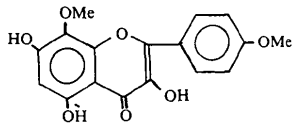
Final atomic coordinates are given in Table 1, and selected bond lengths and angles in Table 2.* A view of prudomestin with the atomic numbering scheme is shown in Fig. 1. The molecular packing is shown in Fig. 2.

Discussion. The X-ray results identify prudomestin as 3,5,7-trihydroxy-8-methoxy-2-(4-methoxyphenyl)-4*H*-1-benzopyran-4-one (I).



(I)

The sample of prudomestin used in this study, and also that of Wollenweber, Dietz, Schilling, Favre-Bonvin & Smith (1985), exhibited a bathochromic shift in its UV spectrum in the presence of NaOAc/H_3BO_3. This observation suggests but does not confirm an *ortho*-dihydroxy system (Harborne, Mabry & Mabry, 1975); definitive evidence for the absence of such a grouping is provided by this investigation.

The C–O skeleton of the molecule is essentially planar with a mean deviation from the plane of 0.05 Å; the methyl C atoms project above and below this plane. Bond lengths and angles are unexceptional. The molecules stack in parallel rows with an alternating pattern of up–down methoxy groups. The main attractive forces between the molecules in a layer involve hydrogen bonding between OH groups and methoxy or C=O groups, Fig. 2.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54876 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0106]

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Structure of (20*R*)-5α-Pregnano[3,4-*c*][1,2,5]oxadiazol-20-ol (HS1011)

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Abstract. $C_{21}H_{32}N_2O_2$, $M_r = 344$, orthorhombic, $P2_12_12_1$, $a = 7.353$ (1), $b = 10.153$ (1), $c = 25.324$ (3) Å, $V = 1891$ Å^3, $Z = 4$, $D_x = 1.21$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 5.30$ cm^{-1}, $F(000) = 752$, room temperature, $R = 0.054$ for 2968 observed reflections. All rings of the steroid skeleton are *trans* connected. Ring *A* is strained and rings *C* and *D* are in chair conformations. Ring *D* has an intermediate envelope–half-chair conformation. The oxadiazole ring is planar. The methyl and hydroxyl side groups linked through C(18) to the steroid skeleton at C(17) are equatorial and axial respectively.

Introduction. Medicinal chemists have modified the structure of testosterone in various ways (Drill & Riegel, 1958) with the object of increasing the anabolic (nitrogen retention) propensity and decreasing its effect as a male hormone. This assumes that the target receptors associated with these two effects are sufficiently different to be sensitive to small changes in the structure of the drug molecule and to react accordingly. One successful approach has been to

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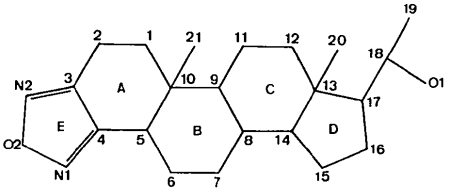
introduce different *A*-ring fused heterocycles [see, for example, Clinton *et al.* (1961), Ohta, Takegoshi, Veno & Shimizu (1965) and Kasahara, Ondera, Mogi, Oshima & Shimizu (1965)]. The compound (20*R*)-5α-pregnano[3,4-*c*][1,2,5]oxadiazol-20-ol (HS1011) (Fig. 1) was prepared during the course of work on the synthesis of steroid oxadiazoles (Jindal, Yadav, Sharma, Agrawal & Singh, 1987). We have determined the structure of HS1011 in order to study the effect of the 5-en-oxadiazole system on the steroid skeleton and to clarify some conformational aspects for future structure–function studies.

