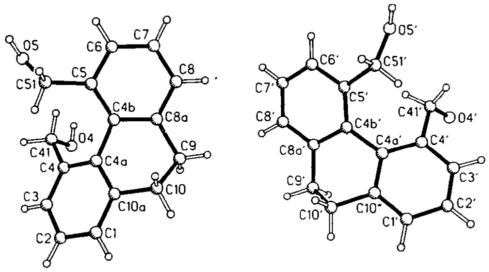

Fig. 1. The two independent molecules of the title compound in the crystal. Atomic radii are arbitrary.

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Structure of 5β-Pregnane-3α,6α,17α-triol Triacetate

By Paweł Grochulski and Zdzisław Wawrzak

Technical University of Łódź, Institute of Physics, ul. Wólczańska 219, 93-005 *Łódź, Poland*

Zdzisław Gałdecki

Technical University of Łódź, Institute of General Chemistry, ul. Zwirki 36, 90-924 *Łódź, Poland*

and William L. Duax and Phyllis D. Strong

Medical Foundation of Buffalo, Inc., 73 *High Street, Buffalo, NY* 14203, *USA*

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Abstract. $C_{27}H_{40}O_7$, $M_r = 476.61$, monoclinic, $P2_1$, $a = 17.440$ (5), $b = 13.267$ (1), $c = 12.168$ (2) Å, $\beta = 110.49$ (8)°, $V = 2637.3$ (9) Å^3, $Z = 4$, $D_x = 1.20$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 7.04$ cm^{-1}, $F(000) = 1032$, $T = 293$ K, $R = 0.048$, $wR = 0.068$ for 5590 observed reflections with $(F_o)^2 > 2\sigma[(F_o)^2]$. The structure contains two crystallographically independent molecules in the asymmetric unit that have almost identical geometry. Rings *A*, *B* and *C* have chair conformations and the *D* ring assumes a half-chair conformation in both molecules. The progesterone side chain has a conformation typical for other 17α-ester steroids; the C(16)—C(17)—C(20)—O(20) torsion angles are −18.2 (5) and −15.0 (4)° for the first and the second molecule respectively.

Experimental. Material for crystallization was provided by Dr F. S. LaBella (Templeton, Sashi Kumar, Bose & LaBella, 1989). A crystal with dimensions 0.44 × 0.64 × 0.80 mm was used for data collection on a CAD-4 diffractometer. Cell dimensions and Laue symmetry were determined from 25 centered reflections ($59.8 < 2\theta < 65.4°$) checked with oscillation photographs. Initial orientation indicated possible *B*-centered orthorhombic space group; however, a check of equivalent reflections indicated the space group to be *B*-centered monoclinic and was subsequently transformed to the current monoclinic $P2_1$ cell. Data were collected using Cu $K\alpha$ radiation, with scan width $(0.80 + 0.20\tan\theta)°$, for $\theta_{\max} = 75°$, $-22 < h < 0$, $0 < k < 16$, $-16 < l < 16$. 6509 reflections were measured using θ–2θ scans. 5665 reflections were unique, $R_{\rm int} = 0.19$. Four standard reflections (217, $7\bar{1}3$, $0\bar{9}1$, 10,1,1) were measured every 196 reflections and varied in intensity by less than 5% during the data collection. Intensity corrections were made with the *DREAM* program (Blessing, 1987).

Direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed positions of all non-H atoms. The positional and anisotropic displacement parameters of all non-H atoms were refined by full-matrix least-squares method on *F* values using 5590 reflections for which $(F_o)^2 > 2\sigma[(F_o)^2]$. The H-atom positions were located in a difference Fourier map and refined with isotropic displacement parameters. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.048$ ($R_{\rm all} = 0.048$), $wR = 0.068$ ($w = 1/\sigma^2$), $S = 2.675$ for 321 variables and 5590 reflections; $(\Delta/\sigma)_{\max}$

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Table 1. *Positional parameters* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^2) *for non*-H *atoms of molecules* (I) *and* (II)

