there is evidently rapid inversion of the *B* rings at room temperature since the 300 MHz ^{1}H NMR spectrum indicates magnetic equivalence of the H atoms on the aliphatic bridge C(15) and C(16) atoms.

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Structure of 13-Ethyl-17β-hydroxy-11-methylene-18,19-dinor-17α-pregna-4,15-dien-20-yn-3-one (11-Methylenegestodene)

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Abstract. $C_{22}H_{26}O_2$, $M_r = 322{\cdot}45$ (×2), monoclinic, $P2_1$, $a = 11{\cdot}611$ (4), $b = 13{\cdot}316$ (3), $c = 12{\cdot}578$ (1) Å, $\beta = 112{\cdot}78$ (2)°, $V = 1793{\cdot}1$ (8) Å^3, $Z = 4$, $D_x = 1{\cdot}194$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 5$ cm^{-1}, $F(000) = 696$, room temperature, $R = 0{\cdot}045$ for 3260 unique reflections with $I \geq 2{\cdot}5\sigma(I)$. The two independent molecules have different conformations with respect to the orientation of the ethyl group and the steroid skeleton in the *A*-ring region. The conformation of the ethyl group, with respect to the *C/D* ring junction, is *trans* and *gauche* above the *D* ring respectively. This observation corresponds to the conformations found for gestodene. The observed conformational variation in the *A*-ring region illustrates the flexibility of this part of the steroid molecule. Molecular mechanics gives a steric energy difference between the relaxed structures of 3 kJ mol^{-1} in favour of the structure with the ethyl group in the *gauche* conformation. Two independent chains of hydrogen-bonded translational-equivalent molecules are formed parallel to **b**.

Introduction. 11-Methylenegestodene is a hybrid molecule of 3-ketodesogestrel (13-ethyl-17β-hydroxy-11-methylene-18,19-dinor-17α-pregn-4-en-20-yn-3-one) and gestodene (13-ethyl-17β-hydroxy-18,19-dinor-17α-pregna-4,15-dien-20-yn-3-one). Gestodene is a new orally active progestogen (*e.g.* Losert, Casals-Stenzel & Buse, 1985), while 3-ketodesogestrel is the active metabolite of desogestrel, which has been marketed for oral contraception for some time. For gestodene two different crystal modifications have been observed (van Geerestein, Duisenberg, Duitz, Kanters & Kroon, 1987), wherein the steroid molecules differ in conformation with respect to the orientation of the ethyl group. The conformation with the ethyl group oriented above the *D* ring is energetically favourable in a Δ^{15} steroid, in contrast to steroids with a saturated *D* ring, where the *trans* conformation with respect to the *C/D* ring junction is more favourable. In the crystal structure, the *A* ring of 3-ketodesogestrel was found to be disordered with respect to the positions of C(1) and C(2) (van Geerestein, Kanters & Kroon, 1987). It has been postulated that the conformation of the *A* ring is closely related to the receptor binding. An inverted 1β,2α-half-chair conformation for the *A* ring should be related to a high affinity for the progestogen receptor (*e.g.* Duax, 1986, and references therein).

