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Structure of *rac*-*D*-Homo-8α-estrone

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Abstract. *rac*-*D*-Homo-3-hydroxy-8α-estra-1,3,5(10)-trien-17a-one, $C_{19}H_{24}O_2$, $M_r = 284\cdot4$, monoclinic, $P2_1/c$, $a = 7\cdot597\ (1)$, $b = 13\cdot814\ (1)$, $c = 14\cdot399\ (1)$ Å, $\beta = 92\cdot05°$, $V = 1510\cdot2\ (4)$ Å^3, $Z = 4$, $D_x = 1\cdot251$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1\cdot5418$ Å, $\mu = 0\cdot627$ mm^{-1}, $F(000) = 616$, $T = 298$ K, $R = 0\cdot045$ for 2413 reflections with $F^2 > 2\cdot0\sigma(F^2)$. The title compound is a synthetic estrogen whose anomalous activity required determination of the configuration at C(8) relative to the configuration at C(13) and the effect of configuration on the conformation of the compound.

Experimental. The title compound was synthesized by the methods reported in Sozokina, Barkova, Zakharychev, Chigir, Ananchenko & Torgov (1973). Crystallization from 90% ethanol, single crystal 0·24 × 0·28 × 0·32 mm. The unit-cell parameters were refined from least-squares analysis of 2θ values for 25 reflections with $50 < 2\theta < 54°$. Intensities for 2519 unique reflections having $2\theta < 114°$ ($-8 \leq h \leq 8$, $0 \leq k \leq 16$, $-16 \leq l \leq 16$) measured on a Syntex $P3$ diffractometer, using a θ–2θ scan mode, Ni-filtered Cu radiation, no monochromator, scan speed from 2·5 to 25° min^{-1} in 2θ, scan width $(1\cdot1 + 1\cdot1\tan\theta)°$. Four standard reflections (008, 080, 500, $\bar{3}44$) were measured every 100 reflections and varied in intensity by ≤ 5% during the data collection.

Direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed positions of all non-H atoms. The positional and anisotropic displacement parameters of all non-H atoms were refined by full-matrix least squares on F using the 2413 reflections for which $I > 2\sigma(I)$. The H-atom positions were located in a difference map and refined with assigned isotropic temperature parameters of the parent atom. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final $R =$

Table 1. *Fractional positional parameters* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10) *for non*-H *atoms with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}
C(1)	−438 (2)	2872 (1)	9764 (1)	35 (1)
C(2)	−1843 (2)	2534 (1)	10260 (1)	37 (1)
C(3)	−3426 (2)	3036 (1)	10200 (1)	34 (1)
C(4)	−3556 (2)	3872 (1)	9680 (1)	33 (1)
C(5)	−2140 (2)	4217 (1)	9185 (1)	31 (1)
C(6)	−2316 (2)	5147 (1)	8647 (1)	38 (1)
C(7)	−1012 (2)	5231 (1)	7872 (1)	35 (1)
C(8)	849 (2)	5033 (1)	8272 (1)	28 (1)
C(9)	991 (2)	3994 (1)	8637 (1)	30 (1)
C(10)	−555 (2)	3699 (1)	9214 (1)	30 (1)
C(11)	1211 (2)	3269 (1)	7836 (1)	42 (1)
C(12)	2808 (3)	3514 (1)	7261 (1)	43 (1)
C(13)	2714 (2)	4536 (1)	6849 (1)	32 (1)
C(14)	2402 (2)	5273 (1)	7645 (1)	27 (1)
C(15)	2427 (2)	6316 (1)	7298 (1)	34 (1)
C(16)	4221 (2)	6558 (1)	6914 (1)	38 (1)
C(17)	4764 (3)	5836 (1)	6175 (1)	42 (1)
C(17a)	4476 (2)	4798 (1)	6435 (1)	35 (1)
C(18)	1357 (3)	4585 (2)	6039 (1)	46 (1)
O(3)	−4885 (2)	2735 (1)	10652 (1)	48 (1)
O(17a)	5597 (2)	4189 (1)	6287 (1)	46 (1)

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Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·386 (2)	C(9)—C(10)	1·518 (2)
C(1)—C(10)	1·392 (2)	C(9)—C(11)	1·541 (2)
C(2)—C(3)	1·388 (2)	C(11)—C(12)	1·531 (3)
C(3)—C(4)	1·379 (2)	C(12)—C(13)	1·531 (2)
C(3)—O(3)	1·370 (2)	C(13)—C(14)	1·557 (2)
C(4)—C(5)	1·395 (2)	C(13)—C(17a)	1·529 (2)
C(5)—C(6)	1·504 (2)	C(13)—C(18)	1·530 (2)
C(5)—C(10)	1·400 (2)	C(14)—C(15)	1·525 (2)
C(6)—C(7)	1·523 (2)	C(15)—C(16)	1·527 (2)
C(7)—C(8)	1·532 (2)	C(16)—C(17)	1·525 (2)
C(8)—C(9)	1·531 (2)	C(17)—C(17a)	1·500 (2)
C(8)—C(14)	1·547 (2)	C(17a)—O(17a)	1·221 (2)

