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9α-Bromo-16α-methylprednisolone* (I), $C_{22}H_{29}BrO_5$, and 9α-Bromoprednisolone (II), $C_{21}H_{27}BrO_5$

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Abstract. (I): $M_r = 453\cdot4$, monoclinic, $P2_1$, $a = 6\cdot550\,(6)$, $b = 14\cdot46\,(1)$, $c = 10\cdot75\,(1)$ Å, $\beta = 100\cdot9\,(5)^\circ$, $V = 999\cdot8$ Å^3, $Z = 2$, $D_m = 1\cdot505\,(3)$, $D_x = 1\cdot506$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1\cdot5418$ Å, $\mu = 28\cdot51$ cm^{-1}, $F(000) = 472\cdot0$, $T = 293$ K, $R = 0\cdot072$ for 1749 unique reflections. (II): $M_r = 439\cdot3$, monoclinic, $P2_1$, $a = 7\cdot737\,(6)$, $b = 14\cdot92\,(1)$, $c = 8\cdot94\,(1)$ Å, $\beta = 103\cdot6\,(5)^\circ$, $V = 1003\cdot1$ Å^3, $Z = 2$, $D_m = 1\cdot452\,(3)$, $D_x = 1\cdot455$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1\cdot5418$ Å, $\mu = 28\cdot27$ cm^{-1}, $F(000) = 456\cdot0$, $T = 293$ K, $R = 0\cdot075$ for 1650 unique reflections. The molecules of both compounds have very similar stereochemistry with minor differences in some torsion angles. Ring *A* is almost planar, rings *B* and *C* have chair conformations while ring *D* is a C(13) envelope. The molecules are packed by O(3)···O(17) (compound I) and O(3)···O(17) and O(3)···O(11) (compound II) intermolecular hydrogen bonds.

Introduction. Samples of the compounds were kindly supplied by the late Dr C. L. Hewett of Organon Laboratories Limited. The study was intended to give information on the stereochemistry of these two closely related compounds which might throw light on their physiological activity.

Experimental. D_m measured by flotation (KI/$Na_2S_2O_3$ in H_2O). Prismatic crystals grown from ethanol. Equi-dimensional crystals approximately 0·3 × 0·3 × 0·3 mm selected for X-ray diffraction measurements. Cell dimensions obtained initially from rotation photographs and more accurately from high-angle reflections on Weissenberg photographs using a local least-squares program. Intensity data: Leeds–Cox integrating Weissenberg camera; multiple-film photographs taken about: (I): *a*(layers 0–4), *b*(layers 0–2), *c*(layers 0–4); (II): *a*(layers 0–5), *b*(layers 0–12). Intensities derived using a Joyce–Loebl microdensitometer, Lorentz and polarization but no absorption corrections applied. (I): 1749, (II): 1650 measured reflections (2290 and 2300 in Cu sphere respectively), Friedel pairs merged; index range (I): $h\,\pm8$, $k\,0/18$, $l\,0/13$; (II): $h\,-8/9$, $k\,0/19$, $l\,0/11$. Heavy-atom methods used to solve structures employing a locally written Fourier synthesis program. Program packages *SHELX76* (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978) also used, with all calculations performed on the Dundee University DEC-10 computer. Refinement on *F* magnitudes, using blocked full-matrix least squares, with *SHELX76*. All atoms except H refined using anisotropic temperature factors. H atoms included as fixed atoms at calculated positions, with isotropic temperature factors fixed at 1·5 times that of the parent atom. Final refinements involved: (I): 252 refined parameters, $R = 0\cdot072$, unit weights, max. shift/e.s.d. = 0·058, av. shift/e.s.d. = 0·005, max. diff. peak = 1·00 e Å^{-3}, min. diff. peak = –0·95 e Å^{-3}. (II): 243 refined parameters, $R = 0\cdot075$, unit weights, max. shift/e.s.d. = 0·055, av. shift/e.s.d. = 0·004, max. diff. peak = 0·73 e Å^{-3}, min. diff. peak = –0·76 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974).