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

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

	Molecule I				Molecule II			
	x	y	z	U_{eq}	x	y	z	U_{eq}
O(3)	0·4998 (2)	0·4408 (2)	0·2951 (2)	0·0806 (8)	0·1059 (4)	0·2095 (2)	0·3356 (3)	0·122 (2)
O(17)	0·4899 (3)	1·2622 (2)	0·1595 (2)	0·078 (1)	0·0394 (2)	1·0244 (2)	0·2150 (2)	0·0628 (8)
C(1)	0·4995 (3)	0·6771 (2)	0·1519 (2)	0·051 (1)	0·1550 (3)	0·4558 (3)	0·2391 (3)	0·068 (1)
C(2)	0·4494 (3)	0·5715 (2)	0·1549 (3)	0·054 (1)	0·1448 (4)	0·3411 (3)	0·2266 (4)	0·085 (2)
C(3)	0·4938 (3)	0·5316 (2)	0·2757 (3)	0·056 (1)	0·0845 (5)	0·2970 (3)	0·3006 (4)	0·091 (2)
C(4)	0·5254 (3)	0·6049 (2)	0·3677 (3)	0·058 (1)	−0·0049 (4)	0·3586 (3)	0·3258 (3)	0·081 (2)
C(5)	0·5120 (3)	0·7039 (2)	0·3512 (2)	0·045 (1)	−0·0368 (4)	0·4522 (3)	0·2847 (3)	0·063 (1)
C(6)	0·5357 (3)	0·7716 (2)	0·4523 (2)	0·054 (1)	−0·1420 (4)	0·5059 (3)	0·3010 (4)	0·076 (2)
C(7)	0·5988 (3)	0·8703 (2)	0·4433 (2)	0·049 (1)	−0·1057 (3)	0·6126 (3)	0·3447 (3)	0·061 (1)
C(8)	0·5213 (3)	0·9231 (2)	0·3306 (2)	0·042 (1)	−0·0630 (3)	0·6716 (2)	0·2632 (2)	0·045 (1)
C(9)	0·5121 (2)	0·8563 (2)	0·2259 (2)	0·0394 (8)	0·0490 (3)	0·6189 (2)	0·2491 (2)	0·045 (1)
C(10)	0·4660 (3)	0·7485 (2)	0·2313 (2)	0·042 (1)	0·0291 (3)	0·5056 (2)	0·2189 (3)	0·051 (1)
C(11)	0·4391 (3)	0·9112 (2)	0·1131 (2)	0·043 (1)	0·0944 (2)	0·6812 (2)	0·1711 (2)	0·045 (1)
C(12)	0·4881 (3)	1·0150 (2)	0·1027 (2)	0·047 (1)	0·1318 (3)	0·7875 (2)	0·2121 (3)	0·045 (1)
C(13)	0·4934 (3)	1·0798 (2)	0·2042 (3)	0·046 (1)	0·0206 (3)	0·8406 (2)	0·2255 (2)	0·042 (1)
C(14)	0·5761 (3)	1·0233 (2)	0·3156 (2)	0·044 (1)	−0·0185 (3)	0·7772 (2)	0·3077 (2)	0·044 (1)
C(15)	0·6117 (3)	1·1052 (2)	0·4047 (3)	0·057 (1)	−0·0928 (3)	0·8512 (3)	0·3469 (3)	0·055 (1)
C(16)	0·6104 (3)	1·1924 (3)	0·3548 (3)	0·063 (1)	−0·0547 (3)	0·9438 (3)	0·3395 (3)	0·057 (1)
C(17)	0·5665 (3)	1·1819 (2)	0·2244 (3)	0·055 (1)	0·0465 (3)	0·9432 (2)	0·2905 (3)	0·049 (1)
C(18)	0·3603 (3)	1·0992 (3)	0·2017 (3)	0·060 (1)	−0·0810 (3)	0·8536 (2)	0·1019 (2)	0·049 (1)
C(20)	0·6780 (3)	1·1733 (3)	0·1958 (3)	0·058 (1)	0·1687 (3)	0·9417 (2)	0·3881 (3)	0·054 (1)
C(21)	0·7697 (4)	1·1631 (3)	0·1783 (4)	0·076 (1)	0·2654 (3)	0·9343 (3)	0·4675 (3)	0·063 (1)
C(22)	0·2604 (3)	1·1315 (3)	0·0857 (3)	0·077 (1)	−0·2134 (3)	0·8884 (3)	0·0832 (3)	0·069 (1)
C(23)	0·3388 (3)	0·8746 (3)	0·0295 (3)	0·061 (1)	0·0938 (3)	0·6498 (3)	0·0707 (3)	0·056 (1)

