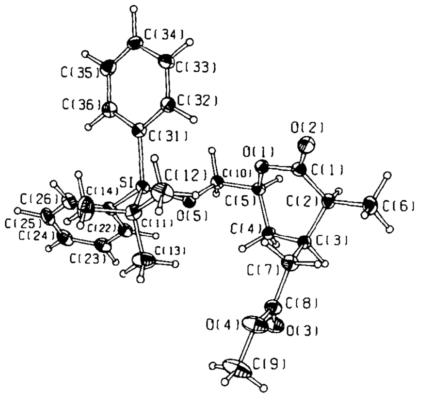

Fig. 1. An *ORTEP* diagram of the title compound with the H atoms and the atomic numbering. Ellipsoids are represented at the 30% probability level.

Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.* Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

Related literature. The intramolecular cyclization of the chloropropionate ester shown above is a rare example of δ-lactone synthesis based on a radical process; see Surzur & Bertrand (1988), Curran (1988), Giese (1986) and Hanessian, Bennani & Di Fabio (1990).

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, distances and angles related to the *tert*-butyldiphenylsilyl substituent, distances to the weighted least-squares planes for the phenyl groups and a stereoview of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52340 (18 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 6-Methylene-4-pregnene-3,20-dione

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Abstract. $C_{22}H_{30}O_2$ (1), $M_r = 326{\cdot}47$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}662$ (3), $b = 23{\cdot}32$ (7), $c = 8{\cdot}167$ (2) Å, $V = 1840{\cdot}3$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}178$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5{\cdot}34$ cm^{-1}, $F(000) = 712$, $T = 293$ K, $R = 0{\cdot}035$, $wR = 0{\cdot}044$, $S = 1{\cdot}954$ for 2147 unique observed reflections with $F_o > 2\sigma(F_o)$. Rings *B* and *C* have chair conformations, the *A* ring assumes an intermediate sofa–half-chair conformation, and the *D* ring is in the half-chair conformation. The progesterone side chain has the same con-

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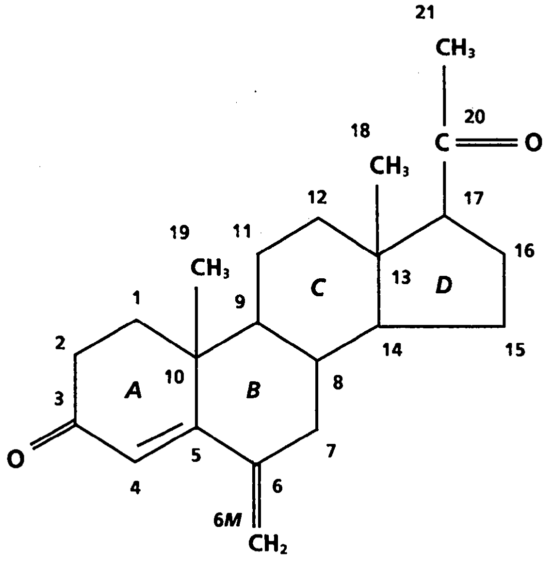
formation as observed in 81 steroids; the C16—C17—C20—O20 torsion angle is −18·9 (3)°; the C4—C5—C6—C6*M* torsion angle is −48·6°.

Experimental. A platelet crystal of (1) with dimensions 0·08 × 0·40 × 0·60 mm. CAD-4 diffractometer, cell dimensions and Laue symmetry from 25 centered reflections ($41 < 2\theta < 60°$) checked with oscillation photographs, Cu $K\alpha$ radiation, Ni-filtered, scan width $(0·8 + 0·2\tan\theta)°$, scan speed from 3 to 30° min^{-1} in 2θ, $\theta_{\max} = 77°$, $0 < h < 9$, $0 < k < 29$, $0 < l < 10$, 2203 reflections measured using θ–2θ scan mode. Five standard reflections (1,13,1, 631, 214, 2,13,3, 353) were measured every 3 h and varied in intensity by < 5% during the data collection. Intensity corrections made with the *DREAM* program (Blessing, 1987). 4064 reflections collected, 2203 unique (R_{int} = 0·018).



