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3β,5α,15α-Trihydroxy-5-androsten-17-one dihydrate

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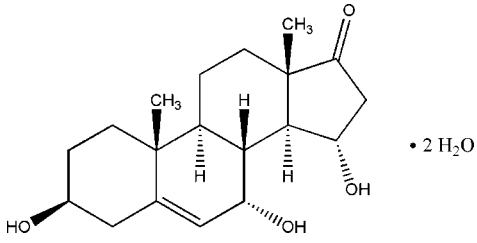
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.099; data-to-parameter ratio = 9.9.

In the steroid skeleton of the title compound, $C_{19}H_{28}O_4\cdot 2H_2O$, ring A assumes a slightly twisted chair conformation, ring C adopts a regular chair conformation, ring B adopts a half-chair conformation and ring D has a somewhat twisted envelope conformation. The crystal packing exhibits an extensive three-dimensional hydrogen-bonding network, formed by intermolecular O—H···O hydrogen bonds between the steroid and water molecules.

Related literature

For related literature, see: Romano *et al.* (2006); Muhn *et al.* (1995).



Experimental

Crystal data

$C_{19}H_{28}O_4\cdot 2H_2O$
M_r = 356.45
Orthorhombic, $P2_12_12_1$
a = 6.0653 (8) Å
b = 12.4906 (16) Å
c = 25.472 (3) Å
V = 1929.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 (2) K
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.975, T_{max} = 0.983
12301 measured reflections
2539 independent reflections
2289 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.099
S = 1.03
2539 reflections
256 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1X···O6	0.830 (18)	1.80 (2)	2.618 (3)	170 (3)
O2—H2X···O3	0.835 (16)	1.98 (2)	2.755 (2)	153 (3)
O3—H3X···O5^{i}	0.844 (17)	1.889 (18)	2.731 (2)	176 (3)
O5—H5X···O1^{ii}	0.852 (18)	1.95 (2)	2.778 (2)	165 (3)
O5—H5Y···O1^{iii}	0.841 (18)	1.867 (19)	2.702 (2)	172 (3)
O6—H6X···O5^{iv}	0.84 (2)	1.92 (2)	2.747 (3)	168 (5)
O6—H6Y···O4^{v}	0.799 (19)	1.99 (2)	2.787 (3)	174 (5)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2322).

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supplementary materials

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3β,5α,15α-Trihydroxy-5-androsten-17-one dihydrate

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Comment

Drospirenone is a new contraceptive drug with the special antimineralocorticoid and antiandrogenic properties (Muhn *et al.*, 1995). The title compound, (I), as a key starting material for the synthesis of drospirenone, was obtained by biotransformation with Colletotrichum lini from dehydroepiandrosterone (DHEA) (Romano *et al.*, 2006). We report here the crystal structure of (I).

In (I) (Fig. 1), ring A assumes a slightly twisty chair conformation and ring C takes a regular chair conformation. In ring B, atoms C8 and C9 deviate at 0.242 and −0.503 Å from the mean plane C5—C7/C10, respectively, thus ring B takes a half-chair conformation. Ring D has a somewhat twisty envelope conformation: atoms C13, C15, C16 and C17 are nearly coplanar and atom C14 deviates from their mean plane at 0.625 Å.

In the crystal (Fig. 2), the crystalline water molecules involved in the formation of hydrogen bonding. The intermolecular and intramolecular O—H···O hydrogen bonding are found in the crystal lattice (Table 1).

Experimental

The title compound was obtained by biotransformation with Colletotrichum lini from dehydroepiandrosterone(DHEA) according to the literature method (Andrea *et al.*, 2006). Dehydroepiandrosterone(DHEA) was kindly offered by Mr. Pan, Jiubang Chemistry Corp. Ltd., Shanghai, China. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from the mixture of tetrahydrofuran and H_2O (9:1).

Refinement

C-bound H atoms were placed in calculated positions (C—H 0.96–0.98 Å) and refined as riding with U_{iso}(H) = 1.2–1.5Ueq of the parent atom. The hydroxy H atoms were located in a difference map and refined isotropically with restaint O—H=0.84 (2) Å. The H atoms in H_2O molecules were located in a difference map and refined isotropically with restaints O—H= 0.83 (2) Å. Due to the absence of any significantanomalous scatterers in the compound, 1937 Friedel pairs were merged before the final refinement.

