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11 β ,17 $\alpha\alpha$ -Dihydroxy-17 β -methyl-D-homoandrosta-1,4-diene-3,17-dione monohydrate

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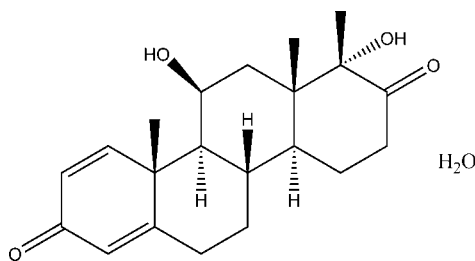
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.042; wR factor = 0.104; data-to-parameter ratio = 8.6.

In the title compound, $\text{C}_{21}\text{H}_{28}\text{O}_4 \cdot \text{H}_2\text{O}$, the cyclohexadienone ring is planar (r.m.s. deviation 0.0186 Å), whereas the two cyclohexane rings and the cyclohexanone ring adopt chair conformations. The crystal structure is stabilized by $\text{O}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds.

Related literature

For general background, see: Conrow (1999). For details of the synthesis, see: Huo (2003).



Experimental

Crystal data

 $\text{C}_{21}\text{H}_{28}\text{O}_4 \cdot \text{H}_2\text{O}$ $M_r = 362.45$

Monoclinic, $P2_1$
 $a = 6.641$ (2) Å
 $b = 18.642$ (6) Å
 $c = 8.017$ (3) Å
 $\beta = 103.797$ (4)°
 $V = 963.9$ (6) Å³

$Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.25 \times 0.20 \times 0.15$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.978$, $T_{\max} = 0.987$

4866 measured reflections
 2182 independent reflections
 1778 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.104$
 $S = 0.97$
 2182 reflections
 254 parameters
 5 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.15$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O2}-\text{H2X} \cdots \text{O5}$	0.821 (19)	2.00 (2)	2.799 (3)	166 (3)
$\text{O4}-\text{H4X} \cdots \text{O1}^{\text{i}}$	0.810 (18)	1.97 (2)	2.758 (3)	164 (3)
$\text{O5}-\text{H5Y} \cdots \text{O3}^{\text{ii}}$	0.86 (2)	1.99 (2)	2.851 (4)	171 (4)
$\text{O5}-\text{H5X} \cdots \text{O4}^{\text{iii}}$	0.85 (2)	1.97 (2)	2.811 (3)	172 (4)

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008b); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2193).

References

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 Huo, S. Q. (2003). *Org. Lett.* **5**, 423–425.
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supplementary materials

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11 β ,17 α -Dihydroxy-17 $\alpha\beta$ -methyl-D-homoandrosta-1,4-diene-3,17-dione monohydrate

Y. Qiu, Y. Chen and P. Xia

Comment

21-Methyl-11 β ,17 α -dihydroxy-1,4-pregnadien-3,20-dione derivatives are intermediates in the synthesis of steroid agents (Conrow, 1999). We tried to prepare 21-methyl-11 β ,17 α -dihydroxy-1,4-pregnadien-3,20-dione by 21-methylation of 11 β ,17 α -dihydroxy-21-chloro-1,4-pregnadien-3,20-dione with organic metal reagent. However, the reaction of 11 β ,17 α -dihydroxy-21-chloro-1,4-pregnadien-3,20-dione with a mixture of iodomethane, zinc dust, I₂(cat.) and Cl₂Ni(PPh₃)₂(cat.) in the solvent DMA offered the title compound as one of the main products. The crystal structure determination of the title compound was carried out in order to determine the exact molecular structure.

The molecular structure of the title compound is shown in Fig.1. The cyclohexadienone ring is planar. The two cyclohexane rings and the cyclohexanone ring adopt chair conformations. Intra- and intermolecular C—H \cdots O and O—H \cdots O hydrogen bonding are observed in the crystal structure.

