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3β,5-Dihydroxy-15β,16β-methylene-5β-androst-6-en-17-one

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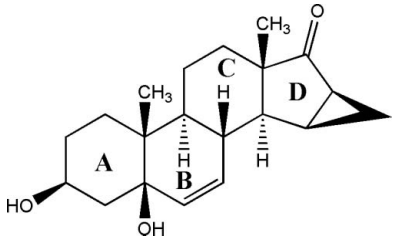
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.110; data-to-parameter ratio = 8.7.

In the title molecule, $C_{20}H_{28}O_3$, ring A has a regular chair conformation, while ring C has a slightly distorted chair conformation. Ring B shows an asymmetric half-chair conformation and ring D is in an envelope conformation. Rings A and B are *cis*-fused, while rings B and C and rings C and D are *trans*-fused. In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules into a ribbon along the b axis.

Related literature

For related literature, see: Bittler *et al.* (1984); Muhn *et al.* (1995); Zhou *et al.* (2006, 2007).



Experimental

Crystal data

$C_{20}H_{28}O_3$
M_r = 316.42
Monoclinic, $P2_1$
a = 6.5158 (9) Å
b = 20.592 (3) Å
c = 6.5877 (9) Å
β = 107.837 (2)°
V = 841.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.976, T_{max} = 0.984
5279 measured reflections
1884 independent reflections
1739 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.110
S = 1.06
1884 reflections
216 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2X···O1	0.85 (5)	1.98 (4)	2.734 (3)	148 (6)
O1—H1X···O3^{i}	0.792 (19)	2.019 (19)	2.811 (3)	178 (4)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2235).

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supplementary materials

Acta Cryst. (2007). E**63**, o4707 [**doi:10.1107/S1600536807057315**]

3*β*,5-Dihydroxy-15*β*,16*β*-methylene-5*β*-androst-6-en-17-one

W. Zhou, G.-X. Zhong, W.-X. Hu and C.-N. Xia

Comment

Drospirenone is a new contraceptive drug with the special antimineralocorticoid and antiandrogenic properties (Muhn *et al.*, 1995). In our attempts to synthesize drospirenone, the title compound, (I), was obtained as an intermediate by the alcoholysis of, 3β-acetoxy-5-hydroxy-15β,16β-methylene-5β-androst-6-en-17-one. Here we report the crystal structure of (I).

In the cyclopropyl ring C15/C16/C20, the bond lengths C16—C20 and C15—C20 are different [1.515 (4) *versus* 1.475 (4) Å], which is also found in 3β-acetoxy-7α-chloro-5,6β-epoxy-15β,16β-methylene-5β-androstan-17-one [1.511 (5) and 1.473 (4) Å; Zhou *et al.*, 2007]. The difference in these bond lengths might be caused by a hyperconjugation interaction between the C=O π-bonding and C16—C20 σ-bonding orbitals. However, in the analogous structure of 3β-acetoxy-17,17-ethylenedioxy-15β,16β-methylene-5-androsten-7β-ol these bond lenghts are almost equal [1.499 (6) and 1.493 (6) Å; Zhou *et al.*, 2006].

In the steroid skeleton, ring A has a regular chair conformation, while ring C has a slightly distorted chair conformation. Ring B shows a asymmetric half-chair conformation and ring D is in an envelope conformation. In ring B, atom C9 and atom C10 deviate by 0.390 (4) and −0.435 (4) Å from the mean plane calculated through the ring C5—C8, respectively. In ring D, C13 deviates by 0.486 (3) Å from the mean plane of C14—C17, which makes a dihedral angle of 64.5 (1)° with the cyclopropyl ring C15/C16/C20. In the crystal structure, an intermolecular O—H···O hydrogen bond is found (Table 1), which links the molecules into ribbons along the *b* axis (Fig. 2). In addition, an intramolecular O—H···O hydrogen bond is found (Table 1).

Experimental

The title compound was synthesized according to the literature method (Bittler *et al.*, 1984). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a tetrahydrofuran solution.

Refinement

C-bound H atoms were placed at calculated positions (C—H = 0.93–0.98 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The hydroxy H atoms were located in a difference map and refined isotropically with restraint O—H = 0.84 (2) Å. Owing to negligible anomalous scattering effects, Friedel pairs were averaged in the refinement. The absolute stereochemistry of (I) was known from the synthetic route (Bittler *et al.*, 1984).

