

localized in the acetamide-oxime group. The short N(1)···N(5) intramolecular distance [2.746 (5) Å] forces an increase in the C(2)–C(3)–N(5) angle.

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References

- CALLERI, M., FERRARIS, G. & VITERBO, D. (1966). *Acta Cryst.* **20**, 73–82.
- CALLERI, M., FERRARIS, G. & VITERBO, D. (1967a). *Acta Cryst.* **22**, 468–475.
- CALLERI, M., FERRARIS, G. & VITERBO, D. (1967b). *Inorg. Chim. Acta*, **1**, 297–299.
- CULLEN, D. L. & LINGAFELTER, E. C. (1970a). *Inorg. Chem.* **9**, 1865–1877.
- CULLEN, D. L. & LINGAFELTER, E. C. (1970b). *Inorg. Chem.* **9**, 1858–1864.
- EDDY, L. P., LEVENHAGEN, W. W. & EWEN, S. K. (1968). *Inorg. Synth.* **11**, 9–11.
- GOZLAN, H. & RICHE, C. (1976). *Acta Cryst.* **B32**, 1662–1665.
- HAMILTON, W. C. (1961). *Acta Cryst.* **14**, 95–100.
- International Tables for X-ray Crystallography* (1968). Vol. III. Birmingham: Kynoch Press.
- LINNET, J. W. (1961). *J. Chem. Soc.* pp. 2643–2654.
- MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1978). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO 78*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- MURMANN, R. K. & SCHLEMPER, E. O. (1967). *Acta Cryst.* **23**, 667–669.
- PANATTONI, C., FRASSON, E. & ZANOTTI, R. (1950). *Gazz. Chim. Ital.* **89**, 2132–2138.
- RAYMOND, K. N., CORFIELD, P. W. R. & IBERS, J. A. (1968). *Inorg. Chem.* **7**, 842–853.
- SHELDRIK, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.
- Tables of Interatomic Distances and Configuration in Molecules and Ions* (1958). Edited by L. E. SUTTON. London: The Chemical Society.
- WILLIAMS, D. E., WOLHAUER, R. & RUNDLE, R. E. (1959). *J. Am. Chem. Soc.* **81**, 755–760.

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17 α -Ethyanyl-17 β -hydroxy-12-methyl-4,9,11-estratrien-3-one

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Abstract. C₂₁H₂₄O₂, orthorhombic, *P*2₁2₁1, *a* = 20.140 (8), *b* = 11.134 (5), *c* = 7.458 (4) Å, *Z* = 4, *d*_x = 1.22 Mg m⁻³. The molecule is bent towards its α side. The additional 12-methyl group does not significantly affect the usual steroid conformation. The final *R* value is 0.036.

Introduction. 17 α -Ethyanyl-17 β -hydroxy-4,9,11-estratrien-3-one presents androgen and progestin properties. Its 12-methylated derivative, the title compound, has a weaker affinity for androgen and progestin receptors (Raynaud, 1979).

The crystals were obtained by slow evaporation from methanol solution. Unit-cell parameters and 1845 independent reflections were measured on an auto-