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

	x	y	z	U_{eq}
Molecule (I)				
C(1)	8586 (1)	9334 (2)	2951 (2)	5 (1)
C(2)	8068 (1)	10290 (2)	2624 (2)	5 (1)
C(3)	8444 (1)	10990 (2)	1972 (2)	5 (1)
C(4)	9326 (1)	11245 (2)	2690 (2)	5 (1)
C(5)	9849 (1)	10286 (2)	3059 (2)	5 (1)
C(6)	10737 (1)	10517 (2)	3778 (2)	5 (1)
C(7)	10839 (1)	10947 (2)	4976 (2)	5 (1)
C(8)	10487 (1)	10229 (2)	5658 (2)	4 (1)
C(9)	9593 (1)	9916 (2)	4948 (1)	4 (1)
C(10)	9490 (1)	9507 (2)	3704 (2)	4 (1)
C(11)	9272 (1)	9197 (2)	5679 (2)	5 (1)
C(12)	9314 (1)	9656 (2)	6858 (2)	5 (1)
C(13)	10192 (1)	9994 (2)	7569 (1)	4 (1)
C(14)	10492 (1)	10709	6801 (1)	4 (1)
C(15)	11290 (1)	11148 (2)	7661 (2)	5 (1)
C(16)	11089 (1)	11309 (2)	8793 (2)	6 (1)
C(17)	10302 (1)	10706 (2)	8644 (2)	5 (1)
C(18)	10751 (2)	9072 (2)	7999 (2)	6 (1)
C(19)	9930 (2)	8493 (2)	3776 (2)	6 (1)
C(20)	10332 (2)	10117 (2)	9741 (2)	6 (1)
C(21)	9542 (2)	9685 (3)	9782 (2)	8 (1)
C(22)	7369 (2)	12009 (3)	747 (3)	7 (1)
C(23)	6992 (2)	13041 (3)	604 (4)	9 (1)
C(24)	11755 (1)	11015 (3)	3012 (2)	6 (1)
C(25)	11993 (2)	11816 (3)	2330 (2)	8 (1)
C(26)	9461 (2)	11996 (3)	9042 (2)	7 (1)
C(27)	8699 (2)	12586 (4)	8500 (4)	10 (1)
O(3*a*)	8007 (1)	11949 (2)	1727 (2)	7 (1)
O(6*a*)	11042 (1)	11235 (2)	3133 (1)	6 (1)
O(17*a*)	9584 (1)	11362 (2)	8262 (1)	6 (1)
O(20)	10975 (2)	9940 (2)	10506 (2)	8 (1)
O(22)	7100 (2)	11316 (3)	96 (3)	13 (1)
O(24)	12146 (1)	10283 (3)	3409 (3)	12 (1)
O(26)	9916 (2)	12058 (3)	10048 (2)	9 (1)
Molecule (II)				
C(1)	4971 (1)	5617 (2)	8304 (2)	6 (1)
C(2)	4163 (1)	6196 (2)	7786 (2)	6 (1)
C(3)	4306 (1)	7140 (2)	7186 (2)	6 (1)
C(4)	4977 (1)	7801 (2)	8005 (2)	5 (1)
C(5)	5773 (1)	7203 (2)	8553 (2)	5 (1)
C(6)	6450 (1)	7849 (2)	9393 (2)	5 (1)
C(7)	6294 (1)	8139 (2)	10498 (2)	6 (1)
C(8)	6148 (1)	7206 (2)	11144 (2)	5 (1)
C(9)	5472 (1)	6528 (2)	10303 (2)	5 (1)
C(10)	5665 (1)	6231 (2)	9189 (2)	5 (1)
C(11)	5266 (2)	5630 (2)	10947 (2)	6 (1)
C(12)	5030 (2)	5959 (2)	12000 (2)	6 (1)
C(13)	5706 (1)	6601 (2)	12849 (2)	5 (1)
C(14)	5890 (1)	7504 (2)	12174 (2)	5 (1)
C(15)	6460 (2)	8171 (3)	13151 (2)	7 (1)
C(16)	6135 (2)	8037 (3)	14171 (2)	7 (1)
C(17)	5495 (1)	7186 (2)	13826 (2)	6 (1)
C(18)	6468 (2)	5946 (3)	13461 (2)	7 (1)
C(19)	6435 (2)	5574 (2)	9496 (2)	7 (1)
C(20)	5491 (2)	6506 (3)	14852 (2)	7 (1)
C(21)	4773 (3)	5799 (3)	14631 (3)	8 (1)
C(22)	2988 (2)	7500 (4)	5784 (3)	9 (1)
C(23)	2276 (2)	8214 (6)	5498 (5)	14 (1)
C(24)	7250 (2)	9122 (3)	8929 (3)	7 (1)
C(25)	7212 (2)	9935 (4)	8053 (4)	9 (1)
C(26)	4331 (2)	8129 (2)	13895 (2)	7 (1)
C(27)	3540 (2)	8567 (4)	13158 (4)	10 (1)
O(3*a*)	3569 (1)	7760 (2)	6811 (2)	7 (1)
O(6*a*)	6514 (1)	8743 (2)	8737 (2)	7 (1)
O(17*a*)	4683 (1)	7601 (2)	13243 (1)	6 (1)
O(20)	6041 (2)	6503 (3)	15772 (2)	10 (1)
O(22)	3040 (2)	6819 (4)	5178 (2)	11 (1)
O(24)	7857 (1)	8851 (3)	9730 (3)	10 (1)
O(26)	4648 (2)	8229 (2)	14944 (2)	8 (1)