Experimental. Colourless needle-shaped crystals (from ethanol), specimen 0.9 × 0.2 × 0.1 mm used for data collection. Preliminary Weissenberg photographs yielded approximate cell dimensions and showed orthorhombic (*mmm*) symmetry. Space group $P2_12_12_1$ determined unambiguously from systematic absences ($h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$; $00l$, $l = 2n + 1$). Enraf–Nonius CAD-4 automated diffractometer, graphite monochromator, Cu $K\alpha$ radiation. 25 high-angle reflections ($25 < 2\theta < 28°$) used to obtain accurate cell dimensions by least-squares fit. ω–2θ scan, scan width $(0.85 + 0.15\tan\theta)°$, vertical aperture 4 mm. 2984 unique reflections ($1 < \theta < 70°$) measured ($-2 < h < 9$, $-12 < k < 12$, $-28 < l < 31$), $R_{int} = 0.014$, 2968 with $I \geq 3\sigma(I)$. Three intensity standards (244, $2\bar{4}4$, $\bar{2}44$) monitored at intervals of 100 measurements showed no significant variations during data collection; intensity data corrected for Lorentz–polarization factors; empirical absorption correction based on φ scans for each of two (412, 812) reflections (North, Phillips & Mathews, 1968) for $\chi = 90°$ measured at 10° intervals from $\varphi = 0$–360°, normalized transmission factors 0.86 to 0.96. Structure solution by direct methods with *SHELX*76 (Sheldrick, 1976). Refinement by full-matrix least squares with anisotropic thermal factors for non-H atoms, isotropic for H atoms, which were placed in calculated positions on the corresponding C atoms (C—H = 1.08 Å) except for H(1) which was identified from the difference map. Function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = (\sigma^2|F_o| + 0.005254|F_o|^2)^{-1}$; $R = 0.054$, $wR = 0.065$, R (all data) = 0.055 for 256 variable parameters, maximum (shift/σ) = 1.090. Final electron density −0.42 to 0.46 e Å^{-3}. Calculations carried out on VAX and AMDAHL 470V/8 computers. Geometrical calculations were performed with *XANADU* (Roberts & Sheldrick, 1975) and molecular illustrations were drawn with *PLUTO* (Motherwell & Clegg, 1978).

Discussion. The refined atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1.* Bond distances and angles are listed in Table 2. The chemical formula with the numbering scheme of the atoms is shown below. Fig. 1 shows the molecular conformation.



HS1011

Most of the bond lengths in HS1011 are close to the expected values. The average C—C single-bond lengths in rings *A*, *B*, *C* and *D* are 1.506 (3), 1.536 (3), 1.531 (3) and 1.543 (3) Å, respectively. The average value of all the C—C single bond lengths in the molecule is 1.529 (3) Å. This is in agreement with the values found in similar steroid structures *e.g.* 17β-hydroxy-17α-methyl-5β-androstano[2,3-*c*]-[1,2,5]oxadiazole (HS804) (El Shora, Palmer, Singh & Paul, 1984), 14β-hydroxy-17α-methyl-5α-androstano[2,3-*c*][1,2,5]oxadiazole (HS805) (El Shora *et al.*, 1984) and 17a-methyl-3β-pyrrolidinyl-17α-aza-*D*-homo-5α-androstane (HS691) (Husain, Tickle, Palmer, Singh, Bhardwaj & Paul, 1982). The shortening in bond length C(2)—C(3) [1.504 (3) Å] is associated with the fusion of the oxadiazole ring with ring *A*; C(3) and C(4) both have C(sp)^3 character. The bond length C(3)—C(4) of 1.409 (3) Å is significantly shorter that those found in HS804 and HS805 (El Shora *et al.*, 1984) whose values are 1.425 (4) and 1.429 (4) Å respectively. The C(3)=N(2) bond length is 1.295 (3) Å which agrees well with those quoted for HS804 and HS805, while the C(4)=N(1) bond length of 1.317 (3) Å is noticeably longer than the corresponding values of 1.300 (5) Å in HS804 and 1.273 (5) Å in HS805. The shortening of bond lengths C(3)—C(4) may be associated with π delocalization in the system N(2)=C(3)—C(4)=N(1), similar to that found for other heterocyclic oxadiazoles [see, for example, Sagebarth & Cox (1965), Calleri, Chiari, Chesi Villa, Gaetani Manfredotti, Guastini & Viterbo (1975), Viterbo & Serafino (1978) and El Shora *et al.* (1984).

The N—O bond lengths N(2)—O(2) of 1.407 (3) Å and N(1)—O(2) of 1.379 (3) Å are comparable to the

* Lists of structure factors, anisotropic thermal parameters, intermolecular close contacts and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54950 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HE0035]

Table 1. *Refined positional parameters* ($\times 10^4$) *and equivalent isotropic temperature factors* ($Å^2 \times 10^4$) *with e.s.d.'s in parentheses*

