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5α-Androst-2-en-17-one

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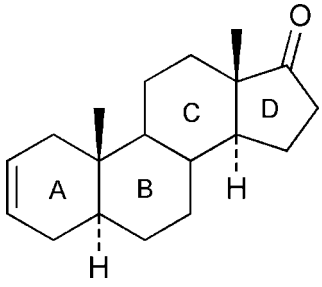
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.122; data-to-parameter ratio = 9.1.

The title compound, $C_{19}H_{28}O$, is a useful intermediate for the synthesis of steroidal compounds. The molecular structure has normal bond lengths and angles. The crystal packing is stabilized by van der Waals forces.

Related literature

For related literature, see: Allen *et al.* (1987); Ambrogio & Paride (2000); D'Onofrio & Scettri (1985); Duax *et al.* (1976); Hanson *et al.* (1999).



Experimental

Crystal data

$C_{19}H_{28}O$
M_r = 272.41
Orthorhombic, $P2_12_12_1$
a = 6.4725 (17) Å
b = 9.003 (2) Å
c = 27.190 (7) Å
V = 1584.4 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
0.41 × 0.38 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.973, T_{max} = 0.993
8053 measured reflections
1650 independent reflections
1404 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.122
S = 1.09
1650 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2261).

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supplementary materials

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5α-Androst-2-en-17-one

D. Zhang and J. Liu

Comment

Steroids occur widely in body tissues and have a large variety of physiological activities. Androsterone, a steroidal hormone, plays an important role in the development of male secondary sex characteristics during puberty and for promoting tissue and muscle growth. After elimination of the hydroxyl group at the 3-position of androsterone, 5α-androst-2-en-17-one was obtained as a useful intermediate for the synthesis of steroidal compounds. (D'Onofrio & Scettri., 1985; Hanson *et al.*, 1999). In this paper, we report the crystal structure of the title compound, (I).

In (I) (Fig. 1), all bond lengths and angles are normal (Allen *et al.*, 1987) and in a good agreement with those reported previously (Duax *et al.*, 1976). The values of torsion angles indicate that the A rings have halfchair conformations, the B and C rings have chair conformations, and the D rings have envelope conformations. The crystal packing is stabilized by van der Waals forces.

Experimental

Androsterone (17 mmol) was protected with *p*-toluene sulfonyl chloride in a solution of pyridine (25 ml)(Ambrogio & Paride, 2000). After aqueous work up, the white solid (16.8 mmol) obtained was dissolved in pyridine (20 ml) and the resulting solution was refluxed for 6 h. The reaction mixture was concentrated, diluted with 20 mL of 10% sulfuric acid solution and kept in the refrigerator overnight. The pale yellow solid was collected by vacuum filtration and washed with water. After crystallized in 95% ethanol, the title compound was obtained in 75% yield. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution in a hexane/ethanol mixture (1: 5 *v*/*v*) at room temperature over a period of one week.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2(1.5 for methyl groups) times U_{eq}(C).

Figures

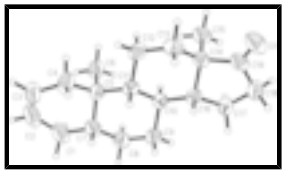


Fig. 1. The molecular structure of (I), with atom labels and 40% probability displacement ellipsoids for non-H atoms.

5α-androst-2-en-17-one

Crystal data

$C_{19}H_{28}O$	F_{000} = 600
M_r = 272.41	D_x = 1.142 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 2794 reflections
a = 6.4725 (17) Å	θ = 2.6–24.3º
b = 9.003 (2) Å	μ = 0.07 mm^{-1}
c = 27.190 (7) Å	T = 298 (2) K
V = 1584.4 (7) Å^3	Block, colourless
Z = 4	0.41 × 0.38 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1650 independent reflections
Radiation source: fine-focus sealed tube	1404 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.033
T = 293(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	h = −7→6
T_{min} = 0.973, T_{max} = 0.993	k = −9→10
8053 measured reflections	l = −32→32

