The torsion angles are given in Fig. 2. The piperidine rings *A, B, C* and *D* have chair, chair, boat and chair conformations, respectively. The position of the phenyl ring can be conveniently described by the torsion angles along C(2)—C(18) (see Fig. 2). The corresponding torsion angles in 2-phenylsparteine *N*(16)-oxide perchlorate (Małuszyńska & Okaya, 1977) have very similar valules of 137·0 (6) and 76·8 (8)°; a similar orientation of the phenyl ring was also observed in 2-(*p*-tolyl)-2-dehydrosparteine [torsion angles 132·9 (7) and −44·6 (9)°].

A stereoview of the unit-cell contents of (I) is presented in Fig. 3. No intermolecular contacts shorter than van der Waals distances were observed.

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Non-Natural 14-Hydroxy Steroids. I. Methyl 14β-Hydroxy-1,7,17-trioxo-5β,8α,9β-androstan-10β-oate

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Abstract. $C_{20}H_{26}O_6$, $M_r = 362{\cdot}42$, monoclinic, $A2/n$, $a = 16{\cdot}6256\ (8)$, $b = 10{\cdot}6293\ (5)$, $c = 20{\cdot}3059\ (6)$ Å, $\beta = 91{\cdot}515\ (3)°$, $V = 3587{\cdot}17\ (7)$ Å^3, $D_x = 1{\cdot}342$ Mg m^{-3}, $Z = 8$, $\lambda(\text{Cu } K\bar{\alpha}) = 1{\cdot}54056$ Å, $\mu = 0{\cdot}77$ mm^{-1}, $F(000) = 1552$, room temperature, final $R = 0{\cdot}039$ for 3207 observed reflections. The non-natural steroid compound bears a methoxycarbonyl group at C(10). The relative stereochemistry is *cis* for the *A/B* ring junction, *syn* between MeO_2C—C(10) and H—C(9), *trans* for the *B/C* ring junction, *anti* between H—C(8) and HO—C(14) and *cis* for the *C/D* ring junction, and an all-chair conformation is adopted.

Introduction As part of a study aimed at the synthesis of various natural and non-natural 14-hydroxy steroids, compound (1) was obtained upon acidic or alkaline treatment of the tetraketone (2) (Ruel & Deslongchamps, 1988). One could predict the stereochemical identity of the C(14) carbon center as that shown in structure (1), based on related work (Yates, Douglas, Datta & Sawyer, 1988) which reported the synthesis of the 14β-hydroxy steriod (3) by a similar approach. Unequivocal assignment of the structural identity of the steroidal compound (3) was made by X-ray analysis (Douglas, Sawyer & Yates, 1987). The present crystallographic analysis was undertaken to confirm the predicted structure of steriod (1) (Fig. 1).

Experimental. Crystal 0·20 × 0·15 × 0·30 mm; Enraf–Nonius CAD-4 diffractometer graphite-monochromator, Cu $K\bar{\alpha}$ radiation; cell dimensions were obtained from 28 reflections with 2θ angles in the range 80·0–120·0°. The $\omega/2\theta$ scan mode was used for data collection at a constant scan speed of 4° min^{-1}.

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A total of 8255 reflections accepted, 7574 symmetry equivalents averaged ($R_{merge} = 0{\cdot}016$), 3507 independent reflections up to $2\theta_{max} = 143{\cdot}5°$ corresponding to $-20 \le h \le 20$, $0 \le k \le 12$, $0 \le l \le 24$. 3207 reflections satisfying $I \ge 2{\cdot}5\sigma(I)$ were considered as observed. No correction was made for absorption. 172 standard reflections, 0·1% intensity variation. The *NRCVAX* system (Gabe, Lee & Le Page, 1985) was used for all calculations. The structure was solved by the application of direct methods and refined by full-matrix least squares on *F*. Anisotropic thermal parameters were refined for non-H atoms. The H atoms were located from a difference map, their positions and isotropic temperature factors were refined. $R = 0{\cdot}039$, $wR = 0{\cdot}031$, $S = 2{\cdot}921$. Weights based on counting statistics were used. the maximum Δ/σ ratio was 0·133. In the last difference Fourier map, the deepest hole was $-0{\cdot}230$ e Å^{-3}, and the highest peak 0·230 e Å^{-3}. Atomic scattering factors stored in the *NRCVAX* program were those of Cromer & Waber (1974).

