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

O(17)–C(17)	1·444 (5)	C(9)–C(10)	1·550 (3)
C(1)–C(2)	1·528 (4)	C(11)–C(22)	1·329 (5)
C(1)–C(10)	1·543 (4)	C(11)–C(12)	1·513 (3)
C(2)–C(3)	1·525 (6)	C(12)–C(13)	1·527 (4)
C(3)–C(4)	1·497 (4)	C(13)–C(18)	1·544 (5)
C(4)–C(5)	1·331 (5)	C(13)–C(17)	1·550 (3)
C(5)–C(10)	1·511 (5)	C(13)–C(14)	1·532 (5)
C(5)–C(6)	1·506 (4)	C(14)–C(15)	1·543 (4)
C(6)–C(7)	1·525 (3)	C(15)–C(16)	1·547 (5)
C(7)–C(8)	1·532 (5)	C(16)–C(17)	1·547 (5)
C(8)–C(9)	1·556 (3)	C(17)–C(20)	1·477 (5)
C(8)–C(14)	1·526 (3)	C(20)–C(21)	1·172 (5)
C(9)–C(11)	1·520 (3)		

C(2)–C(1)–C(10)	110·3 (2)	C(9)–C(11)–C(12)	114·3 (3)
C(1)–C(2)–C(3)	109·1 (3)	C(11)–C(12)–C(13)	108·7 (2)
C(2)–C(3)–C(4)	110·4 (3)	C(14)–C(13)–C(17)	100·1 (3)
C(3)–C(4)–C(5)	126·0 (4)	C(14)–C(13)–C(18)	112·7 (2)
C(4)–C(5)–C(10)	121·9 (3)	C(12)–C(13)–C(18)	109·4 (3)
C(4)–C(5)–C(6)	121·3 (4)	C(17)–C(13)–C(18)	108·1 (3)
C(6)–C(5)–C(10)	116·8 (3)	C(12)–C(13)–C(14)	108·5 (3)
C(5)–C(6)–C(7)	112·1 (2)	C(12)–C(13)–C(17)	117·9 (2)
C(6)–C(7)–C(8)	110·5 (3)	C(13)–C(14)–C(15)	104·4 (2)
C(7)–C(8)–C(14)	111·5 (2)	C(8)–C(14)–C(13)	114·0 (2)
C(7)–C(8)–C(9)	109·8 (2)	C(8)–C(14)–C(15)	118·7 (2)
C(9)–C(8)–C(14)	108·5 (2)	C(14)–C(15)–C(16)	103·6 (3)
C(8)–C(9)–C(11)	108·6 (2)	C(15)–C(16)–C(17)	106·8 (2)
C(10)–C(9)–C(11)	114·8 (2)	C(16)–C(17)–C(20)	110·6 (3)
C(8)–C(9)–C(10)	112·6 (2)	C(13)–C(17)–C(20)	111·3 (3)
C(1)–C(10)–C(5)	109·9 (3)	C(13)–C(17)–C(16)	103·7 (2)
C(1)–C(10)–C(9)	111·3 (2)	O(17)–(C17)–C(20)	108·6 (3)
C(5)–C(10)–C(9)	112·2 (2)	O(17)–C(17)–C(13)	111·8 (3)
C(9)–C(11)–C(22)	125·4 (2)	O(17)–C(17)–C(16)	110·7 (3)
C(12)–C(11)–C(22)	120·1 (3)	C(17)–C(20)–C(21)	178·5 (4)

molecular conformation and the atom-numbering scheme.

Related literature. Other isomorphous Δ^4 steroid structures are 11β-fluorolynestrenol (Rohrer, Duax & Zeelen, 1978) and lynestrenol (Rohrer, Lauffenburger, Duax & Zeelen, 1976). Related structures are reviewed by Griffin, Duax & Weeks (1984). A structure–activity

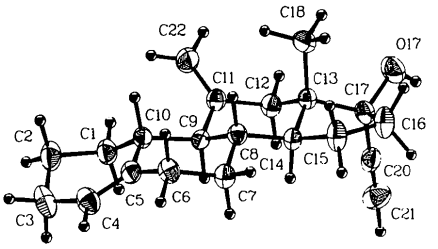

Fig. 1. Thermal-ellipsoid plot of 11-methylenelynestrenol with ellipsoids drawn at the 40% probability level.

study of 11β-substituted lynestrenol derivatives has been published by van der Broek *et al.* (1977).