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.048	H-atom parameters constrained
$wR(F^2)$ = 0.122	$w = 1/[\sigma^2(F_o^2) + (0.0591P)^2 + 0.212P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.09	$(\Delta/\sigma)_{max} < 0.001$
1650 reflections	$\Delta\rho_{max}$ = 0.11 e Å^{-3}
181 parameters	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3020 (3)	0.3005 (3)	−0.01939 (8)	0.0820 (7)
C1	0.8111 (6)	0.8641 (4)	0.22591 (13)	0.0882 (11)
H1A	0.6947	0.8777	0.2479	0.106*
H1B	0.9281	0.8329	0.2457	0.106*
C2	0.8603 (6)	1.0073 (5)	0.2034 (2)	0.1112 (16)
H2A	0.9097	1.0830	0.2235	0.133*
C3	0.8382 (6)	1.0346 (4)	0.15602 (19)	0.0961 (13)
H3A	0.8738	1.1278	0.1439	0.115*
C4	0.7583 (5)	0.9208 (4)	0.12144 (13)	0.0765 (9)
H4A	0.8741	0.8755	0.1045	0.092*
H4B	0.6731	0.9695	0.0969	0.092*
C5	0.6312 (4)	0.7985 (3)	0.14607 (10)	0.0518 (7)
C6	0.4268 (5)	0.8654 (4)	0.16355 (11)	0.0669 (8)
H6A	0.4542	0.9454	0.1860	0.100*
H6B	0.3468	0.7904	0.1799	0.100*
H6C	0.3512	0.9025	0.1358	0.100*
C7	0.7587 (5)	0.7401 (3)	0.18982 (11)	0.0627 (8)
H7A	0.8906	0.7062	0.1761	0.075*
C8	0.6623 (6)	0.6060 (4)	0.21451 (10)	0.0720 (9)
H8A	0.5322	0.6347	0.2295	0.086*
H8B	0.7532	0.5708	0.2404	0.086*
C9	0.6243 (5)	0.4813 (3)	0.17781 (10)	0.0651 (8)
H9A	0.5555	0.3996	0.1943	0.078*
H9B	0.7558	0.4453	0.1655	0.078*
C10	0.4920 (4)	0.5335 (3)	0.13471 (8)	0.0470 (6)
H10A	0.3568	0.5639	0.1474	0.056*
C11	0.5946 (4)	0.6690 (3)	0.11010 (9)	0.0471 (6)
H11A	0.7325	0.6354	0.1002	0.057*
C12	0.4858 (5)	0.7149 (3)	0.06213 (10)	0.0626 (8)
H12A	0.3522	0.7574	0.0701	0.075*
H12B	0.5671	0.7912	0.0460	0.075*
C13	0.4550 (5)	0.5864 (4)	0.02671 (9)	0.0649 (8)
H13A	0.3785	0.6201	−0.0019	0.078*
H13B	0.5882	0.5499	0.0157	0.078*
C14	0.3375 (4)	0.4628 (3)	0.05196 (9)	0.0526 (7)
C15	0.1143 (4)	0.5095 (4)	0.06359 (11)	0.0693 (9)
H15A	0.0468	0.5403	0.0338	0.104*
H15B	0.1156	0.5904	0.0866	0.104*

