CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
DAPPORTO, P. & SEGA, A. (1986). *Acta Cryst.* C**42**, 474–478.
FLORVALL, L. & ÖGREN, S.-O. (1982). *J. Med. Chem.* **25**, 1280–1286.
FORESTI, E., RIVA DI SANSEVERINO, L. & SABATINO, P. (1986). *Acta Cryst.* C**42**, 220–224.
FURUYA, T., FUJITA, S., IWANAMI, S., TAKENAKA, A. & SASADA, Y. (1986). *Acta Cryst.* C**42**, 1345–1347.
FURUYA, T., IWANAMI, S., TAKENAKA, A. & SASADA, Y. (1986*a*). *Acta Cryst.* C**42**, 1071–1073.
FURUYA, T., IWANAMI, S., TAKENAKA, A. & SASADA, Y. (1986*b*). *Acta Cryst.* C**42**, 117–121.
HÖGBERG, T., NORINDER, U., RÄMSBY, S. & STENSLAND, B. (1987). *J. Pharm. Pharmacol.* In the press.
HÖGBERG, T., RÄMSBY, S., DE PAULIS, T., STENSLAND, B., CSÖREGH, I. & WÄGNER, A. (1986). *Mol. Pharmacol.* **30**, 345–351.
HOUTTEMANE, C., BOIVIN, J. C., NOWOGROCKI, G., THOMAS, D. J. & BONTE, J. P. (1981). *Acta Cryst.* B**37**, 981–984.
HOUTTEMANE, C., BOIVIN, J. C., THOMAS, D. J., BERTHELOT, P. & DEBAERT, M. (1983). *Acta Cryst.* C**39**, 1285–1287.
MA, Y. Y., CAMERMAN, N. & CAMERMAN, A. (1982). *Acta Cryst.* B**38**, 2861–2865.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
ÖGREN, S.-O., HALL, H., KÖHLER, C., MAGNUSSON, O., LINDBOM, L.-O., ÄNGEBY, K. & FLORVALL, L. (1984). *Eur. J. Pharmacol.* **102**, 459–474.
PEETERS, O. M., BLATON, N. M., DE RANTER, C. J., DENISOFF, O. & MOLLE, L. (1980). *Cryst. Struct. Commun.* **9**, 851–856.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
TESTA, B., VAN DE WATERBEEMD, H. & CARRUPT, P.-A. (1986). *J. Mol. Struct. Theoret. Chem.* **134**, 351–366.
WÄGNER, A., STENSLAND, B., CSÖREGH, I. & DE PAULIS, T. (1985). *Acta Pharm. Suec.* **22**(2), 101–110.
WATERBEEMD, H. VAN DE & TESTA, B. (1981). *Helv. Chim. Acta.* **64**, 2183–2188.
WATERBEEMD, H. VAN DE & TESTA, B. (1983). *J. Med. Chem.* **26**, 203–207.

Acta Cryst. (1987). C**43**, 2398–2401

Structure of 13-Ethyl-17β-hydroxy-11-methylene-18,19-dinor-17α-pregn-4-en-20-yn-3-one (3-Ketodesogestrel)

BY V. J. VAN GEERESTEIN, J. A. KANTERS AND J. KROON

Laboratorium voor Kristal- en Structuurchemie, Rijksuniversiteit Utrecht, Transitorium 3, *Padualaan* 8, 3584 *CH Utrecht, The Netherlands*

(*Received* 7 *May* 1987; *accepted* 17 *July* 1987)

Abstract. $C_{22}H_{28}O_2$, $M_r = 324{\cdot}46$, monoclinic, $P2_1$, $a = 6{\cdot}653$ (1), $b = 18{\cdot}413$ (1), $c = 7{\cdot}9653$ (8) Å, $\beta = 107{\cdot}49$ (1)°, $V = 930{\cdot}6$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}158$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ (Mo $K\alpha$) = 0·7 cm^{-1}, $F(000) = 352$, room temperature, $R = 0{\cdot}053$ for 1910 unique reflections with $I \geq 2{\cdot}5\sigma(I)$. The ethyl group is in the usual *trans* conformation relative to the *C/D* ring junction. The *A* ring is statistically disordered (1:1) and shows a normal 1α,2β-half-chair as well as an inverted 1β,2α-half-chair conformation. Molecular mechanics gives a steric energy difference of 4 kJ mol^{-1} between the minimized normal and inverted half-chair structures in favour of the normal 1α,2β-half-chair conformation. The steroid molecules are hydrogen-bonded head to tail.

