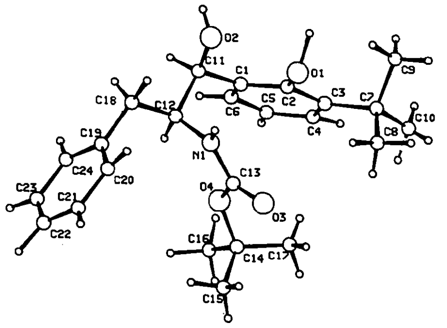


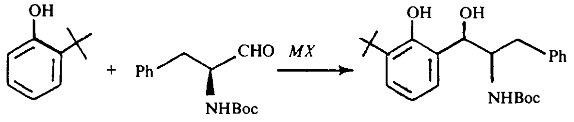
Fig. 1. Projection of the molecule.

rier maps and refined isotropically, $\sum w\Delta F^2$ minimized with weighting scheme $w = 0{\cdot}4035/(\sigma^2F + 0{\cdot}00621F^2)$, seven reflections omitted because of extinction, $(\Delta/\sigma)_{\max} = 0{\cdot}82$, $\Delta\rho_{\max}$ 0·19 e Å^{-3}, 395 variables, final refinement converged to $R = 0{\cdot}046$ and $wR = 0{\cdot}051$. Atomic scattering factors from *SHELX76*.*

Final atomic fractional coordinates are listed in Table 1, bond distances and bond angles are presented in Table 2. A projection of the molecule with the numbering scheme is shown in Fig. 1.

All the calculations were performed on an IBM PS2/80 personal computer with the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreetti, 1987).

Related literature. The synthesis of the title compound forms part of a series of studies on direct diastereoselective synthesis of ephedrine-like compounds by hydroxylalkylation of metal phenolates with N-protected α-amino aldehydes (Bigi, Casnati, Sartori & Araldi, 1989).



Boc = *tert*-butoxycarbonyl *MX* = methylene chloride

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* Lists of structure-factor amplitudes, H-atom coordinates with isotropic thermal parameters and anisotropic thermal parameters of heavy atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52205 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 3β-Hydroxy-5-androsten-17-one (DHEA) Monohydrate

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Abstract. $C_{19}H_{28}O_2.H_2O$, $M_r = 306{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 22{\cdot}545\,(7)$, $b = 22{\cdot}673\,(22)$, $c = 6{\cdot}819\,(2)$ Å, $V = 3485{\cdot}6$ Å^3, $Z = 8$, $D_x = 1{\cdot}17$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}43$ cm^{-1}, $F(000) = 1344$, $T = 293$ K, $R = 0{\cdot}071$ for 1308 observed reflexions. The asymmetric unit contains two steroid molecules of similar conformation and two water molecules. Extensive hydrogen bonding is present

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Table 1. *Fractional coordinates with e.s.d.'s and equivalent values of the anisotropic temperature factor*