Figures

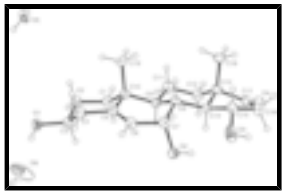

Fig. 1. The structure of (I) with the atomic numbering and 30% probability displacement ellipsoids.

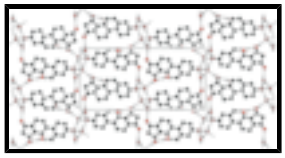

Fig. 2. Packing diagram of (I), viewed along the *a* axis, showing hydrogen bonds as dashed lines. For clarity, H atoms have been omitted except for those involved in hydrogen bonding.

3β,5α,15α-Trihydroxy-5-androsten-17-one dihydrate

Crystal data

$C_{19}H_{28}O_4 \cdot 2H_2O$	F_{000} = 776
M_r = 356.45	D_x = 1.227 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 4751 reflections
a = 6.0653 (8) Å	θ = 2.3–25.8º
b = 12.4906 (16) Å	μ = 0.09 mm^{-1}
c = 25.472 (3) Å	T = 296 (2) K
V = 1929.8 (4) Å^3	Block, colourless
Z = 4	0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	2539 independent reflections
Radiation source: fine-focus sealed tube	2289 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
T = 296(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	h = −7→7
T_{min} = 0.975, T_{max} = 0.983	k = −16→12
12301 measured reflections	l = −33→32

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.099	$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.2939P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2539 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
256 parameters	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
7 restraints	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.015 (2)

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	0.8995 (3)	0.83766 (13)	0.94606 (5)	0.0479 (4)
H1X	0.994 (5)	0.884 (2)	0.9523 (11)	0.083 (11)*
O2	1.0556 (3)	0.96442 (12)	0.69625 (6)	0.0464 (4)
H2X	1.048 (5)	0.9884 (19)	0.6657 (7)	0.060 (8)*
O3	0.8912 (4)	1.01700 (14)	0.59879 (6)	0.0725 (6)
H3X	0.904 (5)	1.054 (2)	0.5714 (8)	0.076 (9)*
O4	0.7005 (4)	0.68704 (14)	0.53143 (6)	0.0679 (5)
O5	0.0560 (3)	0.64593 (14)	0.98689 (6)	0.0547 (4)
H5X	0.027 (6)	0.7099 (16)	0.9776 (11)	0.078 (9)*
H5Y	0.166 (4)	0.657 (2)	1.0062 (10)	0.076 (10)*
O6	1.2334 (5)	0.9667 (2)	0.96135 (14)	0.1160 (11)
H6X	1.325 (7)	0.937 (4)	0.9813 (17)	0.174*
H6Y	1.258 (8)	1.0295 (17)	0.9615 (16)	0.129 (16)*
C1	0.7195 (4)	0.71106 (15)	0.81999 (8)	0.0434 (5)
H1A	0.6202	0.6519	0.8131	0.052*
H1B	0.8654	0.6896	0.8085	0.052*
C2	0.7263 (4)	0.73072 (17)	0.87909 (8)	0.0457 (5)
H2A	0.5794	0.7468	0.8919	0.055*
H2B	0.7791	0.6671	0.8970	0.055*
C3	0.8782 (3)	0.82337 (16)	0.89005 (7)	0.0401 (4)
H3	1.0238	0.8074	0.8753	0.048*
C4	0.7891 (4)	0.92309 (16)	0.86418 (7)	0.0437 (5)
H4A	0.8881	0.9825	0.8710	0.052*
H4B	0.6467	0.9405	0.8793	0.052*
C5	0.7647 (3)	0.90839 (14)	0.80534 (7)	0.0353 (4)
C6	0.8447 (4)	0.98109 (14)	0.77263 (7)	0.0395 (4)
H6	0.9146	1.0401	0.7874	0.047*
C7	0.8322 (3)	0.97645 (14)	0.71421 (7)	0.0370 (4)
H7	0.7756	1.0451	0.7014	0.044*
C8	0.6798 (3)	0.88707 (14)	0.69432 (7)	0.0338 (4)
H8	0.5267	0.9116	0.6970	0.041*