Introduction. The title compound is the biologically active metabolite of the orally active progestogen desogestrel (13-ethyl-11-methylene-18,19-dinor-17α-pregn-4-en-20-yn-17β-ol). The carbonyl function at the 3-position is introduced by metabolic oxidation in the liver. The 4-en-3-one *A* ring is essential for proper binding to the progestogen receptor and for steroids without any further unsaturation this ring shows the greatest conformational flexibility of the steroid backbone. Usually the conformation of the *A* ring is somewhere between a 1α,2β-half-chair and a 1α-sofa, but in a small number of crystal structures an inverted 1β,2α-half-chair has been observed (Duax, Fronckowiak, Griffin & Rohrer, 1982). Most cases of inverted *A*-ring conformations include 2β-substituted steroids where steric effects favour this conformation in the crystal as well as in solution. Medroxyprogesterone acetate (17α-acetoxy-6α-methylprogesterone) was found to have a normal *A*-ring conformation in solution (Barrett, Farrant, Kirk, Mersh, Sanders & Duax, 1982) but an inverted conformation in the crystal (Duax, Cody, Griffin, Hazel & Weeks, 1978). Barrett *et al.* (1982) suggested that the energy difference between the two conformations is small and that packing forces in the crystal which stabilize the inverted conformation may mimic those at the binding site for progestational activity. The energy difference between the different conformers is thought to be in the range of 0 to 10

0108-2701/87/122398-04$01.50

kJ mol^{-1} (Duax, 1986) and may be influenced by specific substitution including short-range effects like a 19-methyl group or a more long-range effect like the introduction of an 11-methylene group.

Experimental. Sample (ORG 3236) was obtained through the Scientific Development Group of Organon, Oss, The Netherlands. Crystals were obtained by slow evaporation from methanol. Data were measured on a crystal with approximate dimensions 1·0 × 0·5 × 0·25 mm on an Enraf–Nonius CAD-4 diffractometer with Zr-filtered Mo $K\alpha$ radiation, lattice parameters refined by least-squares fitting of four alternative settings (de Boer & Duisenberg, 1984) of 12 reflections in the range $20 < 2\theta < 28°$; ω–2θ scan mode, $\Delta\omega = (0{\cdot}50 + 0{\cdot}35\tan\theta)°$. 8064 reflections were measured up to $\theta = 27°$, $\pm h$, $\pm k$, $\pm l$ (max. range 8, 23, 10), Friedel pairs and symmetry-related reflections were merged to 2081 unique observations with $R_{int} = 0{\cdot}028$, of which 1910 with $I \geq 2{\cdot}5\sigma(I)$ were used for structure refinement. Two periodically measured standard reflections ($2\bar{4}\bar{4}$, $2\bar{4}2$) showed a steady decrease in intensity of 8% in 77 h total X-ray exposure time; hence intensities were corrected for this decay as well as for the usual Lp factors.

The structure was solved by Patterson search methods with the *PATSEE* program (Egert & Sheldrick, 1985); two *trans*-fused cyclohexane rings with chair conformations were used as search fragment. The best solution was used in tangent expansion and peak optimization using a preliminary version of *SHELXS*86 (Sheldrick, 1986), resulting in the complete structure, but with relatively low electron densities for atoms C(1) and C(2). H atoms were placed on calculated positions and refined riding on their bonded atoms, except the hydroxyl, ethynyl and methylene group H atoms, which were located on a difference map and refined positionally. The mean square amplitude of vibration for H atoms was fixed at $U = 0{\cdot}08$ Å^2. Anisotropic refinement including H atoms which converged at $R = 0{\cdot}064$ resulted in rather high values for the temperature tensors for C(1) and C(2), describing an unrealistic thermal motion. A difference Fourier map excluding these two atoms showed two maxima for C(1) as well as for C(2), so these two atoms were considered to be statistically disordered over two positions, here labelled *A* and *B*, with the possibility of a superimposed dynamical disorder. Refinement with fixed isotropic thermal parameters for C(1*A*) and C(2*A*) and constrained bond lengths involving these atoms showed two satellite peaks in the difference map, whose coordinates were assigned to C(1*B*) and C(2*B*). During subsequent refinement the bond lengths and geminal distances involving these disordered C atoms were restrained to normal values (Griffin, Duax & Weeks, 1984). In the final cycles of two-block full-matrix least-squares refinement, 121 and 109 parameters were varied respectively: positional parameters, a coupled site occupation factor (s.o.f.) and a coupled isotropic thermal parameter for the disordered C atoms, positional and individual anisotropic parameters for normal C and O atoms and positional parameters for H[O(17)], H[C(21)], H[C(23)*a*] and H[C(23)*b*]. The refinement on F converged at $R = 0{\cdot}053$ and $wR = 0{\cdot}050$, where $w = 1/\sigma^2(F)$. The s.o.f. for conformation *A* refined to 0·571 (4). $\Delta/\sigma = 0{\cdot}03$ (3) (ave.) and 0·4 (max.) for non-H-atom parameters and $\Delta/\sigma = 0{\cdot}1$ (1) (ave.) and 0·5 (max.) for H-atom parameters; final residual electron density $-0{\cdot}2 < \Delta\rho < 0{\cdot}3$ e Å^{-3}. *SHELX*76 (Sheldrick, 1976) was used for structure refinement and scattering factors were taken from this program.