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	U_{eq}
C(1)	−0.1769 (3)	−0.0443 (2)	−0.3907 (1)	0.048
C(2)	−0.1790 (3)	0.0235 (3)	−0.3364 (1)	0.056
C(3)	0.0072 (3)	0.0750 (2)	−0.3231 (1)	0.045
C(4)	0.1518 (3)	0.0758 (2)	−0.3596 (1)	0.040
C(5)	0.1396 (2)	0.0216 (2)	−0.4141 (1)	0.038
C(6)	0.2736 (3)	0.0809 (2)	−0.4531 (1)	0.044
C(7)	0.2621 (3)	0.0080 (2)	−0.5056 (1)	0.042
C(8)	0.0675 (2)	0.0059 (2)	−0.5271 (1)	0.033
C(9)	−0.0692 (2)	−0.0479 (2)	−0.4859 (1)	0.035
C(10)	−0.0607 (2)	0.0269 (2)	−0.4329 (1)	0.036
C(11)	−0.2590 (2)	−0.0556 (2)	−0.5108 (1)	0.041
C(12)	−0.2643 (3)	−0.1373 (2)	−0.5614 (1)	0.042
C(13)	−0.1312 (3)	−0.0856 (2)	−0.6027 (1)	0.036
C(14)	0.0563 (3)	−0.0796 (2)	−0.5761 (1)	0.039
C(15)	0.1873 (3)	−0.0542 (2)	−0.6223 (1)	0.048
C(16)	0.1036 (3)	−0.1340 (2)	−0.6682 (1)	0.049
C(17)	−0.0864 (3)	−0.1799 (2)	−0.6487 (1)	0.037
C(18)	−0.2189 (3)	−0.1924 (2)	−0.6948 (1)	0.044
C(19)	−0.1610 (4)	−0.2952 (2)	−0.7340 (1)	0.060
C(20)	−0.1966 (4)	0.0479 (2)	−0.6242 (1)	0.054
C(21)	−0.1261 (3)	0.1709 (2)	−0.4388 (1)	0.051
N(1)	0.2957 (3)	0.1303 (2)	−0.3377 (1)	0.055
N(2)	0.0588 (3)	0.1279 (3)	−0.2790 (1)	0.060
O(1)	−0.3944 (2)	−0.2239 (2)	−0.6740 (1)	0.056
O(2)	0.2412 (3)	0.1641 (2)	−0.2874 (1)	0.063

Table 2. *Bond lengths* (Å), *bond angles* (°), *selected torsion angles* (°) *and asymmetry parameters* (°)

Bond length and bond angle e.s.d.'s are given in parentheses; e.s.d.'s for torsion angles are in the range 0.3–0.4°.

C(1)—C(2)	1.539 (3)	C(10)—C(21)	1.546 (2)
C(1)—C(10)	1.546 (2)	C(11)—C(12)	1.528 (3)
C(2)—C(3)	1.504 (3)	C(12)—C(13)	1.526 (3)
C(3)—C(4)	1.409 (3)	C(13)—C(14)	1.535 (2)
C(3)—N(2)	1.295 (3)	C(13)—C(17)	1.544 (2)
C(4)—C(5)	1.489 (2)	C(13)—C(20)	1.537 (3)
C(4)—N(1)	1.317 (3)	C(14)—C(15)	1.537 (2)
C(5)—C(6)	1.519 (3)	C(15)—C(16)	1.546 (3)
C(5)—C(10)	1.549 (2)	C(16)—C(17)	1.554 (3)
C(6)—C(7)	1.524 (3)	C(17)—C(18)	1.527 (3)
C(7)—C(8)	1.532 (2)	C(18)—C(19)	1.501 (3)
C(8)—C(9)	1.548 (2)	C(18)—O(1)	1.430 (3)
C(8)—C(14)	1.518 (2)	N(1)—O(2)	1.379 (3)
C(9)—C(10)	1.544 (2)	N(2)—O(2)	1.407 (3)
C(9)—C(11)	1.533 (2)		