C9	0.7080 (3)	0.78640 (13)	0.72853 (7)	0.0332 (4)
H9	0.8660	0.7701	0.7284	0.040*
C10	0.6452 (3)	0.80810 (14)	0.78689 (7)	0.0348 (4)
C11	0.5923 (4)	0.68699 (16)	0.70619 (8)	0.0505 (5)
H11A	0.6357	0.6250	0.7266	0.061*
H11B	0.4342	0.6957	0.7101	0.061*
C12	0.6449 (4)	0.66589 (17)	0.64823 (8)	0.0512 (6)
H12A	0.7997	0.6478	0.6445	0.061*
H12B	0.5582	0.6058	0.6357	0.061*
C13	0.5931 (4)	0.76454 (16)	0.61572 (8)	0.0415 (5)
C14	0.7281 (3)	0.85949 (15)	0.63714 (7)	0.0349 (4)
H14	0.8819	0.8354	0.6367	0.042*
C15	0.7121 (4)	0.94462 (17)	0.59409 (8)	0.0465 (5)
H15	0.5715	0.9830	0.5965	0.056*
C16	0.7217 (5)	0.87818 (19)	0.54340 (8)	0.0547 (6)
H16A	0.6131	0.9035	0.5184	0.066*
H16B	0.8667	0.8831	0.5275	0.066*
C17	0.6729 (4)	0.76470 (18)	0.55922 (8)	0.0470 (5)
C18	0.3422 (4)	0.7862 (2)	0.61283 (10)	0.0593 (6)
H18A	0.2878	0.8033	0.6472	0.089*
H18B	0.2683	0.7236	0.5999	0.089*
H18C	0.3149	0.8452	0.5896	0.089*
C19	0.3950 (4)	0.8263 (2)	0.79409 (9)	0.0526 (6)
H19A	0.3456	0.8802	0.7700	0.079*
H19B	0.3662	0.8492	0.8294	0.079*
H19C	0.3177	0.7606	0.7874	0.079*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0535 (9)	0.0549 (9)	0.0352 (7)	−0.0040 (8)	−0.0004 (7)	0.0027 (6)
O2	0.0432 (8)	0.0525 (8)	0.0436 (7)	−0.0128 (7)	−0.0001 (7)	0.0049 (7)
O3	0.1036 (16)	0.0654 (10)	0.0484 (9)	−0.0366 (11)	−0.0181 (10)	0.0222 (8)
O4	0.0860 (14)	0.0634 (10)	0.0543 (9)	0.0029 (11)	0.0013 (10)	−0.0193 (8)
O5	0.0672 (12)	0.0499 (9)	0.0471 (8)	0.0059 (9)	−0.0137 (8)	−0.0068 (7)
O6	0.0978 (19)	0.0640 (14)	0.186 (3)	−0.0229 (14)	−0.069 (2)	0.0263 (16)
C1	0.0507 (12)	0.0330 (9)	0.0465 (10)	−0.0046 (9)	−0.0029 (10)	0.0049 (8)
C2	0.0508 (12)	0.0432 (11)	0.0430 (10)	−0.0027 (10)	−0.0013 (10)	0.0105 (8)
C3	0.0392 (10)	0.0455 (10)	0.0355 (9)	0.0018 (9)	0.0035 (8)	0.0009 (8)
C4	0.0549 (12)	0.0363 (9)	0.0399 (9)	0.0032 (9)	0.0030 (10)	−0.0026 (8)
C5	0.0380 (10)	0.0296 (8)	0.0383 (8)	0.0062 (8)	0.0009 (8)	−0.0011 (7)
C6	0.0502 (11)	0.0277 (8)	0.0408 (9)	−0.0020 (8)	−0.0053 (9)	−0.0021 (7)
C7	0.0437 (10)	0.0253 (8)	0.0420 (9)	−0.0008 (8)	−0.0021 (8)	0.0047 (7)
C8	0.0306 (9)	0.0316 (8)	0.0390 (8)	0.0019 (8)	−0.0012 (8)	0.0002 (7)
C9	0.0312 (9)	0.0295 (8)	0.0388 (9)	−0.0017 (7)	0.0011 (7)	0.0008 (7)
C10	0.0314 (9)	0.0331 (9)	0.0399 (9)	0.0001 (7)	0.0023 (8)	0.0030 (7)
C11	0.0635 (15)	0.0381 (10)	0.0498 (11)	−0.0165 (11)	0.0010 (11)	−0.0010 (9)
C12	0.0637 (15)	0.0365 (10)	0.0532 (11)	−0.0110 (10)	−0.0012 (11)	−0.0089 (9)