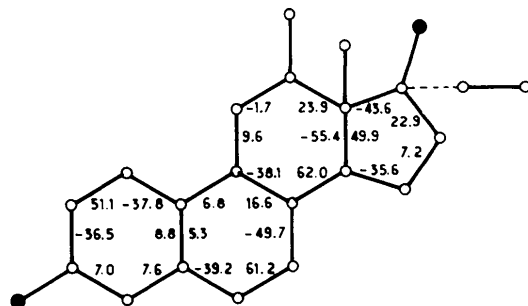
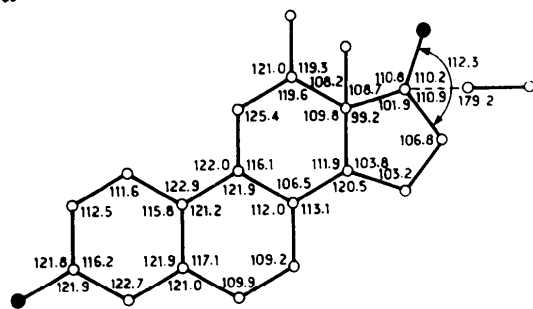
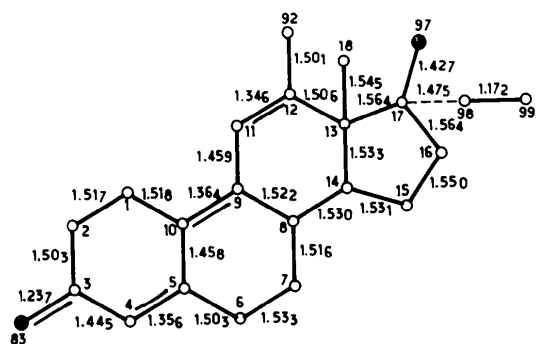
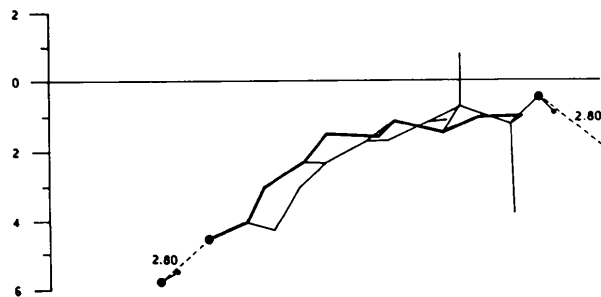
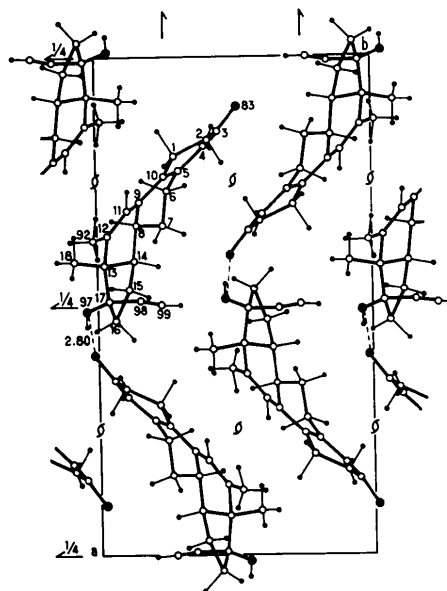
matic diffractometer, using Cu *K* α radiation.* The structure was solved by direct methods, using the *MULTAN* program (Germain, Main & Woolfson, 1971).

All the atomic parameters were refined by a least-squares procedure. The H atom positions were found on a difference Fourier map. The positional and equivalent isotropic thermal parameters of the heavy atoms are listed in Table 1. The diffusion factors used are from *International Tables for X-ray Crystallog-*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35254 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and temperature factors

	x	y	z	B_{eq} (\AA^2)
C(1)	0.1973 (1)	0.2821 (3)	0.9227 (4)	3.5
C(2)	0.1739 (1)	0.4114 (3)	0.9107 (4)	3.9
C(3)	0.1479 (1)	0.4429 (3)	0.7279 (5)	3.1
C(4)	0.1819 (1)	0.3888 (3)	0.5777 (4)	3.6
C(5)	0.2299 (1)	0.3044 (3)	0.5978 (4)	2.7
C(6)	0.2690 (1)	0.2598 (3)	0.4396 (4)	3.1
C(7)	0.3415 (1)	0.2398 (3)	0.4938 (4)	3.2
C(8)	0.3445 (1)	0.1454 (3)	0.6403 (4)	2.9
C(9)	0.2961 (1)	0.1721 (2)	0.7919 (4)	3.0
C(10)	0.2450 (1)	0.2512 (3)	0.7718 (4)	3.0
C(11)	0.3084 (1)	0.1097 (3)	0.9608 (4)	3.1
C(12)	0.3629 (1)	0.0451 (2)	0.9999 (4)	3.1
C(13)	0.4165 (1)	0.0305 (2)	0.8608 (4)	2.6
C(14)	0.4133 (1)	0.1345 (2)	0.7262 (4)	3.1
C(15)	0.4750 (1)	0.1166 (3)	0.6096 (4)	3.5
C(16)	0.5272 (1)	0.0686 (3)	0.7443 (4)	3.5
C(17)	0.4900 (1)	0.0459 (2)	0.9252 (4)	3.0
C(18)	0.4089 (1)	-0.0949 (3)	0.7748 (4)	3.4
C(92)	0.3691 (1)	-0.0201 (3)	1.1752 (4)	3.4
C(98)	0.4958 (1)	0.1507 (3)	1.0451 (4)	3.5
C(99)	0.5002 (2)	0.2349 (3)	1.1387 (5)	4.4
O(83)	0.1021 (1)	0.5156 (2)	0.7069 (3)	4.5
O(97)	0.5127 (1)	-0.0599 (2)	1.0141 (3)	3.9

Fig. 1. Bond lengths (\AA) ($\sigma = 0.004 \text{\AA}$), angles ($^\circ$) ($\sigma = 0.3^\circ$) and dihedral angles ($^\circ$) ($\sigma = 0.8^\circ$).Fig. 2. Projection of the molecule in the C(13)-C(17)-C(18) plane showing the hydrogen bonds (\AA).Fig. 3. Projection of the structure along c showing the hydrogen bonds (\AA).

raphy (1962) for C and O and from Stewart, Davidson & Simpson (1965) for H. The final R value is 0.036.

