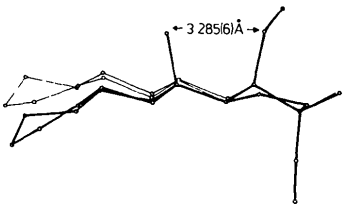


Fig. 3. Superposition of the *D* rings of 13-ethyl-11β-methyl-18-norlynestrenol and lynestrenol (thin lines).

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).

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### References

BROEK, A. J. VAN DER, BROESS, A. I. A., VAN DER HEUVEL, M. J., DE JONGH, H. P., LEEMHUIS, J., SCHÖNEMANN, K., SMITS, J., DE VISSER, J., VAN VLIET, N. P. & ZEELEN, F. J. (1977). *Steroids*, **30**, 481–510.

EGERT, E. & SHELDRICK, G. M. (1985). *Acta Cryst.* A**41**, 262–268.

GEERESTEIN, V. J. VAN, KANTERS, J., DUISENBERG, A. J. M. & KROON, J. (1986). *Acta Cryst.* C**42**, 469–472.

GRIFFIN, J. F., DUAX, W. L. & WEEKS, C. M. (1984). *Atlas of Steroid Structure*, Vol. II. New York: Ronald Press.

ROHRER, D. C., HAZEL, D. C., DUAX, W. L. & ZEELEN, F. J. (1976). *Cryst. Struct. Commun.* **5**, 543–546.

ROHRER, D. C., LAUFFENBURGER, J. C., DUAX, W. L. & ZEELEN, F. J. (1976). *Cryst. Struct. Commun.* **5**, 539–542.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

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

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**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$^{-3}$, $\lambda$(Cu $K\alpha$) = 1·5418 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 {$\Delta C_s$[C(13)] = 2·3 (5)°}, imposed by the $\Delta^{15}$ 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 $2\theta$ 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, $\omega$–$2\theta$ 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

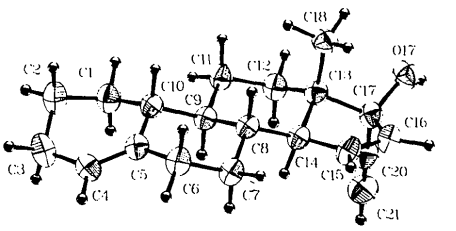


Fig. 1. Thermal-ellipsoid plot of 19-nor-17α-pregna-4,15-dien-20-yn-17β-ol with ellipsoids drawn at 40% probability level.

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

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

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| O(17) | 0·1705 (4) | 0·6398 (5) | 0·3349 (3) | 0·067 (1) |
| C(1) | 0·8660 (5) | 0·8343 (6) | 0·8954 (4) | 0·060 (1) |
| C(2) | 0·9486 (6) | 0·8709 (7) | 1·0287 (4) | 0·070 (2) |
| C(3) | 0·9330 (6) | 1·0089 (6) | 1·0465 (5) | 0·071 (2) |
| C(4) | 0·7413 (5) | 1·0531 (6) | 1·0055 (3) | 0·056 (1) |
| C(5) | 0·6137 (5) | 0·9875 (5) | 0·9350 (3) | 0·048 (1) |
| C(6) | 0·4156 (5) | 1·0315 (6) | 0·9122 (3) | 0·055 (1) |
| C(7) | 0·3394 (5) | 1·0162 (5) | 0·7717 (3) | 0·052 (1) |
| C(8) | 0·3691 (5) | 0·8852 (5) | 0·7243 (3) | 0·044 (1) |
| C(9) | 0·5789 (5) | 0·8522* | 0·7376 (3) | 0·043 (1) |
| C(10) | 0·6565 (5) | 0·8641 (5) | 0·8808 (3) | 0·046 (1) |
| C(11) | 0·6215 (6) | 0·7272 (5) | 0·6798 (4) | 0·054 (1) |
| C(12) | 0·5407 (5) | 0·7108 (5) | 0·5396 (3) | 0·054 (1) |
| C(13) | 0·3332 (5) | 0·7386 (5) | 0·5288 (3) | 0·047 (1) |
| C(14) | 0·3046 (5) | 0·8696 (5) | 0·5817 (3) | 0·046 (1) |
| C(15) | 0·1090 (5) | 0·9003 (6) | 0·5287 (4) | 0·057 (1) |
| C(16) | 0·0713 (5) | 0·8370 (6) | 0·4205 (4) | 0·058 (1) |
| C(17) | 0·2336 (5) | 0·7542 (5) | 0·3903 (3) | 0·052 (1) |
| C(18) | 0·2240 (6) | 0·6420 (6) | 0·6020 (4) | 0·060 (1) |
| C(20) | 0·3567 (6) | 0·8160 (6) | 0·3005 (3) | 0·056 (1) |
| C(21) | 0·4527 (8) | 0·8678 (7) | 0·2292 (4) | 0·074 (2) |

* Kept fixed during refinement.

parameters and $\Delta/\sigma = 0{\cdot}09$ (7) (av.) and 0·2 (max.) for H-atom parameters; $-0{\cdot}3 < \Delta\rho < 0{\cdot}2$ e Å$^{-3}$. Scattering factors used from *SHELX*. Final atomic parameters and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths and bond angles are given in Table 2. Fig. 1 shows the molecular conformation and the atom-numbering scheme.

