Table 3. *Possible hydrogen-bond geometry* (Å, °)

X—H$\cdots Y$	X—H	H$\cdots Y$	$X\cdots Y$	X—H$\cdots Y$	Molecule
O(2*A*)—H(O2*A*)$\cdots$O(3*A*)	0.884 (5)	1.950 (5)	2.573 (5)	126.1 (5)	x, y, z
O(4*B*)—H(O4*B*)$\cdots$O(2*A*)	0.99 (1)	1.73 (1)	2.721 (6)	174.6 (5)	x, y, z
O(3*A*)—H(O3*A*)$\cdots$O(4*B*)	0.816 (5)	1.835 (5)	2.649 (5)	174.5 (6)	$2-x, -\frac{1}{2}+y, -z$
O(2*B*)—H(O2*B*)$\cdots$O(4*A*)	0.984 (7)	1.901 (7)	2.844 (5)	159.7 (5)	$2-x, \frac{1}{2}+y, 1-z$
O(3*B*)—H(O3*B*)$\cdots$O(1*A*)	0.916 (8)	1.936 (6)	2.837 (4)	167.8 (6)	$x, 1+y, z$

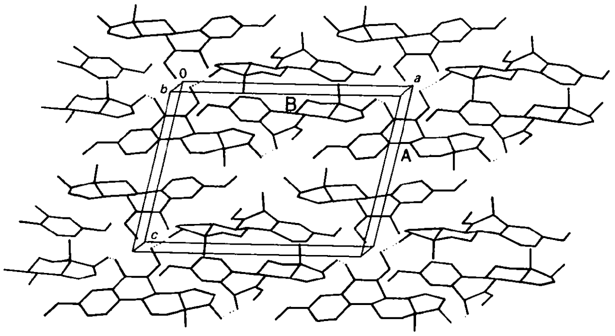

Fig. 3. The packing arrangement viewed down the *b* axis. Intramolecular hydrogen bonds are indicated by dotted lines.

the rather short intramolecular O(2)—H(O2)$\cdots$O(3) hydrogen bond in molecule *A* only (Table 3), forming the six-membered pseudo-ring. The nonbonded distances between O atoms of molecules *A* and *B* are also different: the most important distance for biological activity O(1)$\cdots$O(4) is 10.888 (4) Å in molecule *A* and 10.972 (5) Å in molecule *B*.

The packing arrangement viewed down the *b* axis is presented in Fig. 3. Two independent molecules linked by the O(4*B*)—H(O4*B*)$\cdots$O(2*A*) hydrogen bond lie along the *a* axis. The 6α-hydroxy group of molecule *A* acts simultaneously as a donor of an intramolecular hydrogen bond to O(3). These pairs are then linked to another pair of molecules by short van der Waals contacts* forming columns. The columns are linked by three other hydrogen bonds (Table 3) between the molecules from different asymmetric units.

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* See deposition footnote.

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Structure of a Steriod Fungal Metabolite

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Abstract. 3α,7α,14α-Trihydroxypregn-16-en-20-one, $C_{21}H_{32}O_4$, $M_r = 348.48$, orthorhombic, $P2_12_12_1$, $a = 9.211$ (1), $b = 13.201$ (1), $c = 16.031$ (1) Å, $V = 1949.28$ (29) Å^3, $Z = 4$, $D_x = 1.187$ g cm^{-3}, λ(Cu $K\alpha$), = 1.5418 Å, $\mu = 6.07$ cm^{-1}, $F(000) = 760$, $T = 293$ K, $R = 0.061$ for 1337 observations. The *A*, *B* and *C* rings adopt normal chair conformations with the *D* ring in a 14α-envelope conformation. The molecules are held together by two hydrogen bonds [O(3)$\cdots$O(20) = 2.879 and O(7)$\cdots$O(14) = 2.612 Å].

