

17 α -Hydroxypregna-1,4-diene-3,20-dioneLiu-Qing Sheng,^{a*} Han-Min Pan,^b Chao-Tian Chen^b and Gang Chen^b^aDepartment of Pharmaceutical Science, Jinhua College of Professions and Technology, Jinhua 321007, People's Republic of China, and^bZhejiang Medicine Co. Ltd, Xinchang Pharmaceutical Factory, Xinchang 312500, People's Republic of China

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Key indicators

Single-crystal X-ray study

T = 293 K

Mean σ (C–C) = 0.003 Å

R factor = 0.053

wR factor = 0.127

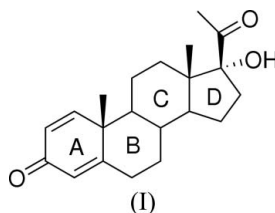
Data-to-parameter ratio = 9.7

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title steroid compound, C₂₁H₂₈O₃, the cyclohexadiene ring has a planar structure, while the cyclopentane ring displays an envelope configuration. Classical O–H···O and weak C–H···O hydrogen bonding helps to stabilize the crystal structure.

Comment

17 α -Hydroxypregna-1,4-diene-3,20-dione, (I), is a useful material for the synthesis of prednisolone because ring A (see scheme) is a fully functionalized 1,4-dien-3-one system (Nitta *et al.*, 1985). In order to understand the relationship between the structure and biological activity, we have determined the structure of (I).



The molecular structure of (I) is shown in Fig. 1. Within the planar ring A, the C1=C2 and C4=C5 bond distances are 1.328 (3) and 1.341 (3) Å, respectively, in agreement with those for reported steroid structures (Rendle & Trotter, 1974; Xia *et al.*, 2005). Rings B and C display the normal chair conformations, while ring D has an envelope conformation, with C13 lying in the flap position and deviating by 0.683 (2) Å from the mean plane formed by the other four atoms.

Classical O–H···O and weak C–H···O hydrogen bonding helps to stabilize the crystal structure (Table 1).

Experimental

To a dichloromethane solution (15 ml) of 20-(acetylamino)pregna-1,4,17(20)-trien-3-one (2.0 g) and sodium acetate (1.3 g), a dichloromethane solution (20 ml) of peroxyacetic acid (1.3 g) was added dropwise at 298 K. After stirring for 1.5 h, the solution was washed with a saturated sodium bicarbonate solution and distilled water in turn. The solution was dried with MgSO₄ and the solvent was evaporated. To the residue, methanol (30 ml) and aqueous sodium hydroxide solution (0.3 M, 28 ml) were added and the mixture was refluxed for 2 h. After cooling to room temperature, the mixture was neutralized with glacial acetic acid solution and then concentrated. The products were extracted with dichloromethane (3 × 20 ml) and the organic layer was washed with distilled water and then dried over anhydrous MgSO₄. Removal of solvents afforded the crude product (I), which was purified on a column of silica gel (eluant: benzene–ethanol 8:2) to obtain pure (I) (yield 0.68 g). Single crystals of (I) were obtained by slow evaporation of an ethanol solution.

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Crystal data

$C_{21}H_{28}O_3$
 $M_r = 328.43$
 Orthorhombic, $P2_12_12_1$
 $a = 7.781(3) \text{ \AA}$
 $b = 9.979(4) \text{ \AA}$
 $c = 22.410(9) \text{ \AA}$
 $V = 1739.9(13) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.254 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.08 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Prism, colorless
 $0.20 \times 0.20 \times 0.15 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 8582 measured reflections

2193 independent reflections
 1871 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.073$
 $\theta_{max} = 27.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.127$
 $S = 1.02$
 2193 reflections
 225 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0832P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.25 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.34 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.040(5)

Table 1

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O2-H2X\cdots O1^i$	0.800 (17)	1.967 (18)	2.754 (3)	167 (3)
$C2-H2\cdots O3^{ii}$	0.93	2.59	3.356 (4)	140

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

The hydroxy H atom was located in a difference Fourier map and refined isotropically. Methyl H atoms were placed in calculated positions with $C-H = 0.96 \text{ \AA}$ and $U_{iso}(H) = 1.5U_{eq}(C)$. Other H

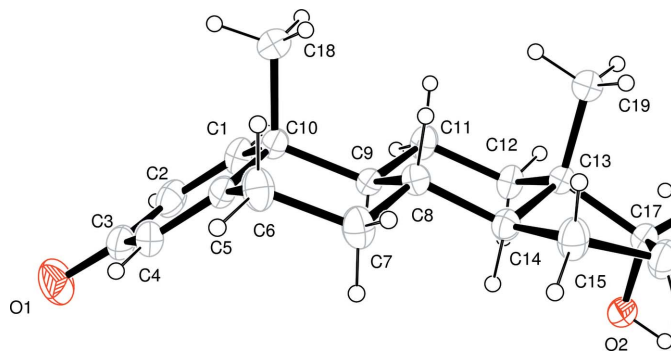


Figure 1

The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

atoms were placed in calculated positions with $C-H = 0.93$ (aromatic), 0.97 (methylene) and 0.98 \AA (methine), and refined in riding mode with $U_{iso}(H) = 1.2U_{eq}(C)$. Because of negligible anomalous scattering effects, Friedel pairs were merged and the absolute configuration was assigned arbitrarily.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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