Related literature. CC-1065 is a powerful antitumor agent (Martin, Chidester, Duchamp & Mizsak, 1980; Chidester, Krueger, Mizsak, Duchamp & Martin, 1981), and the derivatized chiral fragment of CC-1065 is described (Martin, Kelly, Watt, Wicnienski, Mizsak, Nielsen & Prairie, 1988).

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Structure of the Steroid Molecule 17β-Hydroxy-1α-methyl-5α-androstan-3-one (Mesterolone)

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Abstract. $C_{20}H_{32}O_2$, $M_r = 304{\cdot}5$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}123$ (3), $b = 16{\cdot}938$ (4), $c = 18{\cdot}561$ (3) Å, $V = 3497$ (1) Å^3, $Z = 8$, $D_x = 1{\cdot}157$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 5{\cdot}24$ cm^{-1}, $F(000) = 1344$, $T = 293$ K, final $R = 0{\cdot}042$, $wR = 0{\cdot}053$, for 4105 observed reflections and 416 variables. The hexane rings of the two independent molecules in the asymmetric unit have chair conformations, and the *D* rings are intermediate between 13β-envelope and 13β,14α-half-chair conformations.

Experimental. A crystal approximately 0·29 × 0·09 × 0·1 mm was used for the measurements. Throughout the experiment Cu $K\alpha$ radiation was used with a graphite-crystal monochromator on a Nonius CAD-4 single-crystal diffractometer ($\lambda = 1{\cdot}54178$ Å). The unit-cell dimensions were determined from the angular settings of 25 reflections with $10 < \theta < 28°$. The space group was determined from systematic extinctions and the structure determination. The intensity data of 13 671 reflections were measured (half a sphere up to $\theta = 70°$; h −13→0, k −20→20, l −22→22) using the ω–2θ scan technique, with a scan angle of 1·5° and a variable scan rate with a maximum scan time of 20 s per reflection. The intensity of the primary beam was checked throughout the data collection by monitoring three standard reflections every 30 min. The final drift correction factors were between 0·94 and 1·03. A profile analysis (Lehmann & Larsen, 1974; Grant & Gabe, 1978) was performed on all reflections. Lorentz and polarization corrections were applied and the data were reduced to $|F_o|$ values. Symmetry equivalent reflections were averaged, $R_{int} = 0{\cdot}056$ for all reflections and 0·022 for the observed reflections only, resulting in 6636 unique reflections of which 4105 were observed with $F > 6\sigma(F)$.

A misplaced fragment of nine atoms was found with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and this fragment was input to reciprocal translation functions (Beurskens, Gould, Bruins Slot & Bosman, 1987) and automatically expanded by *DIRDIF* (Beurskens, Bosman, Doesburg, van den Hark, Prick, Noordik, Beurskens, Gould & Parthasarathi, 1983) for the location of atoms of the two independent molecules.

Isotropic least-squares refinement, using *SHELX*76 (Sheldrick, 1976), converged to $R = 0{\cdot}10$. At this stage an additional empirical absorption correction was

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applied (Walker & Stuart, 1983), resulting in a further decrease of R to 0·08. Relative absorption correction factors were in the range 0·897–1·311. The Bijvoet coefficient, using 1813 Bijvoet pairs, was −0·063 (30) and therefore the structure was inverted.

The oxygen-bonded H atoms were found by a difference Fourier synthesis and included in the refinement. During the final stages of refinement all positional parameters, and the anisotropic temperature factors of all non-H atoms, were refined. The H atoms had fixed isotropic temperature factors, approximately equal to the isotropic equivalent of the parent C- or O-atom temperature factors.

