viswamitra & Kannan, 1966). The two S–C distances differ significantly at 278 K (Thomas, Shoemaker & Eriks, 1966) and at higher temperature (Dickinson & Holden, 1978).

The crystal structure of the compound reflects some of the common features of steroidal packing. The molecules are packed two thick (along the *a* axis) and two long (along the *b* axis) in a bilayer crystal structure. The layers are approximately perpendicular to the steroid thickness. Fig. 3 gives a view of the packing. The individual layers have a checkerboard pattern of

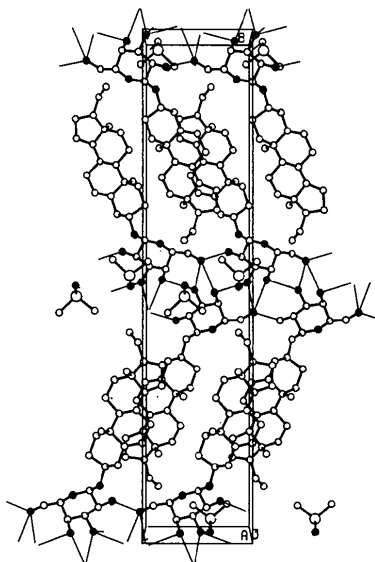


Fig. 3. A view of the packing. Dark circles are oxygen atoms. Hydrogen bonds are indicated by thin lines.

head-to-head and tail-to-tail approaches. The sugar ends of the molecules are extensively linked through hydrogen bonding, while the hydrophobic alkene ends are held together by weak van der Waals interactions. There are four symmetry-independent hydrogen bonds in the structure:  $O(2')-H\cdots O(6')$  ( $x, y, z-1$ ) = 2.729 (10),  $O(3')-H\cdots O(6')$  ( $1.5-x, 2-y, -0.5+z$ ) = 2.839 (10),  $O(4')-H\cdots O(6')$  ( $1.5-x, 2-y, -0.5+z$ ) = 2.908 (10) and  $O(6')-H\cdots O(2')$  ( $x, y, z+1$ ) = 2.729 (10) Å, all of which involve atom O(6'). Besides, there are two short non-bonding O...O contacts:  $O(3')\cdots O(4')$  ( $1.5-x, 2-y, -0.5+z$ ) = 2.884 (10) and  $O(3')\cdots O(D)$  ( $0.5+x, 1.5-y, 2-z$ ) = 2.653 (9) Å. There is no hydrogen bonding between the layers and there is very little overlap of molecules in adjacent layers. Lack of interlayer hydrogen bonding and the presence of hydrophobic regions in the crystal structure may very well explain the rather large crystal mosaic.

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## Structure of 4-Isopropyl-1,6-dimethyl-7-phenylbicyclo[3.2.1]oct-6-en-8-one (Two Crystalline Forms)

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**Abstract.**  $C_{19}H_{24}O$ ,  $M_r = 268.4$ , monoclinic (form I),  $P2_1$ ,  $a = 13.853$  (8),  $b = 7.893$  (5),  $c = 14.855$  (6) Å,  $\beta = 95.91$  (3)°,  $V = 1615.4$  (6.2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.10$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 0.48$  mm<sup>-1</sup>,  $F(000) = 584$ ,  $T = 293$  K. Final  $R = 0.091$  for 1546 observed reflections.  $C_{19}H_{24}O$ ,  $M_r = 268.4$ , orthorhombic (form II),  $Pna2_1$ ,  $a = 17.565$  (18),  $b =$

$11.707$  (14),  $c = 7.806$  (13) Å,  $V = 1605.2$  (6.2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.11$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 0.48$  mm<sup>-1</sup>,  $F(000) = 584$ ,  $T = 293$  K. Final  $R = 0.051$  for 890 observed reflections. The regiochemistry is established of the cyclopentene double bond with respect to the cyclohexane ring and the isopropyl group in the *exo* configuration with respect to the bicyclo[3.2.1] framework. The six-membered ring has a chair conformation. The five-membered ring exists in an envelope conformation with an angle of pucker in the range 42.5 (1.0)–42.9 (1.0)°.

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