(1)

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 method on F values using 2147 reflections for which $F_o > 2\sigma(F_o)$. The H-atom positions were located in a difference Fourier map and refined with isotropic displacement parameters. Atomic scattering factors were taken from the *International Tables for X-ray Crystallography* (1974). Final R = 0·035, wR = 0·044, S = 1·954 for observed reflections, $w = 1/\sigma^2$, $(\Delta/\sigma)_{\max}$ = 0·23. Weighting scheme based on estimates of experimental errors from counting statistics. Final difference map showed maximum negative and positive peaks of −0·19 and 0·13 e Å^{-3}. No corrections for absorption or extinction were made.

Table 1. *Positional parameters* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3) *for non*-H *atoms*

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

	x	y	z	U_{eq}
C1	3422 (3)	6046 (1)	1671 (3)	56 (2)
C2	3439 (3)	5402 (1)	2057 (3)	62 (2)
C3	3896 (2)	5293 (1)	3774 (3)	57 (1)
C4	3531 (2)	5728 (1)	4991 (3)	48 (1)
C5	2857 (2)	6214 (1)	4619 (3)	42 (1)
C6	2359 (2)	6600 (1)	5939 (3)	44 (1)
C7	2637 (2)	7229 (1)	5698 (3)	49 (1)
C8	2215 (2)	7429 (1)	3986 (2)	40 (1)
C9	2884 (2)	7045 (1)	2671 (3)	40 (1)
C10	2526 (2)	6395 (1)	2873 (3)	42 (1)
C11	2592 (2)	7270 (1)	934 (3)	48 (1)
C12	2944 (2)	7907 (1)	703 (3)	50 (1)
C13	2220 (2)	8277 (1)	1985 (3)	41 (1)
C14	2643 (2)	8051 (1)	3686 (3)	43 (1)
C15	2162 (3)	8514 (1)	4870 (3)	57 (1)
C16	2414 (3)	9079 (1)	3938 (3)	63 (1)
C17	2698 (2)	8917 (1)	2127 (3)	50 (1)
C18	643 (2)	8262 (1)	1726 (3)	51 (1)
C19	975 (2)	6276 (1)	2572 (3)	58 (1)
C20	2004 (2)	9316 (1)	900 (3)	57 (1)
C21	2543 (3)	9313 (1)	−817 (3)	70 (2)
C6*M*	1653 (2)	6394 (1)	7205 (3)	58 (1)
O3	4476 (2)	4850 (1)	4168 (3)	85 (2)
O20	1046 (2)	9622 (1)	1281 (3)	82 (1)

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

C1—C2	1·534 (3)	C9—C11	1·537 (3)
C1—C10	1·541 (4)	C10—C19	1·544 (3)
C2—C3	1·492 (4)	C11—C12	1·538 (3)
C3—C4	1·466 (3)	C12—C13	1·526 (3)
C3—O3	1·217 (3)	C13—C14	1·543 (4)
C4—C5	1·345 (3)	C13—C17	1·567 (3)
C5—C6	1·482 (3)	C13—C18	1·538 (3)
C5—C10	1·519 (4)	C14—C15	1·526 (3)
C6—C7	1·506 (3)	C15—C16	1·540 (3)
C6—C6*M*	1·329 (3)	C16—C17	1·550 (4)
C7—C8	1·529 (3)	C17—C20	1·524 (3)
C8—C9	1·540 (3)	C20—C21	1·496 (4)
C8—C14	1·526 (3)	C20—O20	1·211 (3)
C9—C10	1·564 (3)		

