Table 2.* A stereoscopic view of the molecule showing the atomic numbering and the molecular conformation is given in Fig. 1. Fig. 2 shows a stereoview of the molecular packing.

Related literature. Related structures previously reported by Duax, Griffin, Strong & Wood (1989) and Segaloff, Gabbard, Flores, Borne, Baker, Duax, Strong & Rohrer (1980).

A sample of the title compound was provided by the late Dr A. Segaloff of the Alton Ochsner Medical Foundation, New Orleans, LA. This work was supported by the National Institute of Diabetes and Digestive and Kidney Diseases, grant No. DK265 46-22.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53672 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 9β-Estrone

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Abstract. 3-Hydroxy-9β-estra-1,3,5(10)-trien-17-one, $C_{18}H_{22}O_2$, $M_r = 270{\cdot}4$, monoclinic, $P2_1$, $a = 9{\cdot}527\ (2)$, $b = 11{\cdot}182\ (3)$, $c = 7{\cdot}078\ (1)$ Å, $\beta = 108{\cdot}45\ (1)°$, $V = 715{\cdot}3\ (3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}255$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}746$ cm^{-1}, $F(000) = 292$, $T = 298$ K, $R = 0{\cdot}037$ for 2128 reflections with $F > 2{\cdot}0\sigma(F)$. The compound was one in a series of 9β-estrone analogues synthesized to study their estrogenic activity. The *B* ring conformation is a 7β,8α-half chair, due to the configuration at C9. The O3 hydroxy forms a hydrogen bond to O17 at a distance of 2·76 Å.

Experimental. Crystallization from methanol, a thick-plate single crystal, 0·38 × 0·40 × 0·70 mm. The unit-cell parameters were refined from least-squares analysis of 2θ values for 25 reflections from $33 < 2\theta < 38°$. Intensities for 2777 reflections (2195 unique) having $2\theta < 50°$, $-12 < h < 12$, $-1 < k < 14$, $0 < l < 9$, measured on a Syntex *P*3 diffractometer, using a θ–2θ scan mode, Nb-filtered Mo radiation, no monochromator, variable scan speed from 3 to 30° min^{-1} in 2θ, scan width $(1{\cdot}25 + 1{\cdot}25\tan\theta)°$. Four standard reflections ($11\bar{6}$, $17\bar{4}$, $81\bar{1}$, $35\bar{5}$) were measured every 100 reflections and varied in intensity by <1% during data collection.

Direct methods using *MULTAN*78 (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 2128 reflections for which *F* >

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

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
C1	3236 (2)	209 (2)	8018 (2)	4 (1)
C2	4245 (2)	903 (2)	9417 (3)	4 (1)
C3	4572 (2)	631 (2)	11428 (3)	4 (1)
C4	3896 (2)	−350 (2)	11976 (2)	4 (1)
C5	2893 (2)	−1061 (2)	10566 (2)	3 (1)
C6	2250 (2)	−2139 (2)	11284 (3)	4 (1)
C7	1536 (2)	−3021 (2)	9635 (3)	4 (1)
C8	531 (2)	−2385	7787 (2)	4 (1)
C9	1460 (2)	−1542 (2)	6917 (2)	4 (1)
C10	2522 (1)	−776 (2)	8527 (2)	3 (1)
C11	451 (2)	−818 (2)	5136 (2)	4 (1)
C12	−836 (2)	−164 (2)	5555 (2)	4 (1)
C13	−1725 (2)	−1055 (2)	6345 (2)	3 (1)
C14	−685 (2)	−1656 (2)	8223 (2)	3 (1)
C15	−1736 (2)	−2254 (2)	9193 (3)	4 (1)
C16	−2964 (2)	−1311 (2)	8902 (3)	4 (1)
C17	−2905 (2)	−555 (2)	7142 (2)	3 (1)
C18	−2578 (2)	−1948 (2)	4714 (3)	5 (1)
O3	5555 (2)	1350 (2)	12771 (3)	6 (1)
O17	−3700 (1)	296 (2)	6482 (2)	4 (1)

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

C1—C2	1·380 (3)	C9—C10	1·525 (2)
C1—C10	1·400 (3)	C9—C11	1·550 (2)
C2—C3	1·391 (3)	C11—C12	1·535 (3)
C3—C4	1·388 (3)	C12—C13	1·525 (3)
C3—O3	1·365 (3)	C13—C14	1·539 (2)
C4—C5	1·392 (2)	C13—C17	1·515 (2)
C5—C6	1·511 (3)	C13—C18	1·547 (3)
C5—C10	1·410 (2)	C14—C15	1·535 (3)
C6—C7	1·515 (3)	C15—C16	1·540 (3)
C7—C8	1·530 (2)	C16—C17	1·521 (3)
C8—C9	1·548 (3)	C17—O17	1·215 (3)
C8—C14	1·526 (2)		