Experimental

A mixture of CH₃I (568 mg, 4 mmol) (Huo,2003), zinc dust (387 mg, 6 mmol) and I₂ (50 mg, 0.2 mmol) in dry DMA was stirred under N₂ at 5°C until the red color of I₂ disappeared. After stirring further at 30°C for 3 h, the 11 β ,17 α -dihydroxy-21-chloro-1,4-pregnadien-3,20-dione (750 mg, 1.98 mmol) and Cl₂Ni(PPh₃)₂ (35 mg, 0.053 mmol) were added successively. The mixture was stirred at 90°C for 4 h. The TLC showed that the starting material was consumed completely. The mixture was filtered and the filtrate was poured into ice water(150 ml) and extracted with ethyl acetate. The organic layer was washed with brine, dried over Na₂SO₄, filtered and concentrated under reduced pressure. The residue was isolated by chromatography on silicagel column with petroleum ether/EtOAc(1:1) as eluent to afford the pure title compound, which was recrystallized from ethyl acetate to give colorless crystals for the single-crystal X-ray diffraction analysis. Yield: 26.5%.

Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged. All H atoms from C-H groups were positioned geometrically and refined using a riding model with C—H equal 0.93 - 0.98Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, except methyl groups where $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. The H atoms of the OH groups were fully refined.

Figures

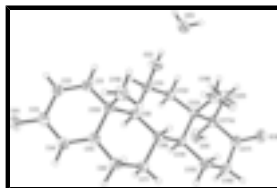


Fig. 1. The molecular structure of the title compound showing displacement ellipsoids at the 30% probability level.

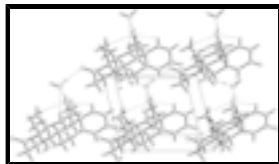


Fig. 2. Crystal packing of the title compound - projection along the *b* axis.

11 β ,17 α -Dihydroxy-17 α β -methyl-D-homoandrosta-1,4-diene-3,17-dione monohydrate

Crystal data

$C_{21}H_{28}O_4 \cdot H_2O$	$F_{000} = 392$
$M_r = 362.45$	$D_x = 1.249 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
Hall symbol: P 2yb	$\lambda = 0.71073 \text{ \AA}$
$a = 6.641 (2) \text{ \AA}$	Cell parameters from 1024 reflections
$b = 18.642 (6) \text{ \AA}$	$\theta = 2.8\text{--}27.1^\circ$
$c = 8.017 (3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 103.797 (4)^\circ$	$T = 293 \text{ K}$
$V = 963.9 (6) \text{ \AA}^3$	Plate, colorless
$Z = 2$	$0.25 \times 0.20 \times 0.15 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2182 independent reflections
Radiation source: fine-focus sealed tube	1778 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.034$
$T = 293 \text{ K}$	$\theta_{\text{max}} = 27.1^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.8^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2008a)	$h = -8 \rightarrow 8$
$T_{\text{min}} = 0.978$, $T_{\text{max}} = 0.987$	$k = -23 \rightarrow 23$
4866 measured reflections	$l = -10 \rightarrow 6$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.104$	$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2]$
$S = 0.97$	where $P = (F_o^2 + 2F_c^2)/3$
2182 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
254 parameters	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$

