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Structure of 11-Methylenelynestrenol

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Abstract. 11-Methylene-19-nor-17α-pregn-4-en-20-yn-17β-ol, $C_{21}H_{28}O$, $M_r = 296{\cdot}45$, monoclinic, $P2_1$, $a = 7{\cdot}143$ (1), $b = 11{\cdot}537$ (2), $c = 10{\cdot}353$ (1) Å, $\beta = 94{\cdot}44$ (1)°, $V = 850{\cdot}6$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}157$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 0·6 cm^{-1}, $F(000) = 324$, $T = 293$ K, $R = 0{\cdot}045$ for 1832 observed reflections. The structure is isomorphous with that of 13-ethyl-11-methylene-28-norlynestrenol [van Soest, van Dijck & Zeelen (1980). *Recl Trav. Chim. Pays Bas*, **99**, 323–325]; a least-squares fit of C(1)–C(22) of both molecules gave an r.m.s. deviation of the fitted atoms of 0·05 Å. All intermolecular contacts are at normal van der Waals separations.

Experimental. Crystal (0·9 × 0·5 × 0·15 mm) obtained from the Scientific Development Group of Organon, Oss, The Netherlands. Lattice parameters refined by *SET*4 method (de Boer & Duisenberg, 1984) from eight reflections in the 2θ range 30–35°. 2354 integrated intensities measured up to $2\theta_{max} = 60°$, $h,k,\pm l$ (max. range 9, 15, 14); Enraf–Nonius CAD-4 diffractometer with Zr-filtered Mo $K\alpha$ radiation, ω–2θ scan mode, $\Delta\omega = (0{\cdot}70 + 0{\cdot}35\tan\theta)°$. Standard reflections showed intensity variations less than 1%; Lp corrections, no correction for absorption. 1833 reflections with $I \geq 2{\cdot}5\sigma(I)$ were considered observed. Initial phases calculated with the coordinates of 13-ethyl-11-methylene-18-norlynestrenol (van Soest, van Dijck & Zeelen, 1980) and refined by tangent recycling with the *SHELXS*86 program (Sheldrick, 1986). H atoms were placed on calculated positions 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. 211 parameters were refined on F with full-matrix least squares using *SHELX*76 (Sheldrick, 1976); reflection $\bar{1}\bar{1}1$ showed severe extinction and was excluded. All non-H atoms were refined anisotropically and for H atoms an overall isotropic thermal parameter was varied [$U = 0{\cdot}067$ (2) Å^2]; convergence at $R = 0{\cdot}047$ and $wR = 0{\cdot}038$, where $w = 1/\sigma^2(F)$ and $S = 0{\cdot}4$; $\Delta/\sigma = 0{\cdot}02$ (2) (av.) and 0·1 (max.) for non-H atom parameters and $\Delta/\sigma = 0{\cdot}1$ (2) (av.) and 0·4 (max.) for H-atom parameters; final electron density within $\Delta\rho = \pm 0{\cdot}2$ 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

* 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 44116 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

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

	x	y	z	U_{eq}
O(17)	0·2018 (3)	0·6283 (3)	0·3277 (2)	0·0631 (6)
C(1)	0·8711 (4)	0·8643 (4)	0·8872 (2)	0·048 (1)
C(2)	0·9476 (4)	0·9010 (4)	1·0230 (3)	0·062 (1)
C(3)	0·9116 (4)	1·0302 (4)	1·0402 (3)	0·070 (1)
C(4)	0·7146 (4)	1·0601 (4)	0·9918 (3)	0·057 (1)
C(5)	0·5964 (4)	0·9920 (4)	0·9210 (2)	0·041 (1)
C(6)	0·3942 (4)	1·0259 (4)	0·8904 (2)	0·046 (1)
C(7)	0·3298 (4)	1·0063 (3)	0·7481 (2)	0·043 (1)
C(8)	0·3629 (3)	0·8801 (3)	0·7098 (2)	0·0363 (8)
C(9)	0·5755 (3)	0·8505 (*)	0·7324 (2)	0·0327 (8)
C(10)	0·6551 (3)	0·8747 (3)	0·8734 (2)	0·0378 (8)
C(11)	0·6062 (4)	0·7277 (3)	0·6851 (2)	0·038 (1)
C(12)	0·5442 (4)	0·7083 (4)	0·5437 (2)	0·043 (1)
C(13)	0·3345 (4)	0·7343 (3)	0·5223 (2)	0·037 (1)
C(14)	0·3005 (3)	0·8584 (3)	0·5675 (2)	0·0363 (8)
C(15)	0·0947 (4)	0·8838 (4)	0·5193 (3)	0·056 (1)
C(16)	0·0662 (4)	0·8137 (4)	0·3919 (3)	0·057 (1)
C(17)	0·2468 (4)	0·7416 (4)	0·3808 (2)	0·046 (1)
C(18)	0·2234 (4)	0·6435 (4)	0·5948 (3)	0·055 (1)
C(20)	0·3741 (5)	0·8005 (4)	0·2962 (3)	0·047 (1)
C(21)	0·4727 (5)	0·8463 (4)	0·2266 (3)	0·066 (1)
C(22)	0·6693 (4)	0·6395 (4)	0·7587 (3)	0·057 (1)

* Kept fixed during the refinement.

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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

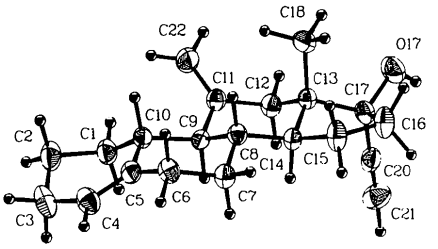

Fig. 1. Thermal-ellipsoid plot of 11-methylenelynestrenol with ellipsoids drawn at the 40% probability level.

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

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

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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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