The Structure of 11β,18-Epoxy-19-nor-17α-pregn-4-en-20-yn-17β-ol (11β,18-Epoxylynestrenol), C20H26O2

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Abstract. $M_r = 298.44$, monoclinic, $P2_1$, $a = 7.584$ (4), $b = 20.644$ (5), $c = 10.497$ (3) Å, $\beta = 92.84$ (4)°, $V = 1641$ (1) Å^3, $Z = 4$, $D_m = 1.20$ (flotation), $D_x = 1.208$ (1) Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, μ(Cu $K\alpha$) = 5.16 cm^{-1}, $F(000) = 648$, $T = 294$ K, $R = 0.070$ for 2796 unique reflections. The crystal used for analysis was twinned by reticular pseudo-merohedry with twin index 7 and twin fraction 0.36 (3). The two independent molecules have nearly the same geometries. The progestational activity of the title compound is much lower than that of other 11β derivatives of lynestrenol which have bent conformations in accordance with the determined correlation between progestational activity and bending of the steroid skeleton. The 11β,18-epoxy bridge exerts a large steric strain on the *D* ring of the steroid, giving rise to an unusual 13β,17α-half-chair conformation for this ring and resulting in a less-bent overall conformation of the steroid backbone compared with lynestrenol and 11β derivatives without the 11β, 18-epoxy bridge. The inferior progestational activity of the epoxy derivative is in accordance with the assumed correlation between activity and degree of flexure.

Introduction. For a series of 11β-substituted lynestrenol derivatives a correlation has been proposed between progestational activity and steric size of the 11β-substituent (van den Broek *et al.*, 1977). The variation in activity seems related to changes in overall shape of the steroid backbone, caused by 1,3-diaxial interaction of the 11β-substituent with the angular 18-methyl group, rather than to direct interaction of the 11β-substituent with the receptor site (van Soest, van Dijck & Zeelen, 1980). The 11β,18-epoxy derivative (*a*), which should serve to distinguish between these two effects, was found to have less progestational activity than lynestrenol (*b*) (van den Broek *et al.*, 1977). As compounds with a higher activity have bent conformations (Rohrer, Hazel, Duax & Zeelen, 1976; Rohrer, Lauffenburger, Duax & Zeelen, 1977; Rohrer, Duax & Zeelen, 1978), we were interested to see whether the inferior activity of the epoxy derivative could be explained in terms of a less-bent shape of the molecule.

(a)

(b)
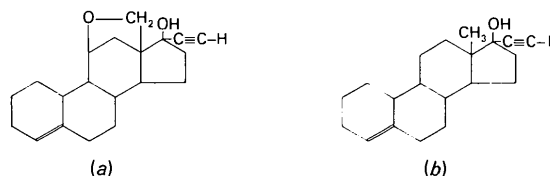
Experimental. Sample obtained through the Scientific Development Group of Organon, Oss, The Netherlands; Weissenberg photographs of crystals mounted along **a** showed systematic extinction of 0*k*0 reflections with $k = 2n + 1$; oscillation photographs around *a* axis showed no symmetry, indicating $P2_1$; Weissenberg photographs showed crystals to be twinned, with a twin fraction of about 2:3 and partially coinciding reciprocal lattices; major individual crystal 0·3 (along **a**) × 0·15 × 0·05 mm, Enraf–Nonius CAD-4 automatic diffractometer, Ni-filtered Cu $K\alpha$ radiation; lattice dimensions determined by least squares of 48 reflections, ω–2θ scan mode, $\Delta\omega = (0.75 + 0.14 \tan \theta)°$, with $\sigma(I)/I < 1\%$ or a maximum scan time of 100s; horizontal and vertical slit widths 1 and 0·3°, respectively (as seen from the crystal), ψ azimuth chosen such that reflection spots with the least separation were separated in height as much as possible; in one half of the reflection sphere 5886 reflections measured up to $\theta = 70°$, $hk \pm l$ (max. range 9,25,12), 3203 independent reflections, 2796 with $I \geq 2.5\sigma(I)$ used for structure determination; one standard reflection, no significant changes, Lp corrections, no correction for absorption, overlapping reflections corrected for twinning, as described below.