Figures

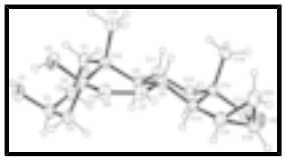

Fig. 1. The molecular structure of (I), with 30% probability displacement ellipsoids.

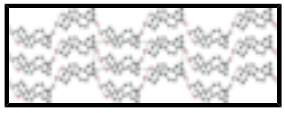

Fig. 2. Packing diagram of (I), viewed along the *a* axis, showing hydrogen bonds as dashed lines. For clarity, H atoms have been omitted except for those involved in hydrogen bonds.

3β,5-Dihydroxy-15β,16β-methylene-5β-androst-6-en-17-one

Crystal data

$C_{20}H_{28}O_3$	F_{000} = 344
M_r = 316.42	D_x = 1.249 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 2477 reflections
a = 6.5158 (9) Å	θ = 3.3–27.4º
b = 20.592 (3) Å	μ = 0.08 mm^{-1}
c = 6.5877 (9) Å	*T* = 296 (2) K
β = 107.837 (2)º	Prism, colorless
V = 841.4 (2) Å^3	0.30 × 0.20 × 0.20 mm
Z = 2	

Data collection

Bruker APEXII CCD area-detector diffractometer	1884 independent reflections
Radiation source: fine-focus sealed tube	1739 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 296(2) K	θ_{max} = 27.0º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	*h* = −7→8
T_{min} = 0.976, T_{max} = 0.984	*k* = −26→26
5279 measured reflections	*l* = −8→6

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.110	$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 0.0701P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
1884 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
216 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	0.0158 (4)	0.57314 (9)	−0.3266 (3)	0.0552 (5)
H1X	−0.091 (4)	0.5628 (18)	−0.416 (4)	0.062 (10)*
O2	0.0413 (3)	0.64237 (9)	0.0343 (3)	0.0509 (5)
H2X	0.030 (10)	0.609 (2)	−0.044 (9)	0.15 (2)*
O3	0.3570 (3)	1.03434 (11)	−0.3503 (3)	0.0623 (5)
C1	−0.1688 (4)	0.70794 (12)	−0.3885 (4)	0.0431 (5)
H1A	−0.2548	0.6747	−0.3486	0.052*
H1B	−0.2674	0.7396	−0.4758	0.052*
C2	−0.0437 (5)	0.67689 (12)	−0.5215 (4)	0.0496 (6)
H2A	0.0307	0.7103	−0.5754	0.060*
H2B	−0.1430	0.6551	−0.6427	0.060*
C3	0.1197 (5)	0.62810 (12)	−0.3921 (4)	0.0494 (6)
H3	0.2077	0.6124	−0.4788	0.059*
C4	0.2652 (4)	0.65956 (11)	−0.1923 (4)	0.0443 (5)
H4A	0.3543	0.6917	−0.2326	0.053*
H4B	0.3602	0.6267	−0.1074	0.053*
C5	0.1422 (4)	0.69255 (10)	−0.0546 (3)	0.0364 (5)
C6	0.3031 (4)	0.72557 (12)	0.1315 (4)	0.0428 (5)
H6	0.3837	0.6995	0.2428	0.051*
C7	0.3376 (4)	0.78913 (12)	0.1477 (4)	0.0427 (5)
H7	0.4451	0.8048	0.2656	0.051*
C8	0.2114 (3)	0.83711 (10)	−0.0151 (3)	0.0342 (4)
H8	0.1020	0.8569	0.0394	0.041*
C9	0.0945 (3)	0.80199 (10)	−0.2272 (3)	0.0331 (4)
H9	0.2088	0.7847	−0.2801	0.040*