Discussion. Bond lengths, angles and dihedral angles of the observed conformation are given in Fig. 1. Fig. 2, which represents the projection of the molecule on to the C(13)-C(17)-C(18) plane, clearly shows the folding of the steroid skeleton towards its α side, whereas the unmethylated compound is described as a flat molecule (Lepicard, Delettré & Moron, 1974).

If we only consider the observed conformations from the X-ray analysis, we should correlate the bending of the molecule with the weaker biological effects. However, the potential flexibility of these unsaturated steroids is well known (Précigoux, Busetta & Geoffre, 1980) and we must also consider the steric hindrance of the 12-methyl group as partly responsible for the decrease of the affinity for the specific receptors.

The crystal packing is illustrated in Fig. 3. In the *b* direction there is a zigzag succession of molecules linked by a hydrogen bond involving the 17 β -ol function: O(97)—H \cdots O(83) [$d = 2.80(1) \text{ \AA}$]. In the other directions crystal cohesion is achieved by attractive van der Waals forces. We observe only one repulsive contact between the C(92) methyl group and the C(10) atom [$d = 3.52(1) \text{ \AA}$].

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3-Acetyl-1-methoxyindole

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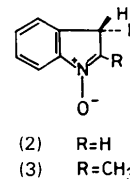
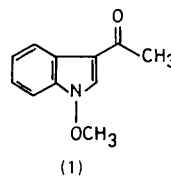
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Abstract. C₁₁H₁₁NO₂, triclinic, $P\bar{1}$, $a = 8.35(1)$, $b = 10.39(1)$, $c = 11.58(1) \text{ \AA}$, $\alpha = 98.50(8)$, $\beta = 97.68(6)$, $\gamma = 91.69(10)^\circ$, $Z = 4$, $\mu = 0.094 \text{ mm}^{-1}$ (Mo $K\alpha$ radiation), $R = 0.054$. The bonds about the N atom lie in one plane and the acetyl group is almost coplanar with the indole ring. Bond lengths suggest π -electron delocalization between the N atom and the carbonyl group.

Introduction. Five derivatives of 1-methoxyindole have been isolated from plant sources (Acheson, 1979), and further members have been synthesized (Acheson, Hunt, Littlewood, Murrer & Rosenberg, 1978). The investigation of the crystal structure of one of these synthetic analogues (1) was undertaken to determine the geometry of the bonds about N and to investigate the conformation about the N—O bond. The ¹³C NMR spectrum of 1-methoxyindole is very similar to that of indole, suggesting a planar N atom, but the ready tautomerism of 1-hydroxyindole and 1-hydroxy-2-methylindole to the nitron tautomers (2) and (3) suggests that there is not a large energy factor in favour of retaining aromaticity in the five-membered ring.



Crystals of (1) were grown by diffusion of petrol vapour into an ethyl acetate solution. Oscillation and Weissenberg photographs and the crystal density showed the crystal system to be triclinic with $Z = 4$. Cell dimensions were determined on an Enraf-Nonius CAD-4 four-circle diffractometer. Intensities were collected with Mo $K\alpha$ radiation to $2\theta \leq 44^\circ$ by an $\omega/2\theta$ scan with standard reflections checked hourly. Lorentz and polarization corrections were applied to the 2429 observed reflections, equivalent reflections merged and structure amplitudes derived for 948 with $I > 3\sigma(I)$. Application of *MULTAN 77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977) gave two solutions, each containing a promising fragment, but neither would serve as a sufficiently good phasing model for the determination of the rest of the structure. The solution (Fig. 1) was finally obtained from *MULTAN 77* by making two changes. The scaling of

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References

- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
International Tables for X-ray Crystallography (1962). Vol. III, pp. 202–207. Birmingham: Kynoch Press.
 LEPICARD, G., DELETTRE, J. & MORNON, J. P. (1974). *Acta Cryst.* **B30**, 2751–2753.
 PRÉCIGOUX, G., Busetta, B. & Geoffre, S. (1980). *Acta Cryst.* In preparation.
 RAYNAUD, J. P. (1979). Private communication.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.