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16α,17-Epoxypregn-4-ene-3,11,20-trione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.109
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.

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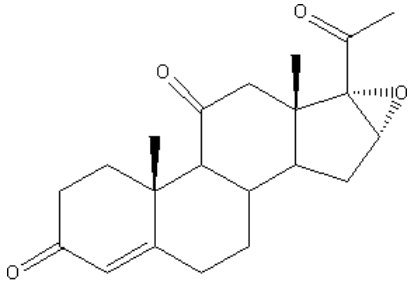
The title compound, $C_{21}H_{26}O_4$, is an important steroid used as an intermediate in the synthesis of hormone pharmaceuticals. No inter- or intramolecular hydrogen bonds were observed in the crystal structure.

Comment

The title compound, (I), is an important steroid compound, which serves as an intermediate in the synthesis of many hormone pharmaceuticals (Xu, 2001). It was first prepared from 11α-hydroxy-16α,17-epoxyprogesterone by oxidation with chromium trioxide by Peterson *et al.* (1955) but, to the best of our knowledge, the single-crystal structure of (I) has not yet been reported.



(I)

The molecular structure of (I) is shown in Fig. 1. The compound has a typical steroid conformation with three six-membered rings and one five-membered ring denoted *A*, *B*, *C* and *D*, respectively. Ring *A* has a 1α-sofa conformation, and rings *B* and *C* are in nearly perfect chair conformations. The presence of the three-membered ring constrains the five-

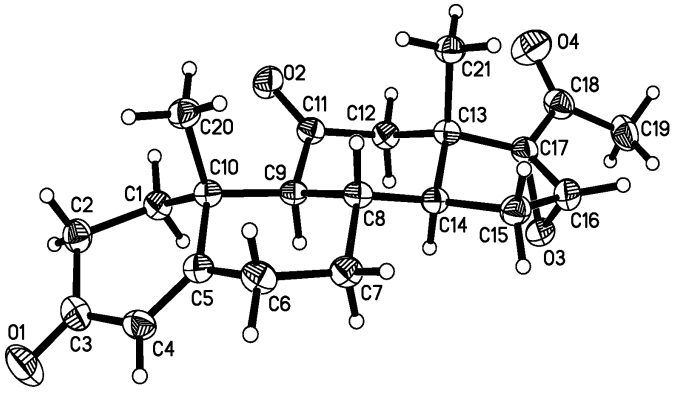


Figure 1
A view of the molecule of compound (I), showing the atom-labeling scheme and displacement ellipsoids drawn at the 30% probability level.

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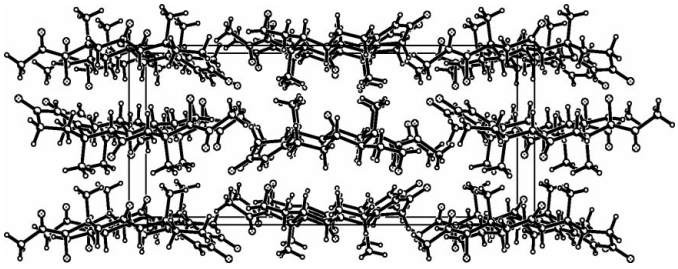

Figure 2
The molecular packing of compound (I), viewed along the *a* axis.

membered *D* ring to have a 14α-envelope conformation as described by Goubitz *et al.* (1984).

The carbonyl group on C18 nearly eclipses the C13—C17 bond. The C13—C17—C18—O4 torsion angle is 7.4 (4)°. The overall conformation is similar to that found in 11α-hydroxy-16α,17-epoxyprogesterone (Wang *et al.*, 2004). The title compound crystallizes in the orthorhombic space group $P2_12_12_1$, similar to 11α-hydroxy-16α,17-epoxyprogesterone. As a result of the lack of hydrogen-bond donors, no inter- or intramolecular hydrogen bonds were observed.

Experimental

11α-Hydroxy-16α,17-epoxyprogesterone, provided by Tianjin Tianyao Pharmaceutical Co. Ltd, was dissolved in pyridine and treated with chromium trioxide at room temperature overnight. The product was chromatographed on silica gel and recrystallized three times from acetone–hexane. The melting point determined by differential scanning calorimetry (DSC) is 465.5 K. Colorless prismatic single crystals suitable for X-ray diffraction were obtained by slow natural evaporation of a chloroform–methanol solution (1:1, 10 ml) at room temperature.

Crystal data

$C_{21}H_{26}O_4$
M_r = 342.42
Orthorhombic, $P2_12_12_1$
a = 7.5364 (12) Å
b = 9.9000 (16) Å
c = 23.288 (4) Å
V = 1737.6 (5) Å^3
Z = 4
D_x = 1.309 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2892 reflections
θ = 2.2–23.9°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colorless
0.32 × 0.28 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
11 707 measured reflections
2396 independent reflections
1925 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 28.0°
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 13$
$l = -30 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.109
S = 1.07
2396 reflections
229 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.0814P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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). The Friedel equivalents were merged prior to the final refinement and the absolute configuration was assigned from the known chiral centers in the precursor molecule, which remained unchanged during the synthesis of the title compound.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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