angles and torsion angles are given in Table 2. The identification of the atoms and the configuration are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1; the packing of the molecules is shown in the unit cell in Fig. 2.

Related literature. Polysubstituted benzenes such as (I) are easily hydrolyzed to acids which serve as precursors to naturally occurring anthraquinones and cyanodontin (de Silva, Watanabe & Snickus, 1979).

The support of Welch Foundation to SSCC (N-495) and ERB (N-118) is greatly appreciated.

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Acta Cryst. (1988). C**44**, 376–378

Structure of the Ethanol Solvate of 13-Ethyl-11β-methyl-18-norlynestrenol

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(*Received* 14 *July* 1987; *accepted* 14 *November* 1987)

Abstract. 13-Ethyl-11β-methyl-18,19-dinor-17α-pregn-4-en-20-yn-17β-ol, $C_{22}H_{32}O.C_2H_6O$, $M_r = 358{\cdot}57$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}888\ (3)$, $b = 10{\cdot}594\ (4)$, $c = 26{\cdot}45\ (2)$ Å, $V = 2210\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}077$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}6$ cm^{-1}, $F(000) = 792$, $T = 293$ K, $R = 0{\cdot}064$ for 1946 observations. The overall molecular conformation is identical to that of 11β-methyllynestrenol [Rohrer, Hazel, Duax & Zeelen (1976). *Cryst. Struct. Commun.* **5**, 543–546]; a least-squares fit of the steroid backbones [C(1)–C(18)] gave a mean deviation of the fitted atoms of 0·033 (4) Å. The steroid backbone is bent by steric interaction between the angular methyl and ethyl groups. The methyl group does not affect the orientation of the ethyl group, which has the preferred *trans* conformation with respect to the *C*/*D* ring junction [van Geerestein, Kanters, Duisenberg & Kroon (1986). *Acta Cryst.* C**42**, 469–472], C(14)–C(13)–C(18)–C(22) = −168·2 (4)°. The ethanol solvent molecule acts as a donor as well as an acceptor in two hydrogen bonds connecting two steroid molecules related by the 2_1 axis parallel to **a**; $\rightarrow O(17)\rightarrow O(e)\rightarrow O(17')$ $(\frac{1}{2}+x, \frac{3}{2}-y, -z)$ with $O(17)\cdots O(e) = 2{\cdot}735\ (6)$, $O(e)\cdots O(17') = 2{\cdot}751\ (6)$ Å, $O(17)\text{–}H\cdots O(e) = 158\ (5)$ and $O(e)\text{–}H\cdots O(17') = 162\ (5)°$. The shortest intermolecular $H\cdots H$ contact distances are approximately 2·3 Å.

Experimental. Sample obtained from the Scientific Development Group of Organon, Oss, The Netherlands. Large crystals were obtained by evaporation of an ethanol solution. An irregular shaped fragment (1·0 × 1·0 × 0·6 mm) was used for data collection. Lattice parameters refined by least-squares fitting of 2θ values of 12 reflections in range 19–24°; Enraf–Nonius CAD-4 diffractometer with Zr-filtered Mo $K\alpha$ radiation; ω–2θ scan mode, $\Delta\omega = (0{\cdot}50 + 0{\cdot}35\tan\theta)°$; 2889 independent reflections measured, $2\theta_{\max} = 55°$, h, k, l (max. range 10, 13, 34), 1946 reflections with $I \geq 2{\cdot}5\sigma(I)$ were considered observed. Four standard reflections showed intensity variations up to 7%; intensities were corrected for these fluctuations. Lp corrections were applied, but no absorption corrections.

Structure solved by Patterson search with a preliminary version of *PATSEE* (Egert & Sheldrick, 1985); the fused $B + C$ rings of the steroid backbone were used as search fragment, whereafter the best solution was used for tangent expansion. E map gave all non-H atoms including those of the solvent molecule. However, the methyl C atom of ethanol was located at very low density.