C2—C1—C10	113·1 (2)	C5—C10—C9	108·7 (2)
C1—C2—C3	111·4 (2)	C5—C10—C19	107·7 (2)
C2—C3—C4	116·6 (2)	C9—C10—C19	111·8 (2)
C2—C3—O3	122·1 (2)	C9—C11—C12	113·6 (2)
C4—C3—O3	121·2 (2)	C11—C12—C13	111·2 (2)
C3—C4—C5	123·3 (2)	C12—C13—C14	107·6 (2)
C4—C5—C6	120·3 (2)	C12—C13—C17	117·0 (2)
C4—C5—C10	123·2 (2)	C12—C13—C18	110·4 (2)
C6—C5—C10	116·5 (2)	C14—C13—C17	100·4 (2)
C5—C6—C7	116·0 (2)	C14—C13—C18	112·2 (2)
C5—C6—C6*M*	120·8 (2)	C17—C13—C18	108·9 (2)
C7—C6—C6*M*	123·2 (2)	C8—C14—C13	113·4 (2)
C6—C7—C8	111·6 (2)	C8—C14—C15	119·4 (2)
C7—C8—C9	110·5 (2)	C13—C14—C15	104·3 (2)
C7—C8—C14	111·2 (2)	C14—C15—C16	104·2 (2)
C9—C8—C14	109·1 (2)	C15—C16—C17	107·0 (2)
C8—C9—C10	113·4 (2)	C13—C17—C16	104·6 (2)
C8—C9—C11	111·6 (2)	C13—C17—C20	113·7 (2)
C10—C9—C11	112·8 (2)	C16—C17—C20	113·6 (2)
C1—C10—C5	109·5 (2)	C17—C20—C21	117·5 (2)
C1—C10—C9	108·7 (2)	C17—C20—O20	121·8 (2)
C1—C10—C19	110·4 (2)	C21—C20—O20	120·7 (2)

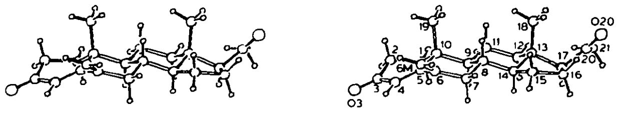


Fig. 1. Stereoview of the molecule with the atomic numbering scheme.

Atomic positional and equivalent displacement parameters for non-H atoms are listed in Table 1. Bond lengths and valency angles are listed in Table 2.* A stereoscopic view (Johnson, 1965) of the molecule showing the atomic numbering and molecular conformation is given in Fig. 1.

Related literature. This compound has been shown to be a good irreversible inhibitor of the steroidal enzyme 5*α*-reductase (Petrow, Wang, Lack & Sandberg, 1981). The overall conformation of the molecule is very similar to the conformation of 17*α*-acetoxy-6-methylene-4-pregnene-3,20-dione (Petrow, Padilla, McPhail, Bruchovsky & Schneider, 1989), another potent 5*α*-reductase inhibitor and to progesterone itself (Campsteyn, Dupont & Dideberg, 1972).

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Structure of an Anhistaminic Drug, Racemic Chlorpheniramine Hydrogen Maleate

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Abstract. γ-(4-Chlorophenyl)-*N*,*N*-dimethyl-2-pyridinepropanaminium hydrogen maleate, $C_{16}H_{20}ClN_2^+.C_4H_3O_4^-$, M_r = 390·87, monoclinic, $P2_1/n$, a = 9·769 (2), b = 10·757 (3), c = 19·139 (5) Å, β = 90·97 (2)°, V = 2010·9 (15) Å^3, Z = 4, D_x = 1·291 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·213 mm^{-1}, $F(000)$ = 824, T = 293 (1) K, R = 0·050 for 2276 observed data with $I > 3\sigma(I)$. The pyridine and the phenyl rings are inclined at an angle of 105·0° and the side chain comprising C(6), C(7), C(8), C(9) and N(2) atoms is fully extended. The maleate anion is hydrogen bonded to the dimethylamino group [O(4)···H(N2) 1·86 (2) Å, O(4)···H(N2)—N(2) 171 (2)°]. The maleate anion also shows a strong intramolecular hydrogen bond [O···H(O) 1·47 (2) Å, O—H(O)···O 174 (2)°].

Experimental. Thin plates of chlorpheniramine hydrogen maleate (Sigma, Inc.) were grown from ethanol solution by slow evaporation at room temperature. A crystal of approximate size 0·47 × 0·52 × 0·20 mm was cut from a large plate for data collection. Accurate cell constants and an orientation matrix were determined by a least-squares refinement of the setting angles of 25 reflections with θ in the range 10–15°. Intensity data in the range $2 < \theta < 25°$ (h 0→11, k 0→12, l −22→22) were collected on an Enraf–Nonius CAD-4 diffractometer using the $\omega/2\theta$ scan method, variable scan speed (1·25–2·35° min^{-1}) and monochromatized Mo $K\alpha$ radiation. The intensities of three standard reflections measured every 2 h of exposure time decayed linearly 3·9% over the period of data collection; this decay was corrected

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