to the values (Δ 22·7°, φ_m 46·8°) found for RMI 12,936 (Cox, Mkandawire & Mallinson, 1981). Both A and D rings are known to be somewhat flexible in steroids related to testosterone (Roberts, Pettersen, Sheldrick, Isaacs & Kennard, 1973). The intramolecular distance between O(1) and O(2) is 10·819 (4) Å [cf. 10·902 (3) Å in RMI 12,936].

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17β-Hydroxy-19-nor-5α,17α-pregn-20-yn-3-one

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Abstract. $C_{20}H_{28}O_2$, $M_r = 300\cdot4$, $\rho_x = 1\cdot159$ Mg m^{-3}, orthorhombic, $P2_12_12_1$, $Z = 4$, $a = 12\cdot391$ (3), $b = 21\cdot066$ (5), $c = 6\cdot597$ (1) Å, $V = 1722\cdot0$ Å^3. Final $R = 0\cdot063$ for 1477 independent reflections with $I \geq 2\sigma(I)$. The C(5)–C(6) bond in the A ring of this molecule (I) is saturated making ring A conformationally rigid and the C(3) carbonyl substituent unconjugated. The parent molecule, 19-norethindrone (II), has an A ring containing a 4-en-3-one conjugated grouping. The result of saturating the A ring is to flatten the steroid backbone and reduce the progestational activity of the molecule. The position of O(3) is shifted 0·53 Å in the β-face direction compared to the position of O(3) in the 19-norethindrone structure [Mornon, Lepicard & Delettré (1976). *C.R. Acad. Sci. Sér. C*, 282, 387–390].

Introduction. The structure determination of the title compound (I), also known as 5α-dihydro-19-norethindrone, was undertaken as part of a study of synthetic progestational agents directed toward identifying the structural features important for activity. Earlier papers (Rohrer, Duax & Zeelen, 1976, 1978) showed that the overall shape of the steroid backbone had a direct relationship to the relative progestational activity of the molecule. In this compound the usual 4-en-3-one A-ring conjugated system has been removed by saturating the C(4)–C(5) bond while retaining the C(3) carbonyl substituent.

(I)   (II)
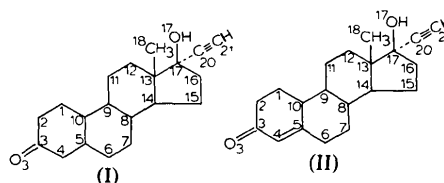
Crystal data were measured on a crystal of dimensions 0·08 × 0·40 × 0·76 mm with a Syntex $P3_1f$ automatic diffractometer with Nb-filtered Mo $K\bar{\alpha}$ radiation. The crystals are orthorhombic and the space group is $P2_12_12_1$. The lattice dimensions were refined by a least-squares fit to a set of measured 2θ values [λ(Mo $K\bar{\alpha}$) = 0·7107 Å] for 25 reflections in the interval 25° < 2θ < 30°. Integrated relative intensities for 1813 independent reflections with 2θ < 50° were measured by ω–2θ scans: 1477 of these reflections were determined to be observed above background ($I > 2\sigma_I$).

The intensities were reduced to structure factor amplitudes and phase angles sufficient to locate the

nonhydrogen atoms were obtained using the *MULTAN* programs (Main, Lessinger, Woolfson, Germain & Declercq, 1977). The H atoms were located on a difference electron density map calculated at an intermediate stage of refinement. In the final cycles of full-matrix least-squares refinement, positional parameters for all atoms, anisotropic thermal-vibration

Table 1. *Atomic coordinates* ($\times 10^5$, *for* H $\times 10^4$) *and isotropic thermal parameters* ($\times 10^2$, *for* H $\times 10$) *for molecule* (I)

$B_{iso} = \frac{4}{3} \sum_i \sum_j b_{ij}(\mathbf{a}_i \cdot \mathbf{a}_j)$ for the nonhydrogen atoms. The estimated standard deviations are given in parentheses.