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* Prednisolone is 11β,17α,21-trihydroxypregna-1,4-diene-3,20-dione.

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Discussion. The atomic numbering is shown in Fig. 1. Atomic coordinates are given in Table 1,* with bond lengths and angles in Table 2. There are no differences

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39873 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
(*a*) 9α-Bromo-16α-methylprednisolone (I)				
C(1)	785 (25)	2137 (11)	−623 (17)	50 (5)
C(2)	83 (31)	2361 (13)	462 (16)	57 (6)
C(3)	923 (28)	1922 (13)	1696 (19)	61 (6)
C(4)	2531 (24)	1222 (11)	1655 (12)	41 (4)
C(5)	3240 (22)	1002 (11)	613 (13)	39 (4)
C(6)	4905 (24)	269 (10)	614 (13)	45 (4)
C(7)	4424 (21)	−424 (10)	−439 (12)	34 (3)
C(8)	3624 (16)	47 (14)	−1733 (9)	26 (2)
C(9)	1837 (20)	708 (9)	−1693 (13)	30 (3)
C(10)	2511 (21)	1473 (9)	−643 (13)	33 (3)
C(11)	744 (25)	1072 (10)	−3000 (12)	37 (4)
C(12)	268 (27)	303 (11)	−4031 (15)	50 (5)
C(13)	2228 (26)	−310 (10)	−4053 (13)	38 (4)
C(14)	2957 (22)	−718 (10)	−2717 (13)	33 (4)
C(15)	4504 (22)	−1454 (11)	−2956 (14)	40 (4)
C(16)	3786 (24)	−1803 (11)	−4351 (12)	36 (4)
C(17)	1801 (22)	−1233 (9)	−4846 (12)	32 (4)
C(18)	3947 (25)	235 (10)	−4536 (14)	45 (5)
C(19)	4386 (27)	2072 (11)	−873 (12)	47 (5)
C(20)	1469 (27)	−1085 (11)	−6226 (13)	45 (5)
C(21)	−678 (29)	−804 (15)	−6931 (16)	58 (6)
C(22)	3493 (29)	−2831 (13)	−4465 (18)	57 (6)
O(3)	419 (24)	2136 (12)	2662 (13)	85 (6)
O(11)	1967 (22)	1797 (8)	−3359 (11)	61 (4)
O(17)	−125 (15)	−1687 (7)	−4655 (8)	37 (3)
O(20)	2804 (22)	−1125 (11)	−6877 (11)	73 (5)
O(21)	−859 (22)	−734 (11)	−8258 (11)	69 (4)
Br(1)	−492 (2)	0	−1107 (1)	40 (1)
(*b*) 9α-Bromoprednisolone (II)				
C(1)	3478 (27)	−1493 (11)	1160 (18)	56 (6)
C(2)	3176 (18)	−1854 (8)	−246 (16)	47 (4)
C(3)	3627 (18)	−1371 (8)	−1519 (15)	44 (4)
C(4)	4368 (19)	−491 (9)	−1157 (14)	47 (4)
C(5)	4533 (17)	−75 (12)	203 (14)	47 (3)
C(6)	5143 (23)	869 (10)	423 (16)	62 (5)
C(7)	3750 (25)	1423 (10)	1003 (21)	52 (5)
C(8)	3353 (19)	1014 (10)	2457 (15)	52 (4)
C(9)	2664 (14)	44 (13)	2166 (11)	37 (3)
C(10)	4086 (17)	−541 (9)	1566 (13)	41 (3)
C(11)	2105 (20)	−383 (8)	3541 (14)	46 (4)
C(12)	757 (18)	223 (7)	4159 (15)	45 (4)
C(13)	1448 (18)	1192 (8)	4466 (14)	43 (4)
C(14)	1910 (19)	1557 (9)	2995 (15)	46 (4)
C(15)	2204 (23)	2545 (10)	3353 (19)	60 (5)
C(16)	951 (20)	2792 (9)	4307 (18)	52 (5)
C(17)	59 (17)	1895 (9)	4647 (14)	42 (3)
C(18)	3059 (21)	1264 (11)	5904 (16)	63 (5)
C(19)	5941 (21)	−674 (13)	2735 (17)	69 (6)
C(20)	−407 (23)	1880 (12)	6179 (17)	61 (5)
C(21)	−1939 (23)	1344 (14)	6411 (21)	75 (7)
O(3)	3380 (13)	−1701 (6)	−2804 (10)	46 (3)
O(11)	3597 (14)	−575 (7)	4718 (10)	59 (3)
O(17)	−1552 (13)	1715 (6)	3479 (10)	49 (3)
O(20)	465 (17)	2321 (10)	7323 (14)	92 (5)
O(21)	−2214 (18)	1392 (11)	7879 (14)	93 (5)
Br(1)	499 (2)	0	451 (1)	50 (1)

