Table 3. *Hydrogen-bond lengths* (Å) *and angles* (°) *with e.s.d.'s*

O(5')–H···O(3')^a	2·74 (3)	C(5')–O(5')–O(3')	103 (2)
O(2')–H···I^b	3·37 (2)	C(2')–O(2')–I	135 (2)
O(3')–H···O(2)^c	2·67 (3)	C(3')–O(3')–O(2)	136 (2)
N(4)–H···O(5')^a	2·90 (3)	C(4)–N(4)–O(5')	158 (2)

Symmetry code: (*a*) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (*b*) $\frac{1}{2}-x, -y, \frac{1}{2}+z$; (*c*) $\frac{1}{2}+x, \frac{1}{2}-y, -z$.

tides (*e.g.* Voet & Rich, 1970). The molecules are linked together by hydrogen bonds (Table 3), but one of the H atoms attached to N(4) is not involved in hydrogen bonding. There appears to be a hydrogen bond between O(2') and an I atom. There is no overlap of bases but the I atom partly overlaps the base of another molecule (Fig. 2). The shortest distance between the I atom and atoms in this base is 3·97 Å and involves C(5). The I atoms are in a zigzag arrangement about the symmetry axes parallel to **b**, with a distance of 4·55 Å between adjacent atoms.

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17β-Hydroxy-7β-methyl-4,14-estradien-3-one

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Abstract. $C_{19}H_{26}O_2$, $M_r = 286{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}8287\,(6)$, $b = 28{\cdot}303\,(1)$, $c = 6{\cdot}0256\,(5)$ Å ($\lambda = 1{\cdot}5418$ Å, $T = 291$ K), $V = 1676{\cdot}2$ Å^3, $Z = 4$, $\rho_x = 1{\cdot}14$ Mg m^{-3}. X-ray analysis of the title compound established unequivocally that the methyl group at C(7) has the β configuration.

Introduction. Introduction of a 7α-methyl function and a Δ^{14} double bond into 19-nortestosterone enhances androgenic activity to 1000 times that of testosterone in the chick comb assay (Segaloff & Gabbard, 1973). The biological activity of the corresponding 7β-methyl isomer has not been investigated and in order to assess its activity this compound was synthesized (the synthesis will be described elsewhere). The crystal structure determination of this product was undertaken in order to unequivocally establish the configuration of the 7-methyl group.

Crystallographic diffraction data were measured on a specimen crystal of dimensions 0·20 × 0·20 × 0·62 mm with an Enraf–Nonius CAD-4 automated diffractometer using Ni-filtered Cu $K\alpha$ radiation. The lattice parameters were refined by a least-squares fit to measured 2θ values for 25 reflections in the interval $50° < 2\theta < 69°$. Integrated relative intensities for 2028 independent reflections with $2\theta < 150°$ were measured as ω–2θ scans; 1643 of these reflections were measured to be observed above background ($I > 2\sigma$).

The intensities were reduced to structure factor amplitudes, and phase angles sufficient for location of the nonhydrogen atoms were derived using the direct-methods program *MULTAN* (Germain, Main & Woolfson, 1971) in conjunction with the negative-quartet figure of merit (DeTitta, Edmonds, Langs & Hauptman, 1975). All H atoms with the exception of H(O17) were located on a difference electron density map prepared at an intermediate stage in the least-squares refinement of the structural parameters. In the final cycles of full-matrix least-squares refinement, positional parameters for all the atoms, anisotropic thermal vibration parameters for the nonhydrogen atoms and isotropic thermal vibration parameters for

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Table 1. *Atomic coordinates of* 17β-*hydroxy*-7β-*methyl*-4,14-*estradien*-3-*one*

Standard deviations are in parentheses.

