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Acta Cryst. (1991). C**47**, 1766–1767

Structure of a 1,4-Dien-3-one-6α-hydroxy Steroid

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(*Received* 10 *January* 1991; *accepted* 6 *February* 1991)

Abstract. 9α,21-Dichloro-6α,11β,17α-trihydroxy-16α-methyl-3,20-dioxopregna-1,4-dien-17-yl 2-furoate, $C_{27}H_{30}Cl_2O_7$, $M_r = 537{\cdot}43$, monoclinic, $P2_1$, $a = 11{\cdot}524$ (2), $b = 15{\cdot}751$ (4), $c = 7{\cdot}975$ (2) Å, $\beta = 111{\cdot}42$ (1)°, $V = 1347{\cdot}7$ (6) Å^3, $Z = 2$, $D_x = 1{\cdot}324$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 2{\cdot}53$ mm^{-1}, $F(000) = 564$, $T = 295$ K, $R = 0{\cdot}047$ for 2298 observed reflections [$F_o > 3\sigma(F_o)$]. The molecules are linked by intermolecular hydrogen bonds; O(26)—HO(27)($1-x$, $y-\frac{1}{2}$, $1-z$) = 2·697 (5) [2·00 (6) for O···H] and O(31)—HO(26) ($2-x$, $\frac{1}{2}+y$, $2-z$) = 2·895 (6) Å [2·04 (8) Å].

Experimental. Colorless plate crystals obtained from methanol. Crystal of dimensions 0·3 × 0·3 × 0·1 mm. Rigaku AFC-5R diffractometer, graphite-monochromatized Cu $K\alpha$. Cell dimensions determined from 2θ angles for 25 reflections in the range $26 < 2\theta < 46°$. Intensities measured up to $\theta = 70°$ in h −13/14, k 0/19 and l −9/0, ω–2θ scans, ω-scan width $(2 + 0{\cdot}2\tan\theta)°$, three standard reflections monitored every 100 measurements showed no significant change. 2571 unique reflections measured, 2298 intensities observed [$F_o \le 3\sigma(F_o)$ and one very strong reflection rejected], no absorption correction. Structures solved by direct methods with *MULTAN*84 (Main, Germain & Woolfson, 1984). H atoms located on a difference density map. Positional parameters of all atoms and anisotropic thermal parameters of non-H atoms refined by block-diagonal least squares. Temperature factor of each H atom equal to B_{eq} of the bonded atom. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0{\cdot}00146|F_o|^2]$, $w = 0$ for 53 reflections with $w^{1/2}|\Delta F| \ge 3$. Final $R = 0{\cdot}047$, $wR = 0{\cdot}055$, $S = 1{\cdot}1150$. Max. Δ/σ in the final cycle 0·03. The highest and lowest peaks in the final difference map are 0·6 and −0·5 e Å^{-3}. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1,...,4$) (*International Tables for X-ray Crystallography*, 1974, Vol. IV). Calculations performed on a VAX station 3100 computer. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* Bond lengths and

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
C(1)	0·4496 (4)	0·2944 (3)	0·3006 (5)	4·1 (1)
C(2)	0·4305 (4)	0·2601 (3)	0·1399 (5)	4·5 (1)
C(3)	0·4636 (5)	0·1717 (3)	0·1247 (5)	4·8 (1)
C(4)	0·5123 (4)	0·1232 (3)	0·2894 (5)	4·2 (1)
C(5)	0·5293 (3)	0·1560 (3)	0·4511 (5)	3·4 (1)
C(6)	0·5800 (4)	0·1035 (3)	0·6214 (5)	3·7 (1)
C(7)	0·6837 (4)	0·1481 (3)	0·7692 (5)	3·7 (1)
C(8)	0·6607 (3)	0·2419 (3)	0·7940 (5)	3·2 (1)
C(9)	0·6162 (3)	0·2900 (3)	0·6137 (4)	3·0 (1)
C(10)	0·4958 (4)	0·2467 (3)	0·4755 (5)	3·5 (1)
C(11)	0·6087 (3)	0·3875 (2)	0·6367 (5)	3·3 (1)
C(12)	0·7241 (4)	0·4244 (2)	0·7837 (5)	3·4 (1)
C(13)	0·7605 (4)	0·3775 (3)	0·9640 (5)	3·4 (1)
C(14)	0·7766 (3)	0·2827 (3)	0·9289 (4)	3·3 (1)
C(15)	0·8322 (4)	0·2440 (3)	1·1144 (5)	4·1 (1)
C(16)	0·9221 (4)	0·3136 (3)	1·2277 (5)	4·2 (1)
C(17)	0·8932 (4)	0·3952 (3)	1·1073 (5)	3·6 (1)
C(18)	0·3848 (4)	0·2431 (3)	0·5421 (6)	4·3 (1)
C(19)	0·6645 (4)	0·3924 (3)	1·0517 (5)	4·1 (1)
C(20)	0·8997 (4)	0·4774 (3)	1·2155 (6)	4·6 (1)
C(21)	0·8873 (6)	0·5591 (4)	1·1128 (8)	6·2 (2)
C(22)	1·0559 (5)	0·2841 (5)	1·3026 (8)	6·9 (2)
Cl(23)	0·7371 (1)	0·2798	0·5149 (1)	3·79 (2)
Cl(24)	0·9575 (3)	0·6477 (1)	1·2487 (3)	10·9 (1)
O(25)	0·4511 (4)	0·1391 (3)	−0·0219 (4)	7·2 (2)
O(26)	0·6146 (3)	0·0219 (2)	0·5876 (4)	5·4 (1)
O(27)	0·4992 (2)	0·4065 (2)	0·6700 (4)	3·9 (1)
O(28)	0·9020 (3)	0·4765 (3)	1·3662 (4)	6·2 (1)
O(29)	0·9740 (2)	0·4019 (2)	1·0035 (4)	3·9 (1)
C(30)	1·0879 (3)	0·4338 (3)	1·0767 (5)	3·9 (1)
O(31)	1·1334 (3)	0·4612 (3)	1·2299 (4)	5·3 (1)
C(32)	1·1485 (4)	0·4369 (3)	0·9464 (6)	4·3 (1)
C(33)	1·1102 (5)	0·4264 (6)	0·7686 (7)	7·5 (2)
C(34)	1·2104 (6)	0·4453 (6)	0·7177 (8)	8·3 (3)
C(35)	1·3070 (5)	0·4622 (4)	0·8680 (7)	5·7 (2)
O(36)	1·2707 (3)	0·4586 (2)	1·0116 (4)	4·5 (1)

