Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

11-Hydroxy-16,17-epoxypregn-4-ene-3,20-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.101
Data-to-parameter ratio = 9.4

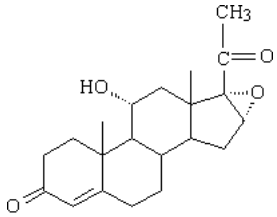
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 16 April 2004
Accepted 18 May 2004
Online 17 July 2004

The title compound, $C_{21}H_{28}O_4$, is a steroid derivative used as an intermediate in medicine. In the crystal structure, symmetry-related molecules are linked by a hydrogen bond between the hydroxy group and the carbonyl group at position 3 [$D\cdots A$ = 2.825 (4) Å].

Comment

The title compound, (I), is a steroid derivative used as an intermediate in medicine (such as a hydrocortisone). It is obtained from the fermentation of 16,17-epoxypregn-4-ene-3,20-dione, (II), by a certain type of fungi (Steroid Research Group, 1981). Although the molecular structure of compound (II) has already been identified (Goubitz *et al.*, 1984), the molecular structure of (I) has not been studied to date.



(I)

The molecular structure of (I) is shown in Fig. 1. Using the typical nomenclature for steroids it can be seen that ring *A* is in the 1α-sofa conformation, rings *B* and *C* are in chair conformations and ring *D* has a 14α-envelope conformation. These conformations are the same as for compound (II) (Goubitz *et al.*, 1984). The O4—C18—C17—C13 torsion angle is 15.0 (5)° and the interatomic distances O2—C11 and O1—C3 are 1.435 (3) and 1.228 (4) Å, respectively.

The molecular structure and the molecular packing of the title compound, (I), are similar to those of (II), but in (I) there is an intermolecular hydrogen bond between the hydroxy group at C11 and the carbonyl group at C3, the $D\cdots A$ distance being 2.825 (4) Å (Table 1 and Fig. 2).

The hydrogen bonds are so strong that they make the separation of the two compounds very difficult. Both (I) and (II) have carbonyl groups at C3, so the hydrogen bonding favours cocrystallization.

Experimental

Compound (I) was supplied by the Tianjin Tianyao Pharmaceutical Co. Ltd, and was characterized by NMR, IR and elemental analyses, which were the same as the reported data. The melting point of (I) is 522.8 K (Xu, 2001), measureed by DSC (differential scanning calorimetry), and the solubility is 61.7 mg ml^{-1} (293 K) in chloroform. Colorless single crystals of (I) suitable for X-ray diffraction

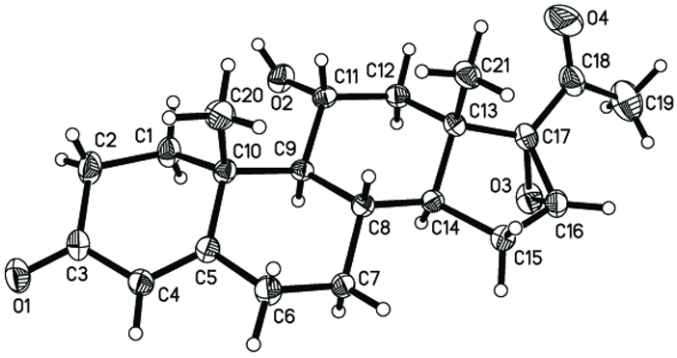


Figure 1
*ORTEP*II (Johnson, 1976) view of the title compound (I), with displacement ellipsoids drawn at the 30% probability level.

were obtained by slow evaporation of a chloroform solution [40 mg ml^{-1} (293 K)] in air.

Crystal data

$C_{21}H_{28}O_4$	Mo $K\alpha$ radiation
M_r = 344.43	Cell parameters from 821 reflections
Orthorhombic, $P2_12_12_1$	θ = 3.2–20.9°
a = 7.229 (3) Å	μ = 0.09 mm^{-1}
b = 13.112 (5) Å	T = 293 (2) K
c = 19.334 (7) Å	Block, colorless
V = 1832.6 (12) $Å^3$	0.38 × 0.20 × 0.14 mm
Z = 4	
D_x = 1.248 Mg m^{-3}	

Data collection

Bruker SMART CCD area-detector diffractometer	2169 independent reflections
	1555 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.054
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	θ_{max} = 26.5°
	$h = -9 \rightarrow 8$
T_{min} = 0.870, T_{max} = 0.990	$k = -16 \rightarrow 14$
10617 measured reflections	$l = -12 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.2106P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.047	
$wR(F^2)$ = 0.102	
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
2169 reflections	$\Delta\rho_{max}$ = 0.15 e $Å^{-3}$
230 parameters	$\Delta\rho_{min}$ = −0.18 e $Å^{-3}$
H-atom parameters constrained	

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.82	2.01	2.825 (4)	175

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

H atoms were placed in calculated positions and were treated as riding atoms, with O—H = 0.82 Å, C—H = 0.93–0.98 Å and U_{iso}(H) = 1.5U_{eq} for H atoms on O and C(CH_3) groups or 1.2U_{eq} for H atoms on other C atoms. In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was assumed arbitrarily.

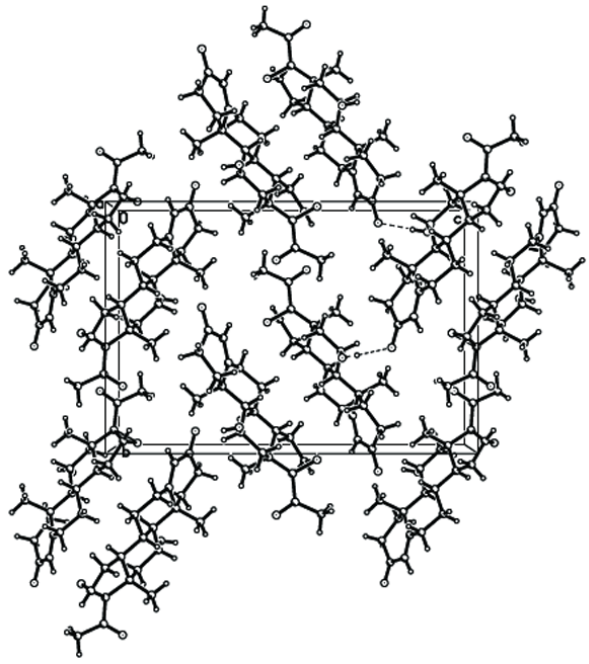


Figure 2
The molecular packing of (I), viewed along the b axis. The thin lines indicate intermolecular hydrogen-bonding interactions.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge the support of the SRCICT of Tianjin University and Tianjin Tianyao Pharmaceutical Co. Ltd for suppling compound (I).

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