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Structure of [20-CH_3],[20-CD_3]-Methylpregnene-3,20-diol Methanolate from Neutron Diffraction at 123 K

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Abstract. $C_{22}H_{33}D_3O_2.CH_3OH$, $M_r = 367.59$, monoclinic, $P2_1$, $a = 13.345\ (3)$, $b = 7.729\ (2)$, $c = 10.471\ (2)$ Å, $\beta = 101.18\ (2)°$, $V = 1059.5\ (4)$ Å^3, $Z = 2$, $D_n = 1.152$ g cm^{-3}, λ_n(Be,002) = 1.0508 Å, $\mu_n = 2.794$ cm^{-1}, $F(000) = 51.93$ fm, $T = 123$ K, $R(F^2) = 0.0390$, $wR(F^2) = 0.0408$, $S = 1.32$ for 3826 unique reflections and 601 variable parameters, including D/H scattering lengths at six sites. The statistical distribution of D between two methyl C20 groups, derived from b_{obs} (e.s.d.'s < 2%), shows the chirality at C20 to be 84.0% *S*, 16.0% *R* within ±0.6 and ±1.0% as estimated from respective averages of three values in each methyl group. The C—H bond lengths of tetrahedral C atoms (uncorrected for thermal motion) have average values and σ values from observed distributions of 1.098 (4) Å for 16 methylene C—H bonds, 1.090 (4) Å for 12 methyl C—H(D) bonds, and 1.105 (6) Å for five methine C—H bonds. The average H(D)—C—H(D) angles are 106.5 (4) and 107.8 (10)° for eight methylene and 15 methyl angles, respectively. The average e.s.d.'s are 0.003 (C—H and C—D) and 0.002 Å (C—C and C—O) for bond lengths and 0.3 [H(D)—C—H(D)], 0.2 [C—C—H(D)] and 0.09° (C—C—C) for bond angles. The H···O hydrogen-bond lengths and O—H···O angles are 1.693 (3) Å, 175.7 (3)°; 1.756 (3) Å, 171.6 (3)°; 1.771 (3) Å, 171.7 (3)°. Together, the three O—H···O bonds form an infinite helical chain about the 2_1 axis at $\frac{1}{2}$, 0. There are five distinct intermolecular H···H distances less than 2.3 Å, one being exceptionally short at 2.020 Å.

Experimental. Deuterated 20-methylpregnenediol was synthesized by the Grignard addition of a deuterated methyl group to pregnenolone and crystallized by slow cooling from a methanol–chloroform solution. The diffraction data from a crystal of dimensions 3.5 × 0.8 × 1.4 mm, 3.9 mm^3, were measured with the four-circle diffractometer at port H6M of the Brookhaven High Flux Beam Reactor. The neutron beam monochromated by reflection from Be(002) planes was of wavelength 1.0504 (1) Å as determined by calibration with a KBr crystal ($a_o = 6.6000$ Å at 295 K). The crystal was maintained at 123.0 (5) K inside a double-stage DISPLEX® helium cryostat. Lattice parameters were determined by a least-squares fit of $\sin^2\theta$ values for 29 reflections within the range $42 < 2\theta < 51°$. 5023 reflections [h, $-k$, $\pm l$; $h \le 20$, $|k| \le 10$, $|l| \le 15$; $\sin\theta/\lambda \le 0.77$ Å^{-1}] were measured by the $\theta/2\theta$ step-scan

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Table 1. *Coordinates* ($x \times 10^5$, $y \times 10^4$, $z \times 10^4$), *anisotropic thermal parameters* (Å$^2 \times 10^4$), *observed* b_{obs} (fm) *and* %D *at methyl sites partially occupied by* D