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(3)	0·1176 (4)	0·1561 (1)	0·0047 (3)	0·092 (1)
O(17)	1·1794 (3)	−0·2862 (1)	0·3015 (3)	0·0661 (6)
C(1*A*)	0·4746 (6)	0·0470 (4)	−0·1596 (5)	0·0726 (9)*
C(1*B*)	0·4287 (7)	0·0214 (3)	−0·097 (1)	0·0726 (9)*
C(2*A*)	0·2729 (8)	0·0561 (3)	−0·1077 (8)	0·0726 (9)*
C(2*B*)	0·296 (1)	0·0892 (4)	−0·1601 (6)	0·0726 (9)*
C(3)	0·2777 (5)	0·1216 (2)	0·0063 (4)	0·075 (1)
C(4)	0·4716 (5)	0·1298 (2)	0·1464 (4)	0·070 (1)
C(5)	0·6475 (5)	0·0946 (2)	0·1524 (4)	0·066 (1)
C(6)	0·8448 (6)	0·1032 (2)	0·3016 (5)	0·080 (1)
C(7)	0·9175 (6)	0·0299 (2)	0·3883 (4)	0·074 (1)
C(8)	0·9464 (5)	−0·0256 (2)	0·2560 (4)	0·057 (1)
C(9)	0·7430 (4)	−0·0329 (2)	0·0979 (4)	0·055 (1)
C(10)	0·6592 (5)	0·0409 (2)	0·0105 (4)	0·072 (1)
C(11)	0·7793 (4)	−0·0915 (2)	−0·0255 (3)	0·0526 (8)
C(12)	0·8249 (4)	−0·1648 (2)	0·0596 (3)	0·0544 (8)
C(13)	1·0276 (4)	−0·1606 (2)	0·2155 (3)	0·0506 (8)
C(14)	0·9987 (4)	−0·1006 (2)	0·3393 (3)	0·057 (1)
C(15)	1·1876 (5)	−0·1094 (2)	0·5043 (4)	0·071 (1)
C(16)	1·2268 (5)	−0·1917 (2)	0·5176 (4)	0·078 (1)
C(17)	1·0753 (5)	−0·2256 (2)	0·3486 (4)	0·062 (1)
C(18)	1·2221 (4)	−0·1461 (2)	0·1509 (3)	0·0593 (8)
C(20)	0·8763 (6)	−0·2512 (2)	0·3792 (5)	0·072 (1)
C(21)	0·7218 (7)	−0·2707 (2)	0·4036 (6)	0·101 (2)
C(22)	1·2451 (5)	−0·1932 (2)	−0·0010 (4)	0·077 (1)
C(23)	0·7852 (5)	−0·0792 (2)	−0·1885 (4)	0·073 (1)

* Coupled isotropic thermal parameter.