C(2)—C(1)—C(10)	122·3 (1)	C(5)—C(10)—C(9)	121·9 (1)
C(1)—C(2)—C(3)	118·8 (1)	C(9)—C(11)—C(12)	111·7 (1)
C(2)—C(3)—C(4)	119·8 (1)	C(11)—C(12)—C(13)	112·7 (1)
C(2)—C(3)—O(3)	122·2 (1)	C(12)—C(13)—C(14)	108·9 (1)
C(4)—C(3)—O(3)	117·9 (1)	C(12)—C(13)—C(17a)	109·9 (1)
C(3)—C(4)—C(5)	121·5 (1)	C(12)—C(13)—C(18)	110·9 (1)
C(4)—C(5)—C(6)	119·9 (1)	C(14)—C(13)—C(17a)	106·9 (1)
C(4)—C(5)—C(10)	119·3 (1)	C(14)—C(13)—C(18)	114·6 (1)
C(6)—C(5)—C(10)	120·8 (1)	C(17a)—C(13)—C(18)	105·5 (1)
C(5)—C(6)—C(7)	113·2 (1)	C(8)—C(14)—C(13)	115·4 (1)
C(6)—C(7)—C(8)	109·1 (1)	C(8)—C(14)—C(15)	114·4 (1)
C(7)—C(8)—C(9)	110·4 (1)	C(13)—C(14)—C(15)	111·9 (1)
C(7)—C(8)—C(14)	117·1 (1)	C(14)—C(15)—C(16)	110·3 (1)
C(9)—C(8)—C(14)	110·9 (1)	C(15)—C(16)—C(17)	112·1 (1)
C(8)—C(9)—C(10)	113·1 (1)	C(16)—C(17)—C(17a)	113·9 (1)
C(8)—C(9)—C(11)	111·1 (1)	C(13)—C(17a)—C(17)	117·5 (1)
C(10)—C(9)—C(11)	110·0 (1)	C(13)—C(17a)—O(17a)	122·0 (1)
C(1)—C(10)—C(5)	118·3 (1)	C(17)—C(17a)—O(17a)	120·4 (1)
C(1)—C(10)—C(9)	119·8 (1)		

Ring *A*		Ring *C*	
C(10)—C(1)—C(2)—C(3)	0·3 (2)	C(14)—C(8)—C(9)—C(11)	−52·5 (2)
C(1)—C(2)—C(3)—C(4)	−2·2 (2)	C(8)—C(9)—C(11)—C(12)	56·5 (2)
C(2)—C(3)—C(4)—C(5)	1·8 (2)	C(9)—C(11)—C(12)—C(13)	−57·8 (2)
C(3)—C(4)—C(5)—C(10)	0·4 (2)	C(11)—C(12)—C(13)—C(14)	53·2 (2)
C(4)—C(5)—C(10)—C(1)	−2·2 (2)	C(12)—C(13)—C(14)—C(8)	−51·1 (2)
C(5)—C(10)—C(1)—C(2)	1·8 (2)	C(13)—C(14)—C(8)—C(9)	51·7 (2)

Ring *B*		Ring *D*	
C(10)—C(5)—C(6)—C(7)	23·7 (2)	C(17a)—C(13)—C(14)—C(15)	57·2 (1)
C(5)—C(6)—C(7)—C(8)	−51·7 (2)	C(13)—C(14)—C(15)—C(16)	−61·6 (2)
C(6)—C(7)—C(8)—C(9)	63·4 (2)	C(14)—C(15)—C(16)—C(17)	53·9 (2)
C(7)—C(8)—C(9)—C(10)	−45·4 (2)	C(15)—C(16)—C(17)—C(17a)	−45·6 (2)
C(8)—C(9)—C(10)—C(5)	17·0 (2)	C(16)—C(17)—C(17a)—C(13)	45·7 (2)
C(9)—C(10)—C(5)—C(6)	−5·9 (2)	C(17)—C(17a)—C(13)—C(14)	−49·8 (2)

0·045, $wR = 0{\cdot}072$, $S = 2{\cdot}34$ for observed reflections and $R = 0{\cdot}047$ for all data, $w = 1/\sigma^2$, $(\Delta/\sigma)_{\max} = 0{\cdot}75$. Weighting scheme based on estimates of experimental errors from counting statistics. Final difference map showed maximum positive and negative peaks of 0·19 and −0·22 e Å^{-3}. No corrections for absorption or extinction were made.

Atomic parameters are listed in Table 1. Distances, angles and selected torsion angles are listed in Table 2.* A stereoscopic view of the molecule showing the atomic numbering and the molecular conformation is given in Fig. 1. We have shown the enantiomorph corresponding to the naturally occurring estrogen absolute configuration at C(13). Fig. 2 shows a stereoview of the molecular packing.

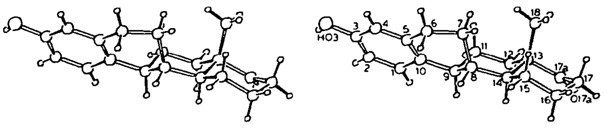

Fig. 1. *ORTEP*II (Johnson, 1976) stereoview of the molecule with atomic numbering.

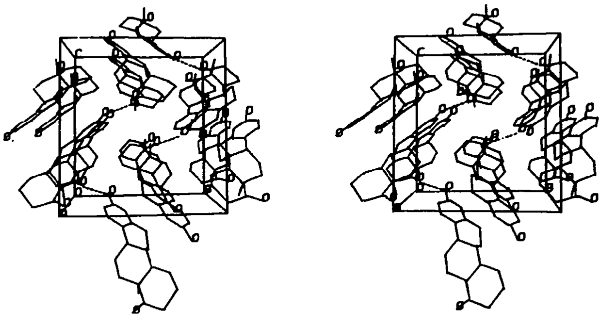

Fig. 2. Stereo packing of the molecule down the *a* axis, showing the hydrogen bonding

Related literature. This structure is one of a series of *D*-homo structures published by Duax, Smith, Swenson, Strong, Weeks, Ananchenko & Egorova (1981) and Chekhlov, Ionov, Egorova & Ananchenko (1982). Biological activity data for the series is reported in Chernyaev, Barkova, Ananchenko, Sorokina, Matarodze & Rozen (1979).

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* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51825 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.