Twinning. Crystals of 11β,18-epoxylynestrenol are twinned by reticular pseudo-merohedry with twin index 7, so that in layers with $h(\bmod 7) = 0$ the reciprocal-lattice points of the twins coincide. An ideal interpretation of this pattern, which is illustrated in Fig. 1, implies a geometric relation which can be expressed in direct-cell parameters as $a + 14c\cos \beta = 0$. From the measured cell parameters and their e.s.d.'s an experimental value for this quantity is calculated to be 0.3 ± 0.1 Å, so the twin condition is not exactly

obeyed. However, experimental conditions did not permit satisfactory resolution of reflections with $h(\text{mod } 7) = 0$. The twin law can equally well be described by a twofold rotation around **a** or by a twofold rotation around **c***, both resulting in an overlap of the $(hkl)_1$ and $(h\bar{k}\bar{l}-h/7)_2$ reflections,† where the subscript refers to the diffracting individual. As the molecule is chiral, a mirror plane cannot be considered as a twin law. No separate twin areas could be observed with a polarizing microscope, so it was not possible to discriminate between these two rotations. Assuming that the individuals independently contribute to each measured intensity I_{hkl}:

$$I_{hkl} = J_{hkl} + KJ_{h\bar{k}\bar{l}-h/7}$$

$$I_{h\bar{k}\bar{l}-h/7} = J_{h\bar{k}\bar{l}-h/7} + KJ_{hkl},$$

where $K = \alpha/(1-\alpha)$ (α is the fraction of the smaller individual) and J_{hkl} and $J_{h\bar{k}\bar{l}-h/7}$ are the intensities which an untwinned crystal with the volume of the larger twin would give. These equations can be solved to yield the unknown J_{hkl}:

$$J_{hkl} = (I_{hkl} - KI_{h\bar{k}\bar{l}-h/7})/(1-K^2).$$

When Friedel's law $(I_{hkl} = I_{\bar{h}\bar{k}\bar{l}})$ is applied for reflections with $h = 0$, this reduces to

$$J_{0kl} = (1-\alpha)I_{0kl}.$$

The constant K can be calculated by collecting for each individual a limited amount of data from layers with $h(\text{mod } 7)\neq 0$. For that purpose 200 intensities of 61 unique reflections for both twins were collected in layers with $h = 3$ and 4, where the peak separation is maximal. For the crystal used in this analysis the factor K was found to be $0\cdot57$ (4), yielding $\alpha = 0\cdot36$ (3).

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† Actually, twofold rotation around **c*** gives overlap with the $(hkl + h/7)_2$ reflection, but for a monoclinic system $I_{hkl} = I_{\bar{h}k\bar{l}}$.
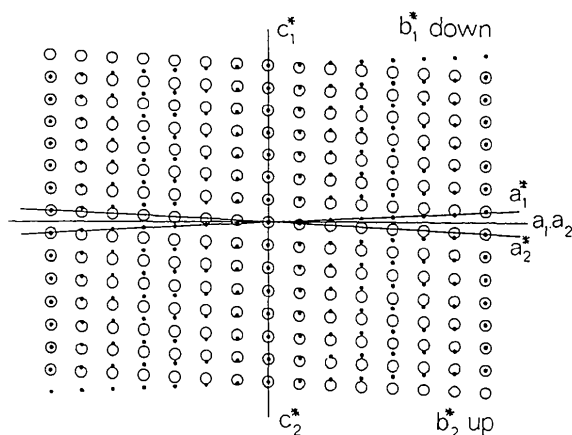

Fig. 1. The superimposed **a*c*** nets of twinned 11β,18-epoxylynestrenol. Points refer to individual 1 and circles to individual 2.

