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1,3,5(10)-Oestratrien-3,17α-diol

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Abstract. $C_{18}H_{24}O_2$, monoclinic, $P2_1$, $a=9{\cdot}148$ (5), $b=23{\cdot}292$ (10), $c=7{\cdot}235$ (5) Å, $\beta=98{\cdot}70$ (7)°, $Z=4$. The two independent molecules do not have exactly the same conformations. With a 17α substituent, the two *D* rings are almost in the half-chair conformation. In the crystal, the molecules are disposed

Table 1. *Atomic coordinates* ($\times 10^4$) *and thermal parameters for non-hydrogen atoms*

	x	*y*	*z*	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(1)	3975 (6)	−3285 (2)	5184 (7)	184 (9)	17 (1)	176 (11)	−3 (5)	97 (15)	0 (6)
C(2)	3865 (6)	−3871 (2)	5506 (7)	190 (9)	17 (1)	216 (13)	−13 (5)	86 (17)	−20 (6)
C(3)	4060 (5)	−4076 (2)	7334 (6)	126 (7)	14 (1)	214 (11)	4 (4)	109 (14)	2 (5)
C(4)	4396 (5)	−3699 (2)	8778 (6)	114 (6)	15 (1)	183 (10)	9 (4)	90 (13)	5 (5)
C(5)	4527 (5)	−3107 (2)	8471 (6)	102 (6)	13 (1)	164 (10)	2 (4)	62 (12)	3 (5)
C(6)	4905 (6)	−2714 (2)	10162 (7)	149 (8)	15 (1)	161 (10)	−2 (4)	−4 (14)	0 (5)
C(7)	5421 (5)	−2123 (2)	9646 (6)	141 (8)	15 (1)	172 (10)	−7 (4)	−14 (14)	−4 (5)
C(8)	4370 (5)	−1883 (2)	7998 (6)	105 (6)	13 (1)	163 (9)	−6 (4)	63 (12)	−7 (5)
C(9)	4467 (5)	−2251 (2)	6264 (6)	115 (7)	15 (1)	168 (10)	−9 (4)	69 (13)	0 (5)
C(10)	4310 (5)	−2894 (2)	6642 (6)	115 (7)	14 (1)	174 (10)	1 (4)	82 (13)	0 (5)
C(11)	3362 (6)	−2032 (2)	4585 (7)	186 (9)	19 (1)	189 (12)	−19 (5)	−20 (16)	9 (6)
C(12)	3672 (6)	−1397 (2)	4154 (7)	191 (9)	19 (1)	213 (12)	−18 (5)	1 (17)	21 (6)
C(13)	3629 (5)	−1024 (2)	5858 (6)	108 (6)	16 (1)	214 (11)	−4 (4)	57 (13)	16 (5)
C(14)	4713 (5)	−1263 (2)	7509 (6)	113 (6)	13 (1)	177 (10)	−1 (4)	70 (12)	−1 (5)
C(15)	4795 (7)	−792 (2)	8983 (7)	217 (10)	16 (1)	229 (13)	3 (5)	120 (18)	−1 (6)
C(16)	4587 (6)	−225 (2)	7840 (8)	173 (9)	17 (1)	274 (14)	20 (5)	119 (18)	0 (6)
