Refinement

Refinement on F
$R = 0.032$
$wR = 0.048$
$S = 3.513$
2219 reflections
194 parameters
All H-atom parameters refined
$w = 4F_o^2/[\sigma^2(I) + (0.02F_o^2)^2]$
$(\Delta/\sigma)_{max} = 0.03$

$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: $(1 + gI_c)^{-1}$ applied to F_c
Extinction coefficient: $1.22\,(4) \times 10^{-5}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O1	0.8821 (2)	0.4411 (1)	0.61592 (3)	4.78 (2)
O2	0.5904 (2)	0.4696 (1)	0.32018 (3)	4.53 (2)
O3	0.8189 (3)	0.2979 (2)	0.27142 (5)	7.97 (3)
C1	0.9240 (2)	0.4735 (1)	0.56055 (4)	3.68 (2)
C2	1.1318 (2)	0.5605 (2)	0.55015 (5)	4.07 (2)
C3	1.1936 (2)	0.6025 (1)	0.49715 (5)	3.94 (2)
C4	1.0500 (2)	0.5637 (1)	0.45061 (4)	3.42 (2)
C5	1.1020 (2)	0.6157 (1)	0.39506 (5)	4.05 (2)
C6	0.9572 (2)	0.5820 (1)	0.35143 (5)	4.13 (2)
C7	0.7536 (2)	0.4933 (1)	0.36270 (5)	3.75 (2)
C8	0.6963 (2)	0.4398 (1)	0.41525 (4)	3.49 (2)
C9	0.8428 (2)	0.4751 (1)	0.46109 (4)	3.21 (4)
C10	0.7835 (2)	0.4291 (1)	0.51684 (4)	3.47 (2)
C11	0.6402 (2)	0.3708 (2)	0.27583 (5)	4.61 (2)
C12	0.6694 (3)	0.3677 (2)	0.63046 (6)	5.36 (3)
C13	0.4466 (3)	0.3680 (2)	0.23515 (5)	5.66 (3)

Table 2. *Selected geometric parameters* (Å, °)

O1—C1	1.363 (1)	C4—C5	1.417 (2)
O1—C12	1.415 (2)	C4—C9	1.421 (1)
O2—C7	1.402 (1)	C5—C6	1.365 (2)
O2—C11	1.346 (2)	C6—C7	1.407 (2)
O3—C11	1.198 (2)	C7—C8	1.362 (1)
C1—C2	1.418 (2)	C8—C9	1.414 (1)
C1—C10	1.370 (1)	C9—C10	1.418 (1)
C2—C3	1.353 (2)	C11—C13	1.488 (2)
C3—C4	1.422 (2)		
C1—O1—C12	118.1 (1)	O2—C7—C6	120.3 (1)
C7—O2—C11	119.7 (1)	O2—C7—C8	116.91 (9)
O1—C1—C2	114.4 (1)	C6—C7—C8	122.6 (1)
O1—C1—C10	125.2 (1)	C7—C8—C9	119.78 (9)
C2—C1—C10	120.4 (1)	C4—C9—C8	118.58 (9)
C1—C2—C3	120.7 (1)	C4—C9—C10	119.94 (9)
C2—C3—C4	121.0 (1)	C8—C9—C10	121.45 (9)
C3—C4—C5	122.42 (9)	C1—C10—C9	119.78 (9)
C3—C4—C9	118.22 (9)	O2—C11—O3	122.5 (1)
C5—C4—C9	119.31 (9)	O2—C11—C13	110.7 (1)
C4—C5—C6	121.3 (1)	O3—C11—C13	126.7 (1)
C5—C6—C7	118.5 (1)		
C10—C1—O1—C12	−5.2 (2)	C7—O2—C11—O3	1.6 (2)
C6—C7—O2—C11	67.2 (2)		

Programs used include *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), *MolEN* (Fair, 1990) and *ORTEP* (Johnson, 1965). The two octants, inequivalent in point group 222, were not averaged. Refinement of the inversion-related structure led to slightly worse agreement, $wR = 0.04803$ *versus* 0.04786.

Support for this work was provided by a grant from the National Science Foundation.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and geometry, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP71678 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1081]

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11-Ketoprogesterone

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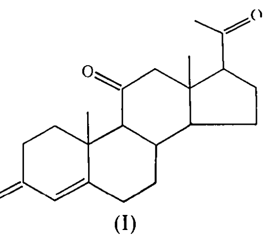
Abstract

In the title compound, 4-pregnen-3,11,20-trione, $C_{21}H_{28}O_3$, ring *A* exists in a sofa conformation.