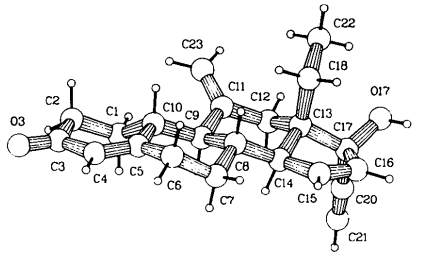
Experimental. Water was slowly added to a 1-propanol solution of 11-methylenegestodene until a slight turbidity was observed. Suitable crystals appeared after 24 h. Data were collected on a crystal of approximate dimensions 0·5 × 0·5 × 0·25 mm on an Enraf–Nonius CAD-4 diffractometer with Ni-filtered Cu $K\alpha$ radiation, lattice parameters refined by least-squares fitting of 2θ values of 6 reflections in the range 64–70°; ω–2θ scan mode, $\Delta\omega = (0{\cdot}6 + 0{\cdot}15\tan\theta)°$, 3556 independent reflections were measured up to $2\theta = 140°$ of which 3260 with $I \geq 2{\cdot}5\sigma(I)$ were considered observed and used for structure refinement; h, k, $\pm l$ (maximum range 14, 15, 15). Three periodically measured reflections (200, 040, 002) showed a maximum variation in intensity of 2%. Data were corrected for this variation in scattering power and for the usual Lp factors.

The structure was solved by direct methods by default run with the *SHELXS*86 program (Sheldrick, 1986). The E map revealed all 46 non-H positions of the two independent molecules. C and O atoms were refined positionally with anisotropic thermal parameters. H atoms bonded to the steroid skeleton were included at calculated positions and refined riding on their bonded atoms. The ethynyl and methylene H atoms were located on a difference map and refined positionally. Even at $R = 0{\cdot}049$, it was not possible to locate the hydroxyl H-atom positions. However, after absorption correction with the *DIFABS* program (Walker & Stuart, 1983) and after convergence was reached in the subsequent refinement on the corrected data set, both H-atom positions were easily found in the difference Fourier map, and included in the refinement. The minimum and maximum applied absorption factors were 0·78 and 0·99 respectively. The isotropic R factor was decreased by the absorption correction from 0·104 to 0·088. The mean-square amplitude of vibration for the H atoms was fixed at $U = 0{\cdot}08$ $Å^2$. Parameters were varied on F in two-block full-matrix refinement with 230 variables contained in both of them. The refinement converged at $R = 0{\cdot}045$ and $wR = 0{\cdot}048$ $[w = 1/\sigma^2(F)]$ with $(\Delta/\sigma)_{av} = 0{\cdot}01$ for all parameters and $(\Delta/\sigma)_{max} = 0{\cdot}1$; $S = 0{\cdot}5$. The electron density in final difference Fourier map was within $\pm 0{\cdot}2$ e $Å^{-3}$. Refinement was carried out with the *SHELX*76 program (Sheldrick, 1976) and geometry calculations were performed with the *EUCLID* package (Spek, 1982). Scattering factors were taken from *SHELX*.

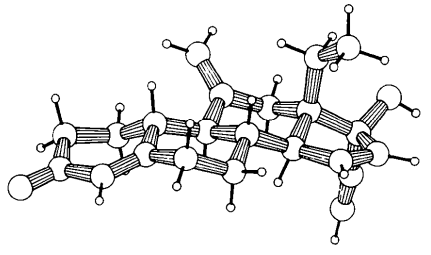
Discussion. The two independent steroid molecules are referred to as molecules I and II respectively. The final atomic parameters are given in Table 1.* Fig. 1 shows the atom-numbering scheme and the conformations of molecules I and II. Bond distances and bond angles involving non-H atoms are given in Table 2 and correspond to those observed in related structures (Griffin, Duax & Weeks, 1984). The mean differences in corresponding bond distances and bond angles for I and II are 0·007 (5) Å and 1 (1)° respectively. The largest differences involve atom C(13) and are believed to be related to the different ethyl-group orientations. The differences in the observed torsion angles for I and II are generally within a few degrees, except for torsion angles involving the A ring and the A/B ring junction and the obviously different torsion angles describing the C(13) ethyl-group orientation. The A/B ring junction is for instance defined by C(4)–C(5)–C(10)–C(9), which