Atoms of methanol are indicated by an asterisk. The temperature factor has the form: $\exp(-2\pi^2\sum_i\sum_j h_i h_j a_i^* a_j^* U_{ij})$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C1	19665 (8)	8189 (2)	2363 (1)	206 (5)	136 (5)	338 (6)	−32 (4)	104 (4)	−61 (4)
C2	29552 (9)	7979 (2)	1841 (1)	215 (5)	154 (5)	346 (6)	−20 (4)	104 (4)	−22 (4)
C3	33936 (8)	6174 (2)	2155 (1)	183 (5)	178 (5)	204 (5)	−4 (4)	35 (4)	−30 (4)
C4	26111 (8)	4816 (2)	1572 (1)	186 (5)	154 (5)	236 (5)	6 (4)	41 (4)	−34 (4)
C5	16042 (8)	5044 (2)	2007 (1)	168 (4)	123 (5)	187 (4)	4 (4)	22 (3)	−21 (4)
C6	11610 (8)	3704 (2)	2489 (1)	184 (4)	126 (4)	244 (5)	15 (4)	26 (4)	18 (4)
C7	1466 (8)	3771 (2)	2902 (1)	200 (5)	124 (5)	253 (5)	7 (4)	33 (4)	29 (4)
C8	−4535 (8)	5424 (0)	2474 (1)	181 (4)	105 (5)	163 (4)	−1 (4)	15 (3)	4 (3)
C9	2595 (8)	7006 (2)	2630 (1)	181 (4)	112 (4)	189 (4)	−10 (4)	48 (3)	−7 (4)
C10	11282 (8)	6844 (2)	1838 (1)	175 (4)	114 (5)	180 (4)	6 (3)	43 (3)	1 (3)
C11	−3543 (9)	8688 (2)	2334 (1)	254 (5)	117 (5)	393 (7)	25 (4)	173 (5)	45 (5)
C12	−12193 (9)	8903 (2)	3096 (1)	235 (5)	120 (5)	339 (6)	−9 (4)	128 (4)	−15 (4)
C13	−19400 (8)	7339 (2)	2919 (1)	185 (4)	130 (5)	163 (4)	5 (4)	48 (3)	8 (4)
C14	−12773 (8)	5708 (2)	3277 (1)	172 (4)	125 (5)	166 (4)	−10 (4)	27 (3)	11 (4)
C15	−20537 (9)	4264 (2)	3326 (1)	223 (5)	130 (5)	297 (6)	−30 (4)	61 (4)	2 (4)
C16	−29379 (8)	5184 (2)	3823 (1)	202 (5)	170 (5)	277 (5)	−33 (4)	66 (4)	34 (4)
C17	−26566 (7)	7136 (2)	3920 (1)	172 (4)	164 (4)	162 (4)	−24 (4)	30 (3)	−1 (4)
C18	−25363 (9)	7253 (2)	1508 (1)	254 (5)	269 (6)	159 (4)	70 (5)	33 (4)	28 (4)
C19	7291 (9)	7157 (2)	376 (1)	246 (5)	258 (6)	193 (4)	64 (5)	58 (4)	62 (5)
C20	−35795 (8)	8346 (2)	3919 (1)	177 (4)	184 (5)	200 (4)	−18 (4)	61 (3)	−10 (4)
C21	−41420 (10)	7772 (2)	4992 (1)	269 (6)	270 (6)	307 (6)	−26 (5)	161 (5)	13 (5)
C22	−32509 (8)	10231 (2)	4139 (1)	220 (5)	170 (5)	263 (5)	−13 (4)	83 (4)	−39 (4)
O3	42913 (10)	5883 (2)	1644 (1)	183 (5)	242 (6)	264 (6)	−11 (5)	58 (4)	−49 (5)
O20	−43252 (9)	8225 (2)	2727 (1)	170 (5)	233 (6)	268 (6)	−27 (5)	15 (4)	0 (5)
C23*	47620 (12)	7284 (2)	8823 (1)	443 (7)	329 (8)	323 (6)	−163 (7)	0 (5)	40 (6)
O23*	41878 (13)	5812 (2)	9021 (1)	470 (9)	267 (7)	244 (7)	−136 (7)	57 (6)	−31 (6)
H11	21503 (21)	8087 (4)	3430 (3)	383 (12)	467 (15)	377 (13)	−86 (12)	91 (10)	−167 (12)
H12	16571 (23)	9500 (4)	2134 (4)	380 (14)	182 (12)	820 (23)	14 (10)	225 (14)	−16 (12)
H21	28184 (21)	8170 (4)	784 (3)	412 (13)	382 (14)	440 (14)	46 (12)	135 (11)	101 (12)
H22	35183 (21)	8956 (4)	2273 (3)	348 (13)	281 (13)	756 (20)	−122 (10)	183 (13)	−147 (12)
H31	35711 (19)	6019 (4)	3218 (2)	349 (11)	436 (13)	227 (9)	−3 (10)	33 (8)	−13 (10)
H41	29194 (19)	3512 (3)	1825 (3)	319 (11)	220 (12)	580 (16)	58 (9)	99 (11)	−21 (11)
H42	25025 (20)	4933 (4)	509 (2)	367 (12)	429 (14)	280 (11)	−11 (11)	77 (9)	−73 (10)
