Related structures: 5,7,4′-trimethoxyflavone (Mariezcurrena, 1978), 3-hydroxyflavone (Etter, Urbanczyk-Lipkowska, Baer & Barbara, 1986), 5,4′-dihydroxy-3,6,7,8-tetramethoxyflavone (Vijayalakshmi, Rajan, Srinivasan & Ramachandran, 1986) and 5-hydroxy-4,7′-dimethoxyflavanone (Miles, Main & Nicholson, 1989).

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Structure of *rac*-3*β*-Hydroxy-18-nor-5*α*-pregn-13(17)-en-20-one

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Abstract. $C_{20}H_{30}O_2$, $M_r = 302{\cdot}46$, orthorhombic, *Fdd*2, $a = 50{\cdot}270\,(9)$, $b = 18{\cdot}018\,(3)$, $c = 7{\cdot}573\,(1)$ Å, $V = 6859{\cdot}6$ Å^3, $Z = 16$, $D_x = 1{\cdot}172$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 5{\cdot}339$ cm^{-1}, $F(000) = 2656$, $T = 298$ K, $R = 0{\cdot}042$ for 1838 unique observed reflections with $I > 2\sigma(I)$. The title compound, a racemic mixture, was synthesized from achiral starting materials. The C20 carbonyl is observed in an unusual conformation, *trans* to the C16—C17 bond. O3*B* and O20 form an intermolecular hydrogen bond: O···O = 2·86 Å, O—H···O = 177·9°, H···O = 2·04 Å.

Experimental. A thin rectangular plate was grown from ethanol. Crystal size 0·80 × 0·30 × 0·08 mm. Enraf–Nonius CAD-4 diffractometer, cell dimensions and Laue symmetry from 25 centered reflections ($50 < 2\theta < 70°$) checked with oscillation photographs, Cu $K\alpha$ radiation, Ni-filtered, no monochromator, scan width variable, scan speed from 0·625 to 4° min^{-1} in 2θ, scan width $(0{\cdot}80 + 0{\cdot}20 \times \tan\theta)$, $2\theta_{\max} = 120°$, $-2 \leq h \leq 56$, $0 \leq k \leq 20$, $0 \leq l \leq 8$, 1900 independent reflections measured using a θ–2θ scan mode, 1838 with $I > 2\sigma(I)$. Four standard reflections (1,11,1, 35,1,1, 19,5,3, 715) were measured every 250 reflections and varied in intensity by ≤ 5% during the data collection.

Direct method 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 1838 reflections for which $I > 2\sigma(I)$. The hydrogen positions were located in a difference map and refined with isotropic temperature parameters. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final $R = 0{\cdot}042$, $wR = 0{\cdot}055$, $S = 2{\cdot}227$ for observed reflections and $R = 0{\cdot}043$ for all data, $w = 1/\sigma^2$, $(\Delta/\sigma)_{\max} = 0{\cdot}27$. Weighting scheme based on estimates of experimental errors from counting statistics. Final difference map showed maximum positive and negative peaks of +0·10 and −0·04 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 view of of the molecule showing the atomic numbering and molecular conformation is given in Fig. 1. Fig. 2 shows a stereoview of the molecular packing. The enantiomer chosen for the

