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16α,17α-Epoxypregna-11α-hydroxy-1,4-diene-3,20-dione

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Key indicators

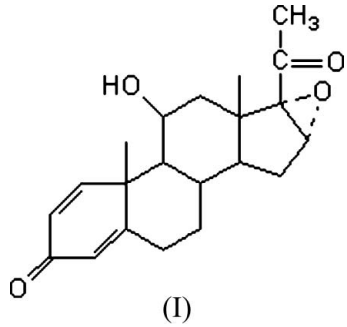
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.055
wR factor = 0.105
Data-to-parameter ratio = 10.5

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

The title compound, $C_{21}H_{26}O_4$, is an important intermediate in the synthesis of corticosteroids. The crystal packing is predominantly stabilized by strong intermolecular O—H···O hydrogen bonds.

Comment

16α,17α-Epoxypregna-11α-hydroxy-1,4-diene-3,20-dione, (I), is used to produce 11β-hydroxypregna-1,4,16-triene-3,20-dione, which is an intermediate in the synthesis of corticosteroids; there has been no previous report of its structure.



(I)

The title compound crystallizes in the orthorhombic space group $P2_12_12_1$. The molecular structure has a typical steroid conformation, with three six-membered rings and one five-membered ring denoted, respectively, as *A*, *B*, *C* and *D*, and is shown in Fig. 1. Ring *A* has a 1α-sofa conformation, and rings *B* and *C* are in nearly perfect chair conformations. Ring *D* is in an envelope conformation. There is a strong hydrogen bond between the 11-OH group and the 3-carbonyl group of adjacent molecules (Table 1), with an H···O distance of 1.94 (2) Å and an O2—H2*B*···O1 angle of 162.1 (4)°.

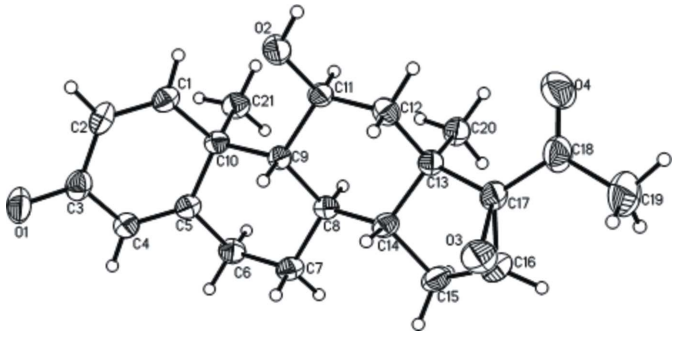


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme and displacement ellipsoids drawn at the 30% probability level.

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Experimental

Crystals of 16α,17α-epoxypregna-11α-hydroxy-1,4-diene-3,20-dione, provided by Tianjin Tianyao Pharmaceutical Co. Ltd, were grown in a 5 ml beaker with 3 ml ethanol at 313 K. The beaker was sealed and single crystals were obtained after 4 d.

Crystal data

$C_{21}H_{26}O_4$
M_r = 342.42
Orthorhombic, $P2_12_12_1$
a = 6.1342 (12) Å
b = 15.433 (3) Å
c = 19.029 (4) Å
V = 1801.5 (6) Å^3
Z = 4
D_x = 1.262 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.11 × 0.11 × 0.08 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.990, T_{max} = 0.993
17631 measured reflections
2371 independent reflections
1444 reflections with $I > 2\sigma(I)$
R_{int} = 0.113
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.105
S = 1.08
2371 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O1^{i}	0.82	1.94	2.731 (3)	162

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

H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C). In the absence of significant anomalous scattering effects, Friedel pairs were merged prior to the final refinement.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

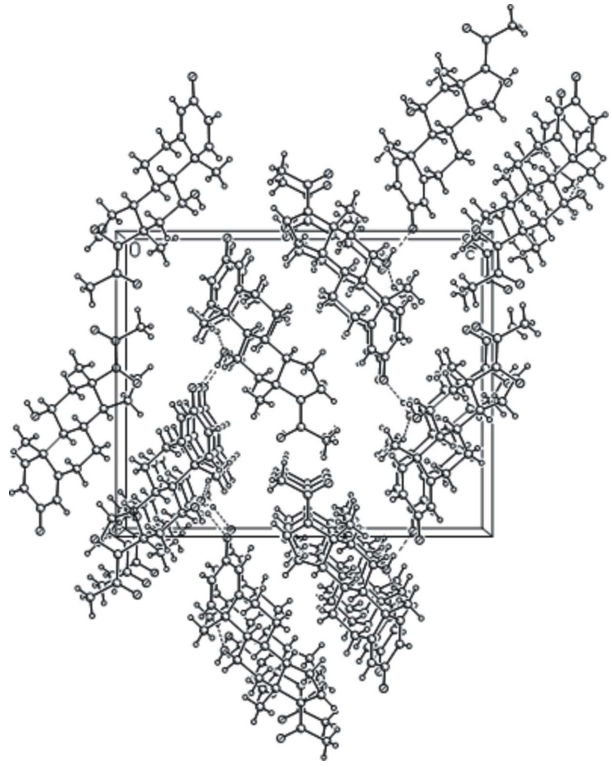

Figure 2
The molecular packing of compound (I), viewed down the *a* axis.

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Rigaku (2004). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1997). *SADABS*, *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.