Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl *N*′-(17*β*-hydroxyandrost-1-en-3-ylidene)-hydrazinedithiocarboxylate

Li-Ming Zhang, Wei-Xiao Hu* and Chun-Nian Xia

College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou 310032, People's Republic of China

Correspondence e-mail: huyang@mail.hz.zj.cn

Key indicators

Single-crystal X-ray study
T = 296 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.108
Data-to-parameter ratio = 20.3

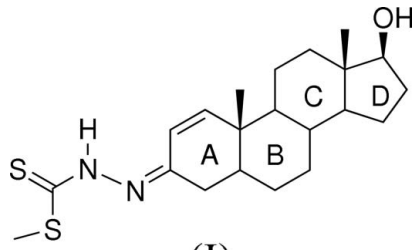
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 28 March 2007
Accepted 4 April 2007

In the title compound, $C_{21}H_{32}N_2OS_2$, the cyclohexene and cyclopentane rings adopt envelope conformations, while the cyclohexane rings are in chair conformations. The molecules are linked into a zigzag chain along the *c* axis by intermolecular N—H···O hydrogen bonds.

Comment

17*β*-Hydroxy-5*α*-androst-1-en-3-one (1-testosterone) is a potent androgen with anabolic properties (Friedel *et al.*, 2006). Thiosemicarbazone and its derivatives have attracted considerable pharmaceutical interest owing to their antiviral, antibacterial and antitumor activites (Hu *et al.*, 2006). As part of our ongoing research on thiosemicarbazones, we present here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The Csp^3—Csp^3 bond lengths show quite a scatter, from 1.519 (3) to 1.554 (3) Å. The shortness of the C1—C2 and C3—N1 bonds reflects their double-bond character, being conjugate to one another. The bond distances (Table 1) are in close agreement with those in a similar steroid structure (Rohrer *et al.*, 1979).

Ring A adopts an envelope conformation; atoms C1, C2, C3, C4 and C10 form a plane with a maximum deviation of 0.041 (2) Å, and atom C5 deviates from the plane by 0.670 (4) Å. Rings B and C have normal chair conformations. Ring D also shows an envelope conformation; atoms C14, C15, C16 and C17 form a plane with a maximum deviation of 0.052 (2) Å, and atom C13 deviates from the plane by 0.680 (4) Å. Atoms S1, S2, N2, C20 and C21 are coplanar.

The crystal packing shows that the molecules are linked into a zigzag chain along the *c* axis by intermolecular N—H···O hydrogen bonds (Fig. 2).

Experimental

A mixture of 1-testosterone (2.7 g, 10 mmol) and methyl hydrazinecarbodithioate (1.2 g, mmol) was dissolved in absolute

ethanol (10 ml) and the solution was refluxed for 3 h, monitored by TLC. After cooling, the precipitated yellow compound was filtered off to give 2.9 g (74%) of crude product. The crude product was recrystallized from absolute ethanol to give the pure product (m.p. 533–535 K). Colourless prism-shaped crystals of (I) were obtained by slow evaporation of a mixture of tetrahydrofuran, acetone and water (*v*/*v* 4:4:2).

Crystal data

$C_{21}H_{32}N_2OS_2$
M_r = 392.61
Orthorhombic, $P2_12_12_1$
a = 9.8693 (10) Å
b = 11.7844 (12) Å
c = 18.0029 (18) Å
V = 2093.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 296 (2) K
0.30 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.924, T_{max} = 0.939
13545 measured reflections
4850 independent reflections
4085 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.108
S = 1.06
4850 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 2031 Friedel pairs
Flack parameter: −0.04 (7)

Table 1
Selected geometric parameters (Å, °).

S1—C20	1.738 (2)	N1—C3	1.291 (2)
S1—C21	1.793 (2)	N1—N2	1.384 (2)
S2—C20	1.654 (2)	N2—C20	1.340 (2)
O1—C17	1.425 (3)	C1—C2	1.330 (3)
N2—C20—S2	120.84 (15)	S2—C20—S1	125.19 (12)
N2—C20—S1	113.96 (14)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*N*···O1^{i}	0.86	2.31	2.937 (3)	130

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

H atoms were placed in calculated positions, with O—H = 0.82 Å, N—H = 0.86 Å and C—H = 0.93–0.98 Å. The U_{iso} values were set at $1.5U_{eq}$ of the carrier atom for hydroxyl and methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the hydroxyl and methyl groups.

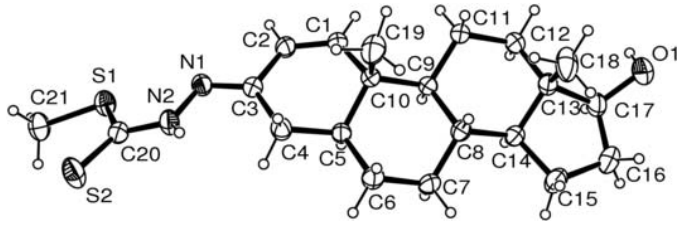


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

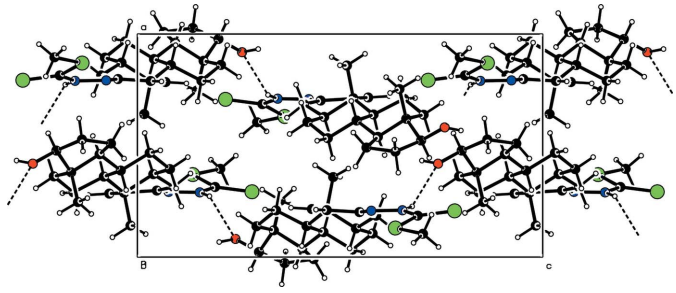


Figure 2
Packing diagram of (I), viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors acknowledge the National Natural and Scientific Foundation (grant No. 20272053) and the Science and Technology Bureau of Zhejiang Province (grant No. 2005 C23022) for financial support.

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