Introduction. The microbial transformation of steroids has been used extensively for the introduction of functional groups in order to obtain biologically useful substances. Fermentation of pregna-4,16-dien-3,20-dione with the fungus *Mucor piriformis* yielded

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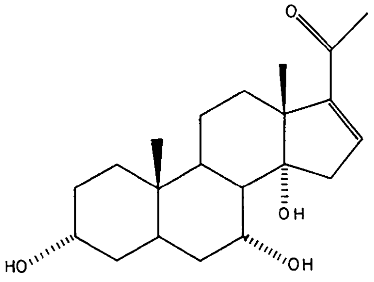
the metabolites 7α,14α-dihydroxypregna-4,16-dien-3,20-dione and 3α,7α,14α-trihydroxy-16-en-20-one (I). We report the X-ray crystallographic analysis of (I) in this paper.



(I)

Experimental. Plate-like single crystals of the title compound were obtained from ethanol. Crystal size approximately 0.4 × 0.2 × 0.15 mm. Lattice parameters refined using 25 reflections in θ range 10–15°. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation; $\theta \le 60°$; scan speed 1° min^{-1}; $\omega/2\theta$ mode. 3 standard reflections (253, 236, 234) monitored after every 3600 s showed no significant intensity variation. 1638 unique reflections collected ($0 \le h \le 10$, $0 \le k \le 14$, $0 \le l \le 18$), 1337 significant [$|F_o| \ge 3\sigma(|F_o|)$]. Data not corrected for absorption or extinction. Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1986). The best E map gave all non-H atoms. Full-matrix least-squares program *SHELX*76 (Sheldrick, 1976) used for refinement. All H atoms except H(O7) were located in difference Fourier map. The positional parameters of all the other H atoms except H(11*A*) were refined and the thermal parameters held fixed during subsequent refinement. No crystallographic assignment of chirality was made, but the reported structure exhibits the standard stereochemistry of the steriod skeleton. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, $w = 1.0/[\sigma(|F_o|)^2 + 0.002267|F_o|^2]$. At the end of the final cycle of refinement using F's, $R = 0.061$, $wR = 0.064$, $S = 1.06$, $\Delta/\sigma = 0.001$ for 318 refinable parameters. Final difference map was featureless ($\Delta\rho \le 0.1$ e Å^{-3}). Atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. Final atomic parameters are given in Table 1,* bond distances and angles are given in Table 2 and a *PLUTO* (Motherwell & Clegg, 1978)

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2 × 10^4) *for non*-H *atoms with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	0.0324 (7)	0.1399 (4)	0.0452 (4)	552 (18)
C(2)	0.1391 (8)	0.2134 (4)	0.0846 (4)	669 (22)
C(3)	0.2722 (7)	0.1609 (5)	0.1199 (4)	712 (22)
C(4)	0.3429 (7)	0.0948 (5)	0.0535 (4)	649 (21)
C(5)	0.2349 (6)	0.0203 (4)	0.0149 (3)	501 (18)
C(6)	0.3055 (6)	−0.0513 (4)	−0.0459 (4)	554 (19)
C(7)	0.2013 (6)	−0.1344 (4)	−0.0727 (3)	493 (17)
C(8)	0.0588 (6)	−0.0910 (4)	−0.1072 (3)	445 (16)
C(9)	−0.0078 (5)	−0.0093 (4)	−0.0494 (3)	451 (18)
C(10)	0.1006 (6)	0.0746 (4)	−0.0239 (3)	501 (20)
C(11)	−0.1530 (7)	0.0294 (4)	−0.0866 (4)	635 (20)
C(12)	−0.2604 (7)	−0.0529 (4)	−0.1119 (4)	617 (22)
C(13)	−0.1914 (6)	−0.1299 (4)	−0.1685 (3)	513 (18)
C(14)	−0.0538 (6)	−0.1710 (4)	−0.1238 (3)	446 (16)
C(15)	−0.0175 (6)	−0.2673 (4)	−0.1726 (4)	594 (20)
C(16)	−0.1698 (8)	−0.3050 (5)	−0.1925 (4)	625 (21)
C(17)	−0.2657 (6)	−0.2306 (5)	−0.1869 (3)	561 (21)
C(18)	−0.1526 (8)	−0.0813 (5)	−0.2539 (4)	714 (23)
C(19)	0.1425 (8)	0.1399 (4)	−0.0977 (4)	677 (22)
C(20)	−0.4213 (7)	−0.2421 (6)	−0.2031 (4)	714 (26)
C(21)	−0.4863 (9)	−0.3453 (6)	−0.2069 (5)	952 (28)
O(3)	0.2387 (5)	0.0960 (4)	0.1890 (3)	810 (18)
O(7)	0.1628 (4)	−0.1952 (3)	−0.0015 (2)	572 (13)
O(14)	−0.1026 (4)	−0.2075 (3)	−0.0433 (2)	477 (12)
O(20)	−0.4967 (6)	−0.1673 (4)	−0.2137 (3)	925 (20)

