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17a-Methyl-4-androstene-3b,17b-diol monohydrate

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In the title compound, $C_{20}H_{32}O_2{\cdot}H_2O$, ring *A* is a slightly distorted half-chair, rings *B* and *C* have regular chair conformations, and ring *D* approximates to an envelope form. Molecules are connected *via* O—H···O hydrogen bonds, generating a three-dimensional network.

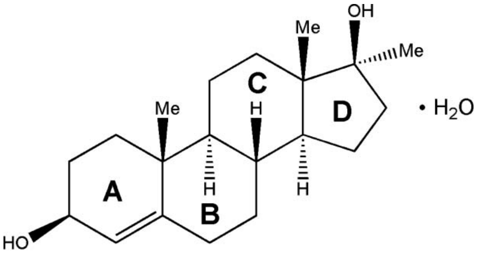
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.096
Data-to-parameter ratio = 8.9

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

Comment

Many 17a-alkyl anabolic steroids have a reduced androgenic side effect (Stanley *et al.*, 1997). Methylandrostenediol can increase the concentration of corticotrophin-releasing factor in the hypothalamus (Paducheva *et al.*, 1969). The molecular structure of the title compound, (I), incorporating one water molecule of crystallization, is illustrated in Fig. 1. Selected bond lengths and angles are listed in Table 1. The C*sp*^{3}—C*sp*^{3} bond lengths show quite a scatter, from 1.483 (3) to 1.561 (3) Å. The C4═C5 distance in ring *A* is 1.323 (3) Å, and the O1—C3 and O2—C17 distances are 1.437 (3) and 1.425 (3) Å, respectively. All these distances are in close agreement with those in similar steroid structures (David & James, 1974; Grochulski & Wawrzak, 1989).



(I)

Bucourt & Hainaut (1965) noted that only the half-chair form among various conformations of cyclohexenes corresponds to the true energy minimum. In (I), ring *A* shows a half-

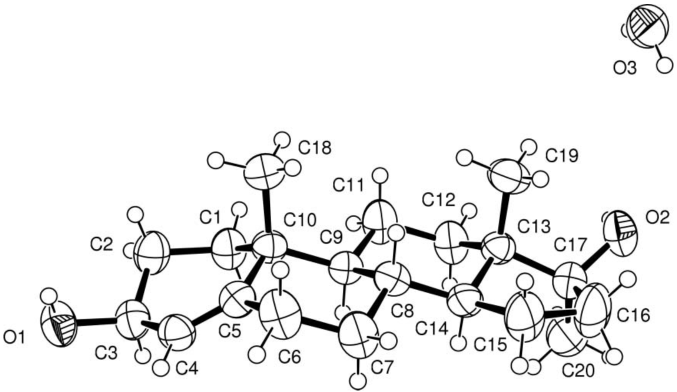


Figure 1
The structure of (I), shown with 50% probability displacement ellipsoids.

chair conformation, which is similar to that reported by Bucourt & Hainaut (1965). In ring *A*, atoms C3/C4/C5/C10 form a good plane [maximum deviation = 0.014 (2) Å], while atoms C1 and C2 lie on opposite sides of the plane, with deviations of 0.288 (4) and 0.476 (5) Å, respectively. Rings *B* and *C* have normal chair conformations. Ring *D* shows an envelope conformation. Atoms C14–C17 deviate from the least-squares plane to a maximum of 0.052 (2) Å, and atom C13 deviates from the plane by 0.656 (4) Å. In the crystal packing, hydrogen bonds involving the water molecule generate a three-dimensional network (Fig. 2 and Table 2).

Experimental

17*a*-Methyl-4-androstene-3*b*,17*b*-diol in the form of a powder, synthesized according to Bernstein *et al.* (1957), was kindly supplied by Mr Pan of Jiubang Chemistry Corporation Ltd, Shanghai, China. Crystals of (I) suitable for structure analysis were obtained by slow evaporation of a solution in tetrahydrofuran, acetone and water (4:4:2) as colourless plates.