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

	x	y	z	U_{eq} (Å^2)
Molecule *A*				
O(1)	0·8941 (4)	−0·4153 (4)	−0·4419 (13)	0·071
O(2)	0·7199 (5)	−0·0061 (4)	0·1760 (12)	0·068
O(3)	0·9915 (4)	0·0291 (4)	−0·0816 (15)	0·078
C(1)	0·7891 (6)	−0·2932 (5)	−0·3054 (20)	0·056
C(2)	0·8081 (6)	−0·3553 (7)	−0·3644 (21)	0·071
C(3)	0·8741 (6)	−0·3588 (6)	−0·4010 (17)	0·051
C(4)	0·9108 (6)	−0·3322 (7)	−0·2346 (17)	0·063
C(5)	0·8882 (6)	−0·2724 (7)	−0·1722 (17)	0·052
C(6)	0·9267 (6)	−0·2301 (7)	−0·1690 (17)	0·054
C(7)	0·9115 (5)	−0·1663 (6)	−0·1102 (19)	0·057
C(8)	0·8527 (6)	−0·1648 (5)	−0·0021 (17)	0·043
C(9)	0·3055 (5)	−0·2001 (6)	−0·1029 (18)	0·043
C(10)	0·8255 (5)	−0·2655 (6)	−0·1281 (15)	0·044
C(11)	0·7423 (6)	−0·1940 (6)	−0·0226 (21)	0·066
C(12)	0·7253 (6)	−0·1294 (6)	0·0124 (21)	0·063
C(13)	0·7722 (6)	−0·0982 (6)	0·1313 (18)	0·050
C(14)	0·8319 (5)	−0·1023 (5)	0·0364 (17)	0·042
C(15)	0·8713 (6)	−0·0578 (7)	0·1403 (20)	0·075
C(16)	0·8263 (6)	−0·0070 (7)	0·1711 (20)	0·067
C(17)	0·7659 (7)	−0·0339 (5)	0·1633 (18)	0·050
C(18)	0·7712 (7)	−0·1235 (7)	0·3511 (20)	0·085
C(19)	0·8085 (7)	−0·3028 (6)	0·0568 (20)	0·078
Molecule *B*				
O(21)	0·9497 (5)	0·0781 (5)	0·2633 (15)	0·077
O(22)	0·8334 (6)	0·5456 (5)	0·2123 (16)	0·105
O(23)	0·9108 (5)	0·0313 (7)	0·6134 (15)	0·127
C(21)	0·9306 (6)	0·2364 (7)	0·4015 (16)	0·060
C(22)	0·9562 (7)	0·1751 (7)	0·4094 (19)	0·073
C(23)	0·9239 (6)	0·1353 (7)	0·2614 (17)	0·055
C(24)	0·9361 (7)	0·1621 (7)	0·0521 (17)	0·064
C(25)	0·9146 (6)	0·2230 (7)	0·0454 (18)	0·057
C(26)	0·8762 (6)	0·2397 (7)	−0·0969 (19)	0·060
C(27)	0·8531 (6)	0·3013 (6)	−0·1274 (18)	0·060
C(28)	0·8888 (6)	0·3493 (6)	−0·0112 (18)	0·051
C(29)	0·9014 (6)	0·3238 (6)	0·1968 (16)	0·052
C(30)	0·9394 (6)	0·2674 (6)	0·1948 (17)	0·055
C(31)	0·9318 (6)	0·3744 (6)	0·3285 (18)	0·062
C(32)	0·8943 (7)	0·4305 (7)	0·3349 (19)	0·066
C(33)	0·8848 (7)	0·4529 (6)	0·1212 (21)	0·066
C(34)	0·8514 (6)	0·4032 (6)	0·0115 (17)	0·054
C(35)	0·8264 (8)	0·4350 (6)	−0·1689 (21)	0·085
C(36)	0·8039 (8)	0·4938 (7)	−0·0898 (20)	0·090
C(37)	0·8421 (8)	0·5021 (8)	0·1020 (21)	0·079
C(38)	0·9436 (7)	0·4763 (7)	0·0343 (21)	0·102
C(39)	1·0054 (5)	0·2802 (7)	0·1546 (20)	0·075

(Add 20 to numbering of molecule *A* to generate atom labels for molecule *B*.)

