Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Re(1)	1664 (1)	9153 (1)	3547 (1)	28 (1)
Re(2)	1734 (1)	9819 (1)	1571 (1)	33 (1)
I(1)	3441 (1)	9053 (1)	2611 (1)	45 (1)
P(1)	2885 (4)	8361 (2)	4424 (2)	30 (1)
P(2)	619 (4)	9991 (2)	2611 (2)	30 (1)
C(1)	2753 (19)	9986 (11)	3967 (9)	52 (7)
O(1)	3387 (17)	10449 (9)	4209 (8)	93 (7)
C(2)	814 (16)	8278 (11)	3081 (8)	43 (7)
O(2)	384 (16)	7754 (8)	2819 (7)	80 (6)
C(4)	2910 (19)	10681 (10)	1732 (9)	42 (7)
O(4)	3644 (15)	11149 (8)	1800 (6)	73 (6)
C(3)	361 (20)	9299 (9)	4068 (9)	47 (7)
O(3)	−470 (12)	9395 (7)	4381 (7)	63 (6)
C(5)	529 (17)	10376 (10)	915 (9)	44 (7)
O(5)	−174 (12)	10694 (8)	506 (6)	62 (5)
C(6)	625 (17)	8936 (9)	1405 (8)	37 (6)
O(6)	−17 (15)	8443 (8)	1280 (7)	76 (6)
C(7)	2601 (16)	9490 (10)	824 (9)	42 (6)
O(7)	3133 (14)	9324 (9)	395 (8)	81 (7)
C(11)	3333 (11)	8714 (5)	5319 (4)	29 (5)
C(12)	2881	9388	5501	47 (7)
C(13)	3213	9639	6187	52 (7)
C(14)	3996	9217	6691	54 (7)
C(15)	4448	8544	6509	70 (9)
C(16)	4117	8292	5823	53 (7)
C(21)	4421 (10)	8077 (6)	4198 (6)	37 (6)
C(22)	4408	7551	3679	46 (7)
C(23)	5550	7369	3452	56 (8)
C(24)	6705	7712	3745	74 (10)
C(25)	6719	8237	4263	92 (12)
C(26)	5577	8420	4490	58 (8)
C(31)	2100 (10)	7495 (5)	4601 (5)	34 (6)
C(32)	2833	6881	4836	43 (6)
C(33)	2226	6254	5020	46 (7)
C(34)	887	6241	4968	57 (8)
C(35)	154	6855	4732	56 (8)
C(36)	761	7481	4549	41 (6)
C(41)	−1138 (7)	9841 (6)	2291 (5)	36 (6)
C(42)	−1784	9233	2483	54 (7)
C(43)	−3078	9116	2186	52 (7)
C(44)	−3726	9607	1698	58 (8)
C(45)	−3080	10216	1506	75 (9)
C(46)	−1786	10333	1803	41 (6)
C(51)	595 (12)	10931 (5)	2970 (6)	43 (6)
C(52)	1688	11369	3013	51 (7)
C(53)	1731	12046	3341	72 (9)
C(54)	680	12285	3624	85 (11)
C(55)	−414	11846	3581	92 (12)
C(56)	−457	11169	3254	67 (9)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Re(1)—I(1)	2·846 (1)	Re(1)—P(1)	2·425 (4)
Re(1)—P(2)	2·482 (4)	Re(2)—I(1)	2·823 (1)
Re(2)—P(2)	2·538 (4)		
I(1)—Re(1)—P(1)	94·9 (1)	I(1)—Re(1)—P(2)	80·0 (1)
P(1)—Re(1)—P(2)	174·3 (1)	I(1)—Re(2)—P(2)	79·5 (1)
Re(1)—I(1)—Re(2)	91·1 (1)	Re(1)—P(2)—Re(2)	107·5 (2)

selected bond lengths and angles in Table 2.* Fig. 1 shows the molecular structure.

Related literature. The structural parameters can be compared with those of $[Re_2(CO)_8(\mu\text{-}PPh_2)_2]$ (Flörke, Woyciechowski & Haupt, 1988), $[Re_2(CO)_8(\mu\text{-}H)(\mu\text{-}PPh_2)]$ (Haupt, Balsaa & Flörke, 1987) and $[Re_2(CO)_7(\mu\text{-}H)(\mu\text{-}PPh_2)(PPh_3)]$ (Haupt, Balsaa & Flörke, 1988). The related compounds $[Mn_2(CO)_8(\mu\text{-}I)(\mu\text{-}PPh_2)]$ and $[Re_2(CO)_8(\mu\text{-}Br)(\mu\text{-}PPh_2)]$ have been reported without structural data (Manning, Peterson, Wada & Dhami, 1986).

* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53742 (24 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 3-Hydroxy-17-oxoestra-1,3,5(10)-trien-11β-yl Acetate

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Abstract. $C_{20}H_{24}O_4$, $M_r = 328{\cdot}41$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}688\ (3)$, $b = 15{\cdot}377\ (5)$, $c = 9{\cdot}466\ (2)$ Å, $V = 1701\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}282$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}823$ cm^{-1}, $F(000) = 704$, $T = 298$ K, $R = 0{\cdot}063$ for 1921 reflections with $F > 2\sigma(F)$. The structure was determined to observe the effect of the 11β-acetate substituent on the conformation of the molecule. The 3-hydroxy is hydrogen bonded to O19 at 2·86 Å. The *B* ring has a 7α,8β-half chair conformation, the most commonly observed *B* ring conformation in estrogen analogues.

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Table 1. *Fractional positional parameters* ($\times 10^4$) *and equivalent isotropic atomic displacement parameters* ($Å^2 \times 10^2$) *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	4538 (3)	−506 (2)	4702 (4)	4 (1)
C2	5051 (3)	−981 (3)	3646 (4)	5 (1)
C3	5008 (3)	−672 (3)	2283 (4)	4 (1)
C4	4460 (3)	92 (2)	2001 (4)	4 (1)
C5	3938 (3)	584 (2)	3057 (4)	4 (1)
C6	3349 (5)	1420 (3)	2659 (4)	5 (1)
C7	2587 (4)	1787 (3)	3811 (5)	5 (1)
C8	3196 (3)	1748 (2)	5228 (4)	4 (1)
C9	3379 (3)	783 (2)	5626 (4)	4 (1)
C10	3983 (3)	285 (2)	4456 (3)	3 (1)
C11	3881 (3)	643 (2)	7095 (4)	4 (1)
C12	3330 (4)	1190 (3)	8252 (4)	4 (1)
C13	3173 (3)	2137 (2)	7826 (4)	4 (1)
C14	2538 (3)	2183 (2)	6411 (4)	4 (1)
C15	2138 (5)	3129 (3)	6324 (6)	6 (1)
C16	1762 (6)	3313 (4)	7846 (7)	7 (2)
C17	2391 (4)	2669 (3)	8753 (5)	6 (1)
C18	4329 (4)	2642 (3)	7801 (6)	6 (1)
C19	5781 (3)	379 (2)	7903 (4)	4 (1)
C20	6997 (4)	663 (4)	7783 (7)	7 (1)
O3	5507 (3)	−1169 (2)	1234 (3)	6 (1)
O11	5110 (2)	824 (1)	7018 (3)	4 (1)
O17	2267 (4)	2594 (2)	10017 (4)	9 (1)
O19	5432 (3)	−175 (2)	8687 (3)	6 (1)

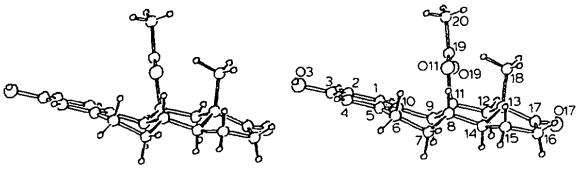

Fig. 1. *ORTEP*II (Johnson, 1976) stereoview of the molecule with atomic numbering.

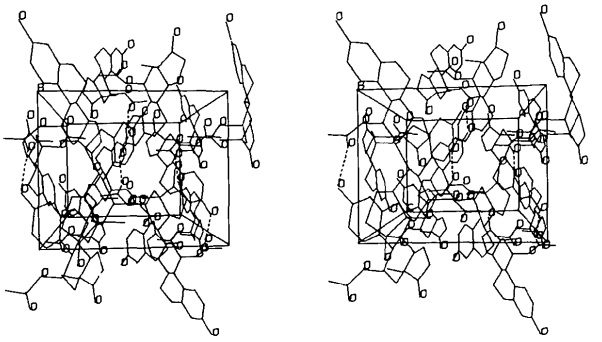

Fig. 2. Stereo packing of the molecule down the *b* axis, showing the hydrogen bonding.