Rings *B* and *C* adopt chair conformations and ring *D* has a 13*β*,14*α* half-chair conformation. The *A*/*B* ring junction is *quasi-trans*, while ring systems *B*/*C* and *C*/*D* are *trans* fused about the C(8)—C(9) and C(13)—C(14) bonds, respectively.

Comment

As part of our crystallographic investigation of a series of progesterone steroids, the crystal structure of the title compound (I) is reported. The isolation of this compound was reported originally by Peterson *et al.* (1952) and it has been re-isolated by the CrO_3 oxidation of 11*α*-hydroxyprogesterone (Bhutani, 1989).



(I)

A general view of the molecule indicating the numbering scheme is shown in Fig. 1, and the unit-cell packing of 11-ketoprogesterone is shown in Fig. 2.

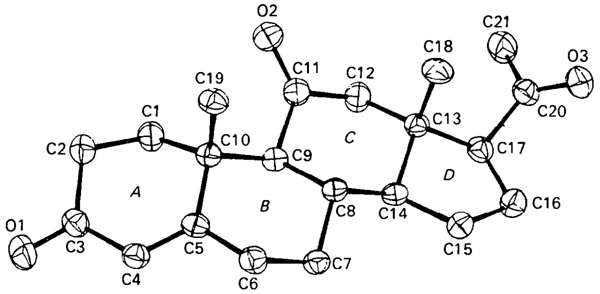


Fig. 1. *ORTEP* (Johnson, 1976) view of the molecule with displacement ellipsoids at 50% probability.

The mean value of the C(sp^3)—C(sp^3) bonds [1.535 (4) Å] is the same as that commonly observed [1.533 Å (Sutton, 1965)], although the bond lengths C(9)—C(10), C(8)—C(14), C(13)—C(17) and C(8)—C(9) show some significant deviations. The bond angles C(8)—C(14)—C(15), C(10)—C(9)—C(11), C(12)—C(13)—C(17), C(14)—C(13)—C(17) and C(13)—C(17)—C(20) show significant deviations from the ideal tetrahedral value of 109.4° (Table 2). These deviations are common in steroids as a result of the strain induced by ring junctions, side chains and unsaturated bonds. The C(4)—C(5) bond length indicates double-bond nature.

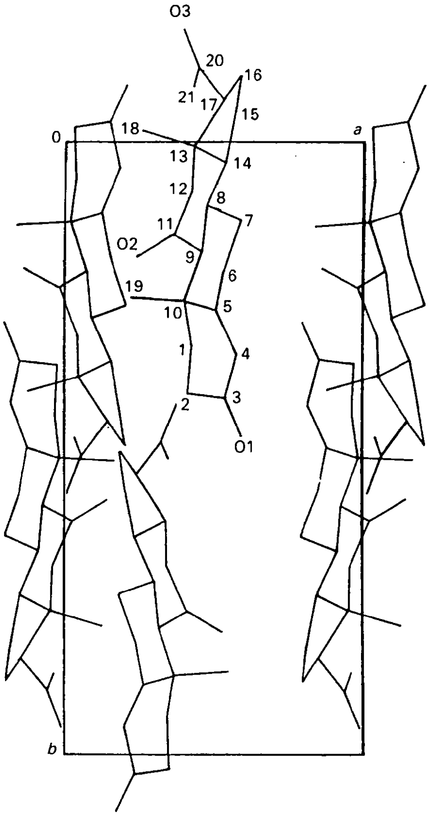


Fig. 2. Packing diagram viewed down the *c* axis.

Ring *A* has a distorted sofa conformation with asymmetry parameters ΔC_s[C(1)—C(4)] = 9.6°, ΔC_2[C(1)—C(2)] = 16.0° (Duax & Norton, 1975). Ring *B* has a normal chair conformation with the best rotational axis bisecting C(5)—C(10) and C(7)—C(8) and asymmetry parameter ΔC_2[C(5)—C(10)] = 3.1°. The best mirror plane passes through C(5) and C(8), with ΔC_s[C(5)—C(8)] = 1.7°. Ring *C* is also in a chair conformation with the best rotational axis bisecting C(8)—C(9) and C(12)—C(13); ΔC_2[C(8)—C(9)] = 4.3°. The best mirror plane passes through C(9) and C(13), with ΔC_s[C(9)—C(13)] = 2.9°.