H61	15596 (21)	2465 (3)	2592 (3)	366 (12)	199 (11)	559 (16)	77 (9)	109 (11)	38 (10)
H71	2687 (21)	3658 (4)	3971 (2)	418 (13)	452 (14)	323 (12)	111 (12)	90 (10)	155 (11)
H72	−3086 (21)	2633 (3)	2521 (3)	355 (13)	199 (11)	594 (17)	−52 (9)	63 (12)	−16 (10)
H81	−8124 (18)	5284 (3)	1441 (2)	321 (10)	314 (11)	210 (9)	14 (10)	1 (8)	−32 (8)
H91	6374 (18)	7038 (4)	3669 (2)	332 (11)	334 (12)	239 (9)	−58 (10)	34 (8)	−55 (9)
H111	−6874 (24)	8737 (4)	1286 (3)	478 (15)	416 (15)	470 (15)	184 (12)	239 (12)	201 (13)
H112	1584 (24)	9803 (4)	2535 (4)	425 (15)	187 (13)	969 (26)	−48 (11)	362 (16)	−29 (13)
H121	−16341 (22)	10106 (4)	2777 (3)	406 (13)	224 (12)	740 (20)	80 (11)	277 (13)	74 (12)
H122	−8952 (22)	9061 (4)	4138 (3)	388 (13)	393 (15)	413 (14)	−79 (11)	111 (11)	−142 (11)
H141	−8618 (18)	5917 (3)	4293 (2)	309 (11)	322 (11)	214 (9)	−4 (9)	0 (8)	21 (9)
H151	−17297 (21)	3199 (4)	3964 (3)	394 (13)	270 (12)	622 (17)	31 (11)	146 (12)	164 (12)
H152	−23230 (21)	3711 (4)	2349 (3)	378 (13)	397 (14)	446 (14)	−89 (11)	54 (10)	−149 (12)
H161	−30330 (23)	4673 (4)	4766 (3)	484 (15)	387 (15)	463 (15)	28 (12)	199 (12)	177 (12)
H162	−36650 (19)	5001 (4)	3157 (3)	264 (11)	335 (13)	546 (16)	−52 (10)	0 (10)	−56 (12)
H171	−21612 (17)	7361 (4)	4885 (2)	316 (10)	345 (12)	220 (9)	−24 (10)	16 (7)	−15 (9)
H181	−29223 (24)	8473 (4)	1227 (3)	500 (15)	471 (16)	403 (13)	233 (13)	87 (12)	134 (12)
H182	−20359 (22)	7020 (5)	819 (2)	443 (14)	716 (19)	291 (11)	203 (15)	125 (10)	30 (13)
H183	−31128 (23)	6242 (4)	1371 (3)	451 (15)	514 (17)	352 (13)	−86 (14)	−48 (11)	−27 (12)
H191	5252 (26)	8510 (4)	177 (3)	652 (19)	357 (15)	450 (15)	191 (14)	150 (14)	180 (12)
H192	13001 (21)	6826 (4)	−202 (2)	426 (14)	632 (19)	307 (11)	163 (13)	157 (10)	51 (12)
H193	533 (25)	6386 (5)	9 (3)	416 (14)	628 (19)	335 (11)	−86 (14)	−14 (10)	43 (13)
D211	−47592 (16)	8668 (3)	5050 (2)	419 (12)	431 (13)	628 (14)	39 (9)	309 (10)	25 (10)
D212	−44667 (17)	6492 (3)	4791 (2)	442 (13)	354 (12)	585 (14)	−98 (9)	249 (10)	9 (9)
D213	−36225 (18)	7744 (3)	5933 (2)	491 (13)	647 (16)	288 (10)	33 (10)	128 (8)	52 (9)
H221	−29843 (41)	10756 (6)	3296 (5)	482 (29)	321 (27)	410 (28)	−9 (21)	208 (22)	50 (20)
H222	−39011 (40)	11032 (7)	4290 (6)	401 (28)	314 (27)	624 (36)	49 (21)	210 (24)	−63 (23)
H223	−26390 (39)	10349 (7)	5003 (5)	455 (28)	452 (30)	456 (28)	−40 (22)	−22 (20)	−92 (21)
H3O	48212 (18)	6698 (4)	2061 (2)	266 (11)	358 (13)	360 (12)	−51 (9)	56 (9)	−72 (10)
H20O	−42122 (19)	9108 (4)	2102 (2)	347 (12)	335 (13)	333 (12)	2 (10)	28 (9)	50 (10)
H231*	44104 (48)	8428 (6)	9106 (6)	1247 (46)	406 (22)	1471 (50)	−136 (24)	607 (38)	−96 (26)
H232*	55134 (34)	7267 (8)	9354 (5)	639 (25)	1154 (38)	1099 (36)	−477 (29)	−248 (24)	454 (33)
H233*	47670 (41)	7436 (7)	7820 (4)	1196 (37)	913 (31)	485 (19)	−570 (29)	143 (21)	110 (20)
H23O*	41932 (21)	5732 (4)	9953 (3)	449 (14)	379 (14)	326 (12)	−69 (12)	92 (10)	−13 (10)

