

11 β -Hydroxypregna-1,4,16-triene-3,20-dioneXiao-Hong Wu,* Yong-Li Wang
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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.042
 wR factor = 0.109
Data-to-parameter ratio = 9.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

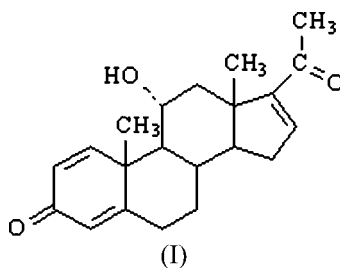
The steroidal title compound, $\text{C}_{21}\text{H}_{26}\text{O}_3$, possesses normal geometrical parameters. Ring *A* has a 1α -sofa conformation, and rings *B* and *C* are in nearly perfect chair conformations. An $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond helps to establish the crystal packing.

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Comment

The title compound (I), which can be made from 16-dehydroprogesterone in a multi-step process (Herzog *et al.*, 1959), is an important steroid derivative. It has been used in the syntheses of some corticosteroids (Conrow, 1999). Its ^1H and ^{13}C NMR spectra have previously been used to establish the atomic connectivity (Kirk *et al.*, 1990). Here, the single-crystal structure of (I) (Fig. 1) is described. It confirms the spectroscopic structure.



A typical steroid conformation is observed, with three six-membered rings and one five-membered ring denoted as rings *A* (C1–C6), *B* (C1, C6–C10), *C* (C9–C14) and *D* (C13–C17), respectively. Ring *A* has a 1α -sofa conformation, and rings *B* and *C* are in nearly ideal chair conformations. Ring *D* is an envelope with C14 in the flap position. The configurations of the chiral C atoms in (I) (C1 *R*; C9 *S*; C10 *S*; C11 *R*; C13 *S*; C14 *S*) are consistent with those of the equivalent atoms in the starting material of known absolute configuration.

An $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond between the 11-OH group of one molecule and the 3-carbonyl group of an adjacent molecule (Table 1 and Fig. 2) helps to establish the crystal packing in (I).

Experimental

Chromium (1.5 g) and hydrochloric acid (40 ml, 8.0%) were reacted under nitrogen at 348 K to generate a divalent chromium solution. A stirred solution of 16 α ,17 α -epoxypregna-11 α -hydroxy-1,4-diene-3,20-dione (5.0 g, 14.7 mmol), provided by Tianjin Tianyao Pharmaceutical Co. Ltd, and the chromium(II) solution were then mixed and heated to 353 K for 30 min. The resulting light-yellow solid was collected by filtration and dried under vacuum at 333 K. The powder

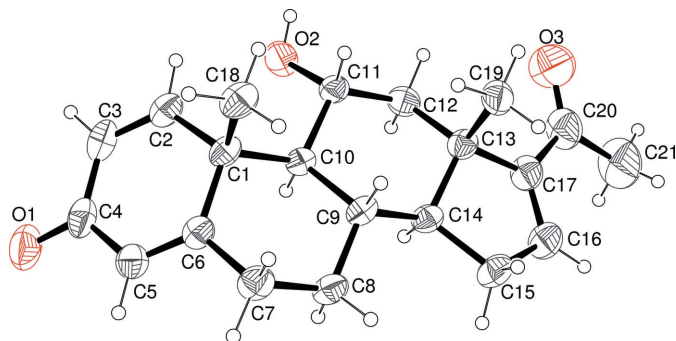


Figure 1
The molecular structure of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms).

was dissolved in ethanol and crystals of (I) grew on slow evaporation of the solvent (m.p. 547 K).

Crystal data

$C_{21}H_{26}O_3$ $Z = 4$
 $M_r = 326.42$ $D_x = 1.198 \text{ Mg m}^{-3}$
 Orthorhombic, $P2_12_12_1$ Mo $K\alpha$ radiation
 $a = 6.2265 (12) \text{ \AA}$ $\mu = 0.08 \text{ mm}^{-1}$
 $b = 14.862 (3) \text{ \AA}$ $T = 293 (2) \text{ K}$
 $c = 19.561 (4) \text{ \AA}$ Blade, light yellow
 $V = 1810.1 (6) \text{ \AA}^3$ $0.37 \times 0.13 \times 0.04 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID IP diffractometer 14741 measured reflections
 1959 independent reflections
 ω scans 1544 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $R_{int} = 0.070$
 $T_{min} = 0.972, T_{max} = 0.998$ $\theta_{max} = 25.5^\circ$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0613P)^2 + 0.0192P]$
 $R[F^2 > 2\sigma(F^2)] = 0.042$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.109$ $(\Delta/\sigma)_{max} = 0.014$
 $S = 1.10$ $\Delta\rho_{max} = 0.16 \text{ e \AA}^{-3}$
 1959 reflections $\Delta\rho_{min} = -0.13 \text{ e \AA}^{-3}$
 218 parameters Extinction correction: *SHELXL97*
 H-atom parameters constrained Extinction coefficient: 0.024 (5)

Table 1
Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O2-H2B \cdots O1^1$	0.82	1.93	2.733 (3)	166

Symmetry code: (i) $-x + 3, y - \frac{1}{2}, -z + \frac{1}{2}$.

In the absence of significant anomalous scattering effects, Friedel pairs were merged prior to refinement. The absolute configuration of (I) was assigned based on that of the well defined starting material. H atoms were placed in calculated positions ($O-H = 0.82 \text{ \AA}$, $C-H =$

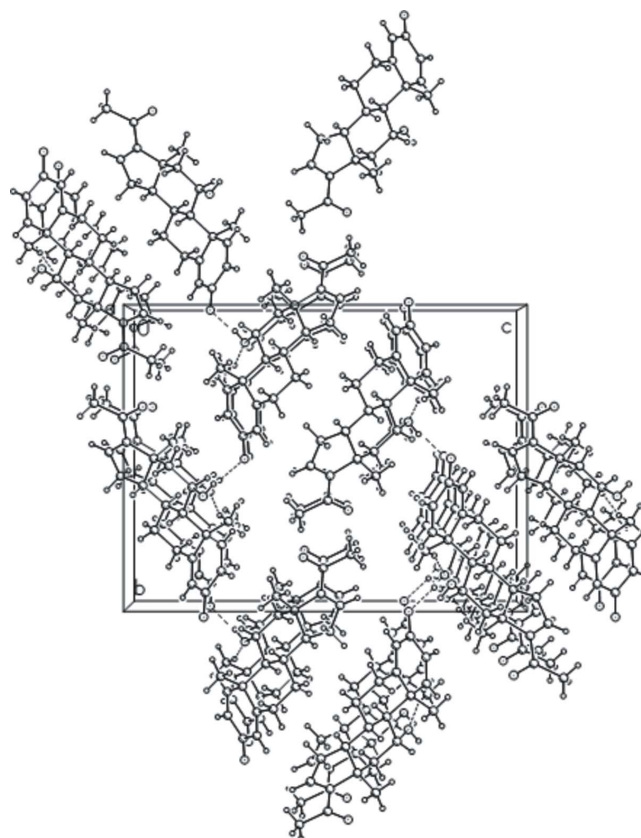


Figure 2
The molecular packing of compound (I), viewed along the a axis. Hydrogen bonds and close contacts are shown as dashed lines.

$0.93\text{--}0.98 \text{ \AA}$) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$ or $1.5U_{eq}(\text{methyl carrier})$.

Data collection: *RAPID-AUTO* (Rigaku, 1999); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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