C10	−0.0304 (3)	0.74189 (10)	−0.1847 (3)	0.0340 (4)
C11	−0.0357 (4)	0.84930 (12)	−0.4003 (4)	0.0448 (5)
H11A	−0.1603	0.8642	−0.3622	0.054*
H11B	−0.0878	0.8262	−0.5346	0.054*
C12	0.0961 (4)	0.90909 (12)	−0.4311 (4)	0.0462 (6)
H12A	0.2075	0.8954	−0.4915	0.055*
H12B	0.0022	0.9395	−0.5291	0.055*
C13	0.1993 (3)	0.94225 (10)	−0.2162 (4)	0.0369 (5)
C14	0.3449 (3)	0.89105 (10)	−0.0685 (3)	0.0357 (5)
H14	0.4209	0.8700	−0.1581	0.043*
C15	0.5202 (4)	0.92742 (12)	0.0985 (4)	0.0464 (6)
H15	0.6546	0.9043	0.1692	0.056*
C16	0.5362 (4)	0.99388 (12)	0.0006 (4)	0.0463 (6)
H16	0.6788	1.0115	0.0109	0.056*
C17	0.3634 (4)	0.99646 (11)	−0.2073 (4)	0.0427 (5)
C18	0.0169 (4)	0.97291 (13)	−0.1433 (5)	0.0508 (6)
H18A	−0.0374	0.9413	−0.0655	0.076*
H18B	−0.0975	0.9869	−0.2658	0.076*
H18C	0.0723	1.0096	−0.0532	0.076*
C19	−0.1823 (4)	0.76125 (14)	−0.0571 (4)	0.0494 (6)
H19A	−0.2584	0.7236	−0.0329	0.074*
H19B	−0.2837	0.7929	−0.1358	0.074*
H19C	−0.0997	0.7794	0.0774	0.074*
C20	0.4718 (5)	0.98657 (15)	0.2017 (4)	0.0568 (7)
H20A	0.5753	0.9999	0.3353	0.068*
H20B	0.3223	0.9948	0.1916	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0676 (13)	0.0364 (9)	0.0532 (11)	−0.0079 (9)	0.0058 (10)	−0.0006 (8)
O2	0.0630 (12)	0.0439 (10)	0.0448 (10)	−0.0119 (8)	0.0151 (9)	0.0087 (8)
O3	0.0712 (12)	0.0485 (11)	0.0662 (12)	−0.0026 (9)	0.0196 (10)	0.0138 (9)
C1	0.0430 (12)	0.0359 (11)	0.0402 (12)	−0.0042 (9)	−0.0022 (10)	0.0013 (10)
C2	0.0714 (18)	0.0402 (13)	0.0338 (11)	−0.0089 (11)	0.0110 (12)	−0.0015 (10)
C3	0.0641 (16)	0.0358 (12)	0.0516 (14)	−0.0005 (11)	0.0227 (12)	−0.0051 (11)
C4	0.0408 (12)	0.0320 (11)	0.0595 (14)	0.0024 (9)	0.0147 (10)	0.0036 (10)
C5	0.0363 (11)	0.0321 (10)	0.0354 (10)	−0.0007 (9)	0.0029 (9)	0.0057 (8)
C6	0.0425 (12)	0.0418 (12)	0.0352 (11)	−0.0021 (10)	−0.0014 (9)	0.0070 (9)
C7	0.0425 (12)	0.0428 (12)	0.0319 (10)	−0.0033 (9)	−0.0046 (9)	0.0013 (9)
C8	0.0345 (10)	0.0328 (10)	0.0304 (10)	0.0013 (8)	0.0027 (8)	−0.0025 (8)
C9	0.0345 (10)	0.0292 (9)	0.0304 (9)	0.0031 (8)	0.0022 (8)	−0.0003 (8)
C10	0.0305 (9)	0.0345 (10)	0.0330 (10)	0.0004 (8)	0.0036 (8)	−0.0009 (8)
C11	0.0498 (13)	0.0354 (11)	0.0365 (11)	0.0010 (10)	−0.0054 (9)	0.0019 (10)
C12	0.0583 (14)	0.0358 (11)	0.0359 (11)	0.0033 (10)	0.0015 (10)	0.0043 (9)
C13	0.0375 (10)	0.0289 (10)	0.0405 (11)	0.0031 (8)	0.0062 (9)	0.0000 (9)
C14	0.0325 (10)	0.0328 (10)	0.0371 (11)	0.0038 (8)	0.0039 (8)	−0.0012 (9)
C15	0.0387 (11)	0.0411 (13)	0.0497 (13)	−0.0025 (10)	−0.0006 (10)	−0.0007 (10)