C13	0.0363 (10)	0.0434 (10)	0.0446 (10)	−0.0041 (9)	−0.0019 (9)	−0.0080 (8)
C14	0.0322 (9)	0.0351 (9)	0.0374 (8)	−0.0001 (8)	−0.0017 (8)	0.0005 (7)
C15	0.0548 (13)	0.0439 (11)	0.0409 (10)	−0.0010 (10)	−0.0081 (10)	0.0043 (8)
C16	0.0642 (15)	0.0610 (13)	0.0389 (10)	0.0004 (13)	−0.0021 (11)	−0.0008 (10)
C17	0.0408 (11)	0.0556 (12)	0.0447 (10)	0.0025 (10)	−0.0063 (9)	−0.0101 (9)
C18	0.0360 (11)	0.0793 (17)	0.0624 (13)	−0.0065 (12)	−0.0042 (10)	−0.0115 (13)
C19	0.0328 (10)	0.0746 (15)	0.0504 (12)	−0.0008 (11)	0.0051 (9)	0.0034 (11)

Geometric parameters (Å, °)

O1—C3	1.443 (2)	C8—C14	1.525 (2)
O1—H1X	0.830 (18)	C8—C9	1.539 (2)
O2—C7	1.438 (3)	C8—H8	0.9800
O2—H2X	0.835 (16)	C9—C11	1.536 (3)
O3—C15	1.418 (3)	C9—C10	1.558 (2)
O3—H3X	0.844 (17)	C9—H9	0.9800
O4—C17	1.212 (3)	C10—C19	1.546 (3)
O5—H5X	0.852 (18)	C11—C12	1.533 (3)
O5—H5Y	0.841 (18)	C11—H11A	0.9700
O6—H6X	0.84 (2)	C11—H11B	0.9700
O6—H6Y	0.799 (19)	C12—C13	1.518 (3)
C1—C2	1.526 (3)	C12—H12A	0.9700
C1—C10	1.544 (3)	C12—H12B	0.9700
C1—H1A	0.9700	C13—C17	1.518 (3)
C1—H1B	0.9700	C13—C14	1.541 (3)
C2—C3	1.505 (3)	C13—C18	1.547 (3)
C2—H2A	0.9700	C14—C15	1.531 (3)
C2—H2B	0.9700	C14—H14	0.9800
C3—C4	1.509 (3)	C15—C16	1.536 (3)
C3—H3	0.9800	C15—H15	0.9800
C4—C5	1.517 (3)	C16—C17	1.503 (3)
C4—H4A	0.9700	C16—H16A	0.9700
C4—H4B	0.9700	C16—H16B	0.9700
C5—C6	1.324 (3)	C18—H18A	0.9599
C5—C10	1.521 (3)	C18—H18B	0.9599
C6—C7	1.491 (2)	C18—H18C	0.9599
C6—H6	0.9300	C19—H19A	0.9599
C7—C8	1.536 (3)	C19—H19B	0.9599
C7—H7	0.9800	C19—H19C	0.9599
C3—O1—H1X	110 (2)	C1—C10—C19	109.70 (18)
C7—O2—H2X	102 (2)	C5—C10—C9	108.76 (15)
C15—O3—H3X	111 (2)	C1—C10—C9	108.25 (15)
H5X—O5—H5Y	100 (3)	C19—C10—C9	112.24 (16)
H6X—O6—H6Y	108 (5)	C12—C11—C9	113.61 (17)
C2—C1—C10	114.86 (16)	C12—C11—H11A	108.8
C2—C1—H1A	108.6	C9—C11—H11A	108.8
C10—C1—H1A	108.6	C12—C11—H11B	108.8
C2—C1—H1B	108.6	C9—C11—H11B	108.8
C10—C1—H1B	108.6	H11A—C11—H11B	107.7