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

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Acta Cryst. (1987). C**43**, 2233–2235

Structure of the Methanol Solvate of 11β-Chloro-13-ethyl-18-norlynestrenol

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(*Received* 7 *May* 1987; *accepted* 2 *June* 1987)

Abstract. 11β-Chloro-13-ethyl-18,19-dinor-17α-pregn-4-en-20-yn-17β-ol methanol solvate, $C_{21}H_{29}ClO.CH_4O$, $M_r = 364{\cdot}95$, monoclinic, $P2_1$, $a = 10{\cdot}234$ (2), $b = 7{\cdot}752$ (1), $c = 13{\cdot}225$ (3) Å, $\beta = 93{\cdot}62$ (1)°, $V = 1047{\cdot}1$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}157$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 1·9 cm^{-1}, $F(000) = 396$, $T = 293$ K, $R = 0{\cdot}049$ for 2532 observations. The molecular conformation is identical to that of 11β-chlorolynestrenol [Rohrer, Lauffenburger, Duax & Zeelen (1977). *Cryst. Struct. Commun.* **6**, 377–380]; a least-squares fit of C(1)–C(17) of both structures gave an r.m.s. deviation of the fitted atoms of 0·05 Å. The

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Table 1. *Positional and equivalent isotropic thermal parameters* (Å²) *for non-*H *atoms with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22}\sin^2\beta + U_{33} + 2U_{13}\cos\beta)/3\sin^2\beta$.

	x	y	z	U_{eq}
Cl	0·05315 (8)	0·5000 (*)	0·67000 (6)	0·0559 (3)
O(17)	−0·4296 (2)	0·2202 (3)	0·5588 (1)	0·0517 (6)
C(1)	0·1599 (3)	0·4425 (5)	0·9313 (3)	0·065 (1)
C(2)	0·2878 (4)	0·4473 (6)	0·9972 (3)	0·083 (2)
C(3)	0·3027 (4)	0·2851 (7)	1·0611 (3)	0·092 (2)
C(4)	0·2741 (3)	0·1282 (6)	0·9995 (3)	0·066 (1)
C(5)	0·2114 (3)	0·1262 (5)	0·9080 (2)	0·049 (1)
C(6)	0·2009 (3)	−0·0332 (5)	0·8443 (3)	0·058 (1)
C(7)	0·0584 (3)	−0·0643 (4)	0·8025 (2)	0·050 (1)
C(8)	0·0032 (3)	0·0930 (4)	0·7463 (2)	0·0396 (8)
C(9)	0·135 (3)	0·2537 (4)	0·8143 (2)	0·0392 (8)
C(10)	0·1552 (3)	0·2879 (4)	0·8587 (2)	0·044 (1)
C(11)	−0·0509 (3)	0·4123 (4)	0·7647 (2)	0·040 (1)
C(12)	−0·1914 (3)	0·3815 (4)	0·7189 (2)	0·0396 (8)
C(13)	−0·2062 (3)	0·2195 (4)	0·6534 (2)	0·0365 (8)
C(14)	−0·1413 (3)	0·0676 (4)	0·7127 (2)	0·0391 (6)
C(15)	−0·1822 (3)	−0·0926 (4)	0·6508 (3)	0·053 (1)
C(16)	−0·3232 (3)	−0·0485 (4)	0·6099 (3)	0·057 (1)
C(17)	−0·3481 (3)	0·1425 (4)	0·6382 (2)	0·045 (1)
C(18)	−0·1520 (3)	0·2417 (4)	0·5466 (2)	0·048 (1)
C(20)	−0·4193 (3)	0·1517 (5)	0·7324 (2)	0·051 (1)
C(21)	−0·4785 (3)	0·1559 (6)	0·8040 (3)	0·073 (1)
C(22)	−0·1899 (3)	0·4062 (5)	0·4879 (2)	0·065 (1)
O(*m*)	−0·4902 (2)	0·5474 (3)	0·6158 (2)	0·066 (1)
C(*m*)	−0·5722 (4)	0·5903 (7)	0·6932 (3)	0·089 (2)

* Kept fixed during the refinement.

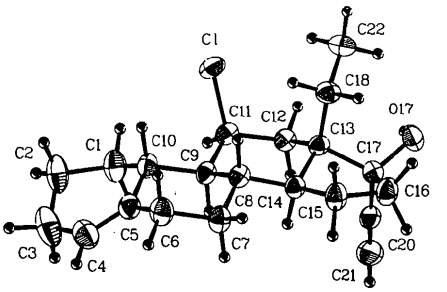

Fig. 1. Thermal-ellipsoid plot of 11β-chloro-13-ethyl-18-norlynestrenol with ellipsoids drawn at the 40% probability level.

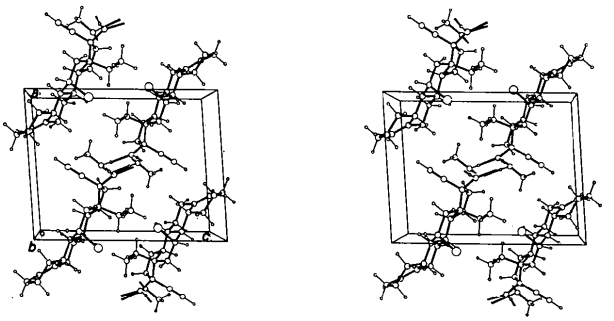

Fig. 2. Stereo packing diagram viewed down **b**.