The H atoms of the secondary CH_2 groups were refined riding on the parent C atoms with a distance of 1·00 Å. The methyl groups were refined as rigid groups. The final conventional agreement factors were $R = 0{\cdot}042$ and $wR = 0{\cdot}053$ for 4105 observed reflections and 416 variables. The function minimized was $\sum w(F_o - F_c)^2$ with $w = 1/[\sigma^2(F_o) + 0{\cdot}001F_o^2]$ with $\sigma(F_o)$ from counting statistics. The maximum shift over e.s.d. in the last full-matrix least-squares cycle was less than 0·04. The final difference Fourier map showed maximum peaks at 0·31 e Å^{-3}. The scattering factors used were those from *International Tables for X-ray Crystallography* (1974). Plots were made with *PLUTO* (Motherwell, 1976).

Final positional and thermal parameters are given in Table 1. The bond lengths of both molecules are collected in Table 2. A stereoview of the molecule, showing the molecular configuration, is given in Fig. 1. The crystallographic numbering scheme is given in Fig. 2.*

Related literature. The title molecule, whose trivial name is mesterolone or 1α-methyl-5α-dihydrotestosterone, is one of a series of steroid molecules. Details of the source and chemical background of mesterolone are given by Danaci & Kendi (1983). It is an androstane derivative and possesses high androgenic activity. Its skeleton consists of three cyclohexane rings and one cyclopentane ring. A hydroxyl group and a methyl group are bonded to the cyclopentane ring, and two methyl groups and a carbonyl group are bonded to the cyclohexane ring. The crystal structure deter-

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, positional and thermal parameters for H atoms, bond angles, torsion angles and asymmetry parameters, geometrical and conformational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51058 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

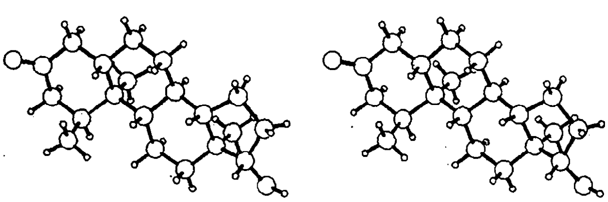

Fig. 1. Stereoview of one molecule showing the molecular configuration.

Table 1. *Fractional positional and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j U_{ij}.$$

