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16α,17-Epoxy-11α-(*p*-tolylsulfonyloxy)pregn-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 = 0.046
wR factor = 0.104
Data-to-parameter ratio = 18.1

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

The title compound, $C_{28}H_{34}O_6S$, is an important intermediate in the synthesis of hormone pharmaceuticals. In the crystal structure, the asymmetric unit consists of two molecules.

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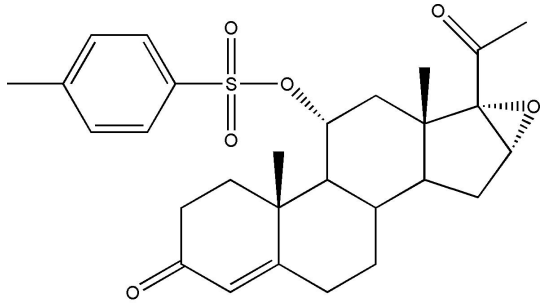
Comment

The title compound, (I), is an important intermediate in the synthesis of hormone pharmaceuticals, such as fluocinonide (Xu, 2001). It was first prepared from 11α-hydroxy-16α,17-epoxyprogesterone by sulfonation with *p*-toluenesulfonyl-chloride (Camerino *et al.*, 1959), but the crystal structure was not reported.



(I)

In the crystal structure, the asymmetric unit consists of two molecules (Figs. 1 and 2). Ring *A* is in a 1α-sofa conformation, rings *B* and *C* are in nearly perfect chair conformations and ring *D* is in a 14α-envelope conformation. The presence of the three-membered ring constrains ring *D* to have a 14α-

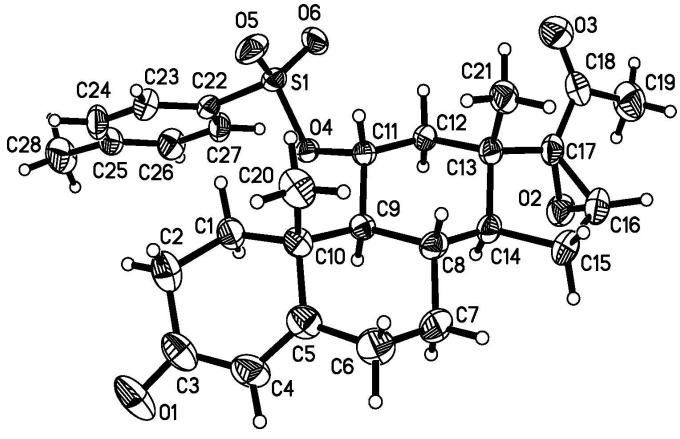


Figure 1
The molecular structure of molecule 1 of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

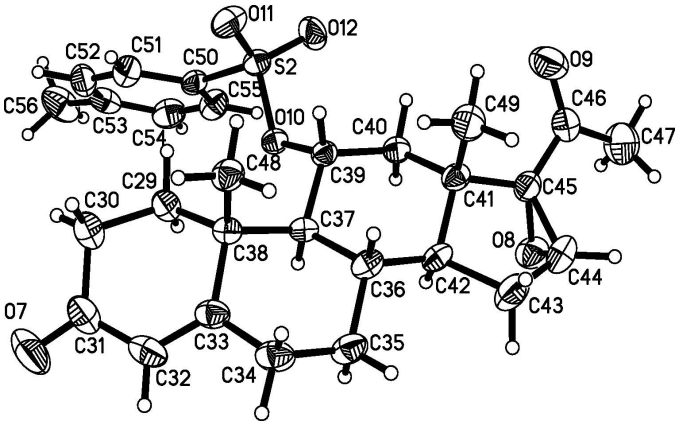


Figure 2
The molecular structure of molecule 2 of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

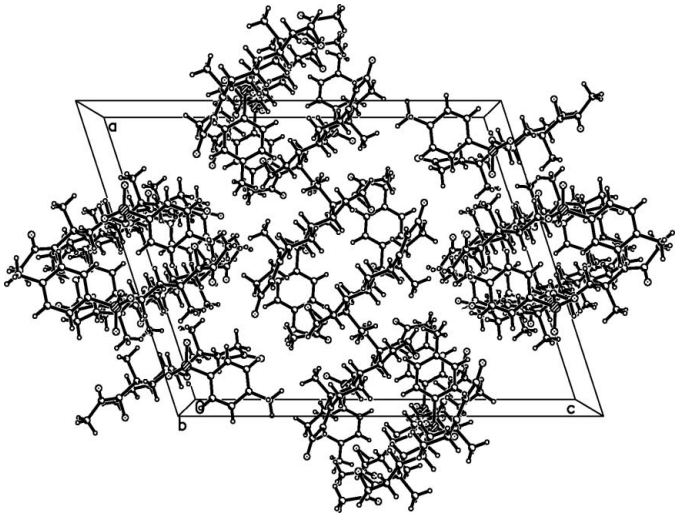


Figure 3
The molecular packing of (I), viewed along the *b* axis.

envelope conformation (Goubitz *et al.*, 1984). The two molecules in the asymmetric unit have similar conformations.

Experimental

11α-Hydroxy-16α,17-epoxyprogesterone (1 g; provided by Tianjin Tianyao Pharmaceutical Co. Ltd) was dissolved in pyridine (20 ml) and treated with *p*-toluenesulfonyl chloride (0.6 g) at room temperature overnight. The product was chromatographed on silica gel and recrystallized from acetone–hexane three times. The melting point, determined by differential scanning calorimetry, is 443.2 K with decomposition. Colourless plate-like single crystals suitable for X-ray diffraction were obtained by slow evaporation of a chloroform–methanol solution (1:1, 10 ml) at room temperature.

Crystal data

$C_{28}H_{34}O_6S$
M_r = 498.61
Monoclinic, *C*2
a = 19.508 (2) Å
b = 10.731 (1) Å
c = 25.920 (4) Å
β = 108.666 (2)°
V = 5140.8 (11) Å^3
Z = 8
D_x = 1.288 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2920 reflections
θ = 2.3–19.4°
μ = 0.17 mm^{-1}
T = 293 (2) K
Thick plate, colourless
0.26 × 0.22 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.927, T_{max} = 0.980
17 613 measured reflections
11 583 independent reflections
7085 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 28.0°
$h = -19 \rightarrow 25$
$k = -13 \rightarrow 14$
$l = -34 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.104
S = 1.01
11583 reflections
639 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: none
Absolute structure: Flack (1983), 4824 Friedel pairs
Flack parameter: 0.08 (5)

H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H distances of 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C). The refinement of the Flack (1983) parameter confirms that the chiral centres retain their original configurations during the synthesis, as expected.

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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