Table 2. *Bond lengths* (Å) *and valency angles* (°) *with e.s.d.'s*

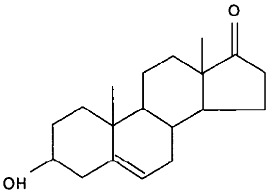
	Molecule *A*	Molecule *B*
O(1)—C(3)	1·39 (2)	1·42 (2)
C(1)—C(2)	1·52 (2)	1·51 (3)
C(2)—C(3)	1·51 (2)	1·54 (2)
C(4)—C(5)	1·51 (2)	1·46 (3)
C(5)—C(10)	1·45 (2)	1·54 (2)
C(7)—C(8)	1·52 (2)	1·57 (2)
C(8)—C(14)	1·52 (2)	1·49 (2)
C(9)—C(11)	1·53 (2)	1·61 (2)
C(11)—C(12)	1·53 (2)	1·53 (3)
C(13)—C(14)	1·50 (2)	1·55 (2)
C(13)—C(18)	1·61 (2)	1·55 (3)
C(15)—C(16)	1·55 (3)	1·52 (3)
O(2)—C(17)	1·22 (2)	1·25 (3)
C(1)—C(10)	1·59 (2)	1·59 (2)
C(3)—C(4)	1·53 (2)	1·58 (2)
C(5)—C(6)	1·29 (3)	1·35 (2)
C(6)—C(7)	1·54 (3)	1·51 (2)
C(8)—C(9)	1·50 (2)	1·56 (2)
C(9)—C(10)	1·56 (2)	1·54 (2)
C(10)—C(19)	1·57 (2)	1·54 (2)
C(12)—C(13)	1·51 (2)	1·56 (2)
C(13)—C(17)	1·48 (2)	1·48 (3)
C(14)—C(15)	1·52 (2)	1·53 (2)
C(16)—C(17)	1·49 (2)	1·58 (3)
C(2)—C(1)—C(10)	114·8 (11)	113·1 (11)
O(1)—C(3)—C(2)	113·8 (12)	109·6 (12)
C(2)—C(3)—C(4)	113·0 (11)	106·6 (12)
C(4)—C(5)—C(6)	116·3 (13)	119·8 (13)
C(6)—C(5)—C(10)	124·8 (15)	121·6 (14)
C(6)—C(7)—C(8)	110·1 (11)	113·4 (11)
C(7)—C(8)—C(14)	112·1 (11)	109·4 (11)
C(8)—C(9)—C(10)	110·7 (10)	113·7 (10)
C(10)—C(9)—C(11)	113·3 (11)	111·1 (11)
C(1)—C(10)—C(9)	108·0 (10)	106·9 (11)
C(5)—C(10)—C(9)	114·0 (12)	110·4 (11)
C(9)—C(10)—C(19)	110·7 (10)	112·4 (12)
C(11)—C(12)—C(13)	110·9 (11)	108·7 (11)
C(12)—C(13)—C(17)	118·3 (12)	114·7 (13)
C(14)—C(13)—C(17)	102·2 (11)	100·9 (13)
C(17)—C(13)—C(18)	102·3 (11)	105·5 (13)
C(8)—C(14)—C(15)	121·4 (11)	120·7 (11)
C(14)—C(15)—C(16)	100·0 (11)	104·5 (11)
O(2)—C(17)—C(13)	127·0 (14)	129·9 (15)
C(13)—C(17)—C(16)	108·7 (12)	109·8 (13)
C(1)—C(2)—C(3)	111·7 (12)	109·6 (12)
O(1)—C(3)—C(4)	109·7 (11)	106·8 (11)
C(3)—C(4)—C(5)	112·3 (11)	109·5 (11)
C(4)—C(5)—C(10)	118·9 (13)	118·5 (12)
C(5)—C(6)—C(7)	123·4 (13)	125·4 (13)
C(7)—C(8)—C(9)	112·6 (10)	107·2 (11)
C(9)—C(8)—C(14)	111·1 (11)	103·2 (11)
C(8)—C(9)—C(11)	116·5 (11)	108·8 (11)
C(1)—C(10)—C(5)	107·6 (10)	104·7 (11)
C(1)—C(10)—C(19)	105·8 (11)	111·2 (11)
C(5)—C(10)—C(19)	110·4 (11)	110·7 (11)
C(9)—C(11)—C(12)	112·0 (11)	111·9 (11)
C(12)—C(13)—C(14)	111·6 (11)	106·3 (12)
C(12)—C(13)—C(18)	109·0 (11)	110·7 (13)
C(14)—C(13)—C(18)	113·2 (11)	118·7 (12)
C(8)—C(14)—C(13)	114·3 (11)	111·8 (12)
C(13)—C(14)—C(15)	106·4 (11)	103·0 (11)
C(15)—C(16)—C(17)	106·8 (12)	102·5 (13)
O(2)—C(17)—C(16)	124·3 (12)	120·3 (15)

but fine details are obscured due to difficulty in locating water H atoms accurately. The ring conformations are *A*: chair, *B*: 8β,9α-half-chair, *C*: chair, *D*: C14α-envelope.

Experimental. Colourless crystal, 1·40 × 0·20 × 0·20 mm. Nicolet *P*3 automated diffractometer, graphite monochromator. Cell dimensions from setting angles of 12 independent reflexions with $2\theta \approx 20°$. Data corrected for Lorentz and polarization effects, but not for absorption. A total of 2738 unique intensities were measured with $2\theta < 50°$ from $\omega - 2\theta$ scans; 1308 reflexions had $F > 5\sigma(F)$. Range of *hkl*: $0 \le h \le 24$, $0 \le k \le 23$, $0 \le l \le 8$. Two reference reflexions monitored periodically showed no significant variation in intensity.