Structure determination and refinement. Structure solved with *MULTAN*80 (Main, 1980); best E map gave 43 out of 44 non-hydrogen atoms. Even after isotropic refinement the remaining C(3) atom of (II) could not be located from a difference map and it was therefore included at a calculated position. Refinement by full-matrix least squares with *SHELX*76 (Sheldrick, 1976); C(sp^3) H atoms included on geometrical considerations, remaining 3 H atoms (per molecule) located from a difference map; former H atoms refined in rigid-body-constraint model (Sheldrick, 1976), latter with constrained bond lengths only; in final stages of refinement an overall scale factor, positional and anisotropic thermal parameters for C and O atoms and an overall thermal parameter for constrained H atoms were varied; $R = 0\cdot070$, $R_w = \sum(w^{1/2}||F_o|-|F_c||)/\sum(w^{1/2}|F_o|) = 0\cdot070$, $w = 10\cdot916/[\sigma^2(F) + 0\cdot00106 \times F_o^2]$; refinement without H constraints did not significantly decrease the R value (Hamilton, 1964); $\Delta/\sigma = 0\cdot07$ (av.) and $0\cdot2$ (max.) for non-H-atom parameters, $0\cdot3$ and 1 for H-atom parameters; $\Delta\rho$ excursions $\leq 0\cdot35$ e Å^{-3}; overall thermal parameter for H atoms refined to $0\cdot073$ (3) Å^2; scattering factors for non-H atoms from Cromer & Mann (1968) and for H atoms from Stewart, Davidson & Simpson (1965).*

Discussion. The final atomic positions and thermal parameters are given in Table 1.

Molecular conformation. The asymmetric unit consists of two independent molecules, which are related by a pseudo screw axis (see below). The conformation and atom numbering of molecule (I) are shown in Fig. 2 and the intramolecular dimensions are given in Table 2. The mean deviations of corresponding distances, angles and torsion angles of the two independent molecules are $0\cdot01$ (1) Å, $0\cdot6$ (5)° and 1 (1)°, respectively. With the exception of bond angles involving atoms C(11)–C(12)–C(13)–C(18)–O(2) of the epoxy ring, which are comparatively small (see Table 2), all distances and angles are similar to those observed in other lynestrenol derivatives (Rohrer, Hazel, Duax & Zeelen, 1976; Rohrer, Lauffenburger, Duax & Zeelen, 1977; Rohrer, Duax & Zeelen, 1978). Because the introduction of the epoxy bridge drastically reduces the progestational activity of the title compound, it is of interest to compare its conformation with that of lynestrenol (Rohrer, Lauffenburger, Duax & Zeelen, 1976). In Table 3 some prominent asymmetry parameters, describing the conformations of these two molecules are compared. The asymmetry parameters of

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* Lists of structure factors, anisotropic thermal parameters and hydrogen-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38678 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

rings A and B of both molecules of the title compound are very similar to those of lynestrenol, ring A having a $1\alpha,2\beta$-half-chair and ring B a distorted chair conformation. On the other hand, the asymmetry parameters of rings C and D show appreciable differences. This difference in conformation is visualized in Fig. 3, which shows a superposition of the A and B rings of the expoxy derivative and lynestrenol, the least-squares fitting resulted in a mean deviation of atoms C(1) through C(12) of 0·020 (2) Å. Whereas in lynestrenol ring C has a highly symmetrical chair conformation, in the epoxy derivative this ring is deformed in the

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^3$) *with e.s.d.'s in parentheses*