C16	0.0411 (12)	0.0372 (12)	0.0586 (14)	−0.0034 (10)	0.0121 (11)	−0.0050 (11)
C17	0.0452 (12)	0.0324 (11)	0.0515 (13)	0.0037 (9)	0.0166 (10)	−0.0030 (10)
C18	0.0396 (11)	0.0418 (13)	0.0692 (17)	0.0079 (10)	0.0142 (12)	−0.0051 (12)
C19	0.0402 (12)	0.0512 (14)	0.0595 (15)	0.0010 (11)	0.0196 (11)	−0.0012 (12)
C20	0.0619 (16)	0.0551 (15)	0.0488 (14)	−0.0132 (13)	0.0101 (12)	−0.0149 (12)

Geometric parameters (Å, °)

O1—C3	1.451 (3)	C9—H9	0.9800
O1—H1X	0.792 (19)	C10—C19	1.535 (3)
O2—C5	1.442 (3)	C11—C12	1.550 (4)
O2—H2X	0.85 (5)	C11—H11A	0.9700
O3—C17	1.214 (3)	C11—H11B	0.9700
C1—C2	1.509 (4)	C12—C13	1.529 (3)
C1—C10	1.537 (3)	C12—H12A	0.9700
C1—H1A	0.9700	C12—H12B	0.9700
C1—H1B	0.9700	C13—C17	1.534 (3)
C2—C3	1.521 (4)	C13—C18	1.547 (3)
C2—H2A	0.9700	C13—C14	1.547 (3)
C2—H2B	0.9700	C14—C15	1.519 (3)
C3—C4	1.512 (4)	C14—H14	0.9800
C3—H3	0.9800	C15—C20	1.475 (4)
C4—C5	1.540 (3)	C15—C16	1.530 (4)
C4—H4A	0.9700	C15—H15	0.9800
C4—H4B	0.9700	C16—C17	1.483 (4)
C5—C6	1.509 (3)	C16—C20	1.515 (4)
C5—C10	1.562 (3)	C16—H16	0.9800
C6—C7	1.327 (4)	C18—H18A	0.9599
C6—H6	0.9300	C18—H18B	0.9599
C7—C8	1.504 (3)	C18—H18C	0.9599
C7—H7	0.9300	C19—H19A	0.9599
C8—C14	1.517 (3)	C19—H19B	0.9599
C8—C9	1.551 (3)	C19—H19C	0.9599
C8—H8	0.9800	C20—H20A	0.9700
C9—C11	1.541 (3)	C20—H20B	0.9700
C9—C10	1.553 (3)		
C3—O1—H1X	112 (3)	C9—C11—H11A	108.9
C5—O2—H2X	107 (4)	C12—C11—H11A	108.9
C2—C1—C10	115.01 (19)	C9—C11—H11B	108.9
C2—C1—H1A	108.5	C12—C11—H11B	108.9
C10—C1—H1A	108.5	H11A—C11—H11B	107.7
C2—C1—H1B	108.5	C13—C12—C11	109.74 (19)
C10—C1—H1B	108.5	C13—C12—H12A	109.7
H1A—C1—H1B	107.5	C11—C12—H12A	109.7
C1—C2—C3	111.4 (2)	C13—C12—H12B	109.7
C1—C2—H2A	109.4	C11—C12—H12B	109.7
C3—C2—H2A	109.4	H12A—C12—H12B	108.2
C1—C2—H2B	109.4	C12—C13—C17	117.64 (19)
C3—C2—H2B	109.4	C12—C13—C18	107.9 (2)

