

Fig. 4. Newman projections around the P(2)–S(4) and P(12)–S(4) bonds, showing torsion angles ($^{\circ}$).

1.449 (11); (II) P=O 1.402 (14) and 1.426 (13); (III) P=O 1.45 (2) and 1.50 (5), and P=S 1.87 (5) and 1.78 (3). The comparatively long phosphoryl P(12)–O(12) and short thiophosphoryl P(2)–S(2) bonds of isomer (IV) are not accompanied by a shortening or elongation of the other P–O and P–S bonds in the PO_3S and PO_2S_2 tetrahedra.

The mutual orientation of both the tetrahedra is shown in Fig. 4 which represents the Newman projection around the bridging P(2)–S(4) and P(12)–S(4) bonds. The bridging plane P(2)–S(4)–P(12) divides nearly symmetrically the largest dihedral angles of the tetrahedra.

The molecular packing did not show a distinct shortening of intermolecular distances.

This research was supported by project MR-I-9 from the Polish Academy of Sciences.

References

- BUKOWSKA-STRZYŻEWSKA, M. & DOBROWOLSKA, W. (1978). *Acta Cryst.* B34, 1357–1360.
- BUKOWSKA-STRZYŻEWSKA, M. & DOBROWOLSKA, W. (1980). *Acta Cryst.* B36, 3169–3172.
- BUKOWSKA-STRZYŻEWSKA, M., MICHALSKI, J., MŁOTKOWSKA, B. & SKOWERANDA, J. (1976). *Acta Cryst.* B32, 2605–2608.
- CORBRIDGE, D. E. C. (1974). *The Structural Chemistry of Phosphorus*, p. 218. Amsterdam: Elsevier.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* 53, 1891–1898.
- CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* 18, 104–109.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A27, 368–376.
- PAULING, L. (1960). *The Nature of the Chemical Bond*. Ithaca: Cornell Univ. Press.
- WIECZOREK, M. W., SHELDRIK, W. S., KAROLAK-WOJCIECHOWSKA, J., MIKOŁAJCZYK, M. & ZIEMNICKA, B. (1979). *Acta Cryst.* B35, 2339–2344.
- WILSON, A. J. C. (1942). *Nature (London)*, 150, 151–152.

Acta Cryst. (1981). B37, 727–729

Structure of 17 β -Hydroxy-7 α -methyl-5-androsten-3-one (RMI 12,936)

BY PHILIP J. COX AND G. J. MKANDAWIRE

School of Pharmacy, Robert Gordon's Institute of Technology, Schoolhill, Aberdeen AB9 1FR, Scotland

AND PAUL R. MALLINSON

Chemistry Department, University of Glasgow, Glasgow G12 8QQ, Scotland

(Received 6 October 1980; accepted 2 December 1980)

Abstract. $\text{C}_{20}\text{H}_{30}\text{O}_2$, $M_r = 302.2$, $D_m = 1.19$, $D_x = 1.19 \text{ Mg m}^{-3}$, orthorhombic, $P2_12_12_1$, $Z = 4$, $a = 5.970$ (2), $b = 14.070$ (3), $c = 20.015$ (3) Å, $U = 1681.2 \text{ Å}^3$. Final $R = 0.029$ for 1294 independent reflexions. Rings *A* and *C* are observed in chair conformations whilst ring *B* approximates to the 8 β ,9 α -half-chair form. Ring *D* approximates to an envelope conformation and an O(2)–H \cdots O(1) hydrogen bond (2.85 Å) provides the head-to-tail linkage between hydroxy and carbonyl O atoms of adjacent molecules.

Introduction. The X-ray crystal structure of the title compound was undertaken as part of a programme to determine the conformational features of a number of related antiprogesterational steroids. RMI 12,936 is an antiprogesterational and an antiestrogenic steroid and its antifertility activity has attracted attention (Kendle, 1979; Geddes, Kendle, Shanks & Steven, 1979). Of particular structural interest is the influence of the 7 α methylation on ring conformation. Crystallographic data were obtained on an Enraf–Nonius CAD-4 automated diffractometer with Ni-filtered Cu $K\alpha$

Table 1. Fractional atomic coordinates ($\times 10^4$) with e.s.d.'s and equivalent values of the anisotropic temperature factor coefficients ($\times 10^3$)