H atoms were placed at calculated positions riding on their bonded atoms, except both hydroxyl-group H atoms and the H atom bonded to the ethynyl group, which were located on a difference map and refined positionally. 245 parameters were varied in full-matrix least-squares refinement on F with *SHELX76* (Sheldrick, 1976); all non-H atoms refined anisotropically and for H atoms an overall isotropic thermal parameter was varied [$U = 0{\cdot}073\ (3)$ Å^2]; H atoms of the

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ethanol solvent molecule were given a fixed thermal parameter ($U = 0{\cdot}1$ Å^2). Convergence was reached at $R = 0{\cdot}064$ and $wR = 0{\cdot}10$, where $w = 1/\sigma^2(F)$ and $S = 0{\cdot}7$; $\Delta/\sigma = 0{\cdot}02$ (3) (av.) and 0·08 (max.) for non-H atom parameters of the steroid molecule and 0·26 (max.) for ethanol parameters; for H-atom parameters, $\Delta/\sigma = 0{\cdot}05$ (3) (av.) and 0·11 (max.); $-0{\cdot}2 < \Delta\rho < 0{\cdot}3$ e Å^{-3}. The methyl group of the ethanol molecule is disordered as indicated by the large thermal motion and the short C(1*e*)—C(2*e*) bond. Scattering factors were taken from *SHELX*. Table 1* gives the final atomic coordinates and equivalent isotropic thermal parameters. Bond lengths and bond angles are given in Table 2. The conformation of the steroid molecule and the atom-numbering scheme are shown in Fig. 1. The packing and hydrogen bonding is illustrated in Fig. 2, which shows a stereoview down **a**.

Table 1. *Positional and equivalent isotropic thermal parameters* (Å^2) *for non*-H *atoms with e.s.d.'s in parentheses*

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

	x	*y*	*z*	U_{eq}
O(17)	0·6668 (5)	0·6679 (3)	0·0311 (1)	0·056 (1)
C(1)	0·9333 (7)	0·1383 (6)	0·2158 (2)	0·068 (2)
C(2)	0·945 (1)	0·0281 (8)	0·2518 (3)	0·095 (3)
C(3)	0·7846 (9)	0·0145 (8)	0·2834 (2)	0·091 (3)
C(4)	0·6334 (8)	0·0230 (6)	0·2499 (2)	0·069 (2)
C(5)	0·6280 (7)	0·0716 (4)	0·2046 (2)	0·051 (1)
C(6)	0·4738 (7)	0·0672 (5)	0·1719 (2)	0·062 (2)
C(7)	0·4310 (6)	0·2023 (4)	0·1528 (2)	0·052 (2)
C(8)	0·5801 (5)	0·2582 (4)	0·1235 (2)	0·039 (1)
C(9)	0·7411 (5)	0·2595 (4)	0·1563 (1)	0·038 (1)
C(10)	0·7841 (6)	0·1291 (4)	0·1795 (2)	0·048 (1)
C(11)	0·8922 (5)	0·3199 (4)	0·1280 (2)	0·042 (1)
C(12)	0·8478 (5)	0·4530 (4)	0·1079 (2)	0·040 (1)
C(13)	0·6850 (5)	0·4580 (4)	0·0773 (1)	0·039 (1)
C(14)	0·5426 (5)	0·3948 (4)	0·1078 (2)	0·041 (1)
C(15)	0·3788 (6)	0·4250 (5)	0·0783 (2)	0·052 (2)
C(16)	0·4113 (6)	0·5604 (5)	0·0583 (2)	0·060 (2)
C(17)	0·6008 (6)	0·5930 (4)	0·0713 (2)	0·047 (1)
C(18)	0·7041 (6)	0·4011 (4)	0·0233 (2)	0·048 (1)
C(20)	0·6054 (7)	0·6647 (5)	0·1186 (2)	0·058 (2)
C(21)	0·606 (1)	0·7281 (6)	0·1557 (2)	0·081 (2)
C(22)	0·8606 (7)	0·4363 (5)	−0·0070 (2)	0·061 (2)
C(23)	0·9709 (6)	0·2294 (4)	0·0891 (2)	0·049 (1)
O(1*e*)	0·9884 (5)	0·7493 (4)	0·0519 (2)	0·075 (1)
C(1*e*)	0·999 (1)	0·8537 (7)	0·0855 (3)	0·100 (3)
C(2*e*)	1·131 (2)	0·839 (1)	0·1229 (4)	0·173 (6)

Table 2. *Bond distances* (Å) *and bond angles* (°) *for non*-H *atoms with e.s.d.'s in parentheses*