* Lists of H-atom coordinates, anisotropic temperature factors of the non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53985 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/91/081766-02$03.00

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1·333 (7)	C(13)—C(14)	1·543 (7)
C(1)—C(10)	1·500 (7)	C(13)—C(17)	1·564 (7)
C(2)—C(3)	1·460 (7)	C(13)—C(19)	1·528 (7)
C(3)—C(4)	1·444 (7)	C(14)—C(15)	1·509 (7)
C(3)—O(25)	1·236 (7)	C(15)—C(16)	1·552 (7)
C(4)—C(5)	1·336 (7)	C(16)—C(17)	1·566 (7)
C(5)—C(6)	1·513 (7)	C(16)—C(22)	1·509 (9)
C(5)—C(10)	1·511 (7)	C(17)—C(20)	1·543 (7)
C(6)—C(7)	1·511 (7)	C(17)—O(29)	1·458 (6)
C(6)—O(26)	1·401 (6)	C(20)—C(21)	1·504 (8)
C(7)—C(8)	1·527 (7)	C(20)—O(28)	1·192 (7)
C(8)—C(9)	1·538 (7)	C(21)—Cl(24)	1·771 (8)
C(8)—C(14)	1·518 (7)	O(29)—C(30)	1·325 (6)
C(9)—C(10)	1·577 (7)	C(30)—O(31)	1·219 (7)
C(9)—C(11)	1·553 (6)	C(30)—C(32)	1·449 (7)
C(9)—Cl(23)	1·841 (5)	C(32)—C(33)	1·333 (11)
C(10)—C(18)	1·555 (7)	C(32)—O(36)	1·355 (6)
C(11)—C(12)	1·530 (6)	C(33)—C(34)	1·389 (13)
C(11)—O(27)	1·413 (5)	C(34)—C(35)	1·331 (11)
C(12)—C(13)	1·533 (7)	C(35)—O(36)	1·356 (7)