Discussion. The final atomic parameters are given in Table 1.* Fig. 1 shows the conformation and disorder in the *A* ring of the steroid molecule and the atom numbering. Bond distances and bond angles are given in Table 2 and their values correspond to those observed in related structures (Griffin, Duax & Weeks,

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44263 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1984), except those involving the disordered atoms. However, the torsion angles show some discrepancies and here we will concentrate on discussion of the conformation of the steroid molecule in relation to calculated and other observed geometries. The symmetry of the rings is illustrated by the use of asymmetry parameters (Duax & Norton, 1975), which measure the degree of departure from ideal mirror or twofold symmetry. The Δ^4 *A* ring has an inverted 1β,2α-half-chair conformation for model *A* {ΔC_2[C(1)–C(2)] = 1·8 (5)°} and a normal 1α,2β-half-chair conformation for model *B* {ΔC_2[C(1)–C(2)] = 2·1 (6)°}. However, these two parameters are not very accurate because it is difficult to refine properly the positions of the disordered atoms. The *B* and *C* rings have the usual chair conformation, where the 11-methylene group enhances the mirror symmetry of the *C* ring illustrated by ΔC_s[C(11)] = 0·3 (2)°, whereas all other relevant asymmetry parameters are in the range of 4·0–4·4°. The *D* ring has an intermediate 13β-envelope/13β,14α-half-chair with ΔC_s[C(13)] = 8·7 (3) and ΔC_2[C(13)–C(14)] = 13·2 (3)°.

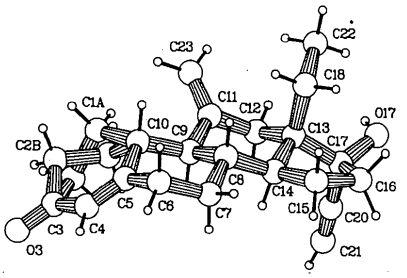

Fig. 1. Molecular structure with atom numbering of 3-ketodesogestrel. Atoms C(1) and C(2) in the *A* ring are disordered.

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

O(3)–C(3)	1·237 (4)	C(8)–C(14)	1·526 (5)
O(17)–C(17)	1·422 (4)	C(9)–C(11)	1·527 (5)
C(1*A*)–C(2*A*)	1·527 (7)*	C(9)–C(10)	1·552 (5)
C(1*B*)–C(2*B*)	1·524 (9)*	C(11)–C(12)	1·500 (5)
C(1*A*)–C(10)	1·535 (5)*	C(11)–C(23)	1·330 (4)
C(1*B*)–C(10)	1·557 (7)*	C(12)–C(13)	1·536 (4)
C(2*A*)–C(3)	1·504 (7)*	C(13)–C(17)	1·567 (5)
C(2*B*)–C(3)	1·491 (6)*	C(13)–C(18)	1·553 (4)
C(3)–C(4)	1·438 (5)	C(14)–C(15)	1·530 (4)
C(4)–C(5)	1·326 (5)	C(15)–C(16)	1·536 (5)
C(5)–C(6)	1·491 (5)	C(16)–C(17)	1·550 (5)
C(5)–C(10)	1·521 (5)	C(17)–C(20)	1·493 (5)
C(6)–C(7)	1·527 (5)	C(18)–C(22)	1·533 (4)
C(7)–C(8)	1·521 (5)	C(20)–C(21)	1·159 (6)
C(8)–C(9)	1·553 (4)		

C(2*A*)–C(1*A*)–C(10)	107·7 (3)	C(5)–C(10)–C(9)	109·1 (3)
C(2*B*)–C(1*B*)–C(10)	111·7 (4)	C(9)–C(11)–C(12)	113·2 (2)
C(1*A*)–C(2*A*)–C(3)	113·0 (5)	C(9)–C(11)–C(23)	124·4 (3)
C(1*B*)–C(2*B*)–C(3)	103·2 (4)	C(12)–C(11)–C(23)	122·2 (3)
O(3)–C(3)–C(2*A*)	122·9 (3)	C(11)–C(12)–C(13)	109·2 (3)
O(3)–C(3)–C(2*B*)	119·8 (4)	C(12)–C(13)–C(14)	107·9 (2)
O(3)–C(3)–C(4)	121·5 (3)	C(12)–C(13)–C(17)	117·3 (3)
C(2*A*)–C(3)–C(4)	113·8 (4)	C(12)–C(13)–C(18)	110·9 (2)
C(2*B*)–C(3)–C(4)	116·0 (4)	C(14)–C(13)–C(17)	98·9 (2)
C(3)–C(4)–C(5)	123·6 (3)	C(14)–C(13)–C(18)	111·6 (3)
C(4)–C(5)–C(6)	122·2 (3)	C(17)–C(13)–C(18)	109·6 (2)
C(4)–C(5)–C(10)	122·2 (3)	C(8)–C(14)–C(13)	115·4 (2)
C(6)–C(5)–C(10)	115·6 (3)	C(8)–C(14)–C(15)	119·4 (3)
C(5)–C(6)–C(7)	110·5 (3)	C(13)–C(14)–C(15)	104·3 (3)
C(6)–C(7)–C(8)	111·7 (3)	C(14)–C(15)–C(16)	104·0 (3)
C(7)–C(8)–C(9)	111·1 (3)	C(15)–C(16)–C(17)	106·5 (3)
C(7)–C(8)–C(14)	111·5 (2)	O(17)–C(17)–C(13)	115·9 (2)
C(9)–C(8)–C(14)	107·7 (3)	O(17)–C(17)–C(16)	107·7 (3)
C(8)–C(9)–C(10)	113·3 (3)	O(17)–C(17)–C(20)	108·8 (3)
C(8)–C(9)–C(11)	108·4 (2)	C(13)–C(17)–C(16)	103·1 (3)
C(10)–C(9)–C(11)	115·8 (3)	C(13)–C(17)–C(20)	110·0 (3)
C(1*A*)–C(10)–C(5)	113·7 (3)	C(16)–C(17)–C(20)	111·3 (3)
C(1*B*)–C(10)–C(5)	107·3 (3)	C(13)–C(18)–C(22)	117·1 (3)
C(1*A*)–C(10)–C(9)	123·0 (4)	C(17)–C(20)–C(21)	179·6 (4)
C(1*B*)–C(10)–C(9)	101·9 (3)		