5 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.1926 (4)	0.41488 (11)	0.9326 (3)	0.0555 (5)
O2	0.6229 (3)	0.61548 (11)	0.4180 (3)	0.0509 (5)
H2X	0.691 (4)	0.6524 (13)	0.440 (4)	0.055 (10)*
O3	0.1121 (4)	0.77632 (12)	-0.2435 (3)	0.0583 (6)
O4	-0.0063 (3)	0.78258 (11)	0.1395 (3)	0.0499 (5)
H4X	-0.050 (4)	0.8214 (11)	0.102 (4)	0.039 (8)*
O5	0.8509 (4)	0.74238 (15)	0.4291 (4)	0.0686 (7)
H5Y	0.936 (5)	0.756 (3)	0.523 (4)	0.089 (15)*
H5X	0.894 (6)	0.750 (2)	0.340 (4)	0.086 (14)*
C1	0.4634 (4)	0.52002 (13)	0.6909 (4)	0.0427 (6)
H1	0.5753	0.5514	0.7111	0.051*
C2	0.4114 (4)	0.49166 (14)	0.8260 (4)	0.0433 (6)
H2	0.4894	0.5028	0.9355	0.052*
C3	0.2351 (4)	0.44350 (13)	0.8073 (4)	0.0409 (6)
C4	0.1154 (4)	0.43065 (13)	0.6331 (4)	0.0403 (6)
H4	-0.0030	0.4023	0.6169	0.048*
C5	0.1682 (4)	0.45781 (13)	0.4951 (3)	0.0371 (6)
C6	0.0458 (4)	0.44352 (13)	0.3178 (4)	0.0437 (6)
H6A	-0.0788	0.4173	0.3230	0.052*
H6B	0.1265	0.4136	0.2591	0.052*
C7	-0.0148 (4)	0.51213 (14)	0.2155 (4)	0.0418 (6)
H7A	-0.1214	0.5364	0.2585	0.050*
H7B	-0.0738	0.4997	0.0964	0.050*
C8	0.1668 (4)	0.56397 (12)	0.2240 (3)	0.0328 (5)
H8	0.2664	0.5417	0.1673	0.039*
C9	0.2742 (4)	0.57722 (12)	0.4142 (3)	0.0332 (5)
H9	0.1632	0.5932	0.4671	0.040*
C10	0.3559 (4)	0.50534 (13)	0.5089 (3)	0.0366 (6)
C11	0.4311 (4)	0.63875 (13)	0.4440 (4)	0.0404 (6)
H11	0.4551	0.6519	0.5655	0.048*

supplementary materials

C12	0.3482 (4)	0.70560 (12)	0.3407 (3)	0.0392 (6)
H12A	0.4596	0.7404	0.3542	0.047*
H12B	0.2406	0.7265	0.3886	0.047*
C13	0.2592 (4)	0.69250 (12)	0.1481 (3)	0.0337 (5)
C14	0.0878 (4)	0.63452 (12)	0.1298 (3)	0.0342 (5)
H14	-0.0177	0.6538	0.1847	0.041*
C15	-0.0196 (5)	0.62098 (16)	-0.0586 (4)	0.0485 (7)
H15A	0.0780	0.5987	-0.1153	0.058*
H15B	-0.1337	0.5879	-0.0644	0.058*
C16	-0.1023 (5)	0.68968 (17)	-0.1538 (4)	0.0535 (7)
H16A	-0.1490	0.6796	-0.2756	0.064*
H16B	-0.2203	0.7067	-0.1135	0.064*
C17	0.0599 (4)	0.74649 (14)	-0.1266 (4)	0.0415 (6)
C17A	0.1594 (4)	0.76373 (13)	0.0615 (3)	0.0396 (6)
C18	0.4297 (4)	0.67001 (16)	0.0598 (4)	0.0453 (6)
H18A	0.4766	0.6226	0.0963	0.068*
H18B	0.5437	0.7030	0.0901	0.068*
H18C	0.3758	0.6704	-0.0625	0.068*
C19	0.5131 (4)	0.46446 (16)	0.4290 (4)	0.0526 (7)
H19A	0.5383	0.4178	0.4805	0.079*
H19B	0.6408	0.4908	0.4493	0.079*
H19C	0.4575	0.4594	0.3075	0.079*
C20	0.3116 (5)	0.82621 (15)	0.0757 (4)	0.0559 (8)
H20A	0.3711	0.8364	0.1946	0.084*
H20B	0.2395	0.8678	0.0213	0.084*
H20C	0.4196	0.8136	0.0201	0.084*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0786 (14)	0.0499 (11)	0.0436 (12)	-0.0146 (10)	0.0255 (10)	0.0001 (9)
O2	0.0356 (9)	0.0403 (11)	0.0753 (15)	-0.0050 (8)	0.0104 (10)	0.0063 (10)
O3	0.0817 (14)	0.0535 (12)	0.0414 (12)	-0.0120 (11)	0.0181 (10)	0.0069 (10)
O4	0.0664 (12)	0.0407 (10)	0.0477 (12)	0.0179 (9)	0.0239 (10)	0.0110 (9)
O5	0.0723 (15)	0.0808 (17)	0.0542 (15)	-0.0300 (13)	0.0177 (13)	0.0031 (13)
C1	0.0380 (13)	0.0372 (13)	0.0484 (16)	-0.0063 (11)	0.0014 (12)	0.0077 (12)
C2	0.0526 (15)	0.0356 (13)	0.0384 (15)	-0.0026 (11)	0.0040 (12)	0.0039 (11)
C3	0.0528 (14)	0.0294 (12)	0.0445 (16)	0.0016 (11)	0.0195 (12)	0.0001 (11)
C4	0.0419 (13)	0.0328 (12)	0.0482 (16)	-0.0065 (10)	0.0147 (12)	0.0030 (11)
C5	0.0410 (12)	0.0267 (11)	0.0430 (15)	-0.0010 (10)	0.0091 (11)	-0.0008 (10)
C6	0.0512 (14)	0.0335 (13)	0.0460 (15)	-0.0118 (11)	0.0111 (13)	-0.0026 (12)
C7	0.0439 (14)	0.0387 (13)	0.0404 (15)	-0.0111 (11)	0.0053 (12)	-0.0011 (12)
C8	0.0346 (12)	0.0295 (11)	0.0329 (13)	-0.0045 (9)	0.0055 (10)	-0.0011 (9)
C9	0.0339 (11)	0.0309 (11)	0.0345 (13)	-0.0027 (9)	0.0079 (10)	0.0011 (9)
C10	0.0339 (12)	0.0348 (13)	0.0414 (14)	-0.0007 (10)	0.0098 (10)	0.0072 (11)
C11	0.0410 (13)	0.0378 (13)	0.0385 (15)	-0.0083 (10)	0.0017 (11)	0.0030 (11)
C12	0.0460 (13)	0.0309 (12)	0.0385 (14)	-0.0086 (10)	0.0056 (11)	-0.0009 (10)
C13	0.0377 (12)	0.0283 (11)	0.0344 (13)	-0.0044 (9)	0.0073 (10)	-0.0003 (9)