	x	*y*	*z*
C(1)	1·6772 (4)	0·78728 (10)	0·6739 (9)
C(2)	1·8211 (4)	0·77788 (13)	0·5869 (9)
C(3)	1·8565 (3)	0·81328 (12)	0·4192 (7)
C(4)	1·8158 (3)	0·86252 (10)	0·4656 (6)
C(5)	1·7350 (3)	0·87352 (9)	0·6350 (6)
C(6)	1·7132 (3)	0·92409 (10)	0·7014 (7)
C(7)	1·5640 (3)	0·93865 (8)	0·7164 (5)
C(8)	1·4818 (2)	0·90269 (8)	0·8527 (4)
C(9)	1·5074 (2)	0·85039 (8)	0·7855 (5)
C(10)	1·6607 (3)	0·83726 (10)	0·7708 (6)
C(11)	1·4316 (3)	0·81795 (9)	0·9481 (6)
C(12)	1·2785 (3)	0·82586 (10)	0·9349 (6)
C(13)	1·2375 (3)	0·87758 (10)	0·9768 (5)
C(14)	1·3292 (2)	0·91064 (8)	0·8474 (4)
C(15)	1·2576 (3)	0·94286 (10)	0·7374 (5)
C(16)	1·1064 (3)	0·93784 (11)	0·7755 (6)
C(17)	1·0954 (3)	0·88857 (11)	0·8753 (5)
C(18)	1·2431 (3)	0·89044 (15)	1·2251 (5)
C(19)	1·5565 (3)	0·98871 (10)	0·8147 (7)
O(3)	1·9273 (3)	0·80316 (9)	0·2577 (5)
O(17*B*)	0·9837 (2)	0·88677 (9)	1·0220 (4)
H(1*A*)	1·615	0·7880 (0)	0·526
H(1*B*)	1·642 (3)	0·7706 (11)	0·784 (5)
H(2*A*)	1·823 (5)	0·7425 (16)	0·529 (9)
H(2*B*)	1·898 (4)	0·7767 (15)	0·769 (6)
H(4)	1·869 (3)	0·8921 (11)	0·383 (5)
H(6*A*)	1·769 (3)	0·9476 (12)	0·608 (6)
H(6*B*)	1·740 (4)	0·9244 (12)	0·854 (7)
H(7*A*)	1·522 (3)	9·9388 (11)	0·563 (5)
H(8*B*)	1·512 (3)	0·9049 (11)	0·998 (5)
H(9*A*)	1·461 (3)	0·8440 (9)	0·637 (5)
H(10*B*)	1·706 (3)	0·8432 (10)	0·923 (4)
H(11*A*)	1·462 (3)	0·7846 (12)	0·893 (6)
H(11*B*)	1·470 (3)	0·8292 (11)	1·124 (6)
H(12*A*)	1·245 (3)	0·8078 (10)	0·766 (5)
H(12*B*)	1·242 (3)	0·8022 (11)	1·036 (6)
H(15)	1·311 (3)	0·9697 (10)	0·646 (6)
H(16*A*)	1·060 (3)	0·9437 (11)	0·612 (5)
H(16*B*)	1·070 (3)	0·9652 (11)	0·887 (6)
H(17)	1·092 (3)	0·8639 (10)	0·762 (5)
H(18*A*)	1·323 (3)	0·8816 (10)	1·306 (5)
H(18*B*)	1·221 (3)	0·9252 (12)	1·241 (6)
H(18*C*)	1·172 (3)	0·8709 (10)	1·316 (6)
H(19*A*)	1·621 (4)	1·0163 (15)	0·713 (7)
H(19*B*)	1·444 (4)	0·9994 (14)	0·851 (8)
H(19*C*)	1·596 (3)	0·9870 (11)	0·937 (5)
H(17O)	0·989 (3)	0·8545 (13)	1·075 (6)

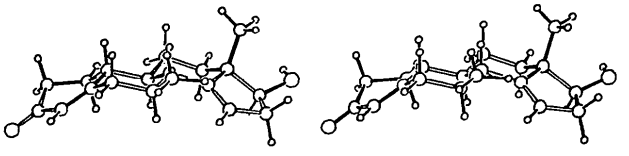

Fig. 1. *ORTEP* (Johnson, 1965) stereodrawing of 17β-hydroxy-7β-methyl-4,14-estradien-3-one.

the H atoms were varied. The quantities $(1/\sigma_F^2)$, where σ_F was as defined by Stout & Jensen (1968, p. 457, equation H14) but with an instrumental instability factor of 0·06, were used to weight the least-squares differences for the observed data; differences for data determined to be unobserved were given zero weight. The final values of the residual ($R = \sum ||F_o| - |F_c|| / \sum |F_o|$) were 0·056 for the observed data and 0·073 for all the measured data. Final positional parameters are listed in Table 1.*

Discussion. The crystallographically observed structure of the title compound is shown in Fig. 1. The intramolecular dimensions involving the nonhydrogen atoms are given in Fig. 2; the largest estimated standard deviation for the bond lengths is 0·006 Å, for

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34676 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