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

	(I)				(II)			
	x	y	z	U_{eq}	x	y	z	U_{eq}
O(1)	9701 (5)	8033 (2)	6007 (3)	66 (1)	6525 (5)	7051 (2)	1169 (3)	65 (1)
O(2)	8890 (4)	6967 (1)	9269 (3)	54 (1)	6673 (4)	8126 (1)	4452 (3)	51 (1)
C(1)	7652 (7)	5284 (3)	10340 (6)	69 (2)	7008 (8)	9862 (3)	5587 (6)	83 (2)
C(2)	7710 (9)	4811 (3)	11455 (5)	78 (1)	6709 (11)	10332 (3)	6631 (7)	97 (3)
C(3)	8779 (9)	4222 (3)	11159 (6)	78 (2)	5369 (12)	10831 (3)	6275 (6)	93 (3)
C(4)	10507 (7)	4412 (2)	10630 (5)	65 (2)	3780 (9)	10529 (3)	5621 (5)	76 (2)
C(5)	10872 (6)	4995 (2)	10193 (4)	53 (1)	3685 (7)	9930 (2)	5175 (4)	59 (1)
C(6)	12696 (6)	5168 (3)	9781 (5)	59 (1)	2001 (7)	9635 (3)	4646 (5)	69 (2)
C(7)	12684 (5)	5503 (2)	8502 (4)	52 (1)	2185 (6)	9307 (3)	3373 (5)	60 (1)
C(8)	11435 (5)	6080 (2)	8496 (4)	41 (1)	3703 (5)	8826 (2)	3465 (4)	42 (1)
C(9)	9557 (5)	5848 (2)	8744 (4)	42 (1)	5421 (5)	9178 (2)	3871 (4)	44 (1)
C(10)	9509 (6)	5533 (2)	10068 (4)	50 (1)	5287 (6)	9491 (2)	5188 (4)	54 (1)
C(11)	8240 (5)	6410 (2)	8529 (4)	50 (1)	7000 (5)	8721 (2)	3770 (4)	48 (1)
C(12)	8188 (5)	6652 (2)	7164 (4)	50 (1)	7224 (5)	8497 (2)	2410 (4)	50 (1)
C(13)	9972 (4)	6991 (2)	7187 (4)	43 (1)	5628 (5)	8044 (2)	2288 (3)	41 (1)
C(14)	11449 (5)	6478 (2)	7272 (4)	43 (1)	3918 (5)	8441 (2)	2223 (4)	41 (1)
C(15)	13141 (5)	6869 (2)	7059 (4)	54 (1)	2464 (5)	7942 (2)	1855 (4)	54 (1)
C(16)	12512 (6)	7510 (3)	6415 (5)	61 (1)	3419 (7)	7366 (2)	1294 (4)	58 (1)
C(17)	10524 (6)	7413 (2)	6069 (4)	52 (1)	5365 (6)	7592 (2)	1135 (4)	51 (1)
C(18)	9852 (6)	7372 (2)	8421 (4)	49 (1)	5861 (6)	7672 (2)	3549 (4)	49 (1)
C(20)	10238 (6)	7070 (2)	4852 (4)	57 (1)	5488 (6)	7947 (2)	-66 (4)	57 (1)
C(21)	9987 (8)	6803 (3)	3868 (5)	70 (2)	5568 (7)	8228 (3)	-1039 (4)	65 (2)

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

	(I)	(II)		(I)	(II)		(I)	(II)
O(1)–C(17)	1·424 (6)	1·421 (6)	C(5)–C(10)	1·519 (6)	1·515 (7)	C(13)–C(14)	1·541 (5)	1·533 (5)
O(2)–C(11)	1·460 (5)	1·449 (5)	C(6)–C(7)	1·510 (7)	1·511 (8)	C(13)–C(17)	1·536 (6)	1·533 (5)
O(2)–C(18)	1·445 (5)	1·449 (5)	C(7)–C(8)	1·522 (6)	1·520 (7)	C(13)–C(18)	1·522 (6)	1·533 (5)
C(1)–C(2)	1·523 (8)	1·489 (9)	C(8)–C(9)	1·537 (5)	1·534 (6)	C(14)–C(15)	1·542 (6)	1·544 (6)
C(1)–C(10)	1·539 (7)	1·553 (8)	C(8)–C(14)	1·526 (6)	1·542 (6)	C(15)–C(16)	1·550 (7)	1·526 (6)
C(2)–C(3)	1·502 (9)	1·48 (1)	C(9)–C(10)	1·536 (6)	1·534 (6)	C(16)–C(17)	1·546 (6)	1·565 (7)
C(3)–C(4)	1·501 (8)	1·49 (1)	C(9)–C(11)	1·540 (6)	1·532 (6)	C(17)–C(20)	1·467 (6)	1·465 (6)
C(4)–C(5)	1·322 (6)	1·323 (7)	C(11)–C(12)	1·516 (6)	1·518 (6)	C(20)–C(21)	1·178 (7)	1·179 (6)
C(5)–C(6)	1·512 (7)	1·497 (7)	C(12)–C(13)	1·522 (5)	1·530 (6)			