Ring *D* has a 13*β*,14*α* half-chair conformation with pseudorotation parameters (Altona, Geise & Romers, 1968) $\Delta = 6.9°$ and $\varphi_m = 46.3°$ {ΔC_2[C(13)—C(14)] = 4.7°, ΔC_s[C(13)] = 14.3°}. The atoms C(13) and C(14) are 0.446 (3) and −0.300 (3) Å, respectively, from the plane formed by atoms C(15), C(16) and C(17). The dihedral angle between the least-squares planes of rings *A* and *D* [29.49 (12)°] indicates a slight folding of the molecule probably due to steric interactions.

Newman projections along the bonds involved in ring fusion are shown in Fig. 3. Atoms H(81) and H(91), as well as C(18) and H(141), have antiperiplanar configurations.

The intra- and intermolecular hydrogen bonds which contribute to the stabilization of the molecular structure are summarized in Table 2.

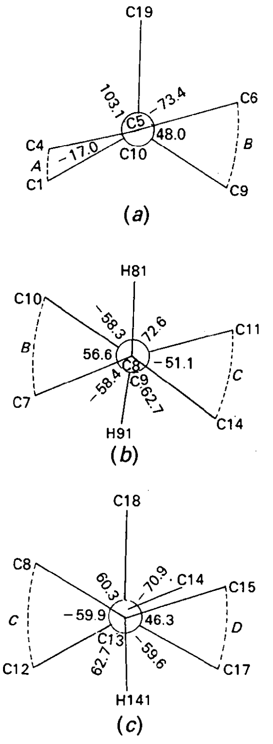


Fig. 3. Newman projections along (*a*) C(5)—C(10), (*b*) C(8)—C(9) and (*c*) C(13)—C(14) (angles given in °).

Experimental

Crystals of the title compound [m.p. 447–449 K, rotation +274° (1% $CHCl_3$)] were grown from acetone by slow evaporation.

Crystal data

$C_{21}H_{28}O_3$
$M_r = 328.45$
Orthorhombic
$P2_12_12_1$
$a = 6.2525$ (4) Å
$b = 12.7576$ (3) Å
$c = 22.4174$ (5) Å
$V = 1788.17$ Å^3
$Z = 4$
$D_x = 1.220$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
$\theta = 16$–$28°$
$\mu = 0.596$ mm^{-1}
$T = 293$ K
Rectangular
0.45 × 0.35 × 0.22 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: none
2116 measured reflections
1953 independent reflections
1810 observed reflections [$I \geq 2.5\sigma(I)$]
$\theta_{max} = 70°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 27$
2 standard reflections monitored every 98 reflections intensity variation: none

Refinement

Refinement on F
$R = 0.064$
$wR = 0.074$
$S = 0.98$
1810 reflections
329 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o) + 0.020(F_o)^2]$
$(\Delta/\sigma)_{max} = 0.71$
$\Delta\rho_{max} = 0.215$ e Å^{-3}
$\Delta\rho_{min} = -0.247$ e Å^{-3}
Atomic scattering factors from *SHELX*76 (Sheldrick, 1976)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
O(1)	0.2692 (8)	−0.1252 (2)	0.6995 (2)	0.1063 (14)
O(2)	−0.1857 (8)	0.2594 (3)	0.5466 (2)	0.1167 (15)
O(3)	0.0074 (7)	0.7498 (2)	0.5280 (2)	0.0993 (11)
C(1)	0.0479 (8)	0.0778 (3)	0.6068 (2)	0.0748 (11)
C(2)	0.0368 (8)	−0.0306 (3)	0.6349 (2)	0.0795 (12)
C(3)	0.1936 (6)	−0.0413 (3)	0.6856 (2)	0.0766 (11)
C(4)	0.2409 (6)	0.0554 (3)	0.7183 (2)	0.0708 (10)
C(5)	0.1595 (5)	0.1484 (3)	0.7054 (1)	0.0633 (9)
C(6)	0.1935 (8)	0.2385 (3)	0.7471 (1)	0.0747 (12)
C(7)	0.2646 (6)	0.3383 (3)	0.7156 (1)	0.0680 (10)
C(8)	0.1141 (5)	0.3667 (2)	0.6640 (1)	0.0554 (8)
C(9)	0.0960 (5)	0.2718 (2)	0.6211 (1)	0.0584 (8)
C(10)	0.0191 (5)	0.1685 (3)	0.6508 (1)	0.0610 (9)
C(11)	−0.0290 (7)	0.3053 (3)	0.5656 (2)	0.0747 (10)
C(12)	0.0535 (8)	0.4017 (3)	0.5338 (1)	0.0780 (13)
C(13)	0.0613 (5)	0.4954 (2)	0.5767 (1)	0.0564 (7)
C(14)	0.1985 (4)	0.4612 (2)	0.6301 (1)	0.0569 (8)
C(15)	0.2374 (7)	0.5631 (3)	0.6639 (2)	0.0720 (11)
C(16)	0.2606 (8)	0.6455 (3)	0.6146 (2)	0.0784 (13)
C(17)	0.1898 (5)	0.5934 (2)	0.5563 (1)	0.0632 (8)
C(18)	−0.1680 (5)	0.5264 (4)	0.5952 (2)	0.0774 (12)
C(19)	−0.2150 (6)	0.1753 (3)	0.6730 (2)	0.0778 (11)
C(20)	0.0743 (6)	0.6647 (3)	0.5134 (2)	0.0723 (10)
C(21)	0.0474 (10)	0.6273 (3)	0.4505 (2)	0.0917 (15)