O(17)—C(17)	1·425 (6)	C(11)—C(12)	1·547 (6)
C(1)—C(2)	1·51 (1)	C(11)—C(23)	1·537 (7)
C(1)—C(10)	1·522 (7)	C(12)—C(13)	1·519 (6)
C(2)—C(3)	1·52 (1)	C(13)—C(14)	1·536 (6)
C(3)—C(4)	1·489 (9)	C(13)—C(17)	1·585 (6)
C(4)—C(5)	1·305 (8)	C(13)—C(18)	1·558 (6)
C(5)—C(6)	1·493 (8)	C(14)—C(15)	1·543 (7)
C(5)—C(10)	1·526 (7)	C(15)—C(16)	1·550 (7)
C(6)—C(7)	1·555 (7)	C(16)—C(17)	1·572 (7)
C(7)—C(8)	1·528 (7)	C(17)—C(20)	1·464 (7)
C(8)—C(9)	1·538 (6)	C(18)—C(22)	1·518 (7)
C(8)—C(14)	1·534 (6)	C(20)—C(21)	1·189 (8)
C(9)—C(10)	1·549 (6)	O(1*e*)—C(1*e*)	1·421 (9)
C(9)—C(11)	1·546 (6)	C(1*e*)—C(2*e*)	1·44 (2)

C(2)—C(1)—C(10)	113·3 (5)	C(11)—C(12)—C(13)	114·0 (3)
C(1)—C(2)—C(3)	111·6 (6)	C(12)—C(13)—C(14)	108·9 (3)
C(2)—C(3)—C(4)	109·5 (5)	C(12)—C(13)—C(17)	116·0 (3)
C(3)—C(4)—C(5)	126·6 (6)	C(12)—C(13)—C(18)	113·2 (3)
C(4)—C(5)—C(6)	123·1 (5)	C(14)—C(13)—C(17)	98·0 (3)
C(4)—C(5)—C(10)	122·1 (5)	C(14)—C(13)—C(18)	112·6 (3)
C(6)—C(5)—C(10)	114·7 (4)	C(17)—C(13)—C(18)	107·3 (3)
C(5)—C(6)—C(7)	109·7 (4)	C(8)—C(14)—C(13)	114·3 (3)
C(6)—C(7)—C(8)	110·8 (4)	C(8)—C(14)—C(15)	119·6 (4)
C(7)—C(8)—C(9)	110·7 (4)	C(13)—C(14)—C(15)	104·9 (4)
C(7)—C(8)—C(14)	110·8 (3)	C(14)—C(15)—C(16)	103·1 (4)
C(9)—C(8)—C(14)	107·7 (3)	C(15)—C(16)—C(17)	106·6 (4)
C(8)—C(9)—C(10)	113·3 (3)	O(17)—C(17)—C(13)	115·1 (4)
C(8)—C(9)—C(11)	111·5 (3)	O(17)—C(17)—C(16)	107·8 (4)
C(10)—C(9)—C(11)	113·1 (3)	O(17)—C(17)—C(20)	109·9 (4)
C(1)—C(10)—C(5)	112·0 (4)	C(13)—C(17)—C(16)	102·8 (3)
C(1)—C(10)—C(9)	111·2 (4)	C(13)—C(17)—C(20)	111·9 (4)
C(5)—C(10)—C(9)	110·6 (4)	C(16)—C(17)—C(20)	108·9 (4)
C(9)—C(11)—C(12)	111·7 (3)	C(13)—C(18)—C(22)	117·9 (4)
C(9)—C(11)—C(23)	112·2 (3)	C(17)—C(20)—C(21)	176·7 (6)
C(12)—C(11)—C(23)	115·5 (4)	O(1*e*)—C(1*e*)—C(2*e*)	112·7 (7)

Related literature. The biological activity of 11*β*-substituted lynestrenol derivatives has been discussed by van der Broek *et al.* (1977). The steric effect of the angular methyl group at C(11) on the bending of the

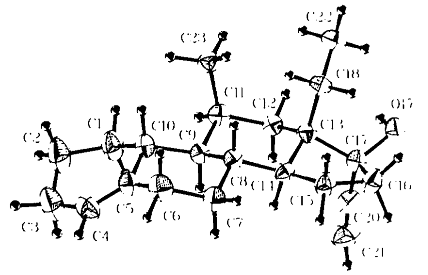

Fig. 1. Thermal-ellipsoid plot of 13-ethyl-11*β*-methyl-18-norlynestrenol with ellipsoids drawn at 40% probability level.

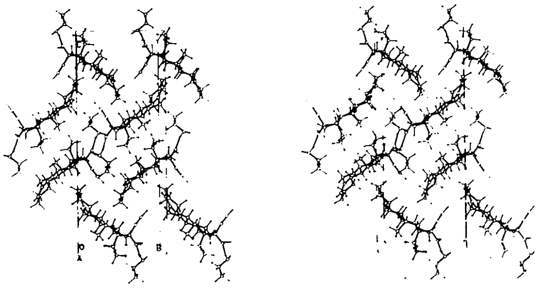

Fig. 2. Stereo packing diagram viewed down **a**.