	(I)	(II)		(I)	(II)		(I)	(II)
C(11)–O(2)–C(18)	107·2 (3)	107·6 (3)	C(8)–C(9)–C(11)	109·9 (3)	110·2 (3)	C(17)–C(13)–C(18)	112·8 (3)	112·4 (3)
C(2)–C(1)–C(10)	111·4 (5)	111·3 (5)	C(10)–C(9)–C(11)	114·0 (3)	114·1 (3)	C(8)–C(14)–C(13)	112·6 (3)	111·3 (3)
C(1)–C(2)–C(3)	110·8 (5)	113·3 (6)	C(11)–C(10)–C(5)	111·4 (4)	111·7 (4)	C(8)–C(14)–C(15)	116·4 (3)	116·8 (3)
C(2)–C(3)–C(4)	110·8 (5)	110·7 (5)	C(1)–C(10)–C(9)	111·8 (4)	111·0 (4)	C(13)–C(14)–C(15)	103·9 (3)	104·3 (3)
C(3)–C(4)–C(5)	124·6 (5)	125·6 (6)	C(5)–C(10)–C(9)	109·9 (3)	109·4 (3)	C(14)–C(15)–C(16)	105·7 (3)	105·6 (3)
C(4)–C(5)–C(6)	121·6 (4)	122·8 (5)	O(2)–C(11)–C(9)	108·4 (3)	109·5 (3)	C(15)–C(16)–C(17)	105·5 (4)	106·1 (3)
C(4)–C(5)–C(10)	122·8 (4)	121·8 (5)	O(2)–C(11)–C(12)	103·6 (3)	103·7 (3)	O(1)–C(17)–C(13)	114·2 (3)	113·7 (3)
C(6)–C(5)–C(10)	115·5 (4)	115·4 (4)	C(9)–C(11)–C(12)	111·9 (3)	112·1 (3)	O(1)–C(17)–C(16)	108·3 (4)	110·4 (3)
C(5)–C(6)–C(7)	113·4 (4)	113·3 (4)	C(11)–C(12)–C(13)	98·9 (3)	98·0 (3)	O(1)–C(17)–C(20)	110·4 (4)	110·4 (4)
C(6)–C(7)–C(8)	109·8 (4)	109·8 (4)	C(12)–C(13)–C(14)	109·1 (3)	109·9 (3)	C(13)–C(17)–C(16)	101·2 (3)	100·6 (3)
C(7)–C(8)–C(9)	109·7 (3)	109·8 (4)	C(12)–C(13)–C(17)	121·6 (3)	120·6 (3)	C(13)–C(17)–C(20)	110·9 (3)	111·3 (3)
C(7)–C(8)–C(14)	113·3 (3)	113·2 (3)	C(12)–C(13)–C(18)	99·2 (3)	100·2 (3)	C(16)–C(17)–C(20)	111·6 (4)	110·0 (4)
C(9)–C(8)–C(14)	110·8 (3)	110·8 (3)	C(14)–C(13)–C(17)	102·1 (3)	102·3 (3)	O(2)–C(18)–C(13)	106·2 (3)	105·5 (3)
C(8)–C(9)–C(10)	110·4 (3)	110·9 (3)	C(14)–C(13)–C(18)	112·3 (3)	111·6 (3)	C(17)–C(20)–C(21)	178·8 (5)	179·1 (5)