* 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 44467 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

is $-152{\cdot}4\,(3)°$ in I and $-141{\cdot}2\,(3)°$ in II. For I the conformation of the angular ethyl group is approximately *trans* with respect to the C(13)–C(14) bond, C(14)–C(13)–C(18)–C(22) = $-166{\cdot}0\,(3)°$. For II this conformation is *gauche* with C(14)–C(13)–C(18)–C(22) = $50{\cdot}8\,(3)°$. These two conformations correspond to those found in modifications I and II for gestodene respectively. The symmetry of the rings is illustrated by the use of asymmetry parameters (*e.g.* Griffin, Duax & Weeks, 1984). Both Δ^4 *A* rings have normal conformations. Mirror symmetry is predominant over twofold symmetry, and the *A*-ring conformations are therefore qualitatively described as sofas, illustrated by $\Delta C_s|C(1)| = 0{\cdot}7\,(4)$ and $8{\cdot}2\,(5)°$ for I and II respectively. The *B* rings have distorted-chair conformations and the *C* rings have a chair conformation with some enhanced mirror symmetry due to the 11-methylene group. The *D* ring has the usual 13β-envelope conformation imposed by the Δ^{15} unsaturation with $\Delta C_s|C(13)| = 4{\cdot}4\,(4)°$ for I and $2{\cdot}9\,(4)°$ for II. The difference in the overall conformation of the steroid backbone of I and II is related to differences in conformation in the *A*- and *B*-ring regions. This is illustrated in Fig. 2, which shows a least-squares superposition of the *D* rings of both molecules. Steric energy relaxation of both structures with the *MMP*2(85) molecular-mechanics program (Allinger & Flanagan, 1983) resulted in a more favourable energy for the structure with the *gauche* conformation of 3 kJ mol^{-1}.



(I)



(II)

Fig. 1. Molecular conformation of 11-methylenegestodene (molecules I and II).

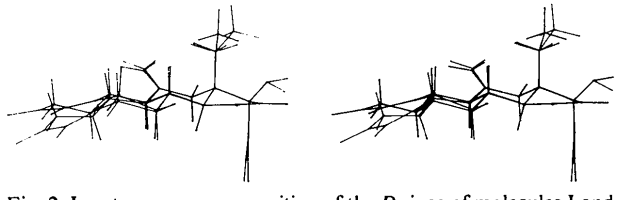


Fig. 2. Least-squares superposition of the *D* rings of molecules I and II.