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* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44464 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

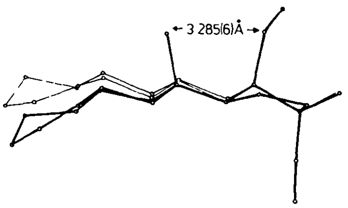

Fig. 3. Superposition of the *D* rings of 13-ethyl-11β-methyl-18-norlynestrenol and lynestrenol (thin lines).

steroid skeleton is illustrated in Fig. 3, which shows a least-squares superposition of the *D* rings of 13-ethyl-11β-methyl-18-norlynestrenol and lynestrenol (Rohrer, Lauffenburger, Duax & Zeelen, 1976). Structural data of several other 4-ene steroids are given by Griffin, Duax & Weeks (1984).

The author thanks A. J. M. Duisenberg for collecting the X-ray data.

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Acta Cryst. (1988). C**44**, 378–379

Structure of 19-Nor-17α-pregna-4,15-dien-20-yn-17β-ol

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(*Received* 14 *July* 1987; *accepted* 14 *October* 1987)

Abstract. $C_{20}H_{26}O$, $M_r = 282{\cdot}42$, monoclinic, $P2_1$, $a = 7{\cdot}152$ (3), $b = 10{\cdot}955$ (6), $c = 10{\cdot}295$ (4) Å, $\beta = 93{\cdot}24$ (3)°, $V = 805{\cdot}3$ (6) Å^3, $Z = 2$, $D_x = 1{\cdot}165$ (1) g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 5$ cm^{-1}, $F(000) = 308$, $T = 293$ K, $R = 0{\cdot}067$ for 1540 observations. The crystal structure is isomorphous to that of lynestrenol (19-nor-17α-pregn-4-en-20-yn-17β-ol) [Rohrer, Lauffenburger, Duax & Zeelen (1976). *Cryst. Struct. Commun.* **5**, 539–542]; a least-squares fit of the steroid backbones [C(1)–C(18)] gave a mean deviation of the fitted atoms of 0·05 Å. The *D* ring has an ideal 13β-envelope conformation $\{\Delta C_s[\mathrm{C}(13)] = 2{\cdot}3\ (5)°\}$, imposed by the Δ^{15} unsaturation. All intermolecular contacts are at normal van der Waals separations.

Experimental. Crystal (0·6 × 0·1 × 0·08 mm) obtained from the Scientific Development Group of Organon, Oss, The Netherlands. Lattice parameters refined by fitting 2θ values of 22 reflections in the range 26–36°. 1611 reflections were measured up to $2\theta_{\max} = 140°$, h, k, $\pm l$ (max. range 8, 13, 12); Enraf–Nonius CAD-4 diffractometer with Ni-filtered Cu $K\alpha$ radiation, ω–2θ scan mode, $\Delta\omega = (0{\cdot}50 + 0{\cdot}15\tan\theta)°$. Standard reflections showed intensity variations less than 2%; Lp corrections, no correction for absorption. 1540 reflections with $I \geq 2{\cdot}5\sigma(I)$ were considered observed.

Initial phases were obtained from the coordinates of the isomorphous crystal structure of lynestrenol and were used to initiate the tangent refinement of *SHELXS*86 (Sheldrick, 1986). H atoms were placed at calculated positions and were refined riding on their bonded atoms, except the hydroxyl-group H atom and the H atom bonded to the ethynyl group, which were located on a difference map and refined positionally. 198 parameters refined on F with full-matrix least squares using *SHELX*76 (Sheldrick, 1976); all non-H atoms refined anisotropically and for H atoms an overall isotropic thermal parameter was varied [$U = 0{\cdot}070$ (3) Å^2]; convergence reached at $R = 0{\cdot}067$ and $wR = 0{\cdot}083$, where $w = 1/\sigma^2(F)$ and $S = 0{\cdot}35$; $\Delta/\sigma = 0{\cdot}01$ (1) (av.) and 0·06 (max.) for non-H-atom

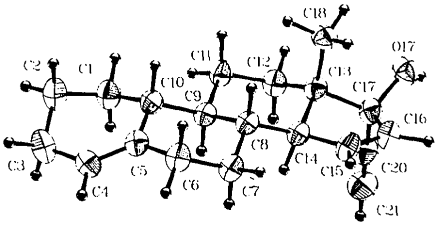

Fig. 1. Thermal-ellipsoid plot of 19-nor-17α-pregna-4,15-dien-20-yn-17β-ol with ellipsoids drawn at 40% probability level.

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