= 1.07. The final difference map showed maximum negative and positive peaks of −0.45 and 0.30 e Å^{-3}. No corrections for absorption or extinction were made.

Atomic positional and equivalent isotropic displacement parameters for non-H atoms are listed in Table 1.* Bond lengths and valency angles are listed in Table 2. A stereoscopic view (Johnson, 1965) of the molecule showing the atomic numbering and molecular conformation is given in Fig. 1.

Related literature. The overall geometry of the title molecule agrees well with the geometry of the mean 5β-saturated steroid molecule, which has been established by using 28 5β-steroid molecules from the Cambridge Structural Database [CSD (Allen, Kennard & Taylor, 1983)], with standard configuration of the steroid skeleton. The *A*, *B* and *C* rings have chair conformations and the *D* ring has a slightly distorted half-chair conformation with asymmetry parameters $\Delta C_s^{12} = 19.2$ and $\Delta C_2^{16} = 1.7$ for the first molecule and $\Delta C_2^{13} = 11.5$ and $\Delta C_2^{16} = 9.0$ for the second (Duax & Norton, 1975). A search of the CSD revealed only one 5β-steroid molecule with 3α and 6α substituents [HXCHOC: 3α,6α-dihydroxy-5β-cholan-24-oic acid (Hall, Maslen & Cooper, 1974)]. The title compound and that found in the CSD have been compared. The result of the comparison of the molecules is presented in Fig. 2. The *A*, *B*, *C* and *D* rings, *i.e.*, atoms from C(1) to C(17), were subjected to a least-squares process to minimize distances between pairs of corresponding atoms (Rohrer &

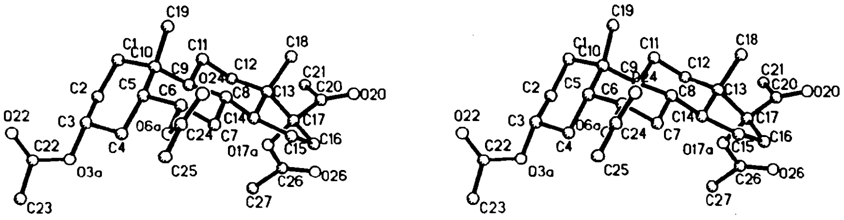

Fig. 1. Stereoview of the molecule with atom numbering.