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

		I	II			I	II
O(3)	C(3)	1·230 (4)	1·236 (5)	C(2)	C(1) C(10)	111·1 (3)	112·5 (3)
O(17)	C(17)	1·428 (4)	1·420 (4)	C(1)	C(2) C(3)	111·5 (3)	111·4 (4)
C(1)	C(2)	1·528 (4)	1·535 (6)	O(3)	C(3) C(2)	121·3 (3)	121·1 (5)
C(1)	C(10)	1·534 (4)	1·534 (5)	O(3)	C(3) C(4)	121·7 (3)	121·1 (5)
C(2)	C(3)	1·500 (5)	1·485 (7)	C(2)	C(3) C(4)	116·9 (2)	117·6 (4)
C(3)	C(4)	1·449 (4)	1·451 (7)	C(3)	C(4) C(5)	124·3 (3)	123·6 (4)
C(4)	C(5)	1·334 (4)	1·345 (6)	C(4)	C(5) C(6)	119·5 (2)	120·5 (4)
C(5)	C(6)	1·494 (4)	1·496 (7)	C(4)	C(5) C(10)	121·2 (2)	121·7 (4)
C(5)	C(10)	1·514 (3)	1·506 (6)	C(6)	C(5) C(10)	119·3 (2)	117·7 (3)
C(6)	C(7)	1·530 (4)	1·523 (6)	C(5)	C(6) C(7)	113·0 (2)	111·6 (4)
C(7)	C(8)	1·525 (4)	1·519 (5)	C(6)	C(7) C(8)	109·7 (2)	110·9 (3)
C(8)	C(9)	1·558 (4)	1·547 (5)	C(7)	C(8) C(9)	110·2 (2)	110·6 (2)
C(8)	C(14)	1·521 (4)	1·528 (4)	C(7)	C(8) C(14)	113·3 (2)	112·4 (2)
C(9)	C(10)	1·543 (4)	1·551 (4)	C(9)	C(8) C(14)	106·9 (2)	106·2 (3)
C(9)	C(11)	1·528 (3)	1·526 (4)	C(8)	C(9) C(10)	113·9 (2)	114·7 (3)
C(11)	C(12)	1·520 (4)	1·512 (4)	C(8)	C(9) C(11)	110·1 (2)	110·0 (2)
C(11)	C(23)	1·323 (5)	1·328 (4)	C(10)	C(9) C(11)	114·1 (2)	115·3 (2)
C(12)	C(13)	1·522 (4)	1·539 (5)	C(1)	C(10) C(5)	109·0 (2)	111·1 (3)
C(13)	C(14)	1·552 (4)	1·533 (4)	C(1)	C(10) C(9)	111·6 (2)	109·7 (3)
C(13)	C(17)	1·571 (4)	1·561 (4)	C(5)	C(10) C(9)	114·5 (2)	112·6 (3)
C(13)	C(18)	1·555 (5)	1·555 (4)	C(9)	C(11) C(12)	115·1 (2)	114·5 (2)
C(14)	C(15)	1·503 (4)	1·513 (5)	C(9)	C(11) C(23)	124·2 (3)	124·4 (3)
C(15)	C(16)	1·317 (5)	1·325 (6)	C(12)	C(11) C(23)	120·7 (3)	120·9 (3)
C(16)	C(17)	1·524 (5)	1·524 (5)	C(11)	C(12) C(13)	109·8 (2)	108·9 (3)
C(17)	C(20)	1·476 (5)	1·474 (5)	C(12)	C(13) C(14)	107·0 (2)	107·5 (2)
C(18)	C(22)	1·533 (5)	1·534 (5)	C(12)	C(13) C(17)	118·0 (3)	117·4 (3)
C(20)	C(21)	1·176 (6)	1·183 (5)	C(12)	C(13) C(18)	111·1 (3)	106·6 (2)
				C(14)	C(13) C(17)	99·6 (3)	100·1 (2)
				C(14)	C(13) C(18)	111·2 (3)	115·3 (3)
				C(17)	C(13) C(18)	109·3 (3)	110·1 (2)
				C(8)	C(14) C(13)	113·3 (2)	114·0 (2)
				C(8)	C(14) C(15)	123·5 (2)	124·4 (3)
				C(13)	C(14) C(15)	102·5 (2)	102·5 (2)
				C(14)	C(15) C(16)	109·6 (3)	109·6 (3)
				C(15)	C(16) C(17)	111·7 (3)	110·7 (3)
				O(17)	C(17) C(16)	114·9 (3)	114·3 (3)
				O(17)	C(17) C(20)	109·3 (3)	110·8 (3)
				C(13)	C(17) C(16)	100·8 (3)	101·2 (3)
				O(17)	C(17) C(13)	112·2 (3)	111·5 (3)
				C(13)	C(17) C(20)	111·3 (3)	110·6 (2)
				C(16)	C(17) C(20)	108·1 (3)	107·9 (3)
				C(13)	C(18) C(22)	116·5 (3)	120·9 (3)
				C(17)	C(20) C(21)	176·3 (4)	175·8 (3)

