Triclinic
$P\bar{1}$
a = 8.608 (3) Å
b = 10.393 (4) Å
c = 12.455 (5) Å
α = 109.36 (4)°
β = 102.24 (3)°
γ = 92.10 (3)°
V = 1020.4 (7) Å^3
Z = 2
D_x = 1.394 Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
θ = 12–21°
μ = 0.106 mm^{-1}
T = 293 (2) K
Prism
0.3 × 0.2 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4261 measured reflections
4219 independent reflections
3636 reflections with $I > 2\sigma(I)$

R_{int} = 0.008
θ_{max} = 29.96°
$h = -12 \rightarrow 11$
$k = -14 \rightarrow 13$
$l = 0 \rightarrow 17$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.141
S = 1.044
4219 reflections
376 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.2731P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.482 e Å^{-3}
$\Delta\rho_{min}$ = −0.210 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O3—C8	1.433 (3)	O5—C4	1.436 (3)
O4—C9	1.446 (3)	C8—C9	1.476 (4)
C17—O6—C21	116.27 (18)	C5—C12—C13	123.38 (18)
C18—O7—C22	113.57 (19)	O6—C17—C18	116.40 (17)
C19—O8—C23	118.33 (18)	O6—C17—C16	123.46 (18)
O1—C1—C14	130.3 (2)	O7—C18—C17	120.15 (18)
C12—C5—C4	123.17 (18)	O7—C18—C19	120.20 (18)
O3—C7—C10	121.45 (19)	O8—C19—C18	115.67 (17)
O4—C10—C7	122.45 (18)	O8—C19—C20	123.95 (18)
C2—O2—C1—C14	−1.8 (3)	O3—C7—C10—O4	−0.4 (3)
C1—O2—C2—C3	21.1 (3)	C4—C5—C12—C13	3.4 (3)
C2—C3—C4—O5	48.1 (3)	C5—C12—C13—C14	−17.8 (2)
C3—C4—C5—C12	−19.7 (3)	C11—C12—C13—C15	−72.3 (2)
C8—O3—C7—C10	−14.3 (3)	O2—C1—C14—C3	−18.5 (2)
C9—O4—C10—C7	−16.8 (3)	C15—C13—C14—C1	48.4 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5O···O3^{i}	0.87 (4)	1.92 (3)	2.770 (3)	166 (3)

Symmetry code: (i) $-x, -1-y, -z$.

The positions of all H atoms were located from a difference map and their atomic coordinates and isotropic displacement parameters were refined.

Data collection: *CAD-4/PC* (Kretschmar, 1996). Cell refinement: *CAD-4/PC*. Data reduction: *CFEO* (Solans, 1978). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *ORTEP* (Brueggemann & Schmid, 1990). Software used to prepare material for publication: *PLATON* (Spek, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1046). Services for accessing these data are described at the back of the journal.

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Androst-4-ene-3,6,17-trione

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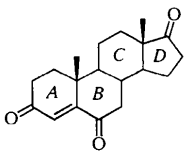
Abstract

The title steroid, $C_{19}H_{24}O_3$, has flattened *A* and *B* rings and a 14α *D*-ring conformation. The crystal structure is stabilized by numerous C—H···O hydrogen bonds.

Comment

Steroids with a Δ^4-3,6-dione functional group occur naturally (Tischler *et al.*, 1988) and are known to be potent inhibitors of aromatase, an enzyme that is the target for curing oestrogen-dependent carcinoma and in the modulation of reproductive processes. Androst-4-ene-3,6,17-trione (AT) is a synthetic androgen and a suicide substrate for aromatase (Covey & Hood,

1981; Numazawa & Tachibana, 1994, and references therein). We synthesized AT as a precusor to *B*-ring functionalized steroids (Nangia & Anthony, 1996; Anthony *et al.*, 1998*a*). The X-ray crystal structure of AT is reported here.