C14	0.0350 (12)	0.0332 (12)	0.0337 (14)	-0.0032 (9)	0.0069 (10)	-0.0012 (10)
C15	0.0567 (16)	0.0434 (14)	0.0386 (15)	-0.0154 (13)	-0.0024 (13)	0.0006 (12)
C16	0.0551 (15)	0.0609 (18)	0.0382 (16)	-0.0120 (14)	-0.0014 (13)	0.0072 (13)
C17	0.0504 (14)	0.0373 (13)	0.0357 (15)	0.0037 (11)	0.0082 (12)	0.0060 (11)
C17A	0.0497 (14)	0.0338 (13)	0.0365 (14)	0.0013 (11)	0.0129 (12)	0.0019 (10)
C18	0.0452 (13)	0.0444 (14)	0.0498 (17)	-0.0029 (12)	0.0180 (12)	-0.0005 (12)
C19	0.0520 (16)	0.0423 (15)	0.068 (2)	0.0121 (13)	0.0239 (15)	0.0125 (14)
C20	0.075 (2)	0.0386 (15)	0.0492 (18)	-0.0124 (14)	0.0058 (15)	0.0027 (13)

Geometric parameters (Å, °)

O1—C3	1.228 (3)	C9—H9	0.9800
O2—C11	1.407 (3)	C10—C19	1.549 (4)
O2—H2X	0.821 (19)	C11—C12	1.525 (3)
O3—C17	1.209 (4)	C11—H11	0.9800
O4—C17A	1.433 (3)	C12—C13	1.535 (4)
O4—H4X	0.810 (18)	C12—H12A	0.9700
O5—H5Y	0.86 (2)	C12—H12B	0.9700
O5—H5X	0.85 (2)	C13—C18	1.531 (4)
C1—C2	1.323 (4)	C13—C14	1.551 (3)
C1—C10	1.489 (4)	C13—C17A	1.570 (3)
C1—H1	0.9300	C14—C15	1.530 (4)
C2—C3	1.454 (4)	C14—H14	0.9800
C2—H2	0.9300	C15—C16	1.524 (4)
C3—C4	1.452 (4)	C15—H15A	0.9700
C4—C5	1.337 (4)	C15—H15B	0.9700
C4—H4	0.9300	C16—C17	1.489 (4)
C5—C6	1.483 (4)	C16—H16A	0.9700
C5—C10	1.512 (3)	C16—H16B	0.9700
C6—C7	1.521 (4)	C17—C17A	1.529 (4)
C6—H6A	0.9700	C17A—C20	1.528 (4)
C6—H6B	0.9700	C18—H18A	0.9599
C7—C8	1.535 (3)	C18—H18B	0.9599
C7—H7A	0.9700	C18—H18C	0.9599
C7—H7B	0.9700	C19—H19A	0.9599
C8—C9	1.541 (3)	C19—H19B	0.9599
C8—C14	1.545 (3)	C19—H19C	0.9599
C8—H8	0.9800	C20—H20A	0.9599
C9—C11	1.530 (3)	C20—H20B	0.9599
C9—C10	1.571 (3)	C20—H20C	0.9599
C11—O2—H2X	101 (2)	C13—C12—H12A	108.6
C17A—O4—H4X	108 (2)	C11—C12—H12B	108.6
H5Y—O5—H5X	114 (4)	C13—C12—H12B	108.6
C2—C1—C10	124.8 (2)	H12A—C12—H12B	107.6
C2—C1—H1	117.6	C18—C13—C12	111.0 (2)
C10—C1—H1	117.6	C18—C13—C14	111.9 (2)
C1—C2—C3	121.5 (3)	C12—C13—C14	107.69 (19)
C1—C2—H2	119.2	C18—C13—C17A	107.9 (2)
C3—C2—H2	119.2	C12—C13—C17A	109.33 (19)