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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 52124 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional positional parameters* ($\times 10^4$) *and equivalent isotropic atomic displacement parameters* ($B_{eq} \times 10$ Å^2) *for non-*H *atoms with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*
C(1)	7940 (1)	11364 (2)	6289 (5)	51 (1)
C(2)	7646 (1)	11288 (2)	6775 (5)	56 (1)
C(3)	7468 (1)	11415 (1)	5189 (5)	51 (1)
C(4)	7547 (1)	10904 (2)	3701 (5)	52 (1)
C(5)	7839 (1)	11002 (1)	3194 (4)	44 (1)
C(6)	7919 (1)	10578 (2)	1545 (5)	62 (1)
C(7)	8199 (1)	10767 (2)	990 (5)	62 (1)
C(8)	8402 (1)	10662 (1)	2478	44 (1)
C(9)	8315 (1)	11055 (1)	4192 (4)	40 (1)
C(10)	8029 (1)	10850 (1)	4774 (4)	39 (1)
C(11)	8526 (1)	10927 (2)	5621 (4)	53 (1)
C(12)	8802 (1)	11210 (2)	5103 (5)	60 (1)
C(13)	8882 (1)	10917 (1)	3328 (4)	45 (1)
C(14)	8676 (1)	10940 (2)	1890 (4)	49 (1)
C(15)	8807 (1)	10490 (3)	399 (5)	69 (1)
C(16)	9103 (1)	10501 (3)	786 (5)	65 (1)
C(17)	9117 (1)	10670 (1)	2741 (4)	47 (1)
C(19)	8014 (1)	10037 (2)	5379 (6)	60 (1)
C(20)	9366 (1)	10606 (1)	3762 (5)	52 (1)
C(21)	9612 (1)	10408 (2)	2763 (6)	63 (1)
O(3*B*)	7198 (1)	11331 (1)	5733 (4)	64 (1)
O(20)	9372 (1)	10702 (2)	5339 (4)	78 (1)

$*B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

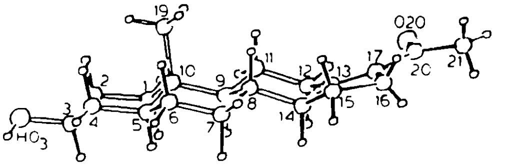

Fig. 1. *ORTEP*II (Johnson, 1976) view of the molecule with atomic numbering.

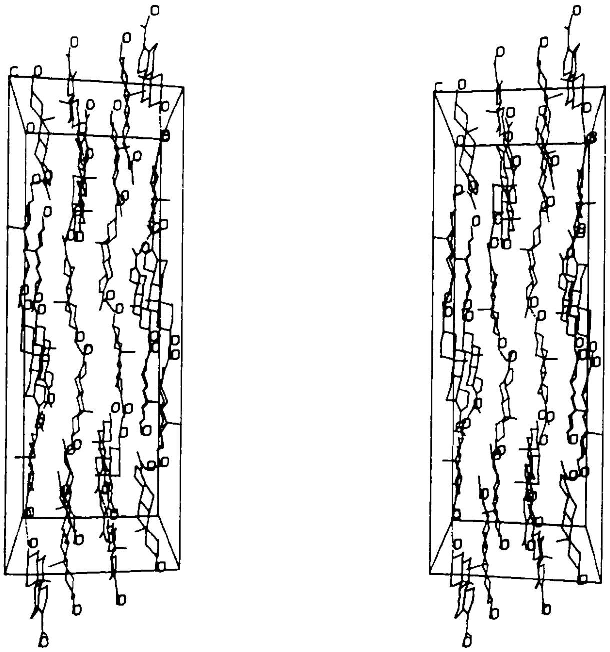

Fig. 2. Stereo packing of the molecule down the c axis, showing the hydrogen bonding.