H1A—C1—H1B	107.5	C13—C12—C11	110.04 (17)
C3—C2—C1	108.85 (16)	C13—C12—H12A	109.7
C3—C2—H2A	109.9	C11—C12—H12A	109.7
C1—C2—H2A	109.9	C13—C12—H12B	109.7
C3—C2—H2B	109.9	C11—C12—H12B	109.7
C1—C2—H2B	109.9	H12A—C12—H12B	108.2
H2A—C2—H2B	108.3	C12—C13—C17	116.87 (18)
O1—C3—C2	109.45 (16)	C12—C13—C14	108.75 (16)
O1—C3—C4	111.19 (16)	C17—C13—C14	99.50 (17)
C2—C3—C4	109.54 (17)	C12—C13—C18	111.8 (2)
O1—C3—H3	108.9	C17—C13—C18	105.57 (19)
C2—C3—H3	108.9	C14—C13—C18	113.90 (19)
C4—C3—H3	108.9	C8—C14—C15	121.00 (16)
C3—C4—C5	111.50 (15)	C8—C14—C13	114.19 (16)
C3—C4—H4A	109.3	C15—C14—C13	104.31 (15)
C5—C4—H4A	109.3	C8—C14—H14	105.3
C3—C4—H4B	109.3	C15—C14—H14	105.3
C5—C4—H4B	109.3	C13—C14—H14	105.3
H4A—C4—H4B	108.0	O3—C15—C14	109.50 (17)
C6—C5—C4	120.17 (17)	O3—C15—C16	112.7 (2)
C6—C5—C10	123.01 (17)	C14—C15—C16	102.98 (17)
C4—C5—C10	116.82 (16)	O3—C15—H15	110.5
C5—C6—C7	125.63 (18)	C14—C15—H15	110.5
C5—C6—H6	117.2	C16—C15—H15	110.5
C7—C6—H6	117.2	C17—C16—C15	106.06 (17)
O2—C7—C6	105.89 (17)	C17—C16—H16A	110.5
O2—C7—C8	112.73 (15)	C15—C16—H16A	110.5
C6—C7—C8	112.84 (16)	C17—C16—H16B	110.5
O2—C7—H7	108.4	C15—C16—H16B	110.5
C6—C7—H7	108.4	H16A—C16—H16B	108.7
C8—C7—H7	108.4	O4—C17—C16	124.8 (2)
C14—C8—C7	111.32 (15)	O4—C17—C13	126.6 (2)
C14—C8—C9	109.55 (14)	C16—C17—C13	108.55 (17)
C7—C8—C9	109.88 (14)	C13—C18—H18A	109.5
C14—C8—H8	108.7	C13—C18—H18B	109.5
C7—C8—H8	108.7	H18A—C18—H18B	109.5
C9—C8—H8	108.7	C13—C18—H18C	109.5
C11—C9—C8	113.58 (15)	H18A—C18—H18C	109.5
C11—C9—C10	112.49 (15)	H18B—C18—H18C	109.5
C8—C9—C10	111.77 (14)	C10—C19—H19A	109.5
C11—C9—H9	106.1	C10—C19—H19B	109.5
C8—C9—H9	106.1	H19A—C19—H19B	109.5
C10—C9—H9	106.1	C10—C19—H19C	109.5
C5—C10—C1	109.81 (15)	H19A—C19—H19C	109.5
C5—C10—C19	108.07 (17)	H19B—C19—H19C	109.5
C10—C1—C2—C3	−57.6 (3)	C11—C9—C10—C19	−59.7 (2)
C1—C2—C3—O1	−175.65 (17)	C8—C9—C10—C19	69.4 (2)
C1—C2—C3—C4	62.2 (2)	C8—C9—C11—C12	49.8 (3)
O1—C3—C4—C5	−179.56 (18)	C10—C9—C11—C12	178.05 (18)