supplementary materials

O1—C3—C4	122.3 (2)	C14—C13—C17A	108.94 (19)
O1—C3—C2	121.3 (3)	C15—C14—C8	111.5 (2)
C4—C3—C2	116.3 (2)	C15—C14—C13	111.5 (2)
C5—C4—C3	122.8 (2)	C8—C14—C13	113.00 (19)
C5—C4—H4	118.6	C15—C14—H14	106.8
C3—C4—H4	118.6	C8—C14—H14	106.8
C4—C5—C6	122.2 (2)	C13—C14—H14	106.8
C4—C5—C10	122.4 (2)	C16—C15—C14	112.5 (2)
C6—C5—C10	115.4 (2)	C16—C15—H15A	109.1
C5—C6—C7	112.3 (2)	C14—C15—H15A	109.1
C5—C6—H6A	109.1	C16—C15—H15B	109.1
C7—C6—H6A	109.1	C14—C15—H15B	109.1
C5—C6—H6B	109.1	H15A—C15—H15B	107.8
C7—C6—H6B	109.1	C17—C16—C15	111.3 (2)
H6A—C6—H6B	107.9	C17—C16—H16A	109.4
C6—C7—C8	113.7 (2)	C15—C16—H16A	109.4
C6—C7—H7A	108.8	C17—C16—H16B	109.4
C8—C7—H7A	108.8	C15—C16—H16B	109.4
C6—C7—H7B	108.8	H16A—C16—H16B	108.0
C8—C7—H7B	108.8	O3—C17—C16	123.0 (3)
H7A—C7—H7B	107.7	O3—C17—C17A	122.1 (3)
C7—C8—C9	108.5 (2)	C16—C17—C17A	114.9 (2)
C7—C8—C14	110.02 (19)	O4—C17A—C20	110.1 (2)
C9—C8—C14	111.72 (19)	O4—C17A—C17	106.6 (2)
C7—C8—H8	108.8	C20—C17A—C17	110.9 (2)
C9—C8—H8	108.8	O4—C17A—C13	107.5 (2)
C14—C8—H8	108.8	C20—C17A—C13	114.2 (2)
C11—C9—C8	114.3 (2)	C17—C17A—C13	107.2 (2)
C11—C9—C10	114.83 (19)	C13—C18—H18A	109.5
C8—C9—C10	111.49 (19)	C13—C18—H18B	109.5
C11—C9—H9	105.0	H18A—C18—H18B	109.5
C8—C9—H9	105.0	C13—C18—H18C	109.5
C10—C9—H9	105.0	H18A—C18—H18C	109.5
C1—C10—C5	111.9 (2)	H18B—C18—H18C	109.5
C1—C10—C19	106.4 (2)	C10—C19—H19A	109.5
C5—C10—C19	107.8 (2)	C10—C19—H19B	109.5
C1—C10—C9	110.3 (2)	H19A—C19—H19B	109.5
C5—C10—C9	106.31 (19)	C10—C19—H19C	109.5
C19—C10—C9	114.1 (2)	H19A—C19—H19C	109.5
O2—C11—C12	113.2 (2)	H19B—C19—H19C	109.5
O2—C11—C9	110.6 (2)	C17A—C20—H20A	109.5
C12—C11—C9	112.6 (2)	C17A—C20—H20B	109.5
O2—C11—H11	106.7	H20A—C20—H20B	109.5
C12—C11—H11	106.7	C17A—C20—H20C	109.5
C9—C11—H11	106.7	H20A—C20—H20C	109.5
C11—C12—C13	114.8 (2)	H20B—C20—H20C	109.5
C11—C12—H12A	108.6		
C10—C1—C2—C3	-1.7 (4)	O2—C11—C12—C13	74.9 (3)
C1—C2—C3—O1	176.9 (3)	C9—C11—C12—C13	-51.5 (3)