H15C	0.0411	0.4270	0.0776	0.104*
C16	0.4598 (4)	0.4119 (3)	0.09699 (9)	0.0492 (6)
H16A	0.5976	0.3852	0.0850	0.059*
C17	0.3560 (5)	0.2663 (3)	0.11169 (11)	0.0661 (8)
H17A	0.2301	0.2842	0.1301	0.079*
H17B	0.4480	0.2050	0.1312	0.079*
C18	0.3094 (6)	0.1936 (4)	0.06180 (12)	0.0723 (9)
H18A	0.4127	0.1190	0.0541	0.087*
H18B	0.1746	0.1465	0.0623	0.087*
C19	0.3143 (4)	0.3164 (4)	0.02465 (11)	0.0620 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0697 (14)	0.1079 (19)	0.0685 (14)	0.0052 (15)	−0.0087 (11)	−0.0295 (13)
C1	0.081 (2)	0.091 (3)	0.092 (2)	−0.010 (2)	−0.015 (2)	−0.024 (2)
C2	0.071 (2)	0.079 (3)	0.184 (5)	−0.009 (2)	−0.019 (3)	−0.057 (3)
C3	0.078 (2)	0.062 (2)	0.149 (4)	−0.019 (2)	−0.018 (3)	0.000 (3)
C4	0.066 (2)	0.0559 (19)	0.107 (2)	−0.0104 (18)	0.0156 (18)	0.0012 (19)
C5	0.0449 (14)	0.0461 (15)	0.0644 (16)	−0.0005 (13)	0.0067 (12)	−0.0033 (13)
C6	0.0615 (19)	0.0570 (18)	0.0823 (19)	0.0043 (15)	0.0080 (15)	−0.0150 (16)
C7	0.0557 (17)	0.0626 (19)	0.0698 (18)	−0.0004 (15)	−0.0044 (15)	−0.0127 (15)
C8	0.085 (2)	0.074 (2)	0.0567 (16)	−0.005 (2)	−0.0179 (16)	0.0033 (16)
C9	0.083 (2)	0.0537 (18)	0.0583 (16)	−0.0008 (17)	−0.0112 (16)	0.0081 (14)
C10	0.0455 (14)	0.0462 (15)	0.0493 (13)	0.0007 (12)	0.0044 (12)	0.0025 (11)
C11	0.0385 (13)	0.0492 (15)	0.0536 (13)	0.0037 (12)	0.0086 (11)	0.0036 (12)
C12	0.0679 (18)	0.0579 (18)	0.0621 (16)	−0.0027 (16)	0.0023 (15)	0.0161 (15)
C13	0.0693 (19)	0.079 (2)	0.0459 (14)	0.0017 (18)	−0.0020 (14)	0.0064 (15)
C14	0.0454 (15)	0.0604 (17)	0.0522 (14)	0.0060 (13)	−0.0016 (13)	−0.0045 (13)
C15	0.0480 (16)	0.078 (2)	0.082 (2)	0.0116 (16)	−0.0071 (14)	−0.0115 (18)
C16	0.0474 (14)	0.0460 (15)	0.0543 (14)	0.0051 (13)	0.0031 (12)	−0.0010 (12)
C17	0.071 (2)	0.0486 (16)	0.0788 (19)	−0.0050 (16)	−0.0074 (17)	0.0013 (15)
C18	0.068 (2)	0.0572 (19)	0.092 (2)	−0.0002 (17)	−0.0068 (18)	−0.0157 (18)
C19	0.0412 (15)	0.077 (2)	0.0679 (19)	0.0089 (16)	−0.0075 (13)	−0.0182 (17)

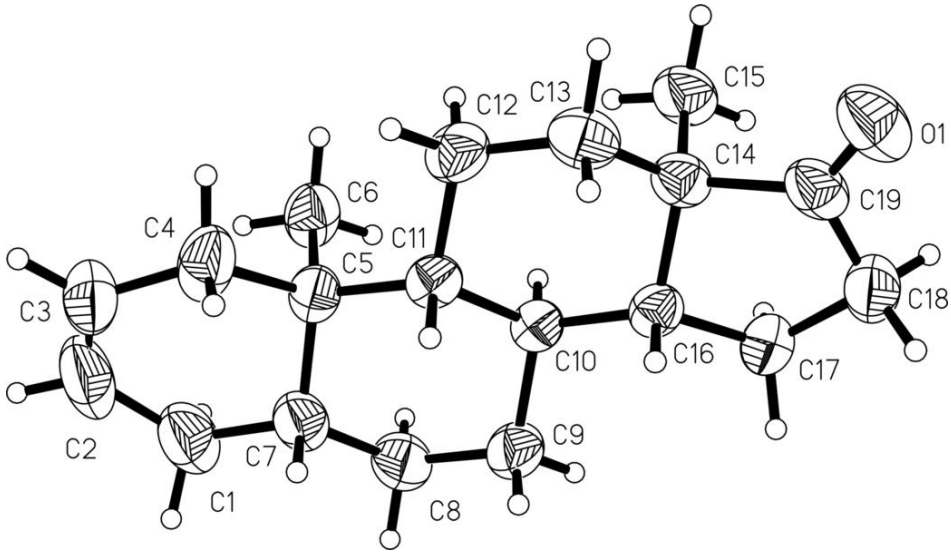
Geometric parameters (Å, °)