Discussion. Table 1 gives the final atomic parameters with their B_{eq} values.* Fig. 1 gives the molecular formula for (1) and (2) and atom numbering, Fig. 2 shows a perspective view of the molecule. Bond angles are given in Table 2.

The crystal structure shows that rings *A*, *B* and *C* have chair conformations, the *C*/*D* ring junction is *cis* and ring *D* is in a C(14) α-envelope conformation.

* Lists of structure factors, thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52047 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

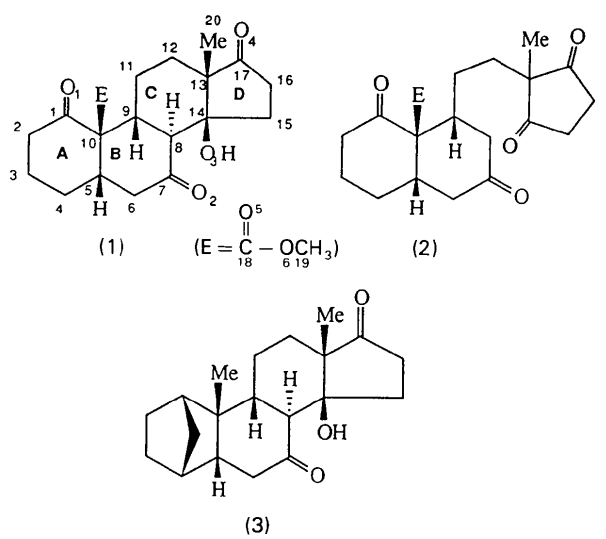

Fig. 1. Molecular formula and atom numbering.

Table 1. *Fractional coordinates and B_{eq} values for non-H atoms with e.s.d.'s in parentheses*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	B_{eq}(Å^2)
O(1)	0·81491 (7)	0·86634 (12)	0·98739 (5)	4·30 (6)
O(2)	0·76842 (7)	1·22615 (9)	0·82744 (5)	3·65 (5)
O(3)	0·89053 (6)	1·12420 (10)	0·76017 (5)	3·16 (5)
O(4)	1·06167 (7)	0·92642 (11)	0·91931 (6)	4·22 (5)
O(5)	0·68138 (7)	0·71950 (10)	0·80177 (5)	3·63 (5)
O(6)	0·75296 (7)	0·63857 (9)	0·88706 (5)	3·53 (5)
C(1)	0·75219 (10)	0·84392 (14)	0·95820 (8)	3·00 (6)
C(2)	0·67769 (11)	0·80471 (18)	0·99406 (8)	3·74 (8)
C(3)	0·60719 (12)	0·89496 (19)	0·98020 (9)	4·14 (8)
C(4)	0·59336 (10)	0·91149 (17)	0·90622 (9)	3·61 (8)
C(5)	0·66980 (9)	0·95299 (14)	0·87187 (8)	2·78 (6)
C(6)	0·69564 (10)	1·08568 (15)	0·89394 (9)	3·30 (7)
C(7)	0·76919 (9)	1·12868 (14)	0·85916 (7)	2·78 (6)
C(8)	0·84179 (9)	1·04228 (13)	0·86477 (7)	2·42 (6)
C(9)	0·81598 (9)	0·90602 (13)	0·84598 (7)	2·48 (6)
C(10)	0·74060 (9)	0·86008 (13)	0·88343 (7)	2·44 (6)
C(11)	0·88845 (10)	0·81746 (15)	0·85112 (9)	3·11 (7)
C(12)	0·95532 (10)	0·86202 (15)	0·80784 (8)	3·04 (7)
C(13)	0·98304 (9)	0·99509 (14)	0·82310 (7)	2·68 (6)
C(14)	0·91440 (9)	1·09258 (13)	0·82635 (7)	2·49 (6)
C(15)	0·95454 (10)	1·20270 (15)	0·86458 (8)	3·01 (7)
C(16)	1·00758 (11)	1·13883 (17)	0·91756 (8)	3·46 (7)
C(17)	1·02325 (9)	1·00818 (16)	0·89119 (8)	3·04 (7)
C(18)	0·71917 (9)	0·73259 (14)	0·85241 (7)	2·86 (6)
C(19)	0·74961 (14)	0·51374 (17)	0·85842 (11)	4·65 (10)
C(20)	1·04625 (11)	1·03411 (18)	0·77267 (9)	3·74 (8)