	(I)	(II)		(I)	(II)		(I)	(II)
C(18)–O(2)–C(11)–C(9)	95·6 (4)	92·2 (4)	C(10)–C(5)–C(6)–C(7)	-47·7 (6)	-49·4 (6)	C(11)–C(12)–C(13)–C(14)	70·2 (4)	71·0 (4)
C(11)–O(2)–C(18)–C(13)	-7·6 (4)	-2·9 (4)	C(5)–C(6)–C(7)–C(8)	52·4 (5)	52·8 (6)	C(12)–C(13)–C(14)–C(8)	-62·5 (4)	63·3 (4)
C(2)–C(1)–C(10)–C(5)	40·2 (6)	39·4 (6)	C(6)–C(7)–C(8)–C(9)	-60·0 (4)	-58·8 (5)	C(9)–C(8)–C(14)–C(13)	46·9 (4)	47·1 (4)
C(10)–C(1)–C(2)–C(3)	-62·2 (6)	-60·2 (7)	C(7)–C(8)–C(9)–C(10)	62·3 (4)	61·3 (5)	C(12)–C(13)–C(14)–C(15)	170·8 (3)	169·9 (3)
C(1)–C(2)–C(3)–C(4)	47·9 (7)	45·6 (8)	C(8)–C(9)–C(10)–C(5)	-54·7 (4)	-54·8 (4)	C(17)–C(13)–C(14)–C(8)	167·6 (3)	167·6 (3)
C(2)–C(3)–C(4)–C(5)	-15·3 (8)	-13·1 (9)	C(7)–C(8)–C(9)–C(11)	-171·1 (3)	-171·4 (4)	C(17)–C(13)–C(14)–C(15)	40·9 (4)	40·5 (4)
C(3)–C(4)–C(5)–C(10)	-5·5 (8)	-5·3 (8)	C(14)–C(8)–C(9)–C(10)	-171·9 (3)	-172·9 (3)	C(14)–C(13)–C(17)–C(16)	-46·3 (4)	45·3 (4)
C(4)–C(5)–C(10)–C(1)	-7·5 (6)	-8·3 (6)	C(14)–C(8)–C(9)–C(11)	-45·3 (4)	45·6 (4)	C(13)–C(14)–C(15)–C(16)	19·1 (4)	18·9 (4)
C(4)–C(5)–C(10)–C(9)	-132·0 (5)	-131·6 (5)	C(8)–C(9)–C(11)–C(12)	62·1 (4)	62·4 (4)	C(14)–C(15)–C(16)–C(17)	9·6 (5)	9·4 (4)
C(6)–C(5)–C(10)–C(1)	172·5 (4)	172·7 (4)	C(9)–C(11)–C(12)–C(13)	-72·0 (4)	71·7 (4)	C(15)–C(16)–C(17)–C(13)	34·5 (4)	33·9 (4)
C(6)–C(5)–C(10)–C(9)	47·9 (5)	49·3 (5)						

direction of a 12α sofa conformation, which is indicated by the decrease of the $\Delta C_2[C(9)–C(11)]$ values (see Table 3). Owing to the steric effect imposed by the epoxy bridge the D ring is forced into a rather unusual 13β,17α-half-chair conformation as indicated by the $\Delta C_2[C(13)–C(17)]$ value, whereas in lynestrenol this ring has a common 13β-envelope conformation, as indicated by the $\Delta C_s[C(13)]$ value. This difference in conformation is also well illustrated by the pseudorotation parameter Δ (Altona, Geise & Romers, 1968); φ_m is equal in the two structures and within the normal range of 46 to 50° (Thomas, 1982), but the phase angle Δ is quite different for the title compound, being far outside the normal range of −36 to 36°, which covers the range from a C(14) to a C(13) envelope. In spite of the deformation of rings C and D induced by the epoxy bridge, the overall shape of the molecule is somewhat flatter than that of lynestrenol, as may be seen in Fig. 3. Therefore, the conclusion is warranted that the degree of flexure is indeed of major importance in predicting progestational activity of 11β derivatives of lynestrenol.