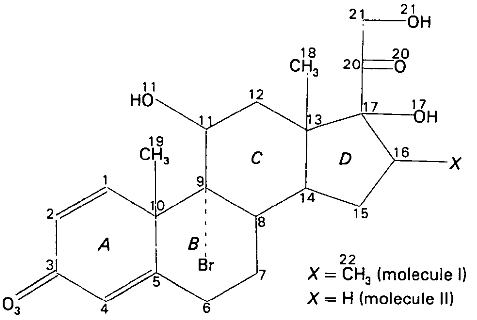

Fig. 1. Atomic numbering for molecules (I) and (II).

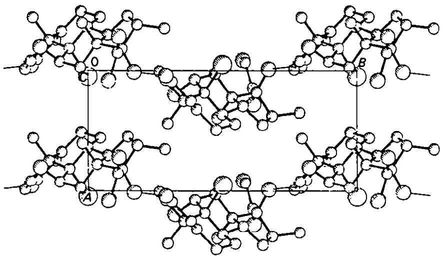

Fig. 2. View of (I) along **c** showing molecular packing and hydrogen bonding.

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

	(I)	(II)		(I)	(II)
C(2)–C(1)	1·37 (3)	1·34 (2)	C(19)–C(10)	1·56 (2)	1·58 (2)
C(10)–C(1)	1·49 (2)	1·51 (2)	C(12)–C(11)	1·56 (2)	1·58 (2)
C(3)–C(2)	1·48 (3)	1·46 (2)	O(11)–C(11)	1·42 (2)	1·40 (2)
C(4)–C(3)	1·47 (3)	1·44 (2)	C(13)–C(12)	1·56 (2)	1·54 (2)
O(3)–C(3)	1·19 (3)	1·22 (2)	C(14)–C(13)	1·54 (2)	1·54 (2)
C(5)–C(4)	1·33 (2)	1·35 (2)	C(17)–C(13)	1·58 (2)	1·54 (2)
C(6)–C(5)	1·52 (2)	1·49 (2)	C(18)–C(13)	1·54 (2)	1·57 (2)
C(10)–C(5)	1·51 (2)	1·51 (2)	C(15)–C(14)	1·52 (2)	1·52 (2)
C(7)–C(6)	1·50 (2)	1·54 (3)	C(16)–C(15)	1·57 (2)	1·48 (3)
C(8)–C(7)	1·55 (2)	1·53 (2)	C(17)–C(16)	1·55 (2)	1·57 (2)
C(9)–C(8)	1·52 (2)	1·54 (2)	C(22)–C(16)	1·50 (2)	
C(14)–C(8)	1·54 (2)	1·54 (2)	C(20)–C(17)	1·47 (2)	1·50 (2)
C(10)–C(9)	1·58 (2)	1·59 (2)	O(17)–C(17)	1·47 (2)	1·45 (1)
C(11)–C(9)	1·54 (2)	1·53 (2)	C(21)–C(20)	1·52 (2)	1·48 (3)
Br(1)–C(9)	2·03 (1)	1·99 (1)	O(20)–C(20)	1·22 (2)	1·27 (2)
			O(21)–C(21)	1·41 (2)	1·38 (2)