C(17)	4211 (5)	−404 (2)	5768 (7)	113 (7)	14 (1)	237 (12)	10 (4)	57 (14)	21 (6)
C(18)	2030 (6)	−981 (3)	6278 (9)	128 (9)	27 (2)	446 (20)	14 (6)	118 (21)	63 (9)
O(83)	3900 (4)	−4645 (1)	7757 (5)	190 (6)	14 (1)	221 (8)	0 (3)	141 (11)	−2 (4)
O(97)	5519 (3)	−374 (1)	4847 (4)	130 (5)	17 (1)	235 (8)	−7 (3)	80 (10)	25 (4)
C(51)	9849 (5)	−1846 (2)	9620 (6)	123 (7)	14 (1)	162 (10)	−6 (4)	14 (13)	−5 (5)
C(52)	9344 (5)	−1287 (2)	9336 (6)	129 (7)	13 (1)	189 (10)	−6 (4)	44 (13)	−26 (5)
C(53)	8657 (5)	−1127 (2)	7578 (6)	91 (6)	13 (1)	207 (11)	1 (4)	49 (12)	−2 (5)
C(54)	8474 (5)	−1526 (2)	6127 (6)	96 (6)	14 (1)	182 (10)	1 (4)	34 (12)	4 (5)
C(55)	8964 (5)	−2085 (2)	6417 (6)	103 (6)	14 (1)	155 (9)	2 (4)	53 (12)	−3 (5)
C(56)	8669 (6)	−2500 (2)	4808 (6)	173 (8)	16 (1)	161 (11)	15 (5)	50 (15)	4 (5)
C(57)	9405 (5)	−3081 (2)	5184 (6)	152 (8)	16 (1)	169 (10)	15 (4)	49 (14)	−8 (5)
C(58)	9287 (5)	−3285 (2)	7142 (6)	119 (6)	12 (1)	172 (10)	3 (4)	23 (13)	0 (5)
C(59)	10211 (5)	−2874 (2)	8559 (6)	90 (6)	13 (1)	156 (10)	−1 (4)	29 (11)	−4 (5)
C(60)	9674 (5)	−2259 (2)	8217 (6)	89 (6)	12 (1)	172 (10)	−4 (3)	63 (12)	−6 (5)
C(61)	10256 (5)	−3070 (2)	10598 (6)	141 (7)	15 (1)	161 (10)	10 (4)	14 (13)	−5 (5)
C(62)	10694 (5)	−3700 (2)	10903 (6)	127 (7)	16 (1)	173 (10)	13 (4)	46 (13)	3 (5)
C(63)	9716 (5)	−4088 (2)	9544 (6)	104 (6)	16 (1)	188 (11)	1 (4)	47 (13)	11 (5)
C(64)	9836 (5)	−3894 (2)	7546 (6)	125 (7)	14 (1)	179 (10)	0 (4)	24 (13)	−4 (5)
C(65)	9116 (7)	−4383 (2)	6316 (8)	239 (11)	16 (1)	265 (15)	0 (6)	−85 (20)	−11 (7)
C(66)	9584 (7)	−4925 (3)	7513 (9)	252 (12)	19 (1)	332 (17)	−9 (7)	−82 (23)	−14 (8)
C(67)	10152 (6)	−4726 (2)	9509 (7)	137 (8)	16 (1)	269 (13)	0 (4)	21 (16)	14 (6)
C(68)	8123 (6)	−4077 (3)	9973 (8)	130 (8)	25 (1)	348 (17)	6 (6)	151 (18)	49 (8)
O(33)	8135 (3)	−581 (1)	7179 (4)	131 (5)	13 (1)	235 (8)	5 (3)	11 (10)	−12 (4)
O(37)	11697 (4)	−4836 (1)	10064 (5)	129 (5)	17 (1)	236 (8)	16 (3)	80 (10)	25 (4)