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

Cl–C(11)	1·826 (3)	C(9)–C(10)	1·553 (4)
O(17)–C(17)	1·432 (3)	C(11)–C(12)	1·543 (4)
C(1)–C(2)	1·527 (5)	C(12)–C(13)	1·528 (4)
C(1)–C(10)	1·534 (5)	C(13)–C(14)	1·542 (4)
C(2)–C(3)	1·517 (7)	C(13)–C(17)	1·571 (4)
C(3)–C(4)	1·483 (7)	C(13)–C(18)	1·559 (4)
C(4)–C(5)	1·333 (5)	C(14)–C(15)	1·531 (4)
C(5)–C(6)	1·496 (5)	C(15)–C(16)	1·547 (5)
C(5)–C(10)	1·510 (5)	C(16)–C(17)	1·552 (4)
C(6)–C(7)	1·546 (4)	C(17)–C(20)	1·484 (4)
C(7)–C(8)	1·518 (4)	C(18)–C(22)	1·530 (5)
C(8)–C(9)	1·536 (4)	C(20)–C(21)	1·156 (5)
C(8)–C(14)	1·530 (4)	O(*m*)–C(*m*)	1·404 (5)
C(9)–C(11)	1·524 (4)		

C(2)–C(1)–C(10)	111·6 (3)	C(11)–C(12)–C(13)	114·0 (3)
C(1)–C(2)–C(3)	110·5 (3)	C(12)–C(13)–C(14)	108·4 (2)
C(2)–C(3)–C(4)	111·4 (3)	C(12)–C(13)–C(17)	116·3 (2)
C(3)–C(4)–C(5)	125·1 (4)	C(12)–C(13)–C(18)	113·2 (2)
C(6)–C(5)–C(10)	115·5 (3)	C(14)–C(13)–C(17)	98·0 (2)
C(4)–C(5)–C(6)	122·3 (4)	C(14)–C(13)–C(18)	112·2 (2)
C(4)–C(5)–C(10)	122·0 (3)	C(17)–C(13)–C(18)	107·7 (2)
C(5)–C(6)–C(7)	111·3 (3)	C(8)–C(14)–C(13)	115·3 (3)
C(6)–C(7)–C(8)	111·3 (3)	C(8)–C(14)–C(15)	119·1 (3)
C(7)–C(8)–C(9)	110·8 (2)	C(13)–C(14)–C(15)	104·7 (2)
C(9)–C(8)–C(14)	107·7 (2)	C(14)–C(15)–C(16)	103·0 (2)
C(7)–C(8)–C(14)	111·2 (3)	C(15)–C(16)–C(17)	106·9 (3)
C(8)–C(9)–C(11)	113·0 (2)	C(13)–C(17)–C(16)	103·2 (2)
C(10)–C(9)–C(11)	113·2 (3)	O(17)–C(17)–C(13)	115·1 (2)
C(8)–C(9)–C(10)	112·8 (2)	C(13)–C(17)–C(20)	111·9 (2)
C(1)–C(10)–C(5)	112·5 (2)	O(17)–C(17)–C(16)	108·7 (2)
C(1)–C(10)–C(9)	111·2 (2)	C(16)–C(17)–C(20)	110·2 (3)
C(5)–C(10)–C(9)	110·0 (3)	O(17)–C(17)–C(20)	107·5 (2)
Cl–C(11)–C(9)	109·9 (2)	C(13)–C(18)–C(22)	117·3 (2)
Cl–C(11)–C(12)	111·0 (2)	C(17)–C(20)–C(21)	177·6 (4)

ethyl group is in the preferred *trans* position relative to the *C/D* ring junction [van Geerestein, Kanters, Duisenberg & Kroon (1986). *Acta Cryst.* C**42**, 469–472], with C(14)–C(13)–C(18)–C(22) = −169·3 (3)°. Infinite chains of hydrogen-bonded steroid and methanol molecules are formed parallel to **b**; →O(17)→O(*m*)→O(17′)(−1−x, $\frac{1}{2}+y$, 1−z)→ with O(17)···O(*m*) = 2·729 (3) Å, O(*m*)···O(17′) = 2·750 (3) Å, O(17)–H···O(*m*) = 172 (3)° and O(*m*)–H···O(17′) = 170 (3)°. Other intermolecular contacts are at normal van der Waals separations.