	(I)	(II)		(I)	(II)
C(10)–C(1)–C(2)	123 (2)	125 (2)	C(12)–C(11)–C(9)	114 (1)	112 (1)
C(3)–C(2)–C(1)	122 (2)	121 (1)	O(11)–C(11)–C(9)	108 (1)	110 (1)
C(4)–C(3)–C(2)	115 (2)	116 (1)	O(11)–C(11)–C(12)	112 (1)	110 (1)
O(3)–C(3)–C(2)	124 (2)	121 (1)	C(13)–C(12)–C(11)	111 (1)	112 (1)
O(3)–C(3)–C(4)	122 (2)	123 (1)	C(14)–C(13)–C(12)	108 (1)	109 (1)
C(5)–C(4)–C(3)	124 (1)	125 (1)	C(17)–C(13)–C(12)	115 (1)	116 (1)
C(6)–C(5)–C(4)	122 (1)	121 (1)	C(17)–C(13)–C(14)	100 (1)	99 (1)
C(10)–C(5)–C(4)	123 (1)	122 (1)	C(18)–C(13)–C(12)	112 (1)	113 (1)
C(10)–C(5)–C(6)	115 (1)	117 (1)	C(18)–C(13)–C(14)	113 (1)	112 (1)
C(7)–C(6)–C(5)	115 (1)	109 (1)	C(18)–C(13)–C(17)	108 (1)	108 (1)
C(8)–C(7)–C(6)	112 (1)	112 (1)	C(13)–C(14)–C(8)	111 (1)	114 (1)
C(9)–C(8)–C(7)	112 (1)	111 (1)	C(15)–C(14)–C(8)	120 (1)	120 (1)
C(14)–C(8)–C(7)	108 (1)	111 (1)	C(15)–C(14)–C(13)	102 (1)	103 (1)
C(14)–C(8)–C(9)	111 (1)	107 (1)	C(16)–C(15)–C(14)	107 (1)	106 (1)
C(10)–C(9)–C(8)	110 (1)	109 (1)	C(17)–C(16)–C(15)	104 (1)	106 (1)
C(11)–C(9)–C(8)	114 (1)	114 (1)	C(22)–C(16)–C(15)	114 (1)	
C(11)–C(9)–C(10)	115 (1)	113 (1)	C(22)–C(16)–C(17)	114 (1)	
Br(1)–C(9)–C(8)	109 (1)	111 (1)	C(16)–C(17)–C(13)	102 (1)	102 (1)
Br(1)–C(9)–C(10)	105 (1)	104 (1)	C(20)–C(17)–C(13)	113 (1)	114 (1)
Br(1)–C(9)–C(11)	102 (1)	105 (1)	C(20)–C(17)–C(16)	112 (1)	114 (1)
C(5)–C(10)–C(1)	113 (1)	111 (1)	O(17)–C(17)–C(13)	112 (1)	108 (1)
C(9)–C(10)–C(1)	111 (1)	113 (1)	O(17)–C(17)–C(16)	114 (1)	111 (1)
C(9)–C(10)–C(5)	109 (1)	109 (1)	O(17)–C(17)–C(20)	104 (1)	108 (1)
C(19)–C(10)–C(1)	106 (1)	103 (1)	C(21)–C(20)–C(17)	119 (1)	121 (1)
C(19)–C(10)–C(5)	106 (1)	104 (1)	O(20)–C(20)–C(17)	126 (2)	122 (2)
C(19)–C(10)–C(9)	113 (1)	116 (1)	O(20)–C(20)–C(21)	115 (1)	117 (2)
			O(21)–C(21)–C(20)	114 (2)	114 (2)