These observations illustrate once more our view that the *A* ring, and *A*/*B* ring junction, in Δ^4-3-one steroids is rather flexible. The normal *A*-ring conformation is observed in the crystal structures almost without exception (Duax, Fronckowiak, Griffin & Rohrer, 1982), which makes us believe that this normal conformation (*versus* the inverted half-chair conformation) is the low-energy conformation. Molecular-mechanics calculations on 3-ketodesogestrel confirmed this view, which showed a more favourable energy for the normal conformation above the inverted conformation of 4 kJ mol^{-1} (van Geerestein, Kanters & Kroon, 1987). A possible conformational adaptation at the receptor site will easily be accomplished within the normal conformation with atom C(1) on the α side of the steroid molecule. The observations on the ethyl-group conformation in the present crystal structure as well as in the structures of both modifications of gestodene indicate a small difference in energy between

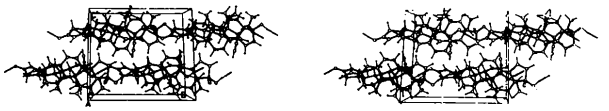


Fig. 3. Stereo packing diagram, viewed down **a**. Hydrogen bonds are indicated.

the *trans* and *gauche* ethyl-group conformers. This is supported by our molecular-mechanics calculations. Both conformers may be found in solution. At the receptor site the ethyl group can adjust itself to its most favourable orientation.

Fig. 3 shows the molecular packing in a stereoview down **a**. The steroid molecules are hydrogen bonded head to tail forming two symmetrically independent chains parallel to **b** for both molecules I and II given by: O(17)→O(3′) (x, $1+y$, z) with distances O···O′ = 2·903 (4) and 2·840 (4), O–H = 0·97 (4) and 1·04 (2), H···O′ = 1·95 (4) and 1·94 (4) Å and angles O–H···O′ = 167 (3) and 174 (3)° respectively. The torsion angle C(16)–C(17)–O(17)–H is different for the two molecules, 17 (2)° for I and 73 (3)° for II. All other intermolecular contacts are at normal van der Waals separations.

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Structure of (1α,2α,4α,6α)-3,7-Dibenzoyl-3,7-diazatricyclo[4.1.0.0^{2,4}]heptane

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Abstract. $C_{19}H_{16}N_2O_2$, $M_r = 304{\cdot}35$, orthorhombic, $P2_12_12_1$, $a = 21{\cdot}150\ (3)$, $b = 11{\cdot}666\ (2)$, $c = 12{\cdot}633\ (2)$ Å, $V = 3117{\cdot}0\ (9)$ Å^3, $Z = 8$, $D_x = 1{\cdot}30$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 6{\cdot}03$ cm^{-1}, $F(000) = 1280$, room temperature, $R = 0{\cdot}0968$ for 1296 reflections with $I > 2{\cdot}5\sigma(I)$. The molecule exists in an *exo, endo* modification having two non-equivalent aziridine N atoms. The central five-membered ring is planar, and the two three-membered rings are *cis* to each other.

Introduction. The stereochemical analysis of a *cis*-diaziridinocyclopentane derivative has been recently carried out (Majchrzak, Kotełko & Lambert, 1983) by means of ^{1}H and ^{13}C NMR and IR spectra. The authors report that the molecule exists in an *exo, endo* modification which might be the first known example of the interconversion of two diastereotopic amine NH groups within the same molecule.

The present X-ray structure determination was undertaken to ascertain the stereochemistry at both N atoms, and provide X-ray evidence for the structure of the molecule.

Experimental. Colourless crystals from chloroform; crystal dimensions ~0·2 × 0·2 × 0·2 mm. Hilger–Watts Y290 diffractometer; unit-cell dimensions from 12 reflections with $\theta_{max} = 45{\cdot}1°$; intensity by $\omega/2\theta$ scan technique; total of 2660 reflections recorded to $\sin\theta/\lambda = 0{\cdot}5559$ Å^{-1}; $0 \le h \le 23$, $0 \le k \le 12$, $0 \le l \le$

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