C2—C3—C4—C5	−58.5 (2)	C9—C11—C12—C13	−54.7 (3)
C3—C4—C5—C6	−130.8 (2)	C11—C12—C13—C17	169.47 (19)
C3—C4—C5—C10	49.5 (3)	C11—C12—C13—C14	57.9 (2)
C4—C5—C6—C7	179.9 (2)	C11—C12—C13—C18	−68.7 (2)
C10—C5—C6—C7	−0.3 (3)	C7—C8—C14—C15	−58.5 (2)
C5—C6—C7—O2	−113.7 (2)	C9—C8—C14—C15	179.81 (17)
C5—C6—C7—C8	10.1 (3)	C7—C8—C14—C13	175.67 (16)
O2—C7—C8—C14	−40.9 (2)	C9—C8—C14—C13	53.9 (2)
C6—C7—C8—C14	−160.83 (17)	C12—C13—C14—C8	−60.2 (2)
O2—C7—C8—C9	80.62 (19)	C17—C13—C14—C8	177.07 (16)
C6—C7—C8—C9	−39.3 (2)	C18—C13—C14—C8	65.2 (2)
C14—C8—C9—C11	−47.6 (2)	C12—C13—C14—C15	165.60 (17)
C7—C8—C9—C11	−170.19 (16)	C17—C13—C14—C15	42.9 (2)
C14—C8—C9—C10	−176.18 (15)	C18—C13—C14—C15	−69.0 (2)
C7—C8—C9—C10	61.2 (2)	C8—C14—C15—O3	72.0 (2)
C6—C5—C10—C1	138.4 (2)	C13—C14—C15—O3	−157.68 (18)
C4—C5—C10—C1	−41.8 (2)	C8—C14—C15—C16	−167.8 (2)
C6—C5—C10—C19	−101.9 (2)	C13—C14—C15—C16	−37.5 (2)
C4—C5—C10—C19	77.8 (2)	O3—C15—C16—C17	134.6 (2)
C6—C5—C10—C9	20.2 (3)	C14—C15—C16—C17	16.7 (3)
C4—C5—C10—C9	−160.08 (17)	C15—C16—C17—O4	−167.5 (2)
C2—C1—C10—C5	46.0 (2)	C15—C16—C17—C13	10.4 (3)
C2—C1—C10—C19	−72.6 (2)	C12—C13—C17—O4	28.5 (3)
C2—C1—C10—C9	164.65 (18)	C14—C13—C17—O4	145.3 (2)
C11—C9—C10—C5	−179.26 (16)	C18—C13—C17—O4	−96.5 (3)
C8—C9—C10—C5	−50.1 (2)	C12—C13—C17—C16	−149.3 (2)
C11—C9—C10—C1	61.5 (2)	C14—C13—C17—C16	−32.6 (2)
C8—C9—C10—C1	−169.36 (16)	C18—C13—C17—C16	85.6 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1X···O6	0.830 (18)	1.80 (2)	2.618 (3)	170 (3)
O2—H2X···O3	0.835 (16)	1.98 (2)	2.755 (2)	153 (3)
O3—H3X···O5^{i}	0.844 (17)	1.889 (18)	2.731 (2)	176 (3)
O5—H5X···O1^{ii}	0.852 (18)	1.95 (2)	2.778 (2)	165 (3)
O5—H5Y···O1^{iii}	0.841 (18)	1.867 (19)	2.702 (2)	172 (3)
O6—H6X···O5^{iv}	0.84 (2)	1.92 (2)	2.747 (3)	168 (5)
O6—H6Y···O4^{v}	0.799 (19)	1.99 (2)	2.787 (3)	174 (5)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) *x*−1, *y*, *z*; (iii) *x*−1/2, −*y*+3/2, −*z*+2; (iv) *x*+3/2, −*y*+3/2, −*z*+2; (v) −*x*+2, *y*+1/2, −*z*+3/2.

Fig. 1

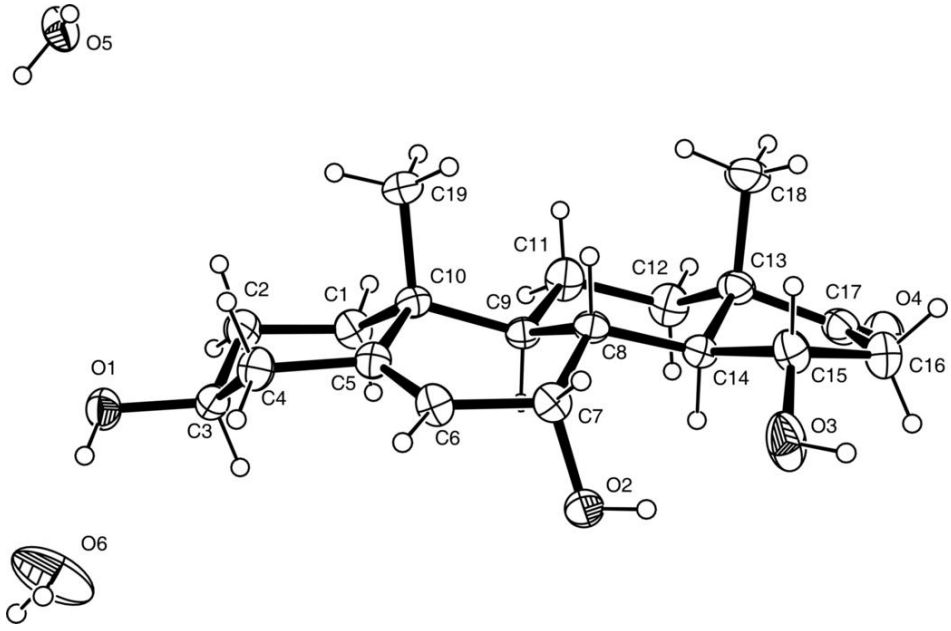
O5
C19
C18
C11
C12
C13
C2
C1
C10
C9
C17
O4
O1
C8
C14
C15
C16
C4
C5
C7
C3
C6
O3
O2
O6

Fig. 2