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3-Hydroxy-17-oxoestra-3,5-dien-3-yl acetate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.009 Å
R factor = 0.050
wR factor = 0.229
Data-to-parameter ratio = 8.6

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

In the title steroid compound, $C_{20}H_{26}O_3$, rings *A* and *B* have screw boat conformations, ring *C* has a regular chair conformation and ring *D* approximates to a distorted half-chair conformation. The molecules are connected *via* C—H···O hydrogen bonds, generating a two-dimensional network.

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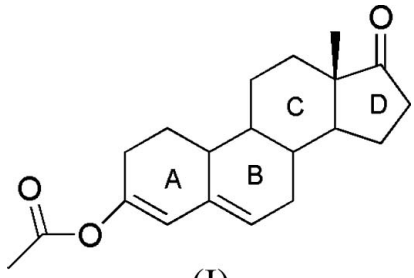
Comment

3-Hydroxyestra-3,5-dien-17-one and its derivatives are common components of widely used hormone regulation and therapy medications (Ekhato *et al.*, 2002).



(I)

In the title compound, (I), rings *A* and *B* both have screw boat conformations (Fig. 1). In ring *A*, atoms C1, C2, C3 and C4 form a plane within a deviation of 0.057 (5) Å, while atoms C5 and C10 lie on the same side of the plane with deviations of 0.354 (12) and 0.819 (12) Å, respectively. In ring *B*, atoms C5, C6, C9 and C10 form a plane within a deviation of 0.052 (4) Å, while atoms C7 and C8 lie on the same side of the plane with deviations of 0.286 (12) and 0.794 (11) Å, respectively. Ring *C* has a normal chair conformation, and atoms C8, C11, C12 and C14 are coplanar within a deviation of 0.023 (3) Å. Ring *D* adopts a distorted half-chair conformation similar to that observed in related compounds (Galdecki, 1989; Andrade *et al.*, 2003). Atoms C13, C16, C17 and O2 are coplanar within a deviation of 0.004 (6) Å, and atoms C14 and C15 deviate from

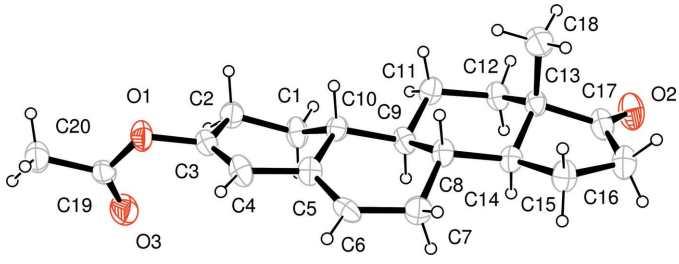


Figure 1
The structure of (I), with 30% probability displacement ellipsoids.

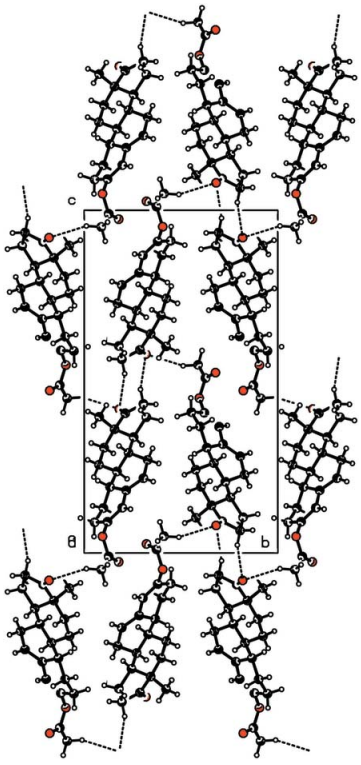


Figure 2
A packing diagram for (I), viewed along the *a* axis, showing the hydrogen bonds as dashed lines.

the plane by 0.806 (10) and −0.258 (12) Å, respectively.

The crystal packing shows that hydrogen bonds generate a two-dimensional network (Fig. 2). C16—H16*A*···O2^{i} and C20—H20*C*···O2^{ii} hydrogen bonds (Table 2; symmetry codes as in Table 2) link two molecules head-to-head and head-to-tail, respectively.

Experimental

3-Hydroxy-17-oxoestra-3,5-dien-3-yl acetate, in the form of a yellow powder, synthesized according to the method of Ekhato *et al.* (2002), was kindly supplied by Mr Pan of Jiubang Chemical Corporation Ltd., Shanghai, China. Crystals of (I) suitable for structure analysis were obtained as colourless plates by slow evaporation of a solution in a mixture of ethanol, dioxane and water (2:2:1).

Crystal data

$C_{20}H_{26}O_3$
M_r = 314.41
Orthorhombic, $P2_12_12_1$
a = 6.3340 (16) Å
b = 12.384 (3) Å
c = 21.911 (6) Å
V = 1718.7 (8) Å^3
Z = 4
D_x = 1.215 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.7–12.5°
μ = 0.08 mm^{-1}
T = 295 (2) K
Plate, colourless
0.40 × 0.35 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.968, T_{max} = 0.998
1910 measured reflections
1811 independent reflections
941 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.2°
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 14$
$l = -1 \rightarrow 26$
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.229
S = 1.04
1811 reflections
211 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1479P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C19	1.354 (8)	O3—C19	1.198 (8)
O1—C3	1.406 (7)	C3—C4	1.323 (9)
O2—C17	1.229 (8)	C5—C6	1.359 (9)
C2—C3—C4—C5	4.9 (10)	C14—C15—C16—C17	16.3 (7)
C3—C4—C5—C10	10.7 (9)	C12—C13—C17—O2	31.4 (10)
C10—C5—C6—C7	3.1 (10)	C3—O1—C19—O3	2.5 (9)
C17—C13—C14—C15	42.6 (6)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16*A*···O2^{i}	0.97	2.52	3.443 (8)	158
C20—H20*C*···O2^{ii}	0.96	2.58	3.453 (9)	152

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

Because of negligible anomalous scattering effects, Friedel pairs were averaged before the refinement. The absolute configuration of compound (I) was known from the synthetic route (Ekhato *et al.*, 2002). H atoms were placed in calculated positions and refined using a riding model, with C—H distances in the range 0.96–0.97 Å and with U_{iso}(H) = 1.2U_{eq}(parent atom) or 1.5U_{eq}(C$_{methyl}$).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *CAD-4 EXPRESS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97*.

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