C(2)—C(1)—C(10)	124·7 (4)	C(12)—C(13)—C(17)	117·3 (4)
C(1)—C(2)—C(3)	120·7 (5)	C(12)—C(13)—C(19)	111·1 (4)
C(2)—C(3)—C(4)	117·1 (5)	C(14)—C(13)—C(17)	98·9 (4)
C(2)—C(3)—O(25)	122·1 (5)	C(14)—C(13)—C(19)	112·8 (4)
C(4)—C(3)—O(25)	120·9 (5)	C(17)—C(13)—C(19)	108·4 (4)
C(3)—C(4)—C(5)	123·0 (5)	C(8)—C(14)—C(13)	113·9 (4)
C(4)—C(5)—C(6)	121·8 (4)	C(8)—C(14)—C(15)	118·2 (4)
C(4)—C(5)—C(10)	122·4 (4)	C(13)—C(14)—C(15)	104·4 (4)
C(6)—C(5)—C(10)	115·7 (4)	C(14)—C(15)—C(16)	103·9 (4)
C(5)—C(6)—C(7)	112·5 (4)	C(15)—C(16)—C(17)	106·0 (4)
C(5)—C(6)—O(26)	111·9 (4)	C(15)—C(16)—C(22)	112·4 (5)
C(7)—C(6)—O(26)	112·2 (4)	C(17)—C(16)—C(22)	117·0 (5)
C(6)—C(7)—C(8)	115·2 (4)	C(13)—C(17)—C(16)	103·5 (4)
C(7)—C(8)—C(9)	111·7 (4)	C(13)—C(17)—C(20)	113·2 (4)
C(7)—C(8)—C(14)	110·6 (4)	C(13)—C(17)—O(29)	104·8 (4)
C(9)—C(8)—C(14)	111·1 (4)	C(16)—C(17)—C(20)	113·0 (4)
C(8)—C(9)—C(10)	110·4 (4)	C(16)—C(17)—O(29)	111·8 (4)
C(8)—C(9)—C(11)	113·0 (4)	C(20)—C(17)—O(29)	110·3 (4)
C(8)—C(9)—Cl(23)	108·1 (3)	C(17)—C(20)—C(21)	116·0 (4)
C(10)—C(9)—C(11)	115·9 (4)	C(17)—C(20)—O(28)	122·2 (5)
C(10)—C(9)—Cl(23)	105·9 (3)	C(21)—C(20)—O(28)	121·5 (5)
C(11)—C(9)—Cl(23)	102·7 (3)	C(20)—C(21)—Cl(24)	114·2 (5)
C(1)—C(10)—C(5)	112·0 (4)	C(17)—O(29)—C(30)	120·8 (4)
C(1)—C(10)—C(9)	110·9 (4)	O(29)—C(30)—O(31)	124·8 (5)
C(1)—C(10)—C(18)	106·2 (4)	O(29)—C(30)—C(32)	110·9 (4)
C(5)—C(10)—C(9)	107·3 (4)	O(31)—C(30)—C(32)	124·3 (5)
C(5)—C(10)—C(18)	106·9 (4)	C(30)—C(32)—C(33)	134·4 (6)
C(9)—C(10)—C(18)	113·6 (4)	C(30)—C(32)—O(36)	116·0 (4)
C(9)—C(11)—C(12)	113·3 (3)	C(33)—C(32)—O(36)	109·5 (5)
C(9)—C(11)—O(27)	108·9 (3)	C(32)—C(33)—C(34)	107·3 (8)
C(12)—C(11)—O(27)	111·2 (3)	C(33)—C(34)—C(35)	106·9 (8)
C(11)—C(12)—C(13)	113·5 (4)	C(34)—C(35)—O(36)	109·7 (6)
C(12)—C(13)—C(14)	107·8 (4)	C(32)—O(36)—C(35)	106·5 (4)

angles are listed in Table 2. A perspective view of the molecule with atom labeling is presented in Fig. 1.

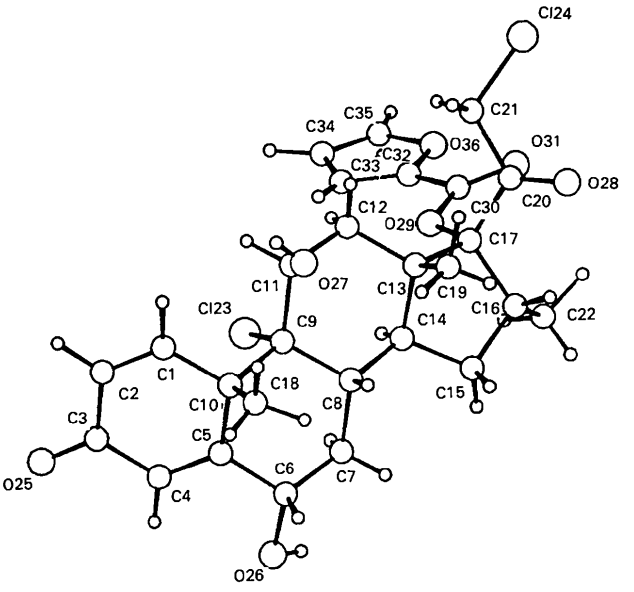

Fig. 1. Perspective view drawn by *PLUTO* (Motherwell & Clegg, 1978).

Related literature. The structure of the title compound reported here has been referred to in the chemical synthesis of 6-hydroxy corticosteroids (Terasawa & Okada, 1991).

The author thanks Drs Terasawa and Okada for supplying the crystals.

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Acta Cryst. (1991). C**47**, 1767–1769

Structure of a 1,4-Dien-3-one-6β-hydroxy Steroid Methanol Solvate

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(*Received* 10 *January* 1991; *accepted* 6 *February* 1991)

Abstract. 9α,21-Dichloro-6β,11β,17α-trihydroxy-16α-methylpregna-1,4-diene-3,20-dione methanol solvate, $C_{22}H_{28}Cl_2O_5.\frac{1}{2}CH_3OH$, $M_r = 459{\cdot}385$, monoclinic, $C2$, $a = 25{\cdot}506\ (5)$, $b = 8{\cdot}371\ (1)$, $c = 15{\cdot}035\ (3)$ Å, $\beta = 136{\cdot}99\ (1)°$, $V = 2189{\cdot}8\ (8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}393$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ =

0108-2701/91/081767-03$03.00