AT

The *ORTEP*II (Johnson, 1976) plot of AT is shown in Fig. 1. The conjugation from the carbonyl-C3 to the carbonyl-C6 atom flattens the *A* and *B* rings. The weighted mean absolute torsion angles in rings *A*, *B* and *C* are 28.3 (1), 48.4 (1) and 55.3 (1)°, respectively. The *D* ring is in the 14α envelope conformation (Duax & Norton, 1975). The C4═C5 bond is in a stronger conjugation with O3 than with O6, as evidenced by the bond distances [C3═O3 1.217 (3), C3—C4 1.452 (4), C4═C5 1.334 (3), C5—C6 1.502 (3), C6═O6 1.215 (3) Å], and this is similar to the situation observed in the C17-dioxolane derivative of AT (Anthony *et al.*, 1998*a*). The overlay plot of the tetracyclic steroid skeleton in AT with that of the 17-ethylenedioxy derivative has an overall r.m.s. deviation of 0.112 Å between the common atoms.

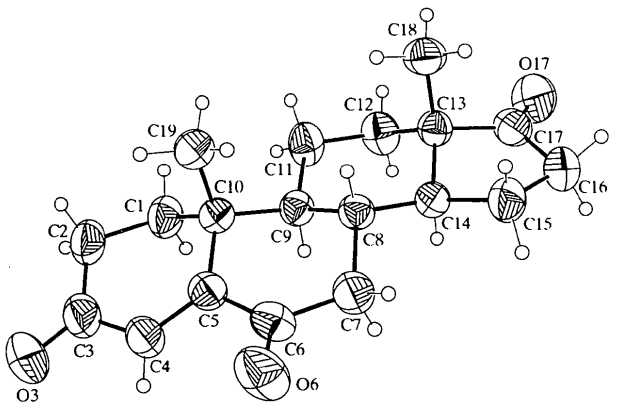


Fig. 1. *ORTEP*II (Johnson, 1976) plot of a molecule of AT. Displacement ellipsoids are drawn at the 50% probability level, except for H atoms which are drawn as spheres of an arbitrary radius.

The crystal structure of AT is stabilized by numerous C—H···O hydrogen bonds. Since the molecule has an abundance of C—H donors, the carbonyl-O atoms are bonded to multiple donor groups (Desiraju *et al.*, 1993). Thus, based on an H···O distance cut-off criterion of 2.8 Å (Desiraju, 1996), O6 is a twofold acceptor (C1—H···O 2.68 and C2—H···O 2.74 Å) and O17 is a fourfold acceptor (C2—H···O 2.50, C16—H···O 2.67, C18—H···O 2.61 and C19—H···O 2.71 Å), while O3 is linked to a single donor atom (C16—H···O 2.63 Å); see Fig. 2 and Table 1. It should be noted that the H atoms involved in hydrogen bonding are either from methylene groups adjacent to a carbonyl group, and as such are activated (Pedireddi & Desiraju, 1992), or are methyl-H atoms on the β face and hence sterically accessible for hydrogen bonding.

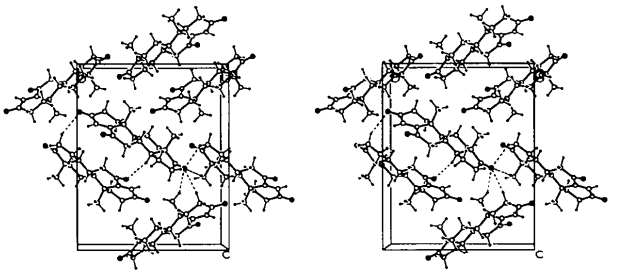


Fig. 2. Stereoview of the packing diagram of AT down [100], showing the C—H···O hydrogen bonds. Notice that O17 is a fourfold acceptor, O6 is bifurcated (the second interaction with a molecule translated along [100] is not shown) and O3 is bonded to a single donor atom.