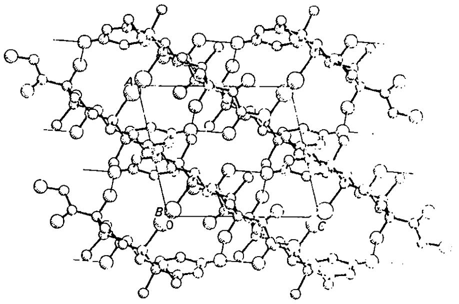

Fig. 3. View of (II) along **b** showing molecular packing and hydrogen bonding.

between the bonds and angles in the two molecules at a significance level of 3σ. There are minor differences in the comparative values of some of the torsion angles in the two molecules.* However, the following conformational details apply to both molecules. Ring *A* is planar within experimental error as is the enol side chain. Rings *B* and *C* have chair conformations. Ring *D* is a C(13) envelope. The ring junctions *A*/*B*, *B*/*C*, *C*/*D* are all *trans*. These structures are in all respects very similar to that of the closely related dexamethasone acetate (Terzis & Theophanides, 1975).

Hydrogen bonding: (I): O(3)···O(17)($-x,y+\frac{1}{2},-z$), 2·77 (2) Å; (II): O(3)···O(17)($-x,y-\frac{1}{2},-z$), 2·75 (2), O(3)···O(11)($x,y,z-1$), 2·82 (2) Å. Figs. 2 and 3 show the hydrogen bonding and molecular packing of compounds (I) and (II) respectively.

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* See deposition footnote.

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Anthranilic Acid I,* $C_7H_7NO_2$, by Neutron Diffraction

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Abstract. $M_r = 137\cdot14$, orthorhombic, $P2_1cn$ (non-standard setting of $Pna2_1$), $a = 12\cdot864\,(2)$, $b = 10\cdot790\,(2)$, $c = 9\cdot314\,(1)$ Å, $V = 1292\cdot81$ Å^3, $Z = 8$, $D_m = 1\cdot405\,(1)$, $D_x = 1\cdot409$ Mg m^{-3}, $\lambda = 1\cdot168$ Å, $\mu = 0\cdot17$ mm^{-1}, $F(000) = 576$, $T = 293$ K, $R = 0\cdot048$ for 1207 observed $[I > \sigma(I)]$ reflexions. There are two molecules per asymmetric unit, one is neutral and the other a zwitterion $C_6H_4.NH_3^+.COO^-$, confirming the X-ray results [Brown (1968). *Proc. R. Soc. London Ser. A*, **302**, 185–199] with significant differences in the C—N and C—O bond lengths in the two molecules. The molecules are linked three-dimensionally in the crystal by a set of six hydrogen bonds of lengths 2·497 (7)–2·894 (6) Å. The H-atom parameters have been refined, giving O—H 1·039 (12), mean C—H 1·081 (11) and mean N—H 1·048 (9) Å.

Introduction. The structure of the title compound has already been determined by X-rays (Brown, 1968), and this work was undertaken principally to confirm the suggested positions of the H atoms which had indicated the novel coexistence of neutral molecules and zwitterions in the same crystal.

Experimental. Large brown triangular crystals approx. 5 × 6 × 3 mm from cold ethanol. Density by flotation in NaI solution. Lattice parameters from 20 high-angle reflexions on a 57·3 mm radius camera. Intensities on a modified Hilger & Watts diffractometer at UKAEA, Harwell, 1207 reflexions observed out of 1264 counted. Corrections for Lp and absorption, transmission factors between 50 and 100% based on calculated path length. Empirical extinction for seven strong reflexions during refinement. Two standard reflexions, no variation other than that due to changes in neutron beam flux (< 5%).

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* 2-Aminobenzoic acid, low-temperature form.

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