Crystal data

$C_{20}H_{32}O_2{\cdot}H_2O$
$M_r = 322.47$
Orthorhombic, $P2_12_12_1$
$a = 6.812$ (3) Å
$b = 12.082$ (5) Å
$c = 22.802$ (3) Å
$V = 1876.7$ (12) Å^3
$Z = 4$
$D_x = 1.141$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.7$–$12.5°$
$\mu = 0.07$ mm^{-1}
$T = 298$ (2) K
Parallelepiped, colourless
$0.50 \times 0.40 \times 0.30$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.944$, $T_{\max} = 0.978$
2283 measured reflections
1957 independent reflections
1440 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.013$
$\theta_{\max} = 25.2°$
$h = -1 \rightarrow 8$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 27$
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.096$
$S = 1.02$
1957 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.2016P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.12$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.437 (3)	C3—C4	1.483 (4)
O2—C17	1.425 (3)	C4—C5	1.323 (3)
C2—C3—C4—C5	−17.1 (3)	C13—C14—C15—C16	−33.2 (3)
C3—C4—C5—C10	−3.4 (4)	C14—C15—C16—C17	8.8 (3)
C4—C5—C10—C1	−9.0 (3)	C15—C16—C17—C13	18.5 (3)
C17—C13—C14—C15	44.6 (2)	C14—C13—C17—C16	−38.3 (2)

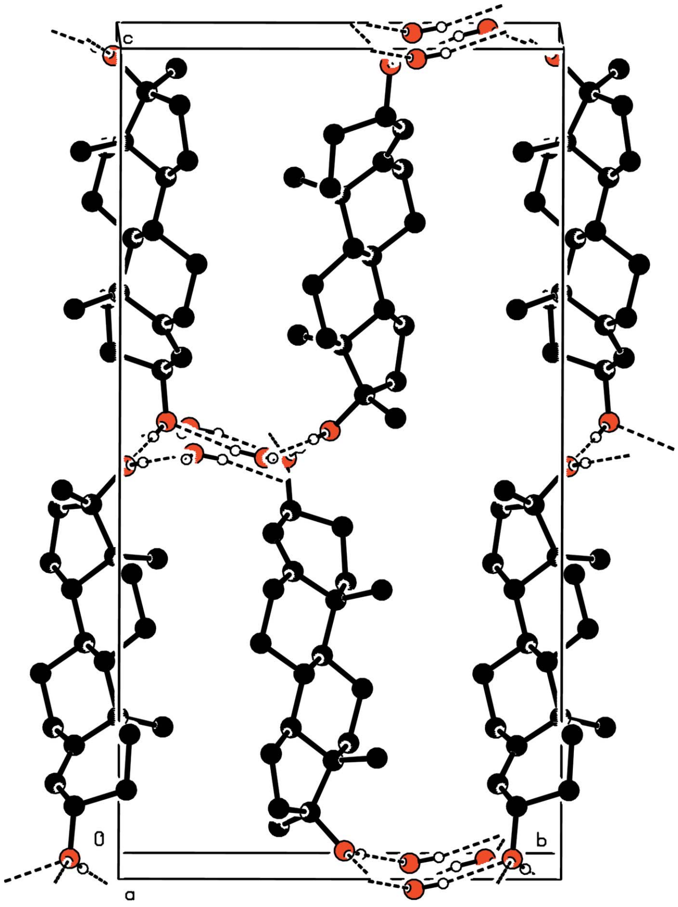


Figure 2
Packing diagram of (I), viewed along the *a* axis, showing hydrogen bonds as dashed lines. H atoms have been omitted except for those involved in hydrogen bonding.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2$^{\rm i}$	0.82	1.87	2.684 (3)	174
O2—H2···O3$^{\rm ii}$	0.82	1.90	2.711 (3)	169
O3—H3*X*···O1$^{\rm iii}$	0.83 (1)	2.09 (1)	2.916 (3)	171 (5)
O3—H3*Y*···O1$^{\rm iv}$	0.83 (1)	1.99 (2)	2.781 (3)	160 (4)

Symmetry codes: (i) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z$; (iii) $-x+\frac{1}{2}, -y+1, z-\frac{1}{2}$; (iv) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Owing to negligible anomalous scattering effects, Friedel pairs were averaged in the refinement. The absolute stereochemistry of the compound (I) was known from the synthetic route (Bernstein *et al.*, 1957). H atoms bonded to the O atoms were located in difference Fourier maps; those of water molecule were refined isotropically with the restraint O—H = 0.83 (1) Å. The H atoms of the hydroxy groups were refined using a riding model, with O—H = 0.82 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm O})$. H atoms bonded to C atoms were placed at calculated positions and refined using a riding model with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ [or $1.5U_{\rm eq}$(methyl C)], with C—H distances of 0.96–0.97 Å.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1994); cell refinement: *CAD-4 Software*; data reduction: *XCAD* (McArdle, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick,

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1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97*.

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