	x	y	z	U_{eq}(Å^2 × 10^2)
Molecule (I)				
C(1)	−0·75293 (23)	−0·30474 (17)	−0·37785 (15)	4·23 (9)
C(2)	−0·85558 (27)	−0·25585 (20)	−0·41135 (18)	5·59 (11)
C(3)	−0·88848 (27)	−0·18657 (18)	−0·36571 (18)	5·01 (10)
C(4)	−0·78680 (26)	−0·13484 (17)	−0·34345 (18)	5·02 (10)
C(5)	−0·68224 (22)	−0·18303 (15)	−0·31121 (15)	3·73 (8)
C(6)	−0·58086 (25)	−0·12961 (16)	−0·28766 (18)	4·78 (10)
C(7)	−0·48241 (24)	−0·17648 (16)	−0·24950 (16)	4·21 (9)
C(8)	−0·43643 (21)	−0·24460 (15)	−0·29610 (14)	3·12 (7)
C(9)	−0·54188 (21)	−0·29795 (14)	−0·31980 (14)	3·05 (8)
C(10)	−0·64122 (23)	−0·25137 (15)	−0·36123 (13)	3·42 (8)
C(11)	−0·49955 (24)	−0·37209 (16)	−0·35968 (16)	4·40 (9)
C(12)	−0·39983 (25)	−0·41768 (16)	−0·32049 (16)	4·40 (9)
C(13)	−0·29524 (23)	−0·36356 (15)	−0·30199 (13)	3·46 (8)
C(14)	−0·34406 (22)	−0·29453 (15)	−0·25695 (14)	3·36 (8)
C(15)	−0·23042 (23)	−0·25543 (17)	−0·22721 (17)	4·51 (9)
C(16)	−0·14246 (25)	−0·32544 (18)	−0·21567 (18)	5·29 (11)
C(17)	−0·20346 (27)	−0·39737 (17)	−0·24883 (15)	4·43 (9)
C(18)	−0·22970 (27)	−0·33491 (18)	−0·37036 (16)	4·82 (10)
C(19)	−0·5907 (3)	−0·21964 (20)	−0·43298 (15)	5·09 (10)
C(20)	−0·80158 (27)	−0·35170 (17)	−0·31420 (18)	5·10 (11)
O(1)	−0·99190 (19)	−0·17343 (14)	−0·34756 (15)	7·15 (9)
O(2)	−0·12502 (19)	−0·45182 (14)	−0·28322 (12)	6·17 (8)
H(1)	−0·068 (3)	−0·4682 (18)	−0·2494 (17)	6·0
Molecule (II)				
C(21)	0·13547 (25)	−0·14558 (19)	0·01966 (16)	5·00 (10)
C(22)	0·2503 (3)	−0·15573 (23)	−0·02638 (20)	6·63 (13)
C(23)	0·2222 (3)	−0·18699 (19)	−0·09955 (19)	5·77 (12)
C(24)	0·12609 (28)	−0·14415 (18)	−0·13985 (16)	5·24 (11)
C(25)	0·01381 (24)	−0·12980 (16)	−0·09342 (14)	3·83 (8)
C(26)	−0·08111 (26)	−0·08312 (17)	−0·13517 (15)	4·36 (9)
C(27)	−0·19741 (26)	−0·07916 (18)	−0·09336 (13)	4·29 (9)
C(28)	−0·17973 (22)	−0·04505 (14)	−0·01760 (13)	3·00 (7)
C(29)	−0·07767 (22)	−0·08732 (15)	0·02351 (13)	3·06 (7)
C(30)	0·04205 (23)	−0·09221 (15)	−0·01954 (15)	3·48 (8)
C(31)	−0·06276 (23)	−0·05189 (18)	0·09919 (14)	4·25 (9)
C(32)	−0·18049 (25)	−0·05203 (17)	0·14267 (13)	4·16 (9)
C(33)	−0·28280 (22)	−0·01270 (14)	0·10174 (13)	3·24 (8)
C(34)	−0·29290 (23)	−0·05216 (15)	0·02828 (13)	3·31 (8)
C(35)	−0·41431 (26)	−0·02357 (20)	−0·00133 (15)	5·17 (11)
C(36)	−0·49296 (27)	−0·01689 (21)	0·06605 (16)	5·51 (11)
C(37)	−0·40886 (25)	−0·02994 (16)	0·13034 (15)	4·09 (9)
C(38)	−0·26217 (27)	0·07687 (15)	0·09700 (18)	4·76 (10)
C(39)	0·0967 (3)	−0·00942 (17)	−0·02842 (19)	5·27 (10)
C(40)	0·0876 (3)	−0·22729 (20)	0·04149 (20)	6·71 (13)
O(21)	0·27559 (25)	−0·24314 (15)	−0·12458 (14)	8·37 (10)
O(22)	−0·44462 (19)	0·01266 (13)	0·19375 (11)	5·01 (7)
H(21)	−0·455 (3)	0·0593 (19)	0·1815 (18)	6·0

Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

	Mol. (I)	Mol. (II)		Mol. (I)	Mol. (II)
C(1)–C(2)	1·541 (4)	1·546 (4)	C(9)–C(10)	1·560 (4)	1·555 (4)
C(1)–C(10)	1·567 (4)	1·558 (4)	C(9)–C(11)	1·532 (4)	1·536 (4)
C(1)–C(20)	1·524 (4)	1·537 (5)	C(10)–C(19)	1·542 (4)	1·537 (4)
C(2)–C(3)	1·493 (5)	1·491 (5)	C(11)–C(12)	1·535 (4)	1·538 (4)
C(3)–C(4)	1·489 (4)	1·493 (5)	C(12)–C(13)	1·520 (4)	1·522 (4)
C(3)–O(1)	1·219 (4)	1·214 (4)	C(13)–C(14)	1·537 (4)	1·523 (3)
C(4)–C(5)	1·542 (4)	1·537 (4)	C(13)–C(17)	1·531 (4)	1·527 (4)
C(5)–C(6)	1·510 (4)	1·530 (4)	C(13)–C(18)	1·542 (4)	1·537 (4)
C(5)–C(10)	1·533 (4)	1·544 (4)	C(14)–C(15)	1·530 (4)	1·536 (4)
C(6)–C(7)	1·527 (4)	1·510 (4)	C(15)–C(16)	1·552 (4)	1·530 (4)
C(7)–C(8)	1·530 (4)	1·533 (3)	C(16)–C(17)	1·524 (4)	1·532 (4)
C(8)–C(9)	1·545 (3)	1·544 (3)	C(17)–O(2)	1·421 (4)	1·437 (3)
C(8)–C(14)	1·516 (4)	1·525 (3)	O(2)–H(1)	0·94 (3)	0·83 (3)

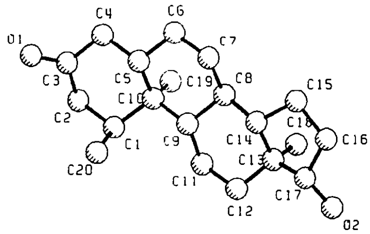

Fig. 2. Crystallographic numbering scheme.

mination was undertaken to determine the effect of the 1α-methyl group in a ring and the configuration at the junction of the *C*, *D* rings. The conformations of the steroid *A*, *B* and *C* rings are close to an ideal chair and that of the *D* ring is intermediate between a 13β envelope and 13β,14α half-chair. The major difference between the two independent molecules (which have the same absolute configuration) is in the direction of the hydroxyl groups. The two independent molecules are linked by one hydrogen bond of the two hydroxyl groups O(2)—H(1)···O(22) with a distance of 2·801 (3) Å. The hydroxyl H(21) atom is not involved in a hydrogen bond. The main conformational difference between the two molecules is a difference of about 6° at the *B*–*C* junction [for molecule (I) C(7)—C(8)—C(9)—C(10) = 56·9 (3)° and C(14)—C(8)—C(9)—C(11) = −50·2 (3)°; these angles for molecule (II) are 51·4 (3) and −56·5 (3)°, respectively].

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Structure of an Indoloquinolizine Derivative

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Abstract. Racemic (2*R*,3*S*,12b*S*)-2-(1,3-dimethyl-2,4,6-trioxo-1,3-diazacyclohexyl)-3-ethyl-1,2,3,4,5,6-hexahydro-12b*H*-indolo[2,3-*a*]quinolizine methanol solvate, $C_{23}H_{28}N_4O_3.CH_3OH$, $M_r = 440·5$, monoclinic, $P2_1/n$, $a = 11·726$ (2), $b = 15·337$ (2), $c = 12·400$ (2) Å, $\beta = 98·23$ (1)°, $V = 2207$ Å^3, $Z = 4$, $D_x = 1·317$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·09$ mm^{-1}, $F(000) = 932$, $T = 298$ K, $R = 0·088$ for 1917 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR. Two different types of intermolecular hydrogen bond link the zwitterionic centres [N(4)···O(17′) 2·70 (1) and N(1)···O(24′) 2·85 (1) Å]. The hydrogens bonded to nitrogen and to the oxygen of the solvent molecule were located in a difference Fourier map and refined with a common temperature factor. The solvent methanol is hydrogen bonded to a carbonyl group [O(12′)···O(1*x*) 2·75 (1) Å].

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Experimental. (I): crystal size 0·2 × 0·2 × 0·6 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 4396 reflections measured, $2\theta_{max} = 45°$, $-12 \le h \le 12$, $-16 \le k \le 16$, $0 \le l \le 13$, three check reflections with