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

C1—C2	1·530 (4)	C9—C11	1·533 (4)
C1—C10	1·541 (4)	C10—C19	1·536 (4)
C2—C3	1·513 (5)	C11—C12	1·527 (4)
C3—C4	1·508 (5)	C12—C13	1·500 (5)
C3—O3*B*	1·427 (3)	C13—C14	1·503 (4)
C4—C5	1·530 (3)	C13—C17	1·338 (3)
C5—C6	1·516 (5)	C14—C15	1·538 (5)
C5—C10	1·553 (4)	C15—C16	1·516 (4)
C6—C7	1·510 (4)	C16—C17	1·513 (5)
C7—C8	1·533 (4)	C17—C20	1·476 (4)
C8—C9	1·541 (3)	C20—C21	1·493 (4)
C8—C14	1·531 (3)	C20—O20	1·208 (5)
C9—C10	1·551 (3)		
C2—C1—C10	113·9 (4)	C5—C10—C9	108·0 (4)
C1—C2—C3	111·4 (4)	C5—C10—C19	111·6 (4)
C2—C3—C4	110·3 (4)	C9—C10—C19	111·0 (4)
C2—C3—O3*B*	108·4 (4)	C9—C11—C12	113·3 (4)
C4—C3—O3*B*	113·6 (4)	C11—C12—C13	111·0 (4)
C3—C4—C5	111·6 (4)	C12—C13—C14	117·0 (4)
C4—C5—C6	113·7 (4)	C12—C13—C17	130·7 (4)
C4—C5—C10	112·1 (4)	C14—C13—C17	112·1 (4)
C6—C5—C10	112·6 (4)	C8—C14—C13	113·6 (4)
C5—C6—C7	111·2 (4)	C8—C14—C15	115·2 (4)
C6—C7—C8	112·9 (4)	C13—C14—C15	102·9 (4)
C7—C8—C9	111·9 (4)	C14—C15—C16	105·7 (4)
C7—C8—C14	110·2 (4)	C15—C16—C17	103·8 (4)
C9—C8—C14	110·5 (4)	C13—C17—C16	110·5 (4)
C8—C9—C10	113·2 (4)	C13—C17—C20	126·9 (4)
C8—C9—C11	109·2 (4)	C16—C17—C20	122·5 (4)
C10—C9—C11	114·0 (4)	C17—C20—C21	117·1 (4)
C1—C10—C5	106·9 (4)	C17—C20—O20	121·9 (4)
C1—C10—C9	109·7 (4)	C21—C20—O20	120·9 (5)
C1—C10—C19	109·7 (4)		
C10—C1—C2—C3	−55·8 (3)	C8—C9—C10—C1	−170·0 (2)
C2—C1—C10—C5	54·4 (3)	C8—C9—C10—C5	−53·9 (2)
C2—C1—C10—C9	171·2 (2)	C8—C9—C10—C19	68·6 (3)
C2—C1—C10—C19	−66·7 (3)	C11—C9—C10—C1	64·3 (3)
C1—C2—C3—C4	54·7 (3)	C11—C9—C10—C5	−179·6 (2)
C1—C2—C3—O3*B*	179·6 (2)	C11—C9—C10—C19	−57·0 (3)
C2—C3—C4—C5	−56·8 (3)	C8—C9—C11—C12	58·6 (3)
O3*B*—C3—C4—C5	−178·7 (2)	C10—C9—C11—C12	−173·7 (2)
C3—C4—C5—C6	−172·1 (2)	C9—C11—C12—C13	−51·3 (3)
C3—C4—C5—C10	58·8 (3)	C11—C12—C13—C14	44·6 (3)
C4—C5—C6—C7	173·3 (3)	C11—C12—C13—C17	−139·9 (3)
C10—C5—C6—C7	−57·9 (3)	C12—C13—C14—C8	−45·7 (3)
C4—C5—C10—C1	−55·4 (3)	C12—C13—C14—C15	−170·9 (3)
C4—C5—C10—C9	−173·3 (2)	C17—C13—C14—C8	138·0 (2)
C4—C5—C10—C19	64·5 (3)	C17—C13—C14—C15	12·8 (3)
C6—C5—C10—C1	175·0 (2)	C12—C13—C17—C16	−174·7 (3)
C6—C5—C10—C9	57·1 (3)	C12—C13—C17—C20	1·6 (5)
C6—C5—C10—C19	−65·1 (3)	C14—C13—C17—C16	0·9 (3)
C5—C6—C7—C8	53·6 (3)	C14—C13—C17—C20	177·2 (3)
C6—C7—C8—C9	−50·7 (3)	C8—C14—C15—C16	−145·1 (3)
C6—C7—C8—C14	−174·0 (2)	C13—C14—C15—C16	−21·0 (3)
C7—C8—C9—C10	51·8 (3)	C14—C15—C16—C17	21·7 (4)
C7—C8—C9—C11	179·9 (2)	C15—C16—C17—C13	−14·5 (4)
C14—C8—C9—C10	175·0 (2)	C15—C16—C17—C20	169·1 (3)
C14—C8—C9—C11	−56·9 (3)	C13—C17—C20—C21	−172·3 (3)
C7—C8—C14—C13	175·1 (2)	C13—C17—C20—O20	8·4 (5)
C7—C8—C14—C15	−66·6 (3)	C16—C17—C20—C21	3·6 (4)
C9—C8—C14—C13	50·9 (3)	C16—C17—C20—O20	−175·8 (3)
C9—C8—C14—C15	169·2 (2)		

values in Table 2 and the view in Fig. 1 is similar to the naturally occurring steroids, for comparison.