	x	y	z	U_{eq} (\AA^2)
O(1)	1885 (5)	3057 (2)	5424 (1)	70
O(2)	-257 (4)	-330 (1)	10282 (1)	54
C(1)	-143 (4)	1432 (2)	6610 (1)	46
C(2)	552 (5)	1599 (2)	5881 (1)	51
C(3)	2127 (5)	2417 (2)	5823 (1)	45
C(4)	4072 (5)	2384 (2)	6294 (1)	47
C(5)	3376 (5)	2174 (2)	7008 (1)	38
C(6)	4092 (5)	2711 (2)	7507 (1)	47
C(7)	3538 (5)	2576 (2)	8232 (1)	42
C(8)	2635 (4)	1563 (2)	8352 (1)	32
C(9)	851 (4)	1300 (2)	7824 (1)	33
C(10)	1842 (4)	1321 (2)	7103 (1)	33
C(11)	-367 (4)	363 (2)	7973 (1)	43
C(12)	-1162 (4)	263 (2)	8701 (1)	41
C(13)	786 (4)	426 (2)	9184 (1)	33
C(14)	1700 (4)	1428 (2)	9054 (1)	34
C(15)	3203 (5)	1627 (2)	9659 (1)	44
C(16)	2069 (5)	1082 (2)	10239 (1)	45
C(17)	116 (5)	526 (2)	9922 (1)	39
C(18)	2567 (5)	-353 (2)	9110 (1)	42
C(19)	3155 (5)	411 (2)	6937 (1)	45
C(20)	2000 (7)	3378 (2)	8464 (2)	61
H(1A)	-1104 (48)	2002 (17)	6770 (15)	
H(1B)	-1107 (49)	853 (16)	6642 (15)	
H(2A)	-780 (51)	1782 (25)	5605 (18)	
H(2B)	1464 (47)	1026 (19)	5684 (14)	
H(4A)	5058 (52)	1853 (19)	6109 (16)	
H(4B)	4960 (56)	3006 (18)	6263 (16)	
H(6)	4980 (52)	3276 (16)	7393 (15)	
H(7)	4958 (42)	2613 (21)	8508 (13)	
H(8)	3918 (38)	1133 (16)	8246 (13)	
H(9)	-300 (44)	1803 (16)	7845 (14)	
H(11A)	659 (44)	-201 (17)	7839 (13)	
H(11B)	-1735 (43)	300 (21)	7669 (14)	
H(12A)	-1822 (52)	-392 (16)	8773 (15)	
H(12B)	-2331 (45)	770 (16)	8789 (13)	
H(14)	316 (35)	1829 (16)	9104 (13)	
H(15A)	4781 (43)	1377 (21)	9571 (18)	
H(15B)	3083 (53)	2329 (16)	9778 (13)	
H(16A)	3058 (52)	606 (18)	10445 (15)	
H(16B)	1459 (52)	1519 (18)	10609 (13)	
H(17)	-1255 (41)	930 (19)	9949 (15)	
H(18A)	3907 (47)	-186 (22)	9412 (14)	
H(18B)	3229 (50)	-341 (19)	8633 (11)	
H(18C)	1847 (55)	-988 (19)	9198 (15)	
H(19A)	2066 (46)	-175 (18)	6880 (15)	
H(19B)	4176 (49)	254 (22)	7332 (13)	
H(19C)	4085 (42)	490 (17)	6511 (11)	
H(20A)	1606 (55)	3374 (21)	8967 (11)	
H(20B)	471 (45)	3319 (22)	8227 (15)	
H(20C)	2747 (52)	3999 (18)	8345 (15)	
HO(2)	-856 (64)	-803 (21)	10005 (17)	

radiation. The systematic absences determined the space group to be $P2_12_12_1$. The cell dimensions were adjusted by least squares from the angular settings of 25 reflexions measured at θ ca 30° . Integrated relative intensities for 1894 independent reflexions with $\theta < 70^\circ$ were measured as 2θ - ω scans; 1294 reflexions had $I > 3\sigma(I)$.

The structure was solved with *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). All H atoms were located in difference maps calculated at intermediate stages of refinement. In the final cycles of full-matrix least-squares refinement the positional parameters for all atoms, anisotropic thermal parameters for the C and O atoms and isotropic for H were varied. Convergence was reached at $R = 2.9\%$ and the weighting scheme in the final calculation was $w = (\sigma^2 F_o + 0.0014F_o^2)^{-1}$. Final positional parameters are listed in Table 1, * torsion angles in Table 2.

Discussion. The molecular structure and the packing of the molecules are shown in Figs. 1 and 2. Fig. 3 shows the bond lengths and angles involving non-hydrogen atoms; e.s.d.'s range from 0.003 to 0.004 \AA for the bond distances and from 0.2 to 0.3 $^\circ$ for the angles.

The conformations of B rings of steroids having a C(5)-C(6) double bond normally vary about the symmetric $8\beta,9\alpha$ -half-chair form (Duax & Norton, 1975). The asymmetry parameters for this ring in RMI

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35776 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

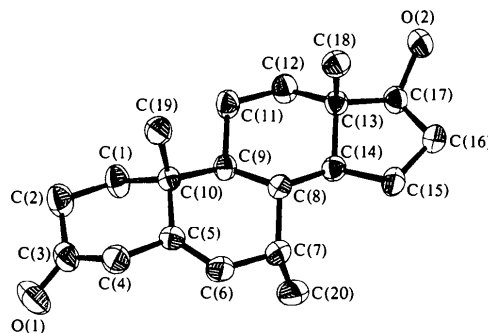


Fig. 1. The atomic arrangement in the molecule.

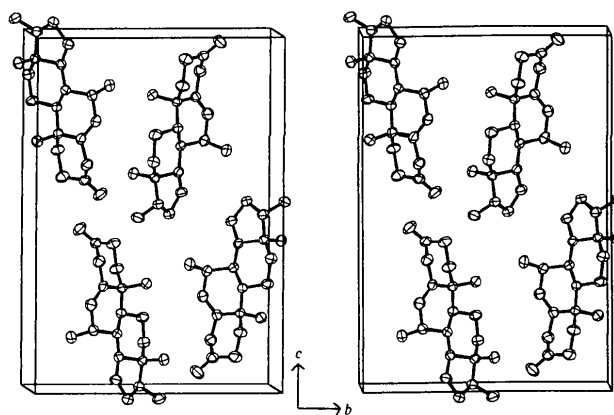


Fig. 2. A stereoscopic view of the molecular packing.