	x	y	z	B_{iso} (Å²)
C(1)	60 (31)	36312 (14)	19585 (58)	405 (8)
C(2)	3075 (29)	43274 (16)	14835 (64)	463 (9)
C(3)	−6342 (29)	47601 (14)	19387 (60)	398 (8)
C(4)	−11464 (31)	46712 (14)	39547 (59)	413 (9)
C(5)	−13700 (25)	39752 (13)	45132 (55)	332 (7)
C(6)	−17515 (31)	39119 (15)	66796 (58)	398 (8)
C(7)	−20125 (27)	32298 (16)	72355 (57)	376 (8)
C(8)	−10776 (25)	27776 (13)	68015 (50)	291 (7)
C(9)	−7016 (25)	28484 (13)	45797 (49)	296 (7)
C(10)	−3915 (23)	35437 (13)	41109 (50)	307 (7)
C(11)	2076 (27)	23762 (14)	40744 (52)	362 (8)
C(12)	−1155 (28)	16858 (13)	45349 (50)	351 (7)
C(13)	−4655 (25)	16096 (13)	67225 (47)	304 (7)
C(14)	−13907 (25)	20855 (13)	72774 (49)	307 (7)
C(15)	−18043 (30)	18799 (16)	92541 (54)	400 (8)
C(16)	−16580 (33)	11528 (16)	92766 (58)	429 (9)
C(17)	−10601 (29)	9785 (13)	71983 (52)	353 (7)
C(18)	4969 (28)	16875 (17)	81887 (61)	433 (9)
C(20)	−18393 (31)	7827 (14)	56922 (55)	384 (8)
C(21)	−24498 (33)	5879 (18)	44945 (64)	505 (10)
O(3)	−9242 (22)	51616 (11)	7284 (43)	510 (7)
O(17B)	−3619 (21)	4540 (9)	76839 (39)	455 (6)
H(1A)	−570 (27)	3474 (17)	959 (54)	61 (9)
H(1B)	600 (30)	3415 (19)	1659 (65)	68 (10)
H(2A)	406 (27)	4382 (15)	19 (57)	51 (8)
H(2B)	982 (29)	4474 (17)	2100 (63)	61 (9)
H(4A)	−1856 (25)	4910 (15)	4050 (51)	42 (8)
H(4B)	−635 (28)	4787 (16)	4595 (55)	55 (9)
H(5A)	−1987 (24)	3812 (14)	3868 (49)	37 (7)
H(6A)	−2484 (19)	4185 (12)	6840 (43)	20 (6)
H(6B)	−1290 (25)	4079 (14)	7468 (57)	50 (8)
H(7A)	−2676 (21)	3090 (14)	6647 (45)	28 (6)
H(7B)	−2065 (23)	3201 (14)	8517 (47)	33 (7)
H(8B)	−512 (21)	2896 (11)	7542 (43)	27 (6)
H(9A)	−1354 (20)	2687 (13)	3997 (41)	29 (6)
H(10B)	224 (25)	3713 (14)	4937 (52)	39 (7)
H(11A)	344 (20)	2423 (11)	2744 (42)	19 (6)
H(11B)	841 (28)	2512 (16)	4887 (57)	50 (8)
H(12A)	−723 (27)	1535 (17)	3670 (55)	53 (8)
H(12B)	505 (24)	1431 (15)	4386 (50)	47 (8)
H(14A)	−1997 (24)	1959 (14)	6471 (47)	34 (7)
H(15A)	−2558 (20)	1976 (13)	9293 (41)	23 (6)
H(15B)	−1414 (25)	2061 (16)	10289 (55)	47 (8)
H(16A)	−2362 (24)	921 (16)	9381 (54)	45 (8)
H(16B)	−1261 (29)	1011 (17)	10333 (62)	59 (9)
H(18A)	854 (34)	2072 (18)	7997 (67)	80 (12)
H(18B)	375 (26)	1625 (16)	9787 (56)	57 (9)
H(18C)	1085 (28)	1393 (16)	7733 (59)	58 (9)
H(20)	−2799 (32)	402 (20)	3516 (68)	69 (11)
H(O17)	247 (33)	474 (18)	7219 (73)	94 (12)

