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(5α,7α)-7-Hydroxy-4,4,7-trimethylcholestan-3-one

Huub Kooijman* and Anthony L. Spek

Bijvoet Center for Biomolecular Research, Department of Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands

Correspondence e-mail: h.kooijman@chem.uu.nl

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Key indicators

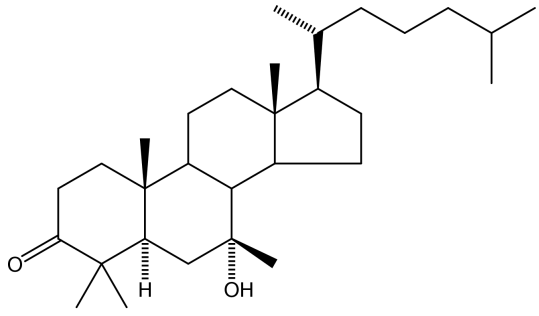
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.005 Å
R factor = 0.065
wR factor = 0.137
Data-to-parameter ratio = 10.8

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

The title compound, $C_{30}H_{52}O_2$, crystallizes with two molecules in the asymmetric unit. The independent molecules differ in the conformation of the side chain attached to C17. Hydrogen bonds link the molecules in an infinite chain, graph-set *C*(8), parallel to the *b* axis.

Comment

The structure determination of the title compound, (I), was carried out to confirm that the 7-hydroxy group is located on the α side of the steroid molecule (Bastiaansen *et al.*, 1996).



(I)

The asymmetric unit of the crystal structure contains two molecules of the title compound (Fig. 1). The crystallographically independent molecules differ only in the conformation of the aliphatic side chain attached to C17 (C57 in molecule 2) of the steroid skeleton. In molecule 1 (atoms C1–C30), the side chain is fully extended (all torsion angles *anti*), whereas in molecule 2 (atoms C51–C80), the torsion angles of the chain C63—C67—C70—C72—C73—C74—C75—C77 (subtract 50 from the numbers in these atom labels to obtain the standard cholesterol numbering scheme) adopt the conformations *anti*, +*gauche*, *anti*, −*gauche*, and *anti*.

The orientation of the hydroxyl group is identical in both molecules. Hydrogen bonds (Table 2) link the molecules into an infinite chain parallel to the *b* axis. The chain can be described with graph-set symbol *C*(8) (Bernstein *et al.*, 1995).

Experimental

The synthesis of the title compound is described by Bastiaansen *et al.* (1996). Crystals were obtained from *n*-hexane.

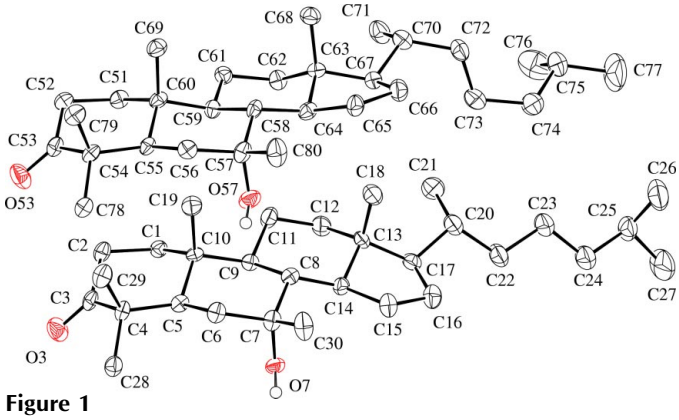


Figure 1
Displacement-ellipsoid plot of the title compound drawn at the 50% probability level (Spek, 2001). H atoms have an arbitrary radius.

Crystal data

$C_{30}H_{52}O_2$
$M_r = 444.72$
Monoclinic, $P2_1$
$a = 11.9017$ (9) Å
$b = 12.0284$ (10) Å
$c = 18.8388$ (11) Å
$\beta = 92.184$ (5)°
$V = 2695.0$ (3) Å^3
$Z = 4$
$D_x = 1.096$ Mg m^{-3}
MoKa radiation
Cell parameters from 25 reflections
$\theta = 10.1$–13.9°
$\mu = 0.07$ mm^{-1}
$T = 150$ K
Block, colourless
0.50 × 0.50 × 0.20 mm

Data collection

Enraf–Nonius CAD-4-Turbo diffractometer
ω scans with $\Delta\omega = 0.67 + 0.35\tan\theta$
7483 measured reflections
6469 independent reflections
4258 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 15$
$k = 0 \rightarrow 15$
$l = -24 \rightarrow 24$
3 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.137$
$S = 1.04$
6469 reflections
599 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.15P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O3—C3	1.215 (5)	O53—C53	1.213 (5)
O7—C7	1.436 (4)	O57—C57	1.446 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7D···O53^{i}	0.82 (5)	2.06 (5)	2.883 (4)	176 (4)
O57—H57D···O3^{i}	0.82 (5)	2.14 (5)	2.949 (4)	171 (5)

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

Due to a lack of significant anomalous scatterers, the absolute configuration could not be determined *ab initio*. The chirality was chosen in agreement with the well known stereochemistry of the cholesterol skeleton. Only one Friedel pair was measured; these Friedel opposites were merged. The hydroxyl-H atoms were located in a difference Fourier map and their coordinates were included as parameters in the refinement. The idealized H atoms of each methyl moiety were included in rigid groups and were allowed to rotate around the C—C bond. All other H atoms were included at calculated positions, riding on their carrier atoms. Isotropic displacement parameters of the H atoms were coupled to the equivalent isotropic displacement parameter of their carrier atoms.

Data collection: Locally modified *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SET*4 (de Boer & Duisenberg, 1984); data reduction: *HELENA* (Spek, 1997); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001); software used to prepare material for publication: *PLATON*.

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