O1—C19	1.209 (3)	C10—C16	1.515 (3)
C1—C2	1.462 (6)	C10—C11	1.541 (3)
C1—C7	1.525 (4)	C10—H10A	0.9800
C1—H1A	0.9700	C11—C12	1.539 (4)
C1—H1B	0.9700	C11—H11A	0.9800
C2—C3	1.320 (6)	C12—C13	1.518 (4)
C2—H2A	0.9300	C12—H12A	0.9700
C3—C4	1.484 (5)	C12—H12B	0.9700
C3—H3A	0.9300	C13—C14	1.513 (4)
C4—C5	1.529 (4)	C13—H13A	0.9700
C4—H4A	0.9700	C13—H13B	0.9700
C4—H4B	0.9700	C14—C19	1.520 (4)

C5—C6	1.529 (4)	C14—C16	1.528 (4)
C5—C11	1.540 (4)	C14—C15	1.538 (4)
C5—C7	1.541 (4)	C15—H15A	0.9600
C6—H6A	0.9600	C15—H15B	0.9600
C6—H6B	0.9600	C15—H15C	0.9600
C6—H6C	0.9600	C16—C17	1.526 (4)
C7—C8	1.516 (4)	C16—H16A	0.9800
C7—H7A	0.9800	C17—C18	1.536 (4)
C8—C9	1.522 (4)	C17—H17A	0.9700
C8—H8A	0.9700	C17—H17B	0.9700
C8—H8B	0.9700	C18—C19	1.498 (5)
C9—C10	1.525 (4)	C18—H18A	0.9700
C9—H9A	0.9700	C18—H18B	0.9700
C9—H9B	0.9700		
C2—C1—C7	115.2 (3)	C11—C10—H10A	108.5
C2—C1—H1A	108.5	C12—C11—C5	113.9 (2)
C7—C1—H1A	108.5	C12—C11—C10	112.6 (2)
C2—C1—H1B	108.5	C5—C11—C10	112.92 (19)
C7—C1—H1B	108.5	C12—C11—H11A	105.5
H1A—C1—H1B	107.5	C5—C11—H11A	105.5
C3—C2—C1	123.3 (4)	C10—C11—H11A	105.5
C3—C2—H2A	118.4	C13—C12—C11	113.1 (2)
C1—C2—H2A	118.4	C13—C12—H12A	109.0
C2—C3—C4	121.9 (4)	C11—C12—H12A	109.0
C2—C3—H3A	119.0	C13—C12—H12B	109.0
C4—C3—H3A	119.0	C11—C12—H12B	109.0
C3—C4—C5	114.0 (3)	H12A—C12—H12B	107.8
C3—C4—H4A	108.7	C14—C13—C12	109.8 (2)
C5—C4—H4A	108.7	C14—C13—H13A	109.7
C3—C4—H4B	108.7	C12—C13—H13A	109.7
C5—C4—H4B	108.7	C14—C13—H13B	109.7
H4A—C4—H4B	107.6	C12—C13—H13B	109.7
C4—C5—C6	108.5 (2)	H13A—C13—H13B	108.2
C4—C5—C11	110.5 (2)	C13—C14—C19	117.8 (2)
C6—C5—C11	111.3 (2)	C13—C14—C16	108.9 (2)
C4—C5—C7	107.2 (2)	C19—C14—C16	100.5 (2)
C6—C5—C7	111.0 (2)	C13—C14—C15	111.3 (3)
C11—C5—C7	108.3 (2)	C19—C14—C15	104.2 (2)
C5—C6—H6A	109.5	C16—C14—C15	113.8 (2)
C5—C6—H6B	109.5	C14—C15—H15A	109.5
H6A—C6—H6B	109.5	C14—C15—H15B	109.5
C5—C6—H6C	109.5	H15A—C15—H15B	109.5
H6A—C6—H6C	109.5	C14—C15—H15C	109.5
H6B—C6—H6C	109.5	H15A—C15—H15C	109.5
C8—C7—C1	112.9 (3)	H15B—C15—H15C	109.5
C8—C7—C5	113.1 (3)	C10—C16—C17	120.3 (2)
C1—C7—C5	111.5 (3)	C10—C16—C14	113.4 (2)
C8—C7—H7A	106.2	C17—C16—C14	103.9 (2)
C1—C7—H7A	106.2	C10—C16—H16A	106.1