Table 2. *Bond distances* (Å) *and angles* (°) *involving non*-H *atoms of the title molecule with e.s.d.'s in parentheses*

C(1)—C(2)	1.518 (8)	C(9)—C(11)	1.550 (7)
C(1)—C(10)	1.537 (7)	C(10)—C(19)	1.513 (7)
C(2)—C(3)	1.517 (9)	C(11)—C(12)	1.524 (8)
C(3)—C(4)	1.522 (9)	C(12)—C(13)	1.503 (7)
C(3)—O(3)	1.434 (8)	C(13)—C(14)	1.553 (7)
C(4)—C(5)	1.529 (8)	C(13)—C(17)	1.524 (8)
C(5)—C(6)	1.505 (7)	C(13)—C(18)	1.553 (8)
C(5)—C(10)	1.559 (7)	C(14)—C(15)	1.529 (7)
C(6)—C(7)	1.519 (7)	C(14)—O(14)	1.449 (6)
C(7)—C(8)	1.535 (7)	C(15)—C(16)	1.522 (9)
C(7)—O(7)	1.439 (6)	C(16)—C(17)	1.324 (9)
C(8)—C(9)	1.548 (7)	C(17)—C(20)	1.464 (8)
C(8)—C(14)	1.503 (7)	C(20)—C(21)	1.489 (11)
C(9)—C(10)	1.546 (7)	C(20)—O(20)	1.219 (9)
C(2)—C(1)—C(10)	113.1 (5)	C(1)—C(10)—C(19)	110.3 (4)
C(1)—C(2)—C(3)	112.7 (4)	C(9)—C(11)—C(12)	115.2 (4)
C(2)—C(3)—O(3)	112.7 (5)	C(11)—C(12)—C(13)	111.6 (4)
C(2)—C(3)—C(4)	110.2 (5)	C(12)—C(13)—C(18)	110.4 (4)
C(4)—C(3)—O(3)	106.8 (5)	C(12)—C(13)—C(17)	121.1 (4)
C(3)—C(4)—C(5)	111.9 (5)	C(12)—C(13)—C(14)	107.6 (4)
C(4)—C(5)—C(10)	112.4 (4)	C(17)—C(13)—C(18)	107.0 (4)
C(4)—C(5)—C(6)	112.6 (4)	C(14)—C(13)—C(18)	111.2 (4)
C(6)—C(5)—C(10)	111.9 (4)	C(14)—C(13)—C(17)	98.6 (4)
C(5)—C(6)—C(7)	111.3 (4)	C(8)—C(14)—C(13)	113.5 (4)
C(6)—C(7)—O(7)	109.5 (4)	C(13)—C(14)—O(14)	105.8 (4)
C(6)—C(7)—C(8)	111.8 (4)	C(13)—C(14)—C(15)	103.4 (4)
C(8)—C(7)—O(7)	106.4 (4)	C(8)—C(14)—O(14)	106.8 (4)
C(7)—C(8)—C(14)	113.0 (4)	C(8)—C(14)—C(15)	121.5 (4)
C(7)—C(8)—C(9)	112.5 (4)	C(15)—C(14)—O(14)	104.3 (4)
C(9)—C(8)—C(14)	108.7 (4)	C(14)—C(15)—C(16)	100.2 (4)
C(8)—C(9)—C(11)	109.9 (4)	C(15)—C(16)—C(17)	110.9 (5)
C(8)—C(9)—C(10)	113.6 (4)	C(13)—C(17)—C(16)	111.1 (5)
C(10)—C(9)—C(11)	115.0 (4)	C(16)—C(17)—C(20)	124.3 (6)
C(5)—C(10)—C(9)	106.7 (4)	C(13)—C(17)—C(20)	124.3 (5)
C(1)—C(10)—C(9)	109.1 (4)	C(17)—C(20)—O(20)	119.8 (6)
C(1)—C(10)—C(5)	107.1 (4)	C(17)—C(20)—C(21)	119.7 (6)
C(9)—C(10)—C(19)	111.4 (4)	C(21)—C(20)—O(20)	120.4 (6)
C(5)—C(10)—C(19)	111.8 (4)		