C(2)—C(1)—C(10)	114.4 (2)	C(9)—C(10)—C(21)	111.6 (1)
C(1)—C(2)—C(3)	110.3 (2)	C(9)—C(11)—C(12)	113.3 (2)
C(2)—C(3)—C(4)	122.8 (2)	C(11)—C(12)—C(13)	111.8 (2)
C(2)—C(3)—N(2)	127.2 (2)	C(12)—C(13)—C(14)	106.8 (1)
C(4)—C(3)—N(2)	110.0 (2)	C(12)—C(13)—C(17)	116.1 (2)
C(3)—C(4)—C(5)	124.1 (2)	C(12)—C(13)—C(20)	110.2 (2)
C(3)—C(4)—N(1)	109.5 (2)	C(14)—C(13)—C(17)	99.4 (1)
C(5)—C(4)—N(1)	126.4 (2)	C(14)—C(13)—C(20)	113.7 (2)
C(4)—C(5)—C(6)	114.7 (1)	C(17)—C(13)—C(20)	110.3 (2)
C(4)—C(5)—C(10)	109.2 (1)	C(8)—C(14)—C(13)	115.5 (1)
C(6)—C(5)—C(10)	113.8 (1)	C(8)—C(14)—C(15)	119.5 (2)
C(5)—C(6)—C(7)	109.8 (2)	C(13)—C(14)—C(15)	103.6 (1)
C(6)—C(7)—C(8)	111.7 (2)	C(14)—C(15)—C(16)	103.6 (2)
C(7)—C(8)—C(9)	111.8 (1)	C(15)—C(16)—C(17)	106.0 (1)
C(7)—C(8)—C(14)	110.5 (1)	C(13)—C(17)—C(16)	104.2 (2)
C(9)—C(8)—C(14)	108.3 (2)	C(13)—C(17)—C(18)	119.5 (2)
C(8)—C(9)—C(10)	112.7 (1)	C(16)—C(17)—C(18)	110.8 (1)
C(8)—C(9)—C(11)	109.4 (1)	C(17)—C(18)—C(19)	112.4 (2)
C(10)—C(9)—C(11)	114.8 (1)	C(17)—C(18)—O(1)	108.1 (1)
C(1)—C(10)—C(5)	107.3 (1)	C(19)—C(18)—O(1)	110.1 (2)
C(1)—C(10)—C(9)	110.4 (1)	C(4)—N(2)—O(2)	105.1 (2)
C(1)—C(10)—C(21)	109.7 (2)	C(3)—N(2)—O(2)	105.0 (2)
C(5)—C(10)—C(9)	106.7 (2)	N(1)—O(2)—N(2)	110.5 (2)
C(5)—C(10)—C(21)	111.0 (2)		

C(1)—C(2)—C(3)—C(4)	8.42	C(12)—C(11)—C(9)—C(8)	55.66
C(2)—C(3)—C(4)—C(5)	−2.56	C(11)—C(9)—C(8)—C(14)	−54.56
C(3)—C(4)—C(5)—C(10)	26.35	C(9)—C(8)—C(14)—C(13)	59.38
C(4)—C(5)—C(10)—C(1)	−53.84	C(14)—C(15)—C(16)—C(17)	9.67
C(5)—C(10)—C(1)—C(2)	64.63	C(15)—C(16)—C(17)—C(13)	19.85
C(10)—C(1)—C(2)—C(3)	−40.00	C(16)—C(17)—C(13)—C(14)	−41.12
C(5)—C(6)—C(7)—C(8)	54.70	C(17)—C(13)—C(14)—C(15)	47.90
C(6)—C(7)—C(8)—C(9)	−53.13	C(13)—C(14)—C(15)—C(16)	−36.04
C(7)—C(8)—C(9)—C(10)	54.47	C(20)—C(13)—C(17)—C(18)	−45.92
C(8)—C(9)—C(10)—C(5)	−54.77	C(13)—C(17)—C(18)—O(1)	−53.97
C(9)—C(10)—C(5)—C(6)	58.30	C(13)—C(17)—C(18)—C(19)	−175.86
C(10)—C(5)—C(6)—C(7)	−59.23	C(16)—C(17)—C(18)—O(1)	−175.17
C(8)—C(14)—C(13)—C(12)	−58.35	C(16)—C(17)—C(18)—C(19)	62.94
C(14)—C(13)—C(12)—C(11)	54.18	C(21)—C(10)—C(9)—C(11)	−59.42
C(13)—C(12)—C(11)—C(9)	−56.79		

Asymmetry parameters

Ring *A*

$\Delta C_1^1 = 32.8$ $\Delta C_2^{1,2} = 53.8$

$\Delta C_1^2 = 43.2$ $\Delta C_2^{2,3} = 37.9$ Sofa/half-chair

$\Delta C_1^3 = 10.6$ $\Delta C_2^{3,4} = 16.0$

Ring *B*

$\Delta C_1^9 = 4.0$ $\Delta C_2^{9,10} = 4.0$

$\Delta C_1^{10} = 3.5$ $\Delta C_2^{5,10} = 2.8$ 8β,9α-Chair

$\Delta C_1^5 = 2.9$ $\Delta C_2^{5,6} = 2.9$

Ring *C*

$\Delta C_1^{12} = 3.3$ $\Delta C_2^{11,12} = 3.5$

$\Delta C_1^{11} = 1.0$ $\Delta C_2^{11,9} = 4.0$ 9α,13β-Chair

$\Delta C_1^9 = 2.6$ $\Delta C_2^{9,8} = 1.8$

Ring *D*

$\Delta C_1^{13} = 12.4$ $\Delta C_2^{16} = 8.0$ 13β-Envelope/13β,14α-half-chair

$\Delta C_1^{14} = 23.8$

values found in 3-amino-4-methylfurazan (Viterbo & Serafino, 1978), in the range 1.380 (3)–1.406 (3) Å, to those found in HS804 and HS805 (El Shora *et al.*, 1984), in the range 1.367 (7)–1.393 (5) Å, and to the average value of N—O bond lengths of 1.380 (3) Å found in furazan (Sagebarth & Cox, 1965).