Table 2. *Molecular geometry*

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

O(1)—C(1)	1.2094 (19)	C(7)—C(8)	1·5185 (21)
O(2)—C(7)	1·2199 (18)	C(8)—C(9)	1·5553 (20)
O(3)—C(14)	1·4311 (17)	C(8)—C(14)	1·5497 (21)
O(4)—C(17)	1·2125 (19)	C(9)—C(10)	1·5613 (22)
O(5)—C(18)	1·1989 (17)	C(9)—C(11)	1·5304 (22)
O(6)—C(18)	1·3373 (18)	C(10)—C(18)	1·5324 (20)
O(6)—C(19)	1·4493 (21)	C(11)—C(12)	1·5114 (25)
C(1)—C(2)	1·512 (3)	C(12)—C(13)	1·5170 (22)
C(1)—C(10)	1·5350 (20)	C(13)—C(14)	1·5442 (21)
C(2)—C(3)	1·535 (3)	C(13)—C(17)	1·5261 (21)
C(3)—C(4)	1·524 (3)	C(13)—C(20)	1·5433 (24)
C(4)—C(5)	1·5306 (24)	C(14)—C(15)	1·5462 (21)
C(5)—C(6)	1·5377 (22)	C(15)—C(16)	1·5314 (23)
C(5)—C(10)	1·5497 (21)	C(16)—C(17)	1·5135 (24)
C(6)—C(7)	1·4996 (24)		

(*b*) Valence angles (°) with e.s.d.'s in parentheses

C(18)—O(6)—C(19)	117·51 (13)	C(5)—C(10)—C(18)	109·50 (11)
O(1)—C(1)—C(2)	121·67 (14)	C(9)—C(10)—C(18)	104·91 (12)
O(1)—C(1)—C(10)	123·14 (15)	C(9)—C(11)—C(12)	110·94 (13)
C(2)—C(1)—C(10)	115·10 (13)	C(11)—C(12)—C(13)	113·42 (13)
C(1)—C(2)—C(3)	111·76 (14)	C(12)—C(13)—C(14)	114·43 (12)
C(2)—C(3)—C(4)	110·33 (15)	C(12)—C(13)—C(17)	113·17 (12)
C(3)—C(4)—C(5)	112·14 (14)	C(12)—C(13)—C(20)	108·88 (13)
C(4)—C(5)—C(6)	111·13 (14)	C(14)—C(13)—C(17)	101·93 (12)
C(4)—C(5)—C(10)	112·58 (13)	C(14)—C(13)—C(20)	111·40 (12)
C(6)—C(5)—C(10)	109·58 (12)	C(17)—C(13)—C(20)	106·67 (13)
C(5)—C)6)—C(7)	111·55 (13)	O(3)—C(14)—C(8)	110·70 (11)
O(2)—C(7)—C(6)	120·68 (14)	O(3)—C(14)—C(13)	107·72 (11)
O(2)—C(7)—C(8)	123·40 (14)	O(3)—C(14)—C(15)	113·52 (12)
C(6)—C(7)—C(8)	115·90 (13)	C(8)—C(14)—C(13)	112·11 (12)
C(7)—C(8)—C(9)	109·34 (12)	C(8)—C(14)—C(15)	109·88 (12)
C(7)—C(8)—C(14)	112·44 (12)	C(13)—C(14)—C(15)	102·70 (12)
C(9)—C(8)—C(14)	114·29 (12)	C(14)—C(15)—C(16)	104·48 (12)
C(8)—C(9)—C(10)	113·05 (12)	C(15)—C(16)—C(17)	105·04 (13)
C(8)—C(9)—C(11)	110·14 (12)	O(4)—C(17)—C(13)	125·03 (15)
C(10)—C(9)—C(11)	114·52 (12)	O(4)—C(17)—C(16)	125·78 (14)
C(1)—C(10)—C(5)	107·22 (12)	C(13)—C(17)—C(16)	109·20 (13)
C(1)—C(10)—C(9)	115·81 (12)	O(5)—C(18)—O(6)	124·60 (14)
C(1)—C(10)—C(18)	109·23 (12)	O(5)—C(18)—C(10)	124·47 (13)
C(5)—C(10)—C(9)	110·08 (11)	O(6)—C(18)—C(10)	110·78 (12)