C2—C1—C10	122·9 (2)	C1—C10—C9	120·7 (1)
C1—C2—C3	119·4 (2)	C5—C10—C9	122·0 (1)
C2—C3—C4	119·1 (1)	C9—C11—C12	114·8 (1)
C2—C3—O3	117·8 (1)	C11—C12—C13	109·3 (1)
C4—C3—O3	123·2 (1)	C12—C13—C14	108·8 (1)
C3—C4—C5	121·6 (1)	C12—C13—C17	117·3 (1)
C4—C5—C6	118·3 (1)	C12—C13—C18	111·7 (1)
C4—C5—C10	119·9 (1)	C14—C13—C17	100·9 (1)
C6—C5—C10	121·9 (1)	C14—C13—C18	113·9 (1)
C5—C6—C7	113·0 (2)	C17—C13—C18	103·9 (1)
C6—C7—C8	111·3 (1)	C8—C14—C13	112·4 (1)
C7—C8—C9	110·0 (1)	C8—C14—C15	120·2 (1)
C7—C8—C14	112·7 (1)	C13—C14—C15	104·1 (1)
C9—C8—C14	108·8 (1)	C14—C15—C16	102·6 (1)
C8—C9—C10	111·9 (1)	C15—C16—C17	105·5 (1)
C8—C9—C11	110·9 (1)	C13—C17—C16	108·9 (1)
C10—C9—C11	114·1 (1)	C13—C17—O17	126·3 (1)
C1—C10—C5	117·2 (1)	C16—C17—O17	124·8 (1)

Ring *A*		Ring *B*	
C10—C1—C2—C3	−0·5 (3)	C10—C5—C6—C7	15·3 (3)
C1—C2—C3—C4	1·3 (3)	C5—C6—C7—C8	−46·6 (2)
C2—C3—C4—C5	−0·5 (3)	C6—C7—C8—C9	63·8 (2)
C3—C4—C5—C10	−1·3 (3)	C7—C8—C9—C10	−47·3 (2)
C4—C5—C10—C1	2·1 (3)	C8—C9—C10—C5	17·0 (2)
C5—C10—C1—C2	−1·2 (3)	C9—C10—C5—C6	−0·5 (3)
Ring *C*		Ring *D*	
C14—C8—C9—C11	−52·0 (2)	C17—C13—C14—C15	41·1 (2)
C8—C9—C11—C12	51·5 (2)	C13—C14—C15—C16	−40·3 (2)
C9—C11—C12—C13	−53·9 (2)	C14—C15—C16—C17	23·2 (2)
C11—C12—C13—C14	57·3 (2)	C15—C16—C17—C13	2·2 (2)
C12—C13—C14—C8	−63·3 (2)	C16—C17—C13—C14	−26·5 (2)
C13—C14—C8—C9	59·9 (2)		

$2\sigma(F)$. The hydrogen positions were located in a difference map and refined with initially assigned isotropic temperature parameters of the parent atom. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0{\cdot}037$, $wR = 0{\cdot}046$, $S = 2{\cdot}19$ for observed reflections and $R = 0{\cdot}038$ for all data, $R_{\rm int} = 0{\cdot}023$, $w = 1/\sigma^2$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}32$. Weighting scheme based on estimates of experimental errors from counting statistics. Final difference map showed maximum positive and negative peaks of $\Delta\rho = +0{\cdot}24$ and $-0{\cdot}19$ 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. Fig. 2 shows a stereoview of the molecular packing.

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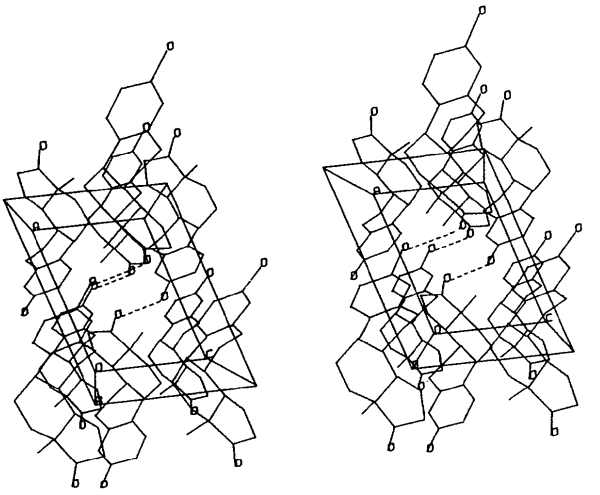

Fig. 1. *ORTEP*II (Johnson, 1976) stereoview of the molecule with atomic numbering.

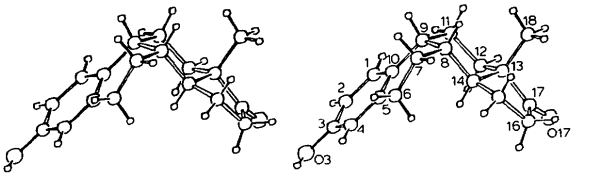

Fig. 2. Stereo packing of the molecule down the *b* axis, showing the hydrogen bonding.

Related literature. This structure is one of a series of 9β-estrone structures published by Segaloff, Gabbard, Flores, Borne, Baker, Duax, Strong & Rohrer (1980) and Duax, Griffin, Strong & Wood (1989).

A sample of the title compound was provided by the late Dr A. Segaloff of the Alton Ochsner Medical Foundation, New Orleans, LA. This work was supported by the National Institute of Diabetes and Digestive and Kidney Diseases, grant No. DK265 46-22.

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