	b_{obs}	%D
D211	5.06 (4)	84.5 (4)
D212	5.03 (5)	84.2 (5)
D213	4.95 (5)	83.4 (5)
H221	−2.02 (4)	16.5 (4)
H222	−2.01 (4)	16.6 (4)
H223	−2.19 (5)	14.9 (5)

method using scan widths $\Delta 2\theta = 3°$ for $\sin\theta/\lambda \leq 0.476$ Å^{-1} and $\Delta 2\theta = (0.95 + 3.29\tan\theta)°$ for $\sin\theta/\lambda > 0.476$ Å^{-1}. The intensities of two reflections, monitored at regular intervals, showed no systematic variations. Integrated intensities I_o and variances $\sigma^2(I_o)$ were derived from the scan profiles as described previously (McMullan, Epstein, Ruble & Craven, 1979). Absorption corrections (de

Meulenaer & Tompa, 1965; Templeton & Templeton, 1973) were applied using the μ/ρ value of 2.390 $m^2\ Kg^{-1}$ for H at $\lambda = 1.0504$ Å. Minimum and maximum transmission factors were 0.66 and 0.81. Averaging F_o^2 values of symmetry-related $(0 -k \pm l)$ reflections gave R_{int} of 2.4% and 3827 unique observations of which 118 had $-F_o^2$ values, but none less than -2σ.

The initial atomic parameters were taken from the 295 K X-ray analysis of Duax & Osawa (1980). Coherent neutron-scattering lengths (fm) for H (−3.7409), D (6.674), C (6.6484) and O (5.803) were from the tabulation of Koester (1977). The H and D atoms were located in a difference map. Refinement was carried out by full-matrix least squares using the program *UPALS* (Lundgren, 1982). A previous analysis with the same data set (Fronckowiak & McMullan, 1980) employed the method of large-block refinement since the computer facilities then available were not adequate for full-matrix procedures. In both cases, the quantity $\sum w|F_o^2 - F_c^2|^2$ was minimized with weights $w = [\sigma_c^2(F_c^2) + (0.02F_o^2)^2]^{-1}$, summing over all 3827 independent observations. The variable parameters were coordinates and anisotropic thermal factors for 66 non-related atoms, scattering lengths at six D/H sites, one scale factor, and the isotropic secondary-extinction parameter for a type I crystal (Becker & Coppens, 1974). The y coordinate of atom C8 was held fixed to define the origin. Extinction corrections greater than 2% were applied to 24 observations, the largest being $1.21 \times F_o^2$ for reflection 201. The refinement converged $[\Delta p_i/\sigma(p_i) < 0.001]$ with fit indices $R(F^2) = 0.0390$, $wR(F^2) = 0.0408$, $S = 1.132$.* In the final ΔF map, the largest $|\Delta\rho|$ e.s.d.'s were < 1% of the peak maximum for C in the ρ_o map.

Nuclear positional and anisotropic thermal parameters, refined scattering lengths and percentages of D in methyl groups at chiral atom C20 are listed in Table 1.† No positional or thermal parameter derived from the present full-matrix refinement differs by more than one e.s.d. from the blocked-refinement value (Fronckowiak & McMullan, 1980). The molecular framework and conformation of 20-methylpregnenediol with atom-numbering scheme is illustrated in Fig. 1. The thermal vibration parameters are depicted in Fig. 2. Bond lengths and angles for the steroid framework are shown on Fig. 1; those involving H and D atoms are listed in Table 2. No thermal motion corrections were applied to the bond length values. Selected torsion angles in the molecule are given in Table 3.