Table 2. *Bond lengths* (Å), *angles* (°), *endocyclic torsion angles* (°) *and hydrogen-bonding geometry* (Å, °)

C(1)—C(2)	1.521 (5)	C(10)—C(19)	1.548 (4)
C(1)—C(10)	1.531 (5)	C(11)—C(12)	1.512 (5)
C(2)—C(3)	1.507 (6)	C(11)—O(2)	1.218 (6)
C(3)—O(1)	1.210 (5)	C(12)—C(13)	1.535 (4)
C(3)—C(4)	1.465 (5)	C(13)—C(14)	1.536 (3)
C(4)—C(5)	1.323 (5)	C(13)—C(17)	1.554 (3)
C(5)—C(6)	1.496 (4)	C(13)—C(18)	1.544 (4)
C(5)—C(10)	1.527 (3)	C(14)—C(15)	1.524 (4)
C(6)—C(7)	1.522 (5)	C(15)—C(16)	1.532 (5)
C(7)—C(8)	1.534 (4)	C(16)—C(17)	1.531 (5)
C(8)—C(9)	1.550 (3)	C(17)—C(20)	1.507 (4)
C(8)—C(14)	1.519 (3)	C(20)—C(21)	1.498 (6)
C(9)—C(10)	1.552 (4)	C(20)—O(3)	1.208 (4)
C(9)—C(11)	1.530 (5)		
C(2)—C(1)—C(10)	114.5 (3)	C(9)—C(10)—C(19)	112.5 (2)
C(1)—C(2)—C(3)	111.4 (3)	C(9)—C(11)—C(12)	115.8 (3)
C(2)—C(3)—C(4)	115.6 (3)	C(9)—C(11)—O(2)	124.1 (3)
C(2)—C(3)—O(1)	121.8 (4)	C(12)—C(11)—O(2)	120.0 (3)
C(4)—C(3)—O(1)	122.4 (4)	C(11)—C(12)—C(13)	110.4 (2)
C(3)—C(4)—C(5)	124.6 (3)	C(12)—C(13)—C(14)	106.5 (2)
C(4)—C(5)—C(6)	119.8 (2)	C(12)—C(13)—C(17)	117.2 (2)
C(4)—C(5)—C(10)	123.1 (3)	C(12)—C(13)—C(18)	109.7 (2)
C(6)—C(5)—C(10)	116.9 (2)	C(14)—C(13)—C(17)	99.7 (2)
C(5)—C(6)—C(7)	113.2 (2)	C(14)—C(13)—C(18)	112.4 (2)
C(6)—C(7)—C(8)	111.6 (2)	C(17)—C(13)—C(18)	110.6 (2)
C(7)—C(8)—C(9)	109.1 (2)	C(8)—C(14)—C(13)	114.9 (2)
C(7)—C(8)—C(14)	110.5 (2)	C(8)—C(14)—C(15)	118.9 (2)
C(9)—C(8)—C(14)	109.5 (1)	C(13)—C(14)—C(15)	103.5 (2)
C(8)—C(9)—C(10)	114.8 (2)	C(14)—C(15)—C(16)	103.9 (3)