Excluding bond angles C(3) to C(4), the average C—C—C bond angle within the steroid skeleton is 109.3°. Ten of the angles are at least 3σ greater than this value [110.3 (2)–115.5 (1)°] and eight are at least 10σ less [99.4 (1)–107.3 (1)°]. The interior angle C(17)—C(13)—C(14) [99.4 (1)°] of ring *D* is significantly less than the same angle in similar compounds [100.8 (3) and 101.1 (3)° in HS804 and HS805 respectively]. All of the bond angles have either central CH or CH_2 substituents, while the small bond angles have either central C(10) or C(13), both bearing CH_3 groups. The average values of the bond angles in these three categories are 110.0 (central CH_2), 111.9 (central CH) and 109.4° (central C bearing CH_3 substituent). The data for the steroid oxadiazoles HS804 and HS805 (El Shora *et al.*, 1984) show a similar effect with average bond angles in these three categories of 110.5 (central CH_2), 111.8 (central CH) and 107.8° (central C bearing CH_3).

Conformational features of the molecule may be described in terms of torsion angles and asymmetry parameters (Table 2). The pseudo-torsion angle C(19)—C(10)···C(13)—C(18) (Duax & Norton, 1975), giving a quantitative measure of the twist about the length of the molecule, has a value of 1.7° in HS1011. Conformation and symmetry in the six-membered rings *A*, *B* and *D* depart, as is to be expected, from the ideal. Following Duax & Norton

(1975) the magnitudes of the asymmetry parameters ΔC_s and ΔC_2 (Table 2), have been calculated to indicate the deviation (about bond directions and bond-angle bisectors) from mirror and twofold symmetry. (A true *m* plane corresponds to $\Delta C_s = 0°$, and a twofold axis to $\Delta C_2 = 0°$.) Ring *A* is an intermediate strained 10β-sofa/10β,1α-half-chair. Rings *B* and *C* have low values for both ΔC_s and ΔC_2 showing good approximation to the ideal chair conformation. Ring *D* is intermediate between 13β-envelope and 13β,14α-half-chair. The oxadiazole ring *E* is planar (r.m.s. deviations 0.001 Å). Ring connections are as follows: *A*/*B trans*, *B*/*C trans* and *C*/*D* quasi-*trans*. The oxadiazole ring is *cis*-fused to ring *A*. The methyl and hydroxyl side groups which are linked through C(18) to the steroid skeleton on C(17) are equatorial and axial (β-oriented) respectively. The molecule is typically β-convex.

Fig. 1. Stereoview of the molecule, edge-on to the steroid nucleus.

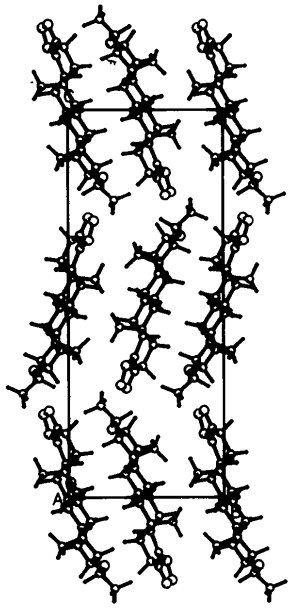


Fig. 2. View illustrating the molecular packing as seen along the *a* axis.

Intermolecular hydrogen bonding is evident between pairs of molecules through the hydroxyl group and the oxadiazole ring N atom [O(1)···N′(2) = 3.079 (3) Å, O(1)—H(1)···N′(2) = 145.1 (3)°, O(1)—H(1) = 0.939 (24) Å and H(1)···N′(2) = 2.262 (3) Å; $-\frac{1}{2}-x$, $-y$, $\frac{1}{2}+z$]. All other intermolecular contacts are very weak van der Waals contacts. No evidence of any disorder was found. The packing of the molecules along the *a* axis is shown in Fig. 2.

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