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* Lists of structure factors, anisotropic thermal parameters and H-atom bond distances, angles and coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55436 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0521]

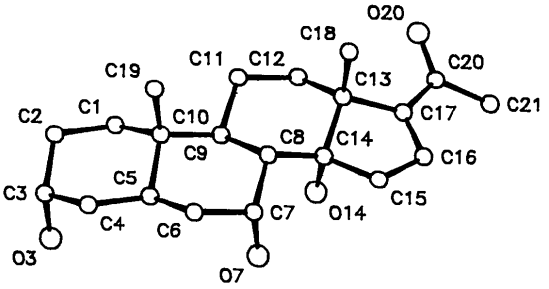

Fig. 1. A *PLUTO* diagram of the title molecule with atom-numbering scheme.

diagram of the title compound with the atomic numbering scheme is shown in Fig. 1. The *A*, *B* and *C* rings adopt normal chair conformations with average endocyclic absolute torsion angles of 55.2, 54.7 and 55.4°, respectively. The *D* ring adopts a 14α-envelope conformation with the asymmetry parameters $\Delta C_s(13) = 23.8\ (32.5)$, $\Delta C_s(14) = 4.2\ (1.4)$, $\Delta C_2(13\text{–}14) = 13.4\ (22.7)$, the values in parentheses being the ideal values for a 14α-envelope conformation (Duax & Norton, 1975). The X-ray structure of the title molecule shows that all the ring junctions adopt a *trans* configuration [H(5)—C(5)—C(10)—C(19) = 178.3 (4), H(8)—C(8)—C(9)—H(9) = 173.8 (4), C(18)—C(13)—C(14)—O(14) = −176.1 (4)°]. The progesterone side-chain orientation is normally restricted to a region over the *D* ring; the torsion angle C(16)—C(17)—C(20)—O(20) in 84 structures having a ketone group at C(20) is observed to be between 0 and −41°, *i.e.* (−)synperiplanar to the C(16)—C(17) bond (Duax, Griffin, Rohrer & Weeks, 1980). In this structure, the carbonyl at C(20) is antiperiplanar to the unsaturated C(16)—C(17) bond [C(16)—C(17)—C(20)—O(20) = 164.6 (6)°] as has been observed in four other 16-en-20-one structures (Khazheeva, Simonov, Kamernitskii, Pavlova-Grishina & Skorova, 1982; Duax, Langs, Strong & Osawa, 1979; Surcouf, 1979; Duax, Weeks & Strong, 1979). The molecules are stabilized in the crystal lattice by hydrogen bonding [O(3)···O(20) = 2.879, O(3)—H(O3) = 0.813, H(O3)···O(20) = 2.110 Å, O(3)—H(O3)···O(20) = 161.01°; O(7)···O(14) = 2.612 Å].

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Acta Cryst. (1993). C**49**, 275–278

Structures of Sobrerol Enantiomers and Racemates. III. (±)-*cis*-5-Hydroxy-α,α,4-trimethyl-3-cyclohexene-1-methanol (Racemic *cis*-Sobrerol)

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Abstract. $C_{10}H_{18}O_2$, $M_r = 170.25$, monoclinic, $P2_1/c$, $a = 16.955\ (1)$, $b = 9.313\ (1)$, $c = 13.380\ (1)$ Å, $\beta = 111.44\ (1)°$, $V = 1966.5\ (3)$ Å^3, $Z = 8$, $D_m = 1.148$, $D_x = 1.150$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.73$ cm^{-1}, $F(000) = 752$, $T = 296$ K, $R = 0.026$ for 1318 unique reflections with $I > 3\sigma(I)$. The asymmetric unit of the crystal contains two molecules joined by O—H···O [2.839 (2) Å] hydrogen bonds and C—H···O [3.432 (3) Å] weak contacts. Enantiomorphic molecules connected by O—H···O [2.810 (2)

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