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

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.043
wR factor = 0.093
Data-to-parameter ratio = 14.5

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

The title compound, $C_{21}H_{29}BrO_3$, is a steroid used as an intermediate in medicine. The asymmetric unit consists of two molecules and the structure contains intermolecular hydrogen bonds.

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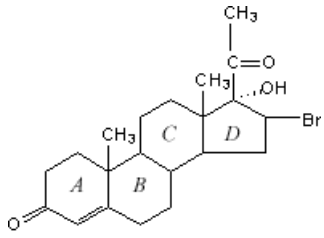
Comment

The title compound, (I), is a steroid derivative used as an intermediate in medicine, and was obtained from the reaction of 16,17-epoxypregn-4-ene-3,20-dione and HBr in acetic acid. In the present paper, we report the crystal structure of (I). 16,17-Epoxypregn-4-ene-3,20-dione does not contain heavy atoms, so it is difficult to determine the absolute configuration; this problem was solved by bromination of the molecule.



(I)

Ring A (see scheme) is in a 1α-sofa conformation, rings B and C are in chair conformations and ring D is in a 14α-envelope conformation (Fig. 1). The conformations are similar to those in 17α-hydroxyprogesterone (Declercq *et al.*, 1972). The title compound crystallizes in the monoclinic space group $C2$, different from 17α-hydroxyprogesterone, which crystallizes in the orthorhombic space group $P2_12_12_1$. The asymmetric unit consists of two molecules and the structure contains intermolecular hydrogen bonds (O3—H3···O4 and

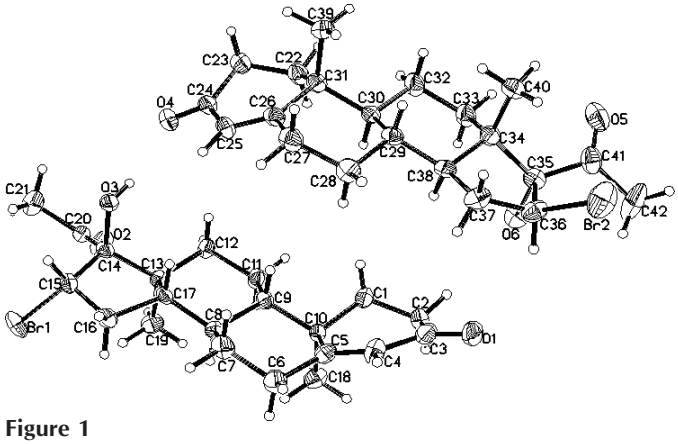


Figure 1
The asymmetric unit of the title compound, with displacement ellipsoids drawn at the 30% probability level.

O6—H6···O1^{i}; Table 1 and Fig. 2). The two molecules are slightly different in shape. For example, the distance between C3 and C21 is 11.146 (7) Å, while the distance between C24 and C42 is 10.971 (9) Å.

Experimental

The title compound (m.p. 469.25 K) was prepared by reaction of 16,17-epoxypregn-4-ene-3,20-dione and HBr in acetic acid. Colorless single crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution in acetone.

Crystal data

$C_{21}H_{29}BrO_3$
M_r = 409.35
Monoclinic, *C*2
a = 22.565 (7) Å
b = 9.393 (3) Å
c = 21.052 (7) Å
β = 115.682 (8)°
V = 4021 (2) Å^3
Z = 8
D_x = 1.352 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2865 reflections
θ = 2.4–21.6°
μ = 2.06 mm^{-1}
T = 293 (2) K
Block, colorless
0.24 × 0.18 × 0.14 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.605, T_{max} = 0.749
10528 measured reflections
6647 independent reflections
4103 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.0°
$h = -23 \rightarrow 26$
$k = -10 \rightarrow 11$
$l = -23 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.093
S = 1.00
6647 reflections
459 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}
Absolute structure: Flack (1983), 2214 Friedel pairs
Flack parameter = 0.012 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O1^{i}	0.82	2.04	2.836 (5)	163
O3—H3···O4	0.82	2.02	2.798 (5)	158

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

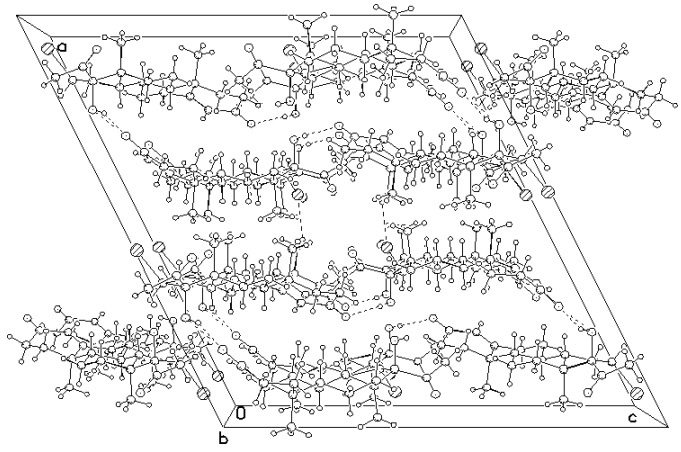


Figure 2
The molecular packing of the title compound, viewed along the *b* axis. Dashed lines indicate the intermolecular hydrogen-bonding interactions.

H atoms were positioned geometricall and treated as riding atoms, with O—H = 0.82 Å and C—H = 0.93–0.98 Å. U_{iso} values were set at $1.5U_{eq}$(parent atom) for H atoms on oxygen and of methyl groups or at $1.2U_{eq}$(parent atom) for H atoms on other C atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998) and *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXTL*.

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