parameters for the nonhydrogen atoms, and isotropic thermal-vibration parameters for the H atoms were varied. The quantities $(1/\sigma_F^2)$ were used to weight the least-squares differences for the observed data, where σ_F was as defined by Stout & Jensen (1968, p. 457, equation H14) but with an instability factor of 0·06; unobserved data were given zero weight. The final values of the residual, $R = \sum||F_o| - |F_c||/\sum|F_o|$, were 0·063 for the observed data and 0·087 for all the unique data measured. The scattering factors used throughout the refinement were generated from coefficients given in Table 2.2B of *International Tables for X-ray Crystallography* (Cromer & Waber, 1974). Final positional parameters and isotropic thermal parameters are given in Table 1.* The equivalent isotropic thermal parameters for the nonhydrogen atoms given in Table 1 were calculated using equation (18) of Hamilton (1959).

Discussion. The crystallographically observed structure of (I) is shown in Fig. 1. The intramolecular dimensions involving the nonhydrogen atoms are given in Fig 2. The C−H bond distances range from 0·80 to 1·07 Å and average 0·95 ± 0·06 Å. Comparison of the conformation of this structure with that of (II) provides information on the effect of unsaturation in the A ring on the steroid conformation.

The A, B and C rings in (I) all have a chair conformation. The A-ring chair is slightly flattened in the region of the carbonyl substituent in order to accommodate the larger valence angles of the sp^2 C(3) atom. The D ring has a distorted C(13) β-envelope conformation. When this conformation is compared to that of (II), very little difference in the B-, C- and D-ring conformations is found. Fig. 3 shows a drawing of the two molecules after the corresponding atoms, C(6) to C(21) and O(17B), are superimposed by minimizing the distance between corresponding atoms in each molecule (Rohrer & Smith, 1980). The 0·20 Å average

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36538 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
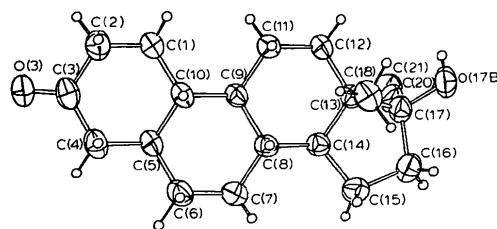

Fig. 1. *ORTEP* II (Johnson, 1965) drawing of molecule (I) with atomic numbering. Thermal ellipsoids for nonhydrogen atoms are scaled at the 60% probability level.

Fig. 3. A *PROPHET/FITMOL* (Rohrer & Smith, 1980) overlay of atoms C(6) to C(21) in molecules (I) and (II).

Fig. 2. Intramolecular dimensions of molecule (I). (a) Bond distances (Å); σ range 0·004 to 0·006 Å. (b) Bond angles (°); σ range 0·2 to 0·3°. (c) Endocyclic torsion angles (°); σ range 0·3 to 0·4°. 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.

separation between these atoms indicates that the conformations of the two molecules in this region are very similar and that the degree of saturation in the A ring has not significantly affected the conformation in the remainder of the molecule. The A-ring conformations are, as expected, very different. Molecule (II) has a C(1)/C(2) α/β-half-chair conformation which bows the molecule in the α direction compared to (I) (see Fig. 3). The carbonyl O location is shifted by 0·53 Å relative to the unchanged portion of the molecule and C(3) is also shifted 0·53 Å.

There are only two nonbonded intermolecular contacts between non-hydrogen atoms less than 3·5 Å. These are C(4) to O(17B), 3·336 (4) Å, and C(12) to

O(3), 3·464 (4) Å. The C(17) hydroxyl also forms a hydrogen bond to O(3), 2·826 (4) Å, of a twofold-screw-related molecule $(-x, \frac{1}{2} + y, \frac{1}{2} - z)$. The H(O17)···O(3) distance is 2·22 (5) Å and the O(17B)—H(O17)···O(3) angle is 132 (3)°.

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