Molecular packing. The molecular packing is illustrated in Fig. 4, which shows the view down **a**. There are two hydrogen bonds connecting (I) and (II) with distances $O(1)_I \cdots O(2)_{II}$ 2·757 (5) and $O(1)_{II} \cdots O(2^i)_I$ 2·753 (4) Å, where (i) = $x,y,z−1$, and O–H$\cdots$O angles of 153 (4) and 163 (4)°, respectively. Infinite chains of hydrogen-bonded molecules are extended parallel to the a axis, with each molecule connected to two neighbours by one hydrogen bond each. The 17β-hydroxyl group and the epoxy O atom act as donor and acceptor, respectively. Adjacent links are related by the pseudo screw axis parallel to **c** (see below), and since the steroid length is perpendicular to the **bc** diagonal, this results in a twisting of the chains with the α face of the steroid away from the screw axis. These twisted chains have a herringbone appearance in the (100) projection. According to the classification of Duax & Norton (1975) the packing arrangement can be described as 212, indicating that the molecules are arranged two thick, one wide and two long.
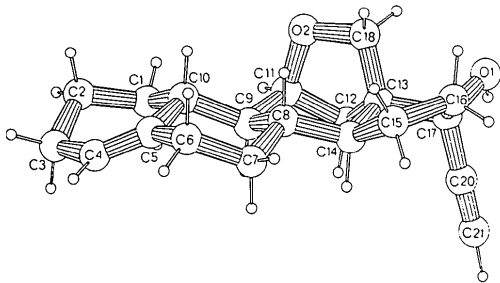

Fig. 2. Molecular structure with atom numbering of 11β,18-epoxylynestrenol.

Table 3. *Selected asymmetry parameters (Duax & Norton, 1975) and D-ring pseudo rotation parameters (Altona et al., 1968) for the title compound and lynestrenol (Rohrer, Lauffenburger, Duax & Zeelen, 1976)*

All parameters in degrees, with e.s.d.'s in parentheses.

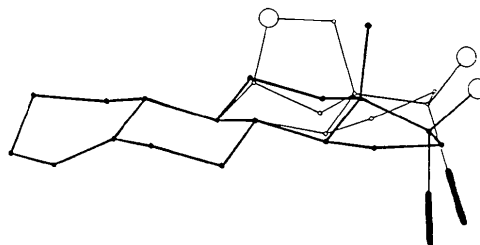|		11β,18-Epoxylynestrenol		Lynestrenol
		(I)	(II)	
$\Delta C_2\|C(1)–C(2)\|$	A	7·8 (4)	5·6 (4)	5·8 (2)
$\Delta C_s\|C(3)\|$		47·4 (3)	45·9 (3)	47·9 (2)
$\Delta C_2\|C(5)\|$	B	1·9 (3)	1·8 (3)	0·9 (2)
$\Delta C_s\|C(6)–C(7)\|$		13·4 (4)	10·7 (4)	8·9 (2)
$\Delta C_2\|C(8)\|$	C	1·5 (3)	1·1 (3)	3·8 (2)
$\Delta C_2\|C(9)–C(11)\|$		25·0 (4)	25·0 (4)	2·0 (2)
$\Delta C_s\|C(13)\|$	D	11·6 (4)	11·1 (4)	6·9 (2)
$\Delta C_2\|C(13)–C(14)\|$		36·6 (4)	35·9 (4)	14·7 (2)
$\Delta C_s\|C(13)–C(17)\|$		8·0 (4)	8·2 (4)	30·8 (2)
Δ		59·0 (6)	58·4 (6)	22·5 (3)
φ_m		47·0 (5)	46·4 (5)	47·0 (2)

Fig. 3. Superposition of the A and B rings in the structures of lynestrenol (thick lines) and the 11β,18-epoxy derivative.
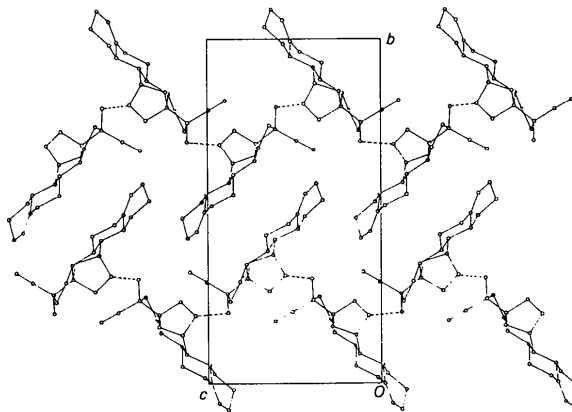

Fig. 4. Packing diagram viewed down **a**. Broken lines indicate hydrogen bonds.