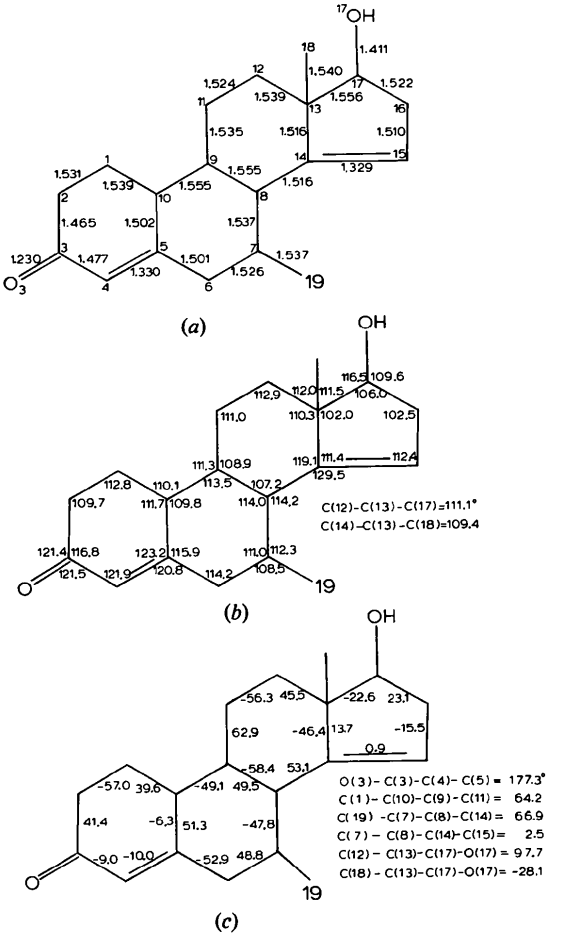

Fig. 2. Intramolecular dimensions of 17β-hydroxy-7β-methyl-4,14-estradien-3-one. (*a*) Bond distances (Å); σ range = 0·004–0·006 Å. (*b*) Bond angles (°); σ range = 0·2–0·3°. (*c*) Endocyclic torsion angles (°). A torsion angle α–β–γ–δ is positive if, when viewed down the β–γ bond, the α–β bond will eclipse the γ–δ bond when rotated less than 180° in a clockwise direction.

bond angles 0·3°, and for torsion angles 0·5°. The C—H bond distances range from 0·833 to 1·33 Å and average 1·05 Å.

The methyl substituent is observed to be in the 7α-position. For Fig. 3 a least-squares process (*FITMOL*, written by D. C. Rohrer for the *PROPHET* system) was used to optimize the overlap of the title compound and 17β-hydroxy-4,14-estradien-3-one (Rohrer, Duax & Segaloff, 1978). The conformations of the two molecules are nearly identical. The principal difference is at C(7) where the interaction between the 7β-methyl substituent and C(15) results in a puckering of the *B* ring. Hydrogen bonding from O(17) to O(3) [O(17)···O(3) = 2·81, O(17)–H = 0·97, O(3)···H = 1·922 Å, ⟨O(17)–H–O(3)⟩ = 152°] links the molecules in chains extending parallel to the *ac* diagonal.

The crystal packing of the title compound is similar to that of 17β-hydroxy-4,14-estradien-3-one. The cell dimensions of that compound are $a = 9{\cdot}7752$, $b = 25{\cdot}519$, $c = 6{\cdot}1158$ Å and the hydrogen-bonded chains are of nearly identical orientations in the two crystals. The staggering of adjacent chains is adjusted and the length of the *b* axis is expanded in order to accommodate the 7β-methyl substituent which is oriented nearly parallel to the *b* axis. Complete stereo packing diagrams will appear in *Atlas of Steroid Structure*, Vol. II (Duax, Griffin & Weeks, 1980).

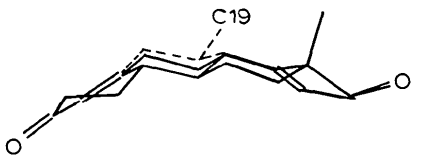

Fig. 3. A *PROPHET/FITMOL* overlap of 17β-hydroxy-7β-methyl-4,14-estradien-3-one (dashed line) and 17β-hydroxy-4,14-estradien-3-one (solid line). The average interatomic separation is 0·05 Å. The maximum deviations are between the corresponding C(7), C(16) and C(15) atoms and have magnitudes of 0·17, 0·11 and 0·09 Å, respectively.

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Benzfurazan 1-Oxide

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Abstract. $C_6H_4N_2O_2$, triclinic, $C\bar{1}$, $a = 14{\cdot}073$ (7), $b = 6{\cdot}772$ (3), $c = 7{\cdot}515$ (4) Å, $\alpha = 67{\cdot}33$ (3), $\beta = 111{\cdot}07$ (3), $\gamma = 90{\cdot}93$ (3)°, $Z = 4$, molecular volume = 152·6 Å^3. (The standard setting has $a = 7{\cdot}515$, $b = 7{\cdot}759$, $c = 6{\cdot}772$ Å, $\alpha = 114{\cdot}94$, $\beta = 112{\cdot}67$, $\gamma = 99{\cdot}08$°. The centered setting can be obtained from the

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