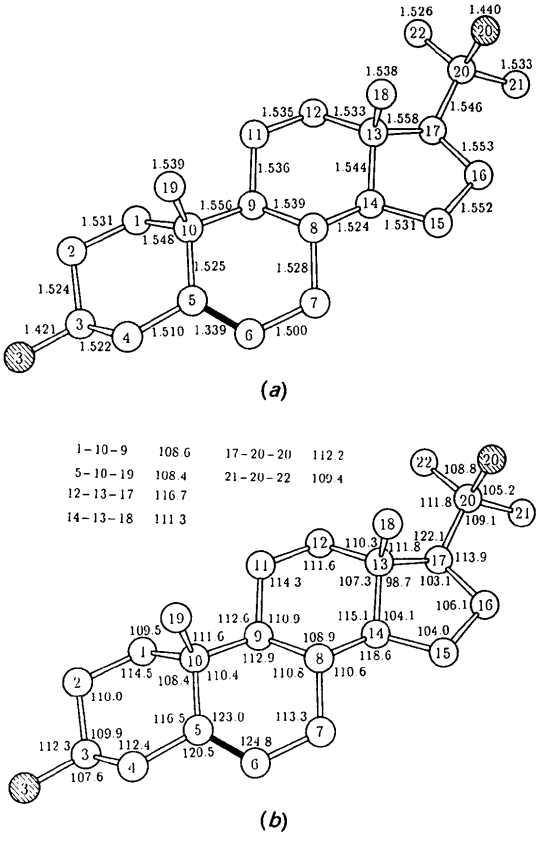

Fig. 1. Molecular framework and numbering scheme of 20-methylpregnenediol viewed approximately normal to plane of steroid nucleus. O atoms are represented by shaded circles; the double bond is indicated by a solid line. (*a*) Bond lengths (Å) have e.s.d.'s of 0.001–0.002 Å. (*b*) Bond angles (°) have e.s.d.'s of 0.09—0.10°.

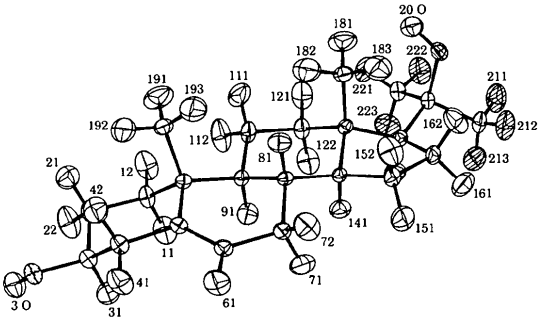

Fig. 2. Perspective view of 20-methylpregnenediol at 123 K with thermal ellipsoids shown at 50% probability level (Johnson, 1976). O-atom and partially occupied D-atom positions are indicated by shading.

* $R(F^2) = \sum\Delta/\sum F_o^2$, $wR(F^2) = [\sum w\Delta^2/\sum(wF_o^2)^2]^{\frac{1}{2}}$, $S = [\sum w\Delta^2/(n-p)]^{\frac{1}{2}}$, where $\Delta = |F_o^2 - F_c^2|$, and n and p are the numbers of observations and parameters, respectively.

† Lists of structure factors, C—C and C—O bond lengths, and C—C—C and C—C—O bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55011 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0374]

Table 2. *Bond lengths* (Å) *and angles* (°) *involving* H/D

Methylene groups

C	C_m	C_n	C—H1	C—H2	H—C—H	C_m—C—H1	C_m—C—H2	C_n—C—H1	C_n—C—H2
C1	C10	C2	1.102 (3)	1.103 (3)	106.7 (3)	108.4 (2)	109.0 (2)	108.5 (2)	109.4 (2)
C2	C1	C3	1.096 (3)	1.099 (3)	106.8 (2)	111.1 (2)	110.4 (2)	108.8 (2)	109.7 (2)
C4	C3	C5	1.102 (3)	1.099 (3)	106.7 (2)	109.9 (2)	107.2 (2)	110.3 (2)	110.2 (2)
C7	C6	C8	1.103 (3)	1.099 (3)	105.5 (2)	109.0 (2)	109.5 (2)	109.3 (2)	109.9 (2)
C11	C9	C12	1.102 (4)	1.095 (3)	106.3 (3)	109.1 (2)	109.8 (2)	108.4 (2)	108.7 (2)
C12	C11	C13	1.100 (3)	1.099 (3)	106.6 (2)	108.5 (2)	109.7 (2)	111.2 (2)	109.0 (2)
C15	C14	C16	1.094 (3)	1.103 (3)	107.1 (3)	112.3 (2)	110.6 (2)	112.1 (2)	110.9 (2)
C16	C15	C17	1.093 (3)	1.089 (3)	106.6 (2)	111.5 (2)	111.5 (2)	111.2 (2)	110.0 (2)