H2A—C2—H2B	108.0	C17—C13—C18	106.49 (18)
O1—C3—C4	107.5 (2)	C12—C13—C14	106.79 (17)
O1—C3—C2	111.8 (2)	C17—C13—C14	100.70 (17)
C4—C3—C2	110.5 (2)	C18—C13—C14	117.7 (2)
O1—C3—H3	109.0	C8—C14—C15	123.38 (18)
C4—C3—H3	109.0	C8—C14—C13	111.09 (17)
C2—C3—H3	109.0	C15—C14—C13	107.45 (18)
C3—C4—C5	113.6 (2)	C8—C14—H14	104.4
C3—C4—H4A	108.8	C15—C14—H14	104.4
C5—C4—H4A	108.8	C13—C14—H14	104.4
C3—C4—H4B	108.8	C20—C15—C14	121.4 (2)
C5—C4—H4B	108.8	C20—C15—C16	60.51 (18)
H4A—C4—H4B	107.7	C14—C15—C16	105.6 (2)
O2—C5—C6	106.38 (17)	C20—C15—H15	117.9
O2—C5—C4	107.91 (19)	C14—C15—H15	117.9
C6—C5—C4	108.69 (19)	C16—C15—H15	117.9
O2—C5—C10	110.33 (17)	C17—C16—C20	118.4 (2)
C6—C5—C10	110.90 (18)	C17—C16—C15	106.9 (2)
C4—C5—C10	112.40 (18)	C20—C16—C15	57.95 (18)
C7—C6—C5	124.6 (2)	C17—C16—H16	119.1
C7—C6—H6	117.7	C20—C16—H16	119.1
C5—C6—H6	117.7	C15—C16—H16	119.1
C6—C7—C8	123.3 (2)	O3—C17—C16	125.1 (2)
C6—C7—H7	118.4	O3—C17—C13	125.4 (2)
C8—C7—H7	118.4	C16—C17—C13	109.4 (2)
C7—C8—C14	114.79 (17)	C13—C18—H18A	109.5
C7—C8—C9	110.28 (17)	C13—C18—H18B	109.5
C14—C8—C9	106.88 (17)	H18A—C18—H18B	109.5
C7—C8—H8	108.2	C13—C18—H18C	109.5
C14—C8—H8	108.2	H18A—C18—H18C	109.5
C9—C8—H8	108.2	H18B—C18—H18C	109.5
C11—C9—C8	112.27 (17)	C10—C19—H19A	109.5
C11—C9—C10	115.91 (17)	C10—C19—H19B	109.5
C8—C9—C10	110.41 (16)	H19A—C19—H19B	109.5
C11—C9—H9	105.8	C10—C19—H19C	109.5
C8—C9—H9	105.8	H19A—C19—H19C	109.5
C10—C9—H9	105.8	H19B—C19—H19C	109.5
C19—C10—C1	106.99 (18)	C15—C20—C16	61.54 (18)
C19—C10—C9	110.70 (19)	C15—C20—H20A	117.6
C1—C10—C9	113.86 (17)	C16—C20—H20A	117.6
C19—C10—C5	110.21 (19)	C15—C20—H20B	117.6
C1—C10—C5	108.32 (17)	C16—C20—H20B	117.6
C9—C10—C5	106.75 (16)	H20A—C20—H20B	114.7
C9—C11—C12	113.41 (19)		
C10—C1—C2—C3	56.5 (3)	C8—C9—C11—C12	50.3 (3)
C1—C2—C3—O1	65.3 (3)	C10—C9—C11—C12	178.5 (2)
C1—C2—C3—C4	−54.4 (3)	C9—C11—C12—C13	−52.8 (3)
O1—C3—C4—C5	−68.1 (3)	C11—C12—C13—C17	171.19 (19)
C2—C3—C4—C5	54.2 (3)	C11—C12—C13—C18	−68.4 (2)

