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16α,17-Epoxy-4-pregnene-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 = 0.038
wR factor = 0.092
Data-to-parameter ratio = 10.9

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

The title compound, $C_{21}H_{28}O_3$, is a steroid derivative used as an intermediate in medicine. The molecular structure corresponds to an earlier determination [Goubitz *et al.* (1984). *Steroids*, **44**, 153–158]. However, the orthorhombic unit-cell parameters of the two determinations are significantly different, and these structures are polymorphs.

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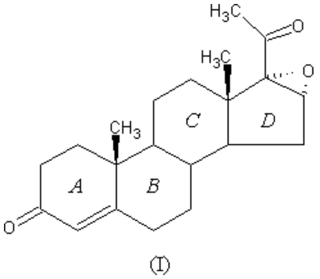
Comment

The title compound, (I), a steroid derivative, was prepared from diosgeum. In the solid state, ring *A* has a 1α-sofa conformation (Fig. 1), rings *B* and *C* have chair conformations and ring *D* has a 14α-envelope conformation. These molecular conformations are similar to those published earlier (Goubitz *et al.*, 1984) for the same compound with the Cambridge Structural Database (Version 5.25; Allen, 2002) refcode DILYEC, (II). However, the orthorhombic unit-cell parameters of (II) [a = 13.8635 Å, b = 17.283 Å and c = 7.424 Å] are markedly different from those for (I), so these are polymorphs. Interestingly, the CSD contains a crystal structure (refcode BUPRGE10; Tseikinskii *et al.*, 1980) with orthorhombic unit-cell parameters [a = 7.53 Å, b = 13.94 Å and c = 17.31 Å] quite close to those for (II). The chemical identity of BUPRGE10, namely 16α,17α-cyclopropano-progesterone, is only slightly different from that of (II), with a CH_2 group instead of the epoxide O atom; the two compounds are essentially isostructural.



(I)

In the crystal structure, there are short intermolecular C—H···O contacts (Table 1). The molecular structure (Fig. 1) and crystal packing (Fig. 2) of (I) are similar to those in 11-hydroxy-16,17-epoxypregn-4-ene-3,20-dione (Wang *et al.*, 2004), which can be obtained from (I) by fermentation.

Experimental

16α,17-Epoxy-4-pregnene-3,20-dione was prepared from diosgeum by hydrolyzation and oxidation, and is a product of the Tianjin Tianyao Pharmaceutical Co. Ltd. The product was characterized by NMR, IR and elemental analyses, and its purity was 99%. The

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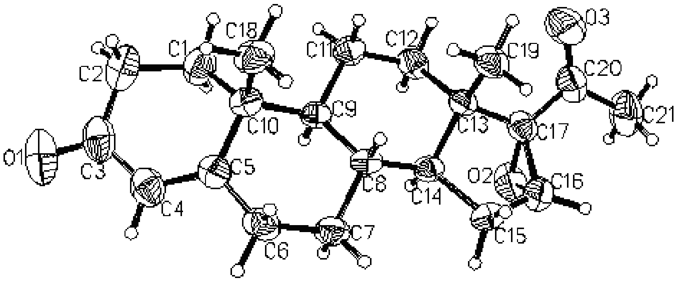

Figure 1
*ORTEP*II (Johnson, 1976) view of the title compound, showing 30% probability displacement ellipsoids.

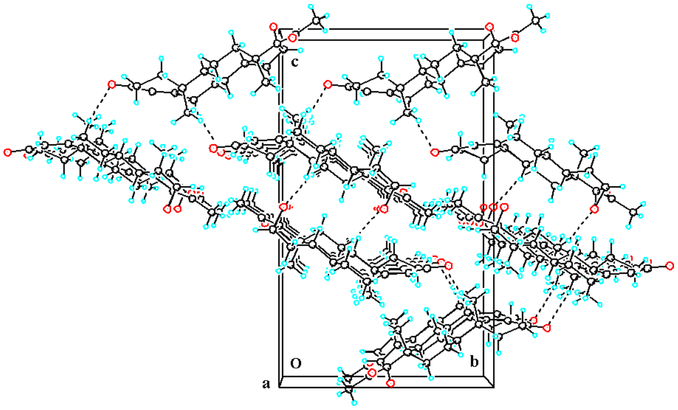

Figure 2
The molecular packing of the title compound, viewed along the *a* axis. Dashed lines indicate the intermolecular short contacts.

melting point of the compound was 482.8 K, measured by differential scanning calorimetry, and its solubility is 0.123 g/g (293 K) in toluene. Colorless single crystals suitable for X-ray diffraction were obtained by slow evaporation in air of a toluene solution with a concentration of 0.1 g/g (293 K).

Crystal data

$C_{21}H_{28}O_3$
$M_r = 328.43$
Orthorhombic, $P2_12_12_1$
$a = 7.3357$ (1) Å
$b = 12.1537$ (2) Å
$c = 20.3204$ (1) Å
$V = 1811.68$ (4) Å^3
$Z = 4$
$D_x = 1.204$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16882 reflections
$\theta = 2.0$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.73 × 0.40 × 0.31 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
ω and φ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.945$, $T_{\max} = 0.976$
16882 measured reflections
2380 independent reflections
1311 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.070$
$\theta_{\max} = 27.5°$
$h = -8 \rightarrow 9$
$k = -14 \rightarrow 15$
$l = -25 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.092$
$S = 0.83$
2380 reflections
218 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.028 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O2$^{\rm i}$	0.97	2.52	3.433 (3)	158
C6—H6B···O1$^{\rm ii}$	0.97	2.43	3.371 (3)	164

Symmetry codes: (i) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$; (ii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$.

All H atoms were placed in geometrically idealized positions (C—H = 0.93–0.98 Å) and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the final refinement and the absolute configuration was assigned to correspond with that of the known chiral centres in a precursor molecule, which remained unchanged during the synthesis of the title compound.

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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