in (b,c) sheets linked to each other by hydrogen bonds.

Introduction. 1,3,5(10)-Oestratriene-3,17α-diol (or iso-oestradiol) was crystallized from ethanol. Cell parameters and intensities were obtained on an automatic Siemens diffractometer with Cu $K\alpha$ radiation. 2866 independent reflexions were collected up to $\theta = 70°$.

The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971) and refined by least squares. The H atoms were located from a difference synthesis. The final R was 0·058. Positional and thermal parameters of the heavy atoms are given in Table 1, the hydrogen coordinates and isotropic thermal factors in Table 2.*

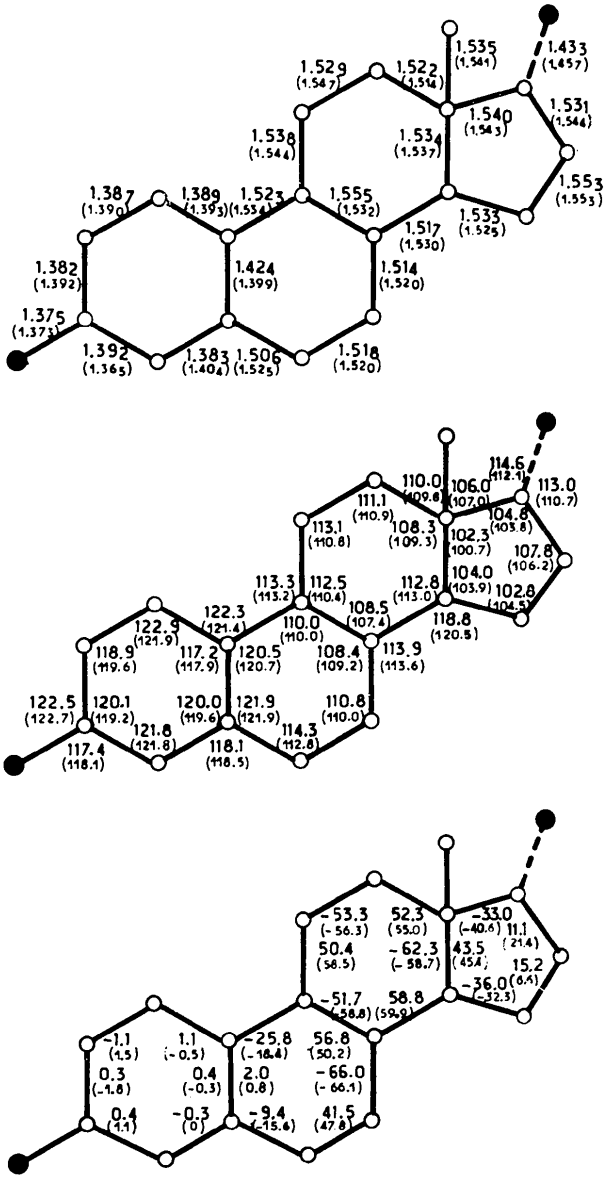

Fig. 1. Conformation of the molecule.

Table 2. *Atomic coordinates* ($\times 10^4$) *and thermal parameters for hydrogen atoms*

	x	y	z	B_i
H(22)	3434 (47)	−4129 (20)	4485 (59)	5·9
H(21)	3870 (48)	−3154 (20)	3888 (62)	5·5
H(24)	4423 (46)	−3815 (19)	10224 (58)	5·4
H(26)	3876 (47)	−2727 (19)	10676 (62)	5·2
H(46)	5594 (47)	−2903 (20)	11359 (61)	5·5
H(27)	5679 (47)	−1922 (20)	10725 (59)	5·4
H(47)	6412 (50)	−2100 (20)	9179 (61)	5·5
H(28)	3389 (44)	−1928 (19)	8258 (55)	4·5
H(34)	5610 (45)	−1241 (18)	6926 (59)	4·6
H(29)	5482 (48)	−2193 (19)	6119 (61)	4·8
H(31)	2196 (48)	−2090 (20)	4856 (61)	5·6
H(41)	3553 (49)	−2203 (20)	3409 (60)	5·6
H(32)	4669 (45)	−1396 (19)	3805 (56)	4·9
H(42)	3036 (46)	−1238 (19)	2981 (60)	5·4
H(35)	3974 (49)	−807 (20)	9508 (59)	4·6
H(45)	5846 (48)	−843 (20)	9962 (61)	5·9
H(36)	3824 (48)	−5 (20)	8240 (59)	5·4
H(76)	5570 (46)	2 (20)	8091 (58)	5·4
H(77)	3562 (48)	−97 (18)	5238 (62)	5·5
H(23)	4096 (46)	−4910 (21)	6787 (57)	5·5
H(78)	2023 (50)	−1058 (20)	7567 (63)	6·8
H(38)	1456 (56)	−1280 (22)	5449 (69)	7·5
H(93)	8289 (46)	−371 (20)	8222 (58)	5·3
H(72)	9421 (46)	−1012 (20)	10498 (60)	5·2
H(71)	10159 (46)	−1973 (20)	11044 (58)	5·2
H(74)	8018 (46)	−1352 (19)	4808 (60)	4·9
H(86)	7686 (48)	−2616 (19)	4930 (62)	5·4
H(96)	8476 (50)	−2338 (20)	3578 (62)	5·9
H(19)	8756 (50)	−3347 (20)	4151 (62)	5·9
H(20)	10269 (47)	−3088 (19)	4980 (59)	5·0
H(48)	8188 (45)	−3228 (19)	7540 (56)	4·4
H(49)	11104 (47)	−2851 (19)	8285 (59)	4·9
H(81)	10908 (48)	−2835 (19)	11531 (61)	4·9
H(91)	9253 (44)	−2995 (18)	11057 (58)	4·2
H(82)	11620 (47)	−3752 (19)	10689 (56)	5·3
H(92)	10424 (47)	−3803 (20)	12120 (57)	5·6
H(84)	10807 (47)	−3904 (20)	7160 (58)	5·3
H(30)	8059 (47)	−4296 (20)	6541 (59)	5·2
H(40)	9515 (48)	−4390 (20)	5110 (63)	5·7
H(25)	12253 (48)	−4818 (20)	9284 (61)	5·7
H(39)	7636 (47)	−3653 (20)	9795 (56)	5·1
H(43)	7975 (46)	−4133 (20)	11062 (62)	5·3
H(44)	7484 (47)	−4355 (20)	9069 (62)	5·4
H(50)	9578 (46)	−4915 (20)	10516 (60)	5·3
H(69)	10403 (49)	−5105 (19)	6937 (62)	5·7
H(70)	8717 (49)	−5173 (18)	7514 (60)	5·2
H(75)	1520 (53)	−612 (21)	6014 (63)	6·9
H(87)	6487 (52)	−497 (21)	5695 (62)	6·9

Discussion. Bond lengths, valency and dihedral angles of the two independent molecules are given in Fig. 1.