C3—C4—C5—O2	68.5 (2)	C11—C12—C13—C14	59.1 (2)
C3—C4—C5—C6	−176.49 (19)	C7—C8—C14—C15	−43.6 (3)
C3—C4—C5—C10	−53.4 (3)	C9—C8—C14—C15	−166.2 (2)
O2—C5—C6—C7	−139.6 (3)	C7—C8—C14—C13	−173.34 (18)
C4—C5—C6—C7	104.5 (3)	C9—C8—C14—C13	64.0 (2)
C10—C5—C6—C7	−19.6 (4)	C12—C13—C14—C8	−67.9 (2)
C5—C6—C7—C8	3.0 (4)	C17—C13—C14—C8	168.77 (17)
C6—C7—C8—C14	−138.5 (3)	C18—C13—C14—C8	53.6 (2)
C6—C7—C8—C9	−17.7 (3)	C12—C13—C14—C15	154.5 (2)
C7—C8—C9—C11	−179.5 (2)	C17—C13—C14—C15	31.1 (2)
C14—C8—C9—C11	−54.1 (2)	C18—C13—C14—C15	−84.1 (2)
C7—C8—C9—C10	49.5 (2)	C8—C14—C15—C20	−89.3 (3)
C14—C8—C9—C10	174.90 (16)	C13—C14—C15—C20	41.9 (3)
C2—C1—C10—C19	−171.9 (2)	C8—C14—C15—C16	−154.0 (2)
C2—C1—C10—C9	65.5 (3)	C13—C14—C15—C16	−22.7 (3)
C2—C1—C10—C5	−53.1 (3)	C20—C15—C16—C17	−113.3 (2)
C11—C9—C10—C19	−75.2 (2)	C14—C15—C16—C17	4.3 (3)
C8—C9—C10—C19	53.9 (2)	C14—C15—C16—C20	117.7 (2)
C11—C9—C10—C1	45.4 (2)	C20—C16—C17—O3	135.8 (3)
C8—C9—C10—C1	174.43 (17)	C15—C16—C17—O3	−161.9 (2)
C11—C9—C10—C5	164.84 (18)	C20—C16—C17—C13	−46.3 (3)
C8—C9—C10—C5	−66.1 (2)	C15—C16—C17—C13	15.9 (3)
O2—C5—C10—C19	46.3 (2)	C12—C13—C17—O3	33.6 (3)
C6—C5—C10—C19	−71.3 (2)	C18—C13—C17—O3	−87.6 (3)
C4—C5—C10—C19	166.8 (2)	C14—C13—C17—O3	149.1 (2)
O2—C5—C10—C1	−70.4 (2)	C12—C13—C17—C16	−144.3 (2)
C6—C5—C10—C1	172.00 (19)	C18—C13—C17—C16	94.6 (2)
C4—C5—C10—C1	50.1 (2)	C14—C13—C17—C16	−28.8 (2)
O2—C5—C10—C9	166.61 (17)	C14—C15—C20—C16	−90.9 (3)
C6—C5—C10—C9	49.0 (2)	C17—C16—C20—C15	93.0 (2)
C4—C5—C10—C9	−72.9 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2X···O1	0.85 (5)	1.98 (4)	2.734 (3)	148 (6)
O1—H1X···O3^{i}	0.792 (19)	2.019 (19)	2.811 (3)	178 (4)

Symmetry codes: (i) −*x*, *y*−1/2, −*z*−1.

Fig. 1

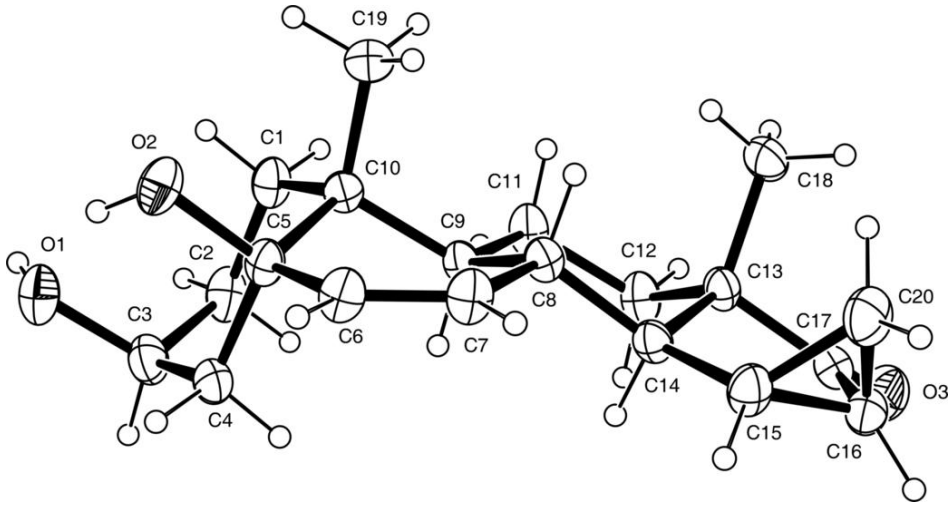
C19
O2
C1
C10
C11
C18
C5
C9
O1
C2
C12
C13
C20
C3
C8
C17
C6
C7
C14
O3
C4
C15
C16

Fig. 2