Related literature. This structure is one of a series of steroid structures related to progesterone. The presence of the C13—C17 double bond and the absence of the C18 methyl group result in a previously unobserved conformation of the C20 carbonyl group. See Duax, Griffin & Rohrer (1981).

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Structure of 4-Nitrobenzaldehyde

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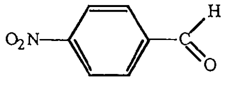
Abstract. $C_7H_5NO_3$, $M_r = 151{\cdot}1$, monoclinic, $P2_1$, $a = 6{\cdot}2076\,(3)$, $b = 5{\cdot}0968\,(4)$, $c = 10{\cdot}6723\,(7)$ Å, $\beta = 95{\cdot}222\,(5)°$, $V = 336{\cdot}3\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}492$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 9{\cdot}74$ cm^{-1}, $F(000) = 156$, $T = 297\,(1)$ K, $R = 0{\cdot}040$ for 1191 reflections with $I > 3\sigma(I)$ (1372 unique observations). The carbonyl O atom exhibits a slight disorder, having 10% occupancy in a site nearly coincident with the aldehydic H atom. The molecule shows only small deviations from overall planarity, with mean deviation 0·014 Å and largest individual deviations 0·032 (1) and −0·022 (2) Å for the O atoms of the nitro group. The N—O distances are 1·213 (2) and 1·227 (2) Å, and the C=O distance for the major conformer is 1·204 (2) Å.

Experimental. The compound (I) was prepared by reducing 4-nitrobenzoyl chloride with tributyltin deuteride (Lusztyk, Lusztyk, Maillard & Ingold, 1984; Four & Guibe, 1981). Crystals for analysis were grown in benzene/hexane (6/1). Deuterium substitution was desired on the aldehydic H atom for observation in low-temperature zero-field NMR spectroscopy. Torsion angles were needed for the NMR study, so the structure of (I) was determined.



(I)

Intensity data for (I) were obtained on an Enraf–Nonius CAD-4 diffractometer with a graphite incident-beam monochromator using Cu $K\alpha$ radiation. The ω–2θ scans were made at variable rate, 0·66–3·30° min^{-1}. Absorption corrections were based on ψ scans. Details of data collection and structural refinement are given in Table 1. The structure was solved by direct methods using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The structure was refined by full-matrix least squares with Enraf–Nonius *SDP* (Frenz, 1985), where the function minimized was $\sum w(|F_o| - |F_c|)^2$ and the weight, w, was defined as $4F_o^2\mathrm{Lp}^2/[S^2(C + R^2B) + (0{\cdot}020F_o^2)^2]$, S = scan rate, C = integrated count, R = scan time/background time, and B = background count. Atomic scattering factors, including those for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974). Except as described below, the C, N and O atoms were refined anisotropically. H atoms were located by difference maps and refined isotropically. The aldehydic H atom initially refined to a position 1·2 Å from C7, with a negative isotropic thermal parameter. This observation was interpreted as a slight disorder (~10%) of the carbonyl O3 atom. Atom O3 was assigned occupancy 90% and refined anisotropically. O3′ was placed in a position related to O3 by 180° rotation about the C4—C7 bond, assigned occupancy 10%, and refined isotropically. Refinement of the occupancies was unsuccessful, and the values used were chosen to yield reasonable thermal parameters for O3. The aldehydic H atom was placed in a calculated position 0·95 Å from C7 and fixed with occupancy 90% and $B = 1{\cdot}3 \times B_{\mathrm{eq}}$ of C7. An extinction parameter, g, was refined where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. The refinement converged with $R =$

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