The crystal packing is shown in Fig. 2. Although we have quite a different set of hydrogen bonds, the iso-oestradiol molecules are lying in sheets parallel to the (b,c) plane as occurred for other oestrogen molecules crystallizing in a similar space group: oestrone (Busetta, Courseille & Hospital, 1973) and oestriol (Cooper,

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31584 (31 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Norton & Hauptman, 1969). As is often the case for steroid molecules, no intermolecular contact less than the sum of the van der Waals radii is observed. Thus, the molecular deformations are due only to the influence of the hydrogen bonds, and may be forecast if we consider the components of the apparent repulsions involved by them (Precigoux, 1973). On molecule 1, the hydrogen bonds exert actions in opposite directions; on molecule 2 the repulsion components are drawn in the same direction, towards the upper face of the steroid molecule. Thus we may forecast that this molecule will be distorted in that direction. Fig. 3, where the projections of both molecules are superimposed onto a plane perpendicular to the aromatic ring *A*, shows the extent of the distortion. The molecule is folded at the level of the cyclohexenic ring *B* with variations of the dihedral angles up to 6°. But the more rigid ring *C* is also affected and the dihedral angles C(8)–C(9) and C(9)–C(11) are closed by 7° and 8°.

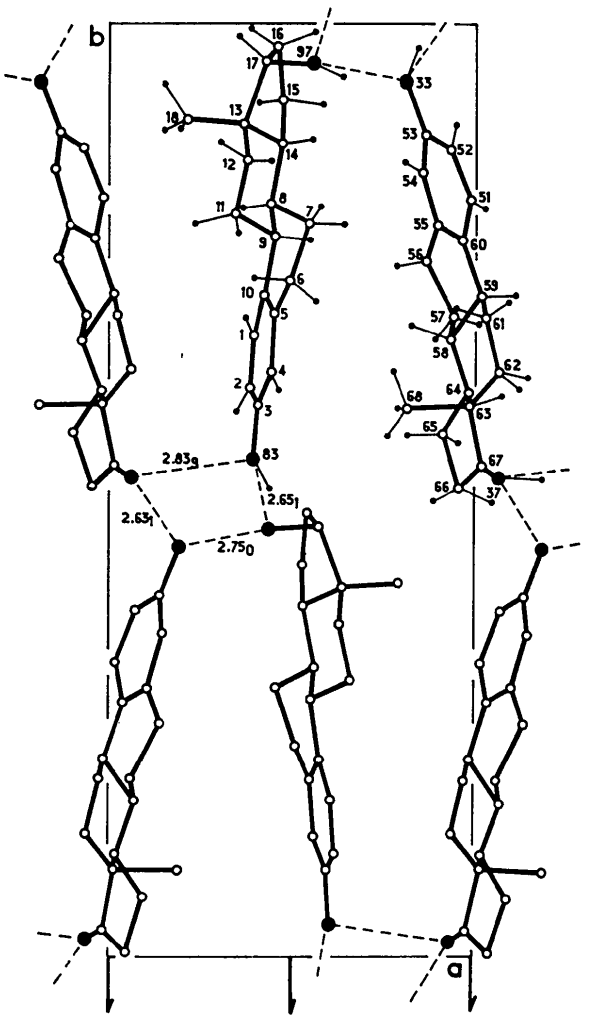

Fig. 2. Projection of the structure along c.

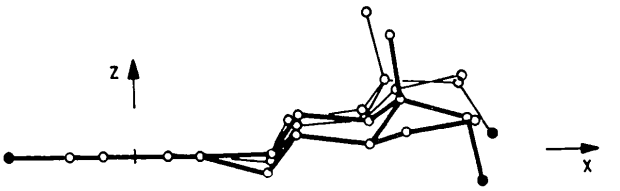

Fig. 3. Projection of the molecule on a plane perpendicular to the *A* ring: molecule 1 in thick line, molecule 2 in thin line.

If we use the Δ and φ_m parameters (Altona, Geise & Romers, 1968) to describe the pentagonal ring *D*, we see that this ring with a conformation close to the β envelope form in molecule 1 ($\Delta = 18{\cdot}8°$, $\varphi_m = 46{\cdot}0°$) is distorted towards the α envelope form in molecule 2 ($\Delta = -5{\cdot}7°$, $\varphi_m = 43{\cdot}5°$).

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