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* Lists of anisotropic displacement parameters, H-atom coordinates, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55317 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0399]

Table 2. *Bond distances* (Å) *and bond angles* (°)

	Molecule (I)	Molecule (II)
C(1)—C(2)	1.527 (4)	1.533 (3)
C(1)—C(10)	1.539 (3)	1.542 (3)
C(2)—C(3)	1.511 (4)	1.513 (4)
C(3)—C(4)	1.518 (3)	1.522 (3)
C(3)—O(3*a*)	1.459 (3)	1.458 (3)
C(4)—C(5)	1.538 (3)	1.534 (3)
C(5)—C(6)	1.520 (3)	1.527 (3)
C(5)—C(10)	1.555 (3)	1.548 (4)
C(6)—C(7)	1.517 (3)	1.512 (4)
C(6)—O(6*a*)	1.449 (3)	1.455 (4)
C(7)—C(8)	1.526 (3)	1.535 (4)
C(8)—C(9)	1.551 (2)	1.549 (3)
C(8)—C(14)	1.527 (3)	1.524 (3)
C(9)—C(10)	1.557 (3)	1.557 (3)
C(9)—C(11)	1.538 (3)	1.537 (4)
C(10)—C(19)	1.536 (4)	1.533 (4)
C(11)—C(12)	1.537 (3)	1.541 (4)
C(12)—C(13)	1.538 (3)	1.526 (3)
C(13)—C(14)	1.546 (3)	1.548 (4)
C(13)—C(17)	1.570 (3)	1.568 (4)
C(13)—C(18)	1.537 (4)	1.543 (4)
C(14)—C(15)	1.533 (2)	1.536 (3)
C(15)—C(16)	1.550 (3)	1.544 (5)
C(16)—C(17)	1.544 (3)	1.538 (4)
C(17)—C(20)	1.532 (3)	1.543 (4)
C(17)—O(17*a*)	1.460 (3)	1.452 (3)
C(20)—C(21)	1.508 (5)	1.512 (5)
C(20)—O(20)	1.204 (3)	1.191 (3)
C(22)—O(3*a*)	1.318 (3)	1.349 (4)
C(22)—C(23)	1.503 (5)	1.502 (7)
C(22)—O(22)	1.197 (5)	1.190 (6)
C(24)—O(6*a*)	1.334 (3)	1.321 (3)
C(24)—C(25)	1.493 (5)	1.502 (6)
C(24)—O(24)	1.189 (5)	1.216 (3)
C(26)—O(17*a*)	1.340 (4)	1.356 (4)
C(26)—C(27)	1.484 (5)	1.476 (5)
C(26)—O(26)	1.206 (3)	1.207 (4)
C(2)—C(1)—C(10)	114.8 (3)	114.0 (2)
C(1)—C(2)—C(3)	108.7 (2)	109.3 (2)
C(2)—C(3)—C(4)	112.0 (3)	112.3 (2)
C(2)—C(3)—O(3*a*)	110.6 (2)	110.1 (2)
C(4)—C(3)—O(3*a*)	106.0 (2)	105.7 (2)
C(3)—C(4)—C(5)	111.2 (3)	111.0 (2)
C(4)—C(5)—C(6)	112.4 (3)	111.7 (2)
C(4)—C(5)—C(10)	113.3 (3)	113.5 (2)
C(6)—C(5)—C(10)	110.7 (3)	109.5 (2)
C(5)—C(6)—C(7)	112.4 (3)	113.6 (2)
C(5)—C(6)—O(6*a*)	108.3 (3)	106.3 (2)
C(7)—C(6)—O(6*a*)	109.4 (3)	110.7 (2)
C(6)—C(7)—C(8)	110.7 (3)	111.4 (2)
C(7)—C(8)—C(9)	112.4 (3)	110.7 (2)
C(7)—C(8)—C(14)	111.1 (3)	111.2 (2)
C(9)—C(8)—C(14)	107.7 (3)	108.6 (2)
C(8)—C(9)—C(10)	112.7 (3)	112.1 (2)
C(8)—C(9)—C(11)	110.0 (3)	111.8 (2)
C(10)—C(9)—C(11)	113.9 (3)	114.1 (2)
C(1)—C(10)—C(5)	107.9 (3)	107.1 (2)
C(1)—C(10)—C(9)	112.1 (3)	112.7 (2)