C(8)—C(9)—C(11)	108.8 (2)	C(15)—C(16)—C(17)	106.8 (3)
C(10)—C(9)—C(11)	115.3 (2)	C(13)—C(17)—C(16)	104.3 (2)
C(1)—C(10)—C(5)	108.7 (2)	C(13)—C(17)—C(20)	115.1 (2)
C(1)—C(10)—C(9)	109.1 (2)	C(16)—C(17)—C(20)	114.8 (2)
C(1)—C(10)—C(19)	111.1 (2)	C(17)—C(20)—C(21)	117.5 (3)
C(5)—C(10)—C(9)	107.9 (2)	C(17)—C(20)—O(3)	122.3 (3)
C(5)—C(10)—C(19)	107.1 (2)	C(21)—C(20)—O(3)	120.1 (3)

C(10)—C(1)—C(2)—C(3)	−54.4 (4)
C(1)—C(2)—C(3)—C(4)	30.2 (5)
C(2)—C(3)—C(4)—C(5)	−1.3 (5)
C(3)—C(4)—C(5)—C(10)	−5.4 (5)
C(4)—C(5)—C(10)—C(1)	−17.0 (4)
C(5)—C(10)—C(1)—C(2)	46.6 (4)
C(10)—C(5)—C(6)—C(7)	−50.3 (4)
C(5)—C(6)—C(7)—C(8)	52.3 (3)
C(6)—C(7)—C(8)—C(9)	−54.7 (3)
C(7)—C(8)—C(9)—C(10)	56.6 (3)
C(8)—C(9)—C(10)—C(5)	−51.5 (3)
C(9)—C(10)—C(5)—C(6)	48.0 (3)
C(14)—C(8)—C(9)—C(11)	−51.1 (3)
C(8)—C(9)—C(11)—C(12)	53.3 (3)
C(9)—C(11)—C(12)—C(13)	−56.9 (4)
C(11)—C(12)—C(13)—C(14)	55.6 (3)
C(12)—C(13)—C(14)—C(8)	−59.9 (2)
C(13)—C(14)—C(8)—C(9)	58.7 (2)
C(17)—C(13)—C(14)—C(15)	46.3 (2)
C(13)—C(14)—C(15)—C(16)	−36.2 (3)
C(14)—C(15)—C(16)—C(17)	11.2 (3)
C(15)—C(16)—C(17)—C(13)	17.4 (3)
C(16)—C(17)—C(13)—C(14)	−38.7 (2)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
C(1)—H(12)···O(2)	2.40 (6)	3.053 (6)	130 (5)
C(19)—H(192)···O(2)	2.43 (4)	3.035 (6)	135 (4)
C(16)—H(161)···O(1^{i})	2.76 (4)	3.490 (5)	129 (3)
C(12)—H(121)···O(2^{ii})	2.44 (4)	3.183 (6)	124 (3)

Symmetry codes: (i) $x, 1 + y, z$; (ii) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$.

Table 3. *Puckering parameters*

Puckering parameters from Cremer & Pople (1975).

Ring *A*		Ring *C*	
q_2 = 0.364 Å	q_3 = 0.251 Å	q_2 = 0.044 Å	q_3 = 0.573 Å
Q = 0.442 Å	φ = 11.6°	Q = 0.574 Å	φ = 260.2°
θ = 55.3°		θ = 4.4°	
Ring *B*		Ring *D*	
q_2 = 0.062 Å	q_3 = −0.530 Å	q_2 = 0.456 Å	φ = 193.9°
Q = 0.534 Å	φ = 64.2°		
θ = 173.3°			

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). Isotropic refinement of the structure by least-squares methods using *SHELX*76 (Sheldrick, 1976) was followed by anisotropic refinement of all the non-H atoms. All H atoms except H(213) were located from a difference Fourier map and their positions and isotropic displacement parameters were refined. H(213) was fixed geometrically.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and dihedral angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71705 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1009]

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2-(2-Hydroxy-5-methylphenyl)-5-chlorobenzotriazole, $C_{13}H_{10}ClN_3O$

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Abstract

Preliminary results of an X-ray crystal structure analysis show that the title molecule [2-(5-chloro-2-benzotriazolyl)-4-methylphenol] is practically planar in the crystalline state. There is an intramolecular hydrogen bond in which the hydroxyl group serves as a donor to the N3 atom of the benzotriazole moiety. Such systems, which undergo intramolecular proton transfer, are widely used as UV stabilizers.

Comment

The 2-(2-hydroxy-5-methylphenyl)-5-chlorobenzotriazole (Cltin) molecule belongs to a class of