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16β-Bromo-17α-hydroxypregn-4-ene-3,20-dione methanol solvate

Shi Wang,* Yongli Wang, Qiang Nie, Aishuang Xiang and Lina Zhou

School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: wangshi04@hotmail.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.012 Å
R factor = 0.066
wR factor = 0.192
Data-to-parameter ratio = 15.0

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

The title compound, $C_{21}H_{29}BrO_3\cdot CH_3OH$, is a steroid and an intermediate for medicinal applications. It is prepared through the reaction of 16,17-epoxypregn-4-ene-3,20-dione and HBr, and the methanol solvate is reported here. The space group is $P2_12_12_1$ and is different from the space group of 16β-bromo-17α-hydroxypregn-4-ene-3,20-dione which is $C2$ [Wang, Wang, Nie, Xiang & Zhou (2005). *Acta Cryst.* E**61**, o1–o2].

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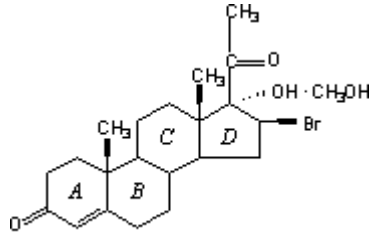
Comment

The title compound, (I), is a steroid derivative with medicinal applications, obtained from the reaction of 16,17-epoxypregn-4-ene-3,20-dione and HBr in acetic acid. It was purified by recrystallization. In the present paper, we report the crystal structure of the title compound, (I).



(I)

Ring A of (I) is in the 1α-sofa conformation, whereas rings B and C are in chair conformations and ring D is in a 14α-envelope conformation. The conformations are similar to those in 17α-hydroxyprogesterone (Declercq *et al.*, 1972; refcode HPRGDO in the Cambridge Structural Database,-Version 5.25; Allen, 2002), and 16α,17-epoxy-4-pregnene-3,20-dione (refcode DILYEC; Goubitz *et al.*, 1984).

The title compound crystallizes with methanol in the space group $P2_12_12_1$. The unit-cell parameters are different in this respect from 16α,17-epoxy-4-pregnene-3,20-dione, which also crystallizes in $P2_12_12_1$. We found that the title compound could

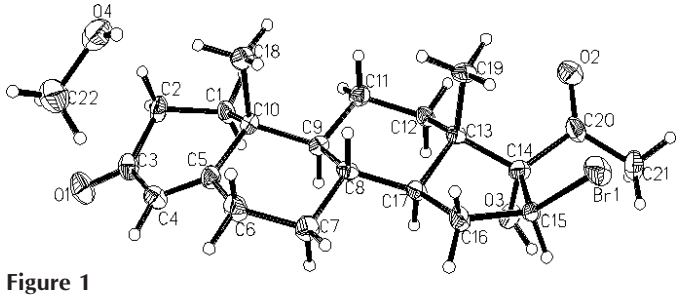


Figure 1
ORTEPII view (Johnson, 1976) of the title compound, shown with 30% probability displacement ellipsoids.

also crystallize in the monoclinic space group *C*2 from an acetone solution.

There is a hydrogen bond between O4 (methanol) and O1 (Table 1). The methanol molecule acts as a bridge linking two steroid molecules and makes a further hydrogen bond with O3 in another molecule.

Experimental

16β-Bromo-17α-hydroxypregn-4-ene-3,20-dione was prepared by the reaction of 16,17-epoxypregn-4-ene-3,20-dione and HBr in acetic acid, and purified by recrystallization. The melting point of 470.65 K was obtained by differential scanning calorimetry, which also showed a peak at 350.0 K due to loss of solvent. Colorless crystals suitable for X-ray diffraction were obtained by slow evaporation of the methanol solution in air.

Crystal data

$C_{21}H_{29}BrO_3\cdot CH_4O$
M_r = 441.39
Orthorhombic, $P2_12_12_1$
a = 12.008 (3) Å
b = 25.192 (6) Å
c = 7.0192 (12) Å
V = 2123.4 (8) Å^3
Z = 4
D_x = 1.381 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3797 reflections
θ = 2.3–25.7°
μ = 1.96 mm^{-1}
T = 293 (2) K
Block, colorless
0.40 × 0.38 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.421, T_{max} = 0.555
10 348 measured reflections
3705 independent reflections
2879 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 25.0°
$h = -6 \rightarrow 14$
$k = -29 \rightarrow 29$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.192
S = 1.05
3705 reflections
247 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2 + 10.8219P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.02 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}
Absolute structure: Flack (1983), 1151 Friedel pairs
Flack parameter = 0.08 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O1^{i}	0.82	2.02	2.757 (11)	150
O3—H3···O4^{ii}	0.85	1.98	2.832 (9)	180

Symmetry codes: (i) $x, y, z-1$; (ii) $x-1, y, z$.

H atoms were positioned geometrically (O—H = 0.85 and C—H = 0.93–0.98 Å) and refined as riding on the parent atom, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

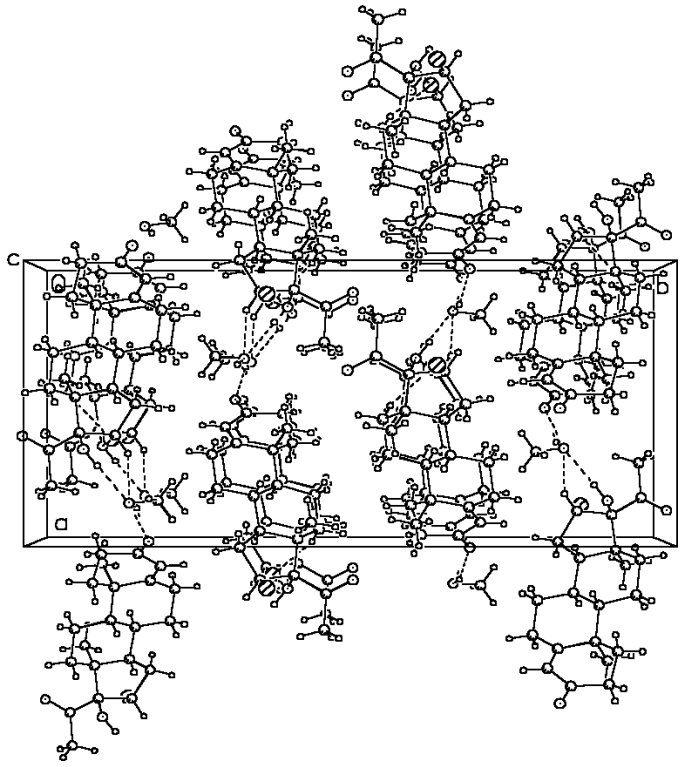


Figure 2
The molecular packing of the title compound, viewed along the *c* axis. Dashed lines indicate the intermolecular hydrogen-bonding interactions

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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