C(1)—C(10)—C(19)	106.9 (3)	106.0 (2)
C(5)—C(10)—C(9)	109.2 (3)	109.0 (2)
C(5)—C(10)—C(19)	109.2 (3)	110.3 (2)
C(9)—C(10)—C(19)	111.4 (3)	111.7 (2)
C(9)—C(11)—C(12)	112.8 (3)	112.5 (2)
C(11)—C(12)—C(13)	110.7 (3)	110.6 (2)
C(12)—C(13)—C(14)	108.7 (3)	109.1 (2)
C(12)—C(13)—C(17)	116.8 (3)	117.3 (2)
C(12)—C(13)—C(18)	110.4 (3)	110.1 (3)
C(14)—C(13)—C(17)	99.5 (3)	99.6 (2)
C(14)—C(13)—C(18)	112.6 (3)	112.9 (3)
C(17)—C(13)—C(18)	108.4 (3)	107.7 (3)
C(8)—C(14)—C(13)	113.5 (3)	114.2 (2)
C(8)—C(14)—C(15)	119.6 (3)	119.3 (3)
C(13)—C(14)—C(15)	103.9 (3)	103.5 (2)
C(14)—C(15)—C(16)	103.0 (3)	103.6 (3)
C(15)—C(16)—C(17)	107.0 (3)	107.5 (3)
C(13)—C(17)—C(16)	104.7 (3)	103.3 (3)
C(13)—C(17)—C(20)	112.1 (3)	113.1 (3)
C(13)—C(17)—O(17*a*)	104.6 (3)	105.4 (2)
C(16)—C(17)—C(20)	113.9 (3)	114.6 (3)
C(16)—C(17)—O(17*a*)	110.9 (3)	110.2 (2)
C(20)—C(17)—O(17*a*)	110.2 (3)	109.7 (2)
C(17)—C(20)—C(21)	118.0 (3)	117.2 (3)
C(17)—C(20)—O(20)	120.7 (3)	122.2 (3)
C(21)—C(20)—O(20)	121.0 (3)	120.6 (3)
O(3*a*)—C(22)—C(23)	110.9 (3)	110.2 (3)
O(3*a*)—C(22)—O(22)	124.3 (3)	124.9 (3)
C(23)—C(22)—O(22)	124.7 (3)	124.9 (3)
O(6*a*)—C(24)—C(25)	110.9 (3)	110.4 (3)
O(6*a*)—C(24)—O(24)	123.9 (3)	123.3 (3)
C(25)—C(24)—O(24)	125.2 (4)	126.2 (3)
O(17*a*)—C(26)—C(27)	110.9 (3)	111.5 (3)
O(17*a*)—C(26)—O(26)	123.4 (3)	122.9 (3)
C(27)—C(26)—O(26)	125.7 (3)	125.7 (3)
C(3)—O(3*a*)—C(22)	117.0 (3)	116.1 (2)
C(6)—O(6*a*)—C(24)	116.7 (3)	118.0 (2)
C(17)—O(17*a*)—C(26)	118.8 (3)	118.3 (2)

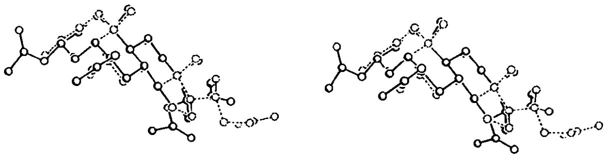

Fig. 2. Least-squares superposition of the title molecule (solid line) with HXCHOC (dashed line). The fit was made with C(1) to C(17) atoms.

Smith 1980). As Fig. 2 suggests, both molecules have similar overall conformations. The side-chain orientations of the substituents at the C(3) and C(6) atoms are very similar in both molecules.

It has been shown that for thirteen 17α-ester substituted steroid structures the average value of the C(16)—C(17)—C(20)—O(20) torsion angle is − 21 (8)° (Gałdecki, Grochulski, Wawrzak, Duax & Strong, 1990). The C(16)—C(17)—C(20)—O(20) torsion angle is − 18.2 (5)° for the first molecule and − 15.0 (4)° for the second in the present study, in the range observed previously.

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