The structure of AT is similar to that of androst-4-ene-3,17-dione reported by Busetta *et al.* (1972). The conformations of the two steroids are identical (overall r.m.s. deviation 0.090 Å). The unit cell similarity index Π is 0.0015 and the isostructurality index I_D^7 is 93.2% (Kálmán & Párkányi, 1997; Anthony *et al.*, 1998*b*).

With the increasing awareness of the role of weak hydrogen bonds in ligand–receptor complexes (Wahl & Sundaralingam, 1997), and in view of the opinion that the approach geometry of hydrogen-bonding donor and acceptor groups in these complexes can be derived from the ligand crystal structures (Klebe, 1994), the crystal structures of steroids stabilized by C—H···O hydrogen bonding are a source of valuable information.

Experimental

The title compound was prepared according to the procedure described by Nangia & Anthony (1996) and recrystallized from an EtOAc/hexane solvent mixture.

Crystal data

$C_{19}H_{24}O_3$
M_r = 300.38
Orthorhombic
$P2_12_12_1$
a = 7.2842 (6) Å
b = 13.450 (1) Å
c = 16.503 (2) Å
V = 1616.8 (3) Å^3
Z = 4
D_x = 1.234 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 52 reflections
θ = 16–84°
μ = 0.652 mm^{-1}
T = 293 (2) K
Prism
0.5 × 0.4 × 0.3 mm
Light yellow

Data collection

Kuma KM-4 diffractometer	$\theta_{max} = 68.10°$
$\omega/2\theta$ scans	$h = 0 \rightarrow 8$
Absorption correction: none	$k = 0 \rightarrow 16$
1603 measured reflections	$l = 0 \rightarrow 19$
1603 independent reflections	2 standard reflections
1436 reflections with $I > 2\sigma(I)$	every 100 reflections
	intensity decay: <2.1%

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.169$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.035$	$\Delta\rho_{min} = -0.166$ e Å^{-3}
$wR(F^2) = 0.101$	Extinction correction: *SHELX*97 (Sheldrick, 1997)
$S = 1.010$	Extinction coefficient: 0.0053 (7)
1603 reflections	Scattering factors from *International Tables for Crystallography* (Vol. C)
200 parameters	
H atoms fixed	
$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 0.2880P]$ where $P = (F_o^2 + 2F_c^2)/3$	
$(\Delta/\sigma)_{max} < 0.001$	

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H12···O6^{i}	0.97	2.68	3.340 (3)	125
C2—H21···O17^{ii}	0.97	2.50	3.464 (3)	172
C2—H22···O6^{i}	0.97	2.74	3.409 (4)	127
C16—H161···O17^{iii}	0.97	2.67	3.434 (4)	136
C16—H162···O3^{iv}	0.97	2.63	3.525 (4)	154
C18—H181···O17^{iii}	0.96	2.61	3.514 (4)	156
C19—H192···O17^{ii}	0.96	2.71	3.515 (4)	141

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

Data collection: *KM-4 Software* (Kuma Diffraction, 1989). Cell refinement: *KM-4 Software*. Data reduction: *KM-4 Software*. Program(s) used to solve structure: *SHELX*97 (Sheldrick, 1997). Program(s) used to refine structure: *SHELX*97. Molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELX*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1312). Services for accessing these data are described at the back of the journal.

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1,11-Undecanediol

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Abstract

The crystal structure analysis of 1,11-undecanediol, $C_{11}H_{24}O_2$, has been carried out by X-ray diffraction. The hydrocarbon skeleton has an all-*trans* conformation. One of the hydroxyl groups, which are located at both ends of the hydrocarbon chain, has a *gauche* conformation with respect to the skeleton, whereas the other has a *trans* conformation. The molecules lie parallel to the *b* axis and layers are formed with a thickness of *b*/2. The molecules are arranged in an antiparallel fashion along the *a* axis in these layers. These features are similar to those of the homologues with an odd number of C atoms, but different from those with an even number.

Comment

Generally, long-chain aliphatic compounds have been studied as basic models of polymers. These compounds