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

C1 C2	1·375 (5)	C11—C12	1·524 (5)
C1—C10	1·399 (5)	C11—O11	1·466 (4)
C2—C3	1·376 (5)	C12—C13	1·522 (5)
C3—C4	1·363 (5)	C13—C14	1·533 (6)
C3—O3	1·382 (5)	C13—C17	1·508 (6)
C4—C5	1·395 (5)	C13—C18	1·559 (6)
C5—C6	1·506 (6)	C14—C15	1·530 (6)
C5—C10	1·403 (5)	C15—C16	1·533 (8)
C6—C7	1·517 (7)	C16—C17	1·502 (8)
C7—C8	1·520 (6)	C17—O17	1·211 (6)
C8—C9	1·545 (5)	C19—C20	1·491 (6)
C8—C14	1·514 (5)	C19—O11	1·336 (4)
C9—C10	1·520 (5)	C19—O19	1·201 (5)
C9—C11	1·524 (5)		

C2—C1—C10	122·9 (3)	C9—C11—O11	107·8 (2)
C1—C2—C3	118·8 (3)	C12—C11—O11	110·2 (3)
C2—C3—C4	119·9 (3)	C11—C12—C13	112·9 (3)
C2—C3—O3	117·8 (3)	C12—C13—C14	109·5 (3)
C4—C3—O3	122·3 (3)	C12—C13—C17	116·0 (3)
C3—C4—C5	122·2 (3)	C12—C13—C18	112·1 (3)
C4—C5—C6	118·9 (3)	C14—C13—C17	100·9 (3)
C4—C5—C10	118·9 (3)	C14—C13—C18	112·5 (3)
C6—C5—C10	122·2 (3)	C17—C13—C18	105·3 (3)
C5—C6—C7	113·9 (3)	C8—C14—C13	112·3 (3)
C6—C7—C8	110·2 (3)	C8—C14—C15	122·4 (3)
C7—C8—C9	108·5 (3)	C13—C14—C15	103·8 (3)
C7—C8—C14	113·4 (3)	C14—C15—C16	102·2 (4)
C9—C8—C14	108·4 (3)	C15—C16—C17	106·0 (4)
C8—C9—C10	111·7 (3)	C13—C17—C16	108·8 (4)
C8—C9—C11	114·3 (3)	C13—C17—O17	126·5 (3)
C10—C9—C11	114·5 (3)	C16—C17—O17	124·7 (4)
C1—C10—C5	117·3 (3)	C20—C19—O11	111·2 (3)
C1—C10—C9	122·2 (3)	C20—C19—O19	125·4 (3)
C5—C10—C9	120·4 (3)	O11—C19—O19	123·5 (3)
C9—C11—C12	114·5 (3)	C11—O11—C19	116·6 (2)

Ring *A*		Ring *B*	
C10—C1—C2—C3	−0·7 (6)	C10—C5—C6—C7	−13·7 (6)
C1—C2—C3—C4	−0·2 (6)	C5—C6—C7—C8	44·8 (5)
C2—C3—C4—C5	0·5 (6)	C6—C7—C8—C9	−65·3 (4)
C3—C4—C5—C10	0·2 (6)	C7—C8—C9—C10	53·5 (4)
C4—C5—C10—C1	−1·1 (5)	C8—C9—C10—C5	−23·1 (4)
C5—C10—C1—C2	1·4 (5)	C9—C10—C5—C6	2·9 (5)

Ring *C*		Ring *D*	
C14—C8—C9—C11	−50·9 (4)	C17—C13—C14—C15	41·4 (4)
C8—C9—C11—C12	45·2 (4)	C13—C14—C15—C16	−40·2 (4)
C9—C11—C12—C13	−45·8 (4)	C14—C15—C16—C17	23·0 (5)
C11—C12—C13—C14	52·6 (4)	C15—C16—C17—C13	2·7 (5)
C12—C13—C14—C8	−61·5 (4)	C16—C17—C13—C14	−27·1 (5)
C13—C14—C8—C9	59·6 (4)		

Experimental. Thick-plate-shaped crystals, 0·2 × 0·4 × 0·8 mm, Nicolet *P*3 diffractometer. ω-scan method, lattice parameters from the 2θ values of 25 reflections with $20 < 2\theta < 30°$, intensity measurements performed up to $2\theta = 60°$; range of h, k, l 0 to 17, 0 to 22, −1 to 14; variable scan speed from 3 to 30° min^{-1} (2θ). Four standard reflections (125, 081, 710, 353) varied in intensity by 3% throughout the experiment. 3287 reflections measured, 2828 unique reflections and 1921 with $F > 2\sigma(F)$ used in refinement.

Structure solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). H atoms found from difference map and refined with isotropic displacement parameters; final $R = 6·3\%$, $wR = 6·0\%$, $S = 2·12$, $(\Delta/\sigma)_{max} = 0·20$. w was calculated using a weighting scheme based on estimates of experimental errors from counting statistics. $(\Delta\rho)_{max} = 0·27$, $(\Delta\rho)_{min} = -0·26$ e $Å^{-3}$ from final difference Fourier synthesis. No corrections for absorption or extinction were made. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

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.

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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