**Related literature.** Structural data of several other 4-ene steroids have been reviewed by Griffin, Duax & Weeks (1984). A receptor binding study of 15-ene steroids has been reported by Bergink & Kloosterboer (1985).

* 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 44465 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

| | | | |
|---|---|---|---|
| O(17)–C(17) | 1·439 (7) | C(9)–C(10) | 1·551 (4) |
| C(1)–C(2) | 1·517 (6) | C(9)–C(11) | 1·531 (5) |
| C(1)–C(10) | 1·533 (5) | C(11)–C(12) | 1·535 (5) |
| C(2)–C(3) | 1·53 (1) | C(12)–C(13) | 1·513 (5) |
| C(3)–C(4) | 1·492 (6) | C(13)–C(14) | 1·553 (7) |
| C(4)–C(5) | 1·342 (6) | C(13)–C(17) | 1·567 (5) |
| C(5)–C(6) | 1·502 (6) | C(13)–C(18) | 1·538 (7) |
| C(5)–C(10) | 1·501 (7) | C(14)–C(15) | 1·510 (5) |
| C(6)–C(7) | 1·526 (5) | C(15)–C(16) | 1·327 (7) |
| C(7)–C(8) | 1·534 (7) | C(16)–C(17) | 1·519 (7) |
| C(8)–C(9) | 1·542 (5) | C(17)–C(20) | 1·477 (6) |
| C(8)–C(14) | 1·523 (4) | C(20)–C(21) | 1·179 (7) |
| C(2)–C(1)–C(10) | 111·2 (4) | C(11)–C(12)–C(13) | 111·2 (3) |
| C(1)–C(2)–C(3) | 110·0 (5) | C(12)–C(13)–C(14) | 108·0 (4) |
| C(2)–C(3)–C(4) | 111·1 (4) | C(12)–C(13)–C(17) | 118·8 (3) |
| C(3)–C(4)–C(5) | 124·4 (5) | C(12)–C(13)–C(18) | 110·4 (4) |
| C(4)–C(5)–C(6) | 121·3 (5) | C(14)–C(13)–C(17) | 99·0 (4) |
| C(4)–C(5)–C(10) | 122·5 (4) | C(14)–C(13)–C(18) | 112·6 (3) |
| C(6)–C(5)–C(10) | 116·2 (4) | C(17)–C(13)–C(18) | 107·7 (3) |
| C(5)–C(6)–C(7) | 113·3 (3) | C(8)–C(14)–C(13) | 113·8 (4) |
| C(6)–C(7)–C(8) | 110·8 (4) | C(8)–C(14)–C(15) | 123·2 (3) |
| C(7)–C(8)–C(9) | 110·0 (3) | C(13)–C(14)–C(15) | 102·5 (4) |
| C(7)–C(8)–C(14) | 111·8 (4) | C(14)–C(15)–C(16) | 109·1 (4) |
| C(8)–C(9)–C(10) | 111·0 (3) | C(15)–C(16)–C(17) | 111·3 (3) |
| C(9)–C(8)–C(14) | 107·4 (3) | O(17)–C(17)–C(13) | 112·5 (4) |
| C(8)–C(9)–C(11) | 112·9 (3) | O(17)–C(17)–C(16) | 112·0 (3) |
| C(10)–C(9)–C(11) | 112·1 (3) | O(17)–C(17)–C(20) | 109·6 (3) |
| C(1)–C(10)–C(5) | 112·0 (4) | C(13)–C(17)–C(16) | 101·1 (3) |
| C(1)–C(10)–C(9) | 111·8 (3) | C(13)–C(17)–C(20) | 111·3 (3) |
| C(5)–C(10)–C(9) | 111·1 (3) | C(16)–C(17)–C(20) | 110·1 (4) |
| C(9)–C(11)–C(12) | 113·4 (4) | C(17)–C(20)–C(21) | 178·5 (7) |

We thank A. J. M. Duisenberg for collecting the X-ray data.

### References

BERGINK, E. W. & KLOOSTERBOER, H. J. (1985). *Adv. Contraception*, **1**, 256–257.

GRIFFIN, J. F., DUAX, W. L. & WEEKS, C. M. (1984). *Atlas of Steroid Structure*, Vol. II. New York: Ronald Press.

ROHRER, D. C., LAUFFENBURGER, J. C., DUAX, W. L. & ZEELEN, F. J. (1976). *Cryst. Struct. Commun.* **5**, 539–542.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1986). *SHELXS*86. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.

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# Structure of 17$\beta$-Hydroxy-11-methylene-19-nor-17$\alpha$-pregna-4,15-dien-20-yn-3-one

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

**Abstract.** $C_{21}H_{24}O_2$, $M_r = 308{\cdot}42$, monoclinic, $P2_1$, $a = 8{\cdot}190$ (2), $b = 11{\cdot}705$ (2), $c = 9{\cdot}326$ (3) Å, $\beta = 98{\cdot}56$ (2)°, $V = 875{\cdot}5$ (3) Å$^3$, $Z = 2$, $D_x = 1{\cdot}170$ g cm$^{-3}$, $\lambda$(Cu $K\alpha$) = 1·54184 Å, $\mu = 5$ cm$^{-1}$, $F(000) = 332$, $T = 293$ K, $R = 0{\cdot}067$ for 1682 observations. The $A$ ring has a 1$\alpha$-sofa conformation

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