Pseudo symmetry. Monoclinic 11β,18-epoxylynestrenol is pseudo-orthorhombic, with space group $P2_12_12_1$. In the standard $P2_1$ space-group setting the two pseudo screw axes parallel to **a** and **c** give rise to the following equivalent positions: $(x + T_a, \bar{y} + 2C_{ya}, \bar{z} + 2C_{za})$ and $(\bar{x} + 2C_{xc}, \bar{y} + 2C_{yc}, z + T_c)$, where C_{ya},

C_{za} and C_{xc}, C_{yc} are the coordinates, and T_a and T_c the translation components of the screw axes parallel to **a** and **c**, respectively. By means of the y coordinates of the two independent molecules, the y origin in $P2_1$ was chosen such that the pseudo 2_1 axes parallel to **a** and **c** lie at $y = 0$ and $y = \frac{1}{4}$, respectively, expressed by the calculated values for C_{ya} and C_{yc}, giving 0·001 (1) and 0·250 (5), respectively. The x and z coordinates of the independent molecules were used to calculate the other unknowns, giving $T_a = 0·52$ (6), $C_{za} = 0·25$ (1) and $T_c = 0·50$ (2) and $C_{xc} = 0·26$ (3). Although the pseudo 2_1 axis parallel to **c** is not exactly at $x = \frac{1}{4}$, it has a mean translation of $\frac{1}{2}$**c** and this ought to give rise to very weak 00l reflections with $l = 2n + 1$, in accordance with experimental observations. The mean translation for the pseudo 2_1 axis parallel to **a** is significantly different from $\frac{1}{2}$**a**, and consequently no relative weak $h00$ reflections with $h = 2n + 1$ have been observed. Assuming exact $P2_12_12_1$ symmetry results in a mean deviation of atom positions of 0·52 (4) Å for both molecules.

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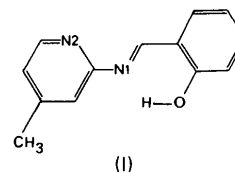# Structure of 2-(4-Methyl-2-pyridyliminomethyl)phenol, C₁₃H₁₂N₂O

BY C. ESCOBAR AND M. T. GARLAND

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Abstract. $M_r = 212·0$, monoclinic, $P2_1/c$, $a = 11·772$ (4), $b = 7·5386$ (7), $c = 15·154$ (5) Å, $\beta = 124·89$ (2)° (JCPDS Diffraction File No. 33–1999) [reduced cell: $P2_1/n$, $a' = a$, $b' = b$, $c' = 12·812$ (5) Å, $\beta' = 104·02$ (5)°], $V = 1103·1$ (4) Å³, $Z = 4$, $D_m = 1·24$ (3) (flotation in dioxane and bromoform), $D_x = 1·276$ (1) Mg m⁻³, $\lambda(Mo\,K\alpha) = 0·7107$ Å, $\mu = 0·09$ mm⁻¹, $F(000) = 448·0$. The final R is 0·079 for 875 observed reflections. The molecule has an O—H···N hydrogen bond contained in the plane of the salicylidene ring. The pyridine ring is twisted by ~12 (1)° from the rest of the molecule. The H atom of the exocyclic C atom is *cis* with respect to the N atom of the pyridine ring.

Introduction. The structure of 2-(4-methyl-2-pyridyliminomethyl)phenol (I) has been determined as a preliminary step in the synthesis and structural study of copper(II) complexes with Schiff bases derived from salicylaldehyde and 2-aminopyridine as ligands.

(I)