* Restrained bond distance.

An identical disorder of ring *A* was observed for one of the two independent molecules in the crystal structure of nortestosterone (Precigoux, Busetta, Courseille & Hospital, 1975). Ring *A* was found to have a normal half-chair conformation as well as an inverted half-chair conformation in a 2:1 ratio, whereas in the ordered molecule the *A* ring has an intermediate 1α,2β-half-chair/2α-sofa conformation.

Duax, Fronckowiak, Griffin & Rohrer (1982) compared structures observed in X-ray determinations with calculated structures using molecular mechanics and they concluded that the observed torsion angles are not adequately reproduced. For 4-en-3-one steroids different patterns of torsion-angle variation were observed in X-ray and minimized structures, possibly because of unrealistically weighted constraints upon the conjugated 4-en-3-one system in the *A* ring. We performed energy minimizations of both models *A* and *B* with the *MMP*2 program (Allinger & Flanagan, 1983). Fig. 2 shows a fit of rings *C* and *D* of these minimized structures with their corresponding X-ray structures.

Additional calculations indicated that for the *A* ring an extensive range of conformational space is accessible at the expense of only a few kJ mol^{-1}. The difference in steric energy amounts to 4 kJ mol^{-1} in favour of the normal half-chair conformation, which is in accordance with the far larger number of steroids

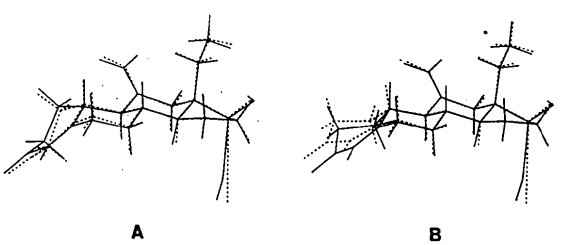

Fig. 2. Fit of *C* and *D* rings of observed (solid lines) and minimized structures (dashed lines). The *A* ring of the steroid molecule has an inverted half-chair conformation in model *A* and a normal half-chair conformation in model *B*.

with normal instead of inverted half-chair conformations in the Cambridge Crystallographic Database. However, receptor binding may favour an inverted conformation, implying a more favourable interaction to compensate for this small loss of energy.