Experimental. Crystal (1·0 × 0·32 × 0·25 mm) obtained from the Scientific Development Group of Organon, Oss, The Netherlands. Lattice parameters refined by fitting 2θ values of 21 reflections in the range of 6–34°. 3258 reflections measured up to $2\theta_{max} = 60°$, $h,k,\pm l$ (max. range 14, 10, 18); Enraf–Nonius CAD-4 diffractometer with Zr-filtered Mo $K\alpha$ radiation, ω–2θ scan mode, $\Delta\omega = (0{\cdot}60 + 0{\cdot}35\tan\theta)°$. Standard reflections showed intensity variations less than 2%; Lp corrections, no correction for absorption. 2532 reflections with $I \geq 2{\cdot}5\sigma(I)$ were considered observed. The structure was solved by automatic heavy-atom methods using a preliminary version of the *SHELXS*86 program (Sheldrick, 1986). Tangent expansion followed by peak optimization gave an E map revealing all C and O

atoms. H atoms were placed on 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. 235 parameters were 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 = 0·082 (2) Å^2]; convergence at $R = 0{\cdot}049$ and $wR = 0{\cdot}044$, where $w = 1/\sigma^2(F)$ and $S = 0{\cdot}4$; $\Delta/\sigma =$ 0·01 (1) (av.) and 0·06 (max.) for non-H-atom parameters and $\Delta/\sigma = 0{\cdot}09$ (7) (av.) and 0·19 (max.) for H-atom parameters; final electron density within $\Delta\rho = \pm 0{\cdot}4$ e Å^{-3}. Scattering factors from *SHELX*76. Final atomic parameters are given in Table 1, and bond lengths and angles in Table 2.* Fig. 1 shows the molecular conformation and the atom-numbering scheme. The packing and hydrogen bonding is illustrated in Fig. 2, which shows a stereoview down **b**.

* 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 44117 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Structural data of several other Δ^4 steroids have been reported in literature, *e.g.* lynestrenol (Rohrer, Lauffenburger, Duax & Zeelen, 1976) and have been reviewed by Griffin, Duax & Weeks (1984). A study concerning the biological activity of 11β-substituted lynestrenol derivatives has been published by van der Broek *et al.* (1977).

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

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Acta Cryst. (1987). C**43**, 2235–2236

Diphenylphosphino-*N*-methylthioformamid

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(*Eingegangen am* 1. *Juli* 1985; *angenommen am* 17. *Juni* 1987)

Abstract. $C_{14}H_{14}NPS$, $M_r = 259{\cdot}31$, monoclinic, $P2_1/n$, $a = 16{\cdot}882$ (2), $b = 9{\cdot}786$ (2), $c = 17{\cdot}128$ (2) Å, $\beta = 107{\cdot}66$ (2)°, $V = 2696{\cdot}3$ Å^3, $Z = 8$, $D_x = 1{\cdot}278$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 30{\cdot}428$ cm^{-1}, $F(000) = 1088$, $T = 293$ K, $R = 0{\cdot}065$ for 2389 observed reflections. In the asymmetric unit are two independent molecules with the same configuration but different conformation of the phenyl rings.

Experimentelles. Nadelförmige Einkristalle, 0,15 × 0,20 × 0,50 mm; monokline Raumgruppe $P2_1/n$ aus Buerger-Präzessionsaufnahmen, Verfeinerung der Gitterkonstanten auf dem Enraf–Nonius Vierkreisdiffraktometer CAD-4 anhand von 25 Reflexen hoher Beugungswinkel ($15 < \theta < 26°$); Cu $K\alpha$-Strahlung, Graphitmonochromator; mit ω/θ scan 5175 Reflexe gemessen ($\theta < 52°$), $h + 17$, $k \pm 9$, $l \pm 17$; drei Kontrollreflexe mit keiner signifikanten Änderung der Intensität; empirische Absorptionskorrektur (Walker & Stuart, 1983), Mittelung ($R_{int} = 0{,}028$) ergab 3009 symmetrieunabhängige Reflexe, davon 2389 mit $I > 3\sigma(I)$. Lösung der Struktur mit direkten Methoden (Sheldrick, 1984). H-Atome der Phenylgruppen in berechneten Positionen mit d(C—H) = 0,95 Å, die anderen Wasserstoffpositionen wurden einer Differenzfouriersynthese entnommen; alle Atome (außer H) mit anisotropen Temperaturparametern, H-Atomlagen der NH-Gruppe mit individuellen isotropen Temperaturfaktoren verfeinert, H-Atome der Phenyl- und Methylgruppe nur in die Strukturfaktorrechnung einbezogen; Full-matrix Verfeinerung (F) (Frenz, 1978; Version 1984) führte zu $R = 0{,}065$, $wR = 0{,}078$; 316 Parameter, $w^{-1} = \sigma^2(F)$, $\sum w|\Delta F|^2$ minimalisiert, $(\Delta/\sigma)_{max} = 0{,}01$, $\Delta\rho < |0{,}53|$ e Å^{-3}; $S = 2{,}76$. Atom-

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