Methyl groups

C	C_m	C—H1	C—H2	C—H3	C_m—C—H1	C_m—C—H2	C_m—C—H3	H1—C—H2	H1—C—H3	H2—C—H3
C18	C13	1.087 (3)	1.088 (3)	1.086 (3)	110.6 (2)	112.0 (2)	112.3 (2)	106.5 (2)	107.6 (3)	107.7 (3)
C19	C10	1.090 (3)	1.093 (3)	1.087 (3)	111.5 (2)	111.8 (2)	111.7 (2)	107.1 (2)	107.1 (3)	107.4 (3)
C21	C20	1.086 (2)	1.084 (2)	1.089 (3)	109.9 (2)	110.8 (2)	110.6 (2)	108.3 (2)	108.6 (2)	108.5 (2)
C22	C20	1.092 (5)	1.102 (5)	1.099 (5)	111.2 (3)	110.3 (3)	110.4 (3)	107.3 (4)	109.2 (4)	108.3 (4)
C23	O23	1.069 (6)	1.048 (5)	1.058 (4)	110.3 (3)	113.8 (3)	110.1 (3)	106.8 (5)	105.7 (5)	109.8 (4)

Methine groups

C	C_l	C_m	C_n	C—H	C_l—C—H	C_m—C—H	C_n—C—H
C3	C2	C4	O3	1.099 (3)	108.5 (2)	109.1 (2)	109.4 (2)
C6	C5	C7		1.090 (3)	118.3 (2)	116.9 (2)	
C8	C7	C9	C14	1.100 (2)	108.1 (2)	109.0 (2)	109.5 (1)
C9	C8	C10	C11	1.108 (2)	106.1 (2)	106.3 (1)	107.6 (2)
C14	C8	C13	C15	1.112 (2)	105.7 (1)	105.7 (1)	106.8 (1)
C17	C13	C16	C20	1.109 (2)	105.2 (1)	107.8 (2)	103.9 (2)

Hydroxyl groups

O	C	O—H	C—O—H
O3	C3	0.984 (3)	108.5 (2)
O20	C20	0.978 (3)	111.3 (2)
O23	C23	0.976 (3)	107.4 (2)

Table 3. *Selected bond torsion angles* (°)

C2—C3—O3—H31O	63.0 (2)	C4—C5═C6—C7	−177.6 (1)
C10—C1—C2—C3	−57.5 (1)	C3—C4—C5═C6	−129.6 (1)
C1—C2—C3—C4	57.8 (1)	C5═C6—C7—C8	11.6 (2)
C2—C3—C4—C5	−55.1 (1)	C1—C10—C5═C6	134.3 (1)
C3—C4—C5—C10	51.6 (1)	C13—C17—C20—C21	178.3 (1)
C4—C5—C10—C1	−47.0 (1)	C13—C17—C20—C22	−60.5 (1)
C5—C10—C1—C2	50.0 (1)	C12—C13—C18—H183	−173.9 (2)
C2—C1—C10—C9	169.9 (1)	C1—C10—C19—H193	−170.3 (2)
C10—C5═C6—C7	1.1 (2)	C17—C20—C22—H222	−170.9 (3)
C10—C5═C6—H61	−179.0 (2)	C17—C20—C21—D211	175.8 (2)
C4—C5═C6—H61	2.2 (2)	C17—C20—O20—H201	−93.9 (2)

Related literature. One other steroid structure, that of cholesteryl acetate (Weber, Craven, Sawzik & McMullan, 1991), has been determined from neutron-diffraction data. The tables and figures reported here and for the cholesteryl acetate structure provide ready comparisons of bond lengths, bond angles and conformations of the two steroid molecules. The biochemical implications of statistical populations of $—CD_3$ and $—CH_3$ groups at the chiral C20 center (Table 1), observed on deuteromethylation of pregnenolone, have been discussed by Fronckowiak (1982). The molecular dimensions and conformation of 20-methylpregnenediol (Fronckowiak & McMullan, 1980; Fronckowiak, 1982) and the side chain and backbone conformations of other steroids from X-ray analyses have been compared with results obtained by molecular mechanics calculations (Duax, Fronckowiak, Griffin & Rohrer, 1982).

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