The structure (I) was determined with *MITHRIL* (Gilmore, 1984) and completed with *SHELX*76 (Sheldrick, 1976). Blocked full-matrix least-squares calculations on *F* with anisotropic thermal parameters for C and O atoms and isotropic thermal parameters for H atoms converged at $R = 0·071$. Each hydrated steroid molecule was refined in alternate cycles of least-squares calculations such that the total number of refined parameters was 423. Unit weights were used. The positions of the hydroxy and water H atoms, which were associated with high error, were refined but the remaining H atoms were only allowed to ride on their attached atoms. All

C—H and O—H bond distances were constrained to 1·00 (2) Å and the H atoms were given one of four common temperature factors (methyl or non-methyl in molecule *A* or *B*). Atomic scattering factors from *SHELX76*. Final $\Delta/\sigma \leq 0{\cdot}5$, $\Delta\rho$ max. = 0·1, $\Delta\rho$ min. = $-0{\cdot}1$ e Å^{-3}. Molecular geometries were generated by the *GX* package (Mallinson & Muir, 1985).



(I)

Atomic coordinates are listed in Table 1, bond lengths and valency angles in Table 2 and short intermolecular O···O contacts in Table 3.* The atomic arrangement is shown in Fig. 1.

Related literature. DHEA may be obtained commercially or can be synthesized in high yield (Hosoda, Fukushima & Fishman, 1973). The hydrate of DHEA was isolated by us during a synthesis of 3,5-cycloandrost-6β-ol-17-one. Its conformation may be compared with crystal structures of other 5-ene steroids such as androst-5-ene-3,17-dione (Carrell, Glusker, Covey, Batzold & Robinson, 1978) and 17β-hydroxy-7α-methyl-5-androsten-3-one (Cox, Mkandawire & Mallinson, 1981).

Table 3. *Short* O···O *contacts* (Å) *with e.s.d.'s*

O(1)···O(3^{i})	2·87 (1)
O(1)···O(2^{ii})	2·87 (1)
O(2)···O(23^{iii})	3·03 (2)
O(3)···O(21)	2·77 (1)
O(3)···O(23^{iv})	2·76 (2)
O(21)···O(23)	2·76 (2)

Coordinates transposed by: (i) $2-x$, $-\frac{1}{2}-y$, $-\frac{1}{2}-z$; (ii) x, $-1+y$, $-1+z$; (iii) $1\frac{1}{2}-x$, $-y$, $-\frac{1}{2}+z$; (iv) x, y, $-1+z$.

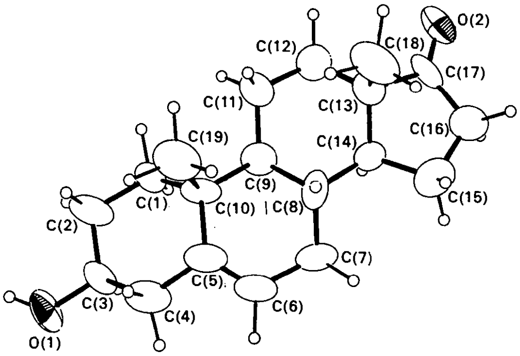


Fig. 1. The atomic arrangement in molecule *A* (50% probability ellipsoids).

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* Lists of structure factors, anisotropic thermal parameters, H-atom positions and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52309 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diisopropylammonium Chloride

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Abstract. $C_6H_{16}N^+.Cl^-$, $M_r = 137{\cdot}65$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}825\,(4)$, $b = 8{\cdot}257\,(1)$, $c = 13{\cdot}268\,(2)$ Å, $V = 857{\cdot}3\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}066$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 3·6 cm^{-1}, $F(000) = 304$, $T = 295$ K, $R = 0{\cdot}030$ for 556 observations (of 901 unique data). Each H on the N atom is hydrogen bonded to a Cl atom. N—H···Cl bond lengths (N—Cl distance) are 3·180 (3) and 3·163 (3) Å with N—H···Cl angles 176 (2) and 175 (2)° respectively. Each Cl atom is involved in two

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