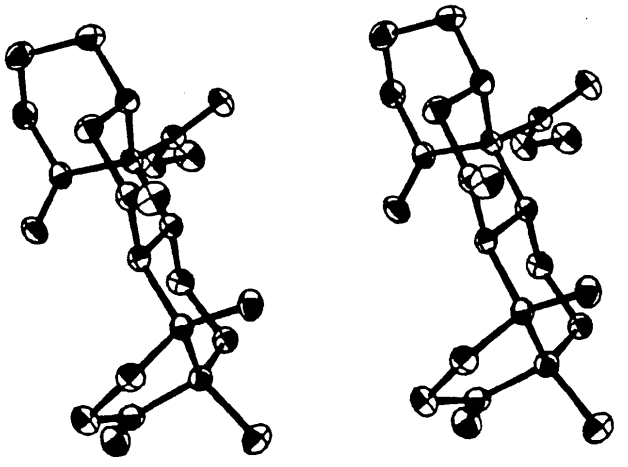

Fig. 2. *ORTEP* stereoview (Johnson, 1976).

Rings *C* and *D* are very similar to their homologues in compound (3) (Douglas *et al.*, 1987) leading to the conclusion that the main influence of the 1β,4β-methano bridge present in compound (3) is to force a boat conformation on rings *A* and *B*.

The chair conformation of ring *B* contributes to the folding of the molecule with rings *A* and *D* facing towards each other. The resulting overall conformation of the steroid (Fig. 3) is rather similar to that of compound (3). An intramolecular hydrogen bond between the C(7)—O(2) carbonyl group and the O(3)—H contributes to the stabilization of the molecular structure and particularly the chair conformation: *d*[O(2)···O(3)] = 2·703 (1), *d*[O(2)···HO(3)] = 1·98 (1), *d*[O(3)—HO(3)] = 0·86 (1) Å, ∠[O(2)···HO(3)—O(3)] = 141 (1°).

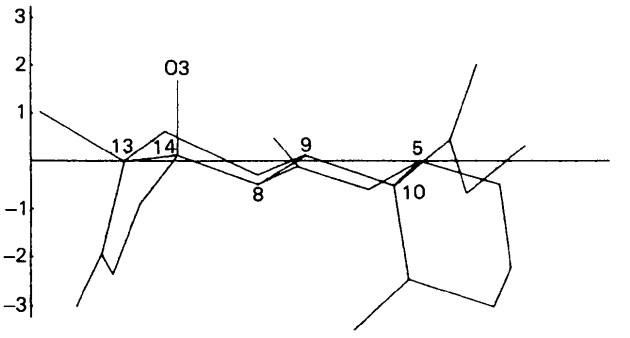

Fig. 3. Overall conformation of the steroid, viewed parallel to the least-squares mean plane through rings *B* and *C* (scale in Å).

The distance between these two functions is much greater in compound (3) where ring *B* is in a boat conformation (Douglas *et al.*, 1987). No abnormally short intermolecular contacts were observed in the crystal packing.

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Structure of 2-Cyclohexylamino-4,6-dimethoxy-1,3,5-triazine

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Abstract. $C_{11}H_{18}N_4O_2$, $M_r = 238{\cdot}4$, triclinic, $P\bar{1}$, $a = 6{\cdot}894$ (4), $b = 8{\cdot}138$ (1), $c = 12{\cdot}201$ (6) Å, $\alpha = 108{\cdot}67$ (2), $\beta = 90{\cdot}42$ (3), $\gamma = 105{\cdot}41$ (1)°, $V = 621{\cdot}97$ Å^3, $Z = 2$, $D_x = 1{\cdot}272$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}8$ mm^{-1}, $F(000) = 256$. The structure was solved by direct methods and refined to $R = 0{\cdot}069$ for 2225 observed intensities. Delocalization of the *s*-triazine π electrons ranges over adjacent O and N atoms resulting in the formation of a planar 2-amino-4,6-dimethoxy-1,3,5-triazine system with stacking of triazine rings and perpendicular orientation of the cyclohexane mean plane in relation to the triazine plane.

Introduction. Derivatives of alkylamino-1,3,5-triazines have been used as herbicides. It has been suggested that a possible mechanism by which triazines are absorbed by soil mineral matter is the formation of coordination complexes with exchangeable cations of clay minerals (Hance, 1969).

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