C5—C7—H7A	106.2	C17—C16—H16A	106.1
C7—C8—C9	111.3 (2)	C14—C16—H16A	106.1
C7—C8—H8A	109.4	C16—C17—C18	102.8 (2)
C9—C8—H8A	109.4	C16—C17—H17A	111.2
C7—C8—H8B	109.4	C18—C17—H17A	111.2
C9—C8—H8B	109.4	C16—C17—H17B	111.2
H8A—C8—H8B	108.0	C18—C17—H17B	111.2
C8—C9—C10	111.5 (2)	H17A—C17—H17B	109.1
C8—C9—H9A	109.3	C19—C18—C17	106.1 (2)
C10—C9—H9A	109.3	C19—C18—H18A	110.5
C8—C9—H9B	109.3	C17—C18—H18A	110.5
C10—C9—H9B	109.3	C19—C18—H18B	110.5
H9A—C9—H9B	108.0	C17—C18—H18B	110.5
C16—C10—C9	112.0 (2)	H18A—C18—H18B	108.7
C16—C10—C11	109.71 (19)	O1—C19—C18	125.4 (3)
C9—C10—C11	109.6 (2)	O1—C19—C14	126.4 (3)
C16—C10—H10A	108.5	C18—C19—C14	108.2 (2)
C9—C10—H10A	108.5		
C7—C1—C2—C3	7.8 (6)	C9—C10—C11—C5	−57.1 (3)
C1—C2—C3—C4	0.7 (7)	C5—C11—C12—C13	178.8 (2)
C2—C3—C4—C5	22.1 (6)	C10—C11—C12—C13	−51.0 (3)
C3—C4—C5—C6	70.0 (4)	C11—C12—C13—C14	55.8 (3)
C3—C4—C5—C11	−167.8 (3)	C12—C13—C14—C19	−172.6 (2)
C3—C4—C5—C7	−50.0 (4)	C12—C13—C14—C16	−59.1 (3)
C2—C1—C7—C8	−166.8 (3)	C12—C13—C14—C15	67.3 (3)
C2—C1—C7—C5	−38.1 (4)	C9—C10—C16—C17	59.4 (3)
C4—C5—C7—C8	−173.6 (2)	C11—C10—C16—C17	−178.7 (2)
C6—C5—C7—C8	68.1 (3)	C9—C10—C16—C14	−176.9 (2)
C11—C5—C7—C8	−54.4 (3)	C11—C10—C16—C14	−55.0 (3)
C4—C5—C7—C1	57.8 (3)	C13—C14—C16—C10	60.9 (3)
C6—C5—C7—C1	−60.5 (3)	C19—C14—C16—C10	−174.8 (2)
C11—C5—C7—C1	177.1 (3)	C15—C14—C16—C10	−64.0 (3)
C1—C7—C8—C9	−176.6 (3)	C13—C14—C16—C17	−166.9 (2)
C5—C7—C8—C9	55.6 (4)	C19—C14—C16—C17	−42.5 (3)
C7—C8—C9—C10	−55.8 (4)	C15—C14—C16—C17	68.3 (3)
C8—C9—C10—C16	177.9 (2)	C10—C16—C17—C18	167.3 (2)
C8—C9—C10—C11	55.9 (3)	C14—C16—C17—C18	39.1 (3)
C4—C5—C11—C12	−57.5 (3)	C16—C17—C18—C19	−19.9 (3)
C6—C5—C11—C12	63.1 (3)	C17—C18—C19—O1	173.4 (3)
C7—C5—C11—C12	−174.6 (2)	C17—C18—C19—C14	−6.6 (3)
C4—C5—C11—C10	172.5 (2)	C13—C14—C19—O1	−31.7 (4)
C6—C5—C11—C10	−66.9 (3)	C16—C14—C19—O1	−149.8 (3)
C7—C5—C11—C10	55.4 (3)	C15—C14—C19—O1	92.1 (3)
C16—C10—C11—C12	48.9 (3)	C13—C14—C19—C18	148.2 (3)
C9—C10—C11—C12	172.2 (2)	C16—C14—C19—C18	30.1 (3)
C16—C10—C11—C5	179.5 (2)	C15—C14—C19—C18	−88.0 (3)

Fig. 1


C15
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