The *MMP*2 program gives rather promising results in describing the conformation, *i.e.* torsion angles, of the steroid molecule. More extensive calculations on other 3-oxo-4-ene steroids are necessary to confirm these findings. Table 3 shows a comparison of some selected torsion angles of calculated and observed structures. The torsion angles for 3-ketodesogestrel are midway between the mean values observed in the Database for normal half-chair conformations and the values observed for 17α-acetoxy-6α-methylprogesterone. This observation is consistent with the averaged electron density obtained by X-ray diffraction and the 1:1 occupancy. The values of the minimized structures correspond to the literature values. It seems that the *MMP*2 program gives better results in describing 4-en-3-one steroids than the *MM2/P* program [*MM*2 with π-electron handling from *MMP*1; see Duax *et al.* (1982)], which does not reproduce the observed patterns of torsion-angle variation as was concluded by Duax *et al.* (1982). However, it has to be stressed that though the steric energy differences may be over- or underestimated, the inverted half-chair conformation is observed only exceptionally, so it may be assumed that this conformation really has a higher energy. The conformation of the angular ethyl group is approximately *trans* with respect to the C(13)–C(14) bond. The torsion angle C(14)–C(13)–C(18)–C(22) [−168·3 (3)°] is close to the mean value of 165 (3)° observed for 13-ethyl steroids with a saturated *D* ring (van Geerestein, Kanters, Duisenberg & Kroon, 1986). Fig. 3 shows the molecular packing in a stereoview down **a**. The steroid molecules are hydrogen-bonded from head to tail forming infinite chains parallel to **b**, O(17)→O(3)($1-x,-\frac{1}{2}+y,-z$) with O···O = 2·843 (3) Å and O–H···O = 164 (2)°. Two unrealistic short intermolecular H···H contact distances of 2·01 and 1·94 Å, involving disordered H atoms at C(1*A*) and C(1*B*) respectively, indicate the poor accuracy of the disordered C-atom coordinates, which were used to calculate the H-atom positions. One other intermolecular H···H contact between H atoms at C(12) and C(22) is less than 2·1 Å.

Table 3. *Comparison of selected torsion angles* (°) *for observed structures and structures minimized with MMP*2

	A-ring* conformation		*T*1	*T*2	*T*3
3-Ketodesogestrel	X-ray	*D*	−0·8 (6)	121·8 (4)	−125·5 (3)
	*MMP*2	*N*	−3·5	130·3	−137·7
	*MMP*2	*I*	3·7	114	−117·9
19-Nortestosterone	X-ray	*D*	−5·0	123·9	−127·9
	X-ray	*N*	−9·0	133·0	−136·7
Database (n = 20)	X-ray	*N*	−6 (2)	132 (3)	−135 (3)
Medroxyprogesterone acetate	X-ray	*I*	7·9	112·5	−114·5

*T*1 = C(3)–C(4)–C(5)–C(10); *T*2 = C(4)–C(5)–C(6)–C(7); *T*3 = C(4)–C(5)–C(10)–C(9).

* *D*: disordered structure; *N*: normal half-chair; *I*: inverted half-chair.

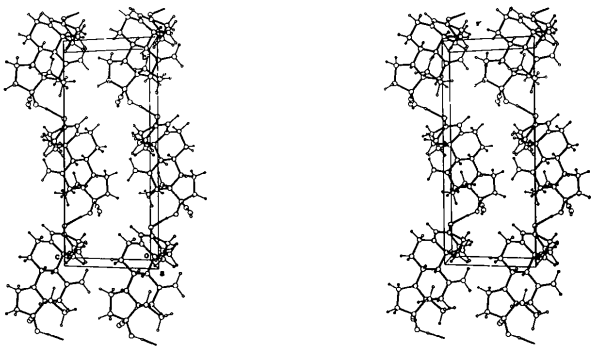

Fig. 3. Stereo packing diagram viewed down **a**.

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

References

ALLINGER, N. C. & FLANAGAN, H. L. (1983). *J. Comput. Chem.* **4**, 399–403.

BARRETT, M. W., FARRANT, R. D., KIRK, D. N., MERSH, J. D., SANDERS, J. K. M. & DUAX, W. L. (1982). *J. Chem. Soc. Perkin Trans.* 2, pp. 105–110.

BOER, J. L. DE & DUISENBERG, A. J. M. (1984). *Acta Cryst.* A**40**, C410.

DUAX, W. L. (1986). Collected Abstracts, 10th Eur. Crystallogr. Meet., Wrocław, pp. 30–31.

DUAX, W. L., CODY, V., GRIFFIN, J. F., HAZEL, J. & WEEKS, C. M. (1978). *J. Steroid Biochem.* **9**, 901–907.

DUAX, W. L., FRONCKOWIAK, M. D., GRIFFIN, J. F. & ROHRER, D. C. (1982). In *Intramolecular Dynamics*, edited by J. JORTNER & B. PULLMAN, pp. 508–524. Dordrecht: Reidel.

DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, Vol. I, pp. 16–22. New York: IFI/Plenum Data Co.

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

GEERESTEIN, V. J. VAN, KANTERS, J. A., DUISENBERG, A. J. M. P. & 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: IFI/Plenum Data Co.

PRECIGOUX, G., BUSETTA, B., COURSEILLE, C. & HOSPITAL, M. (1975). *Acta Cryst.* B**31**, 1527–1532.

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.