C1—C2—C3—C4	-2.3 (4)	C11—C12—C13—C18	-66.8 (3)
O1—C3—C4—C5	-175.5 (3)	C11—C12—C13—C14	56.0 (3)
C2—C3—C4—C5	3.7 (4)	C11—C12—C13—C17A	174.3 (2)
C3—C4—C5—C6	179.3 (2)	C7—C8—C14—C15	-59.1 (3)
C3—C4—C5—C10	-1.0 (4)	C9—C8—C14—C15	-179.7 (2)
C4—C5—C6—C7	127.3 (3)	C7—C8—C14—C13	174.4 (2)
C10—C5—C6—C7	-52.5 (3)	C9—C8—C14—C13	53.8 (3)
C5—C6—C7—C8	49.2 (3)	C18—C13—C14—C15	-61.1 (3)
C6—C7—C8—C9	-52.6 (3)	C12—C13—C14—C15	176.6 (2)
C6—C7—C8—C14	-175.2 (2)	C17A—C13—C14—C15	58.1 (3)
C7—C8—C9—C11	-168.7 (2)	C18—C13—C14—C8	65.4 (3)
C14—C8—C9—C11	-47.2 (3)	C12—C13—C14—C8	-56.9 (3)
C7—C8—C9—C10	59.0 (2)	C17A—C13—C14—C8	-175.36 (19)
C14—C8—C9—C10	-179.54 (19)	C8—C14—C15—C16	179.1 (2)
C2—C1—C10—C5	4.1 (4)	C13—C14—C15—C16	-53.6 (3)
C2—C1—C10—C19	-113.5 (3)	C14—C15—C16—C17	50.2 (4)
C2—C1—C10—C9	122.3 (3)	C15—C16—C17—O3	124.4 (3)
C4—C5—C10—C1	-2.7 (3)	C15—C16—C17—C17A	-54.6 (4)
C6—C5—C10—C1	177.0 (2)	O3—C17—C17A—O4	125.0 (3)
C4—C5—C10—C19	114.0 (3)	C16—C17—C17A—O4	-56.0 (3)
C6—C5—C10—C19	-66.2 (3)	O3—C17—C17A—C20	5.2 (4)
C4—C5—C10—C9	-123.2 (3)	C16—C17—C17A—C20	-175.8 (3)
C6—C5—C10—C9	56.6 (3)	O3—C17—C17A—C13	-120.1 (3)
C11—C9—C10—C1	46.5 (3)	C16—C17—C17A—C13	58.9 (3)
C8—C9—C10—C1	178.55 (19)	C18—C13—C17A—O4	177.4 (2)
C11—C9—C10—C5	168.0 (2)	C12—C13—C17A—O4	-61.7 (2)
C8—C9—C10—C5	-59.9 (3)	C14—C13—C17A—O4	55.7 (3)
C11—C9—C10—C19	-73.2 (3)	C18—C13—C17A—C20	-60.2 (3)
C8—C9—C10—C19	58.8 (3)	C12—C13—C17A—C20	60.7 (3)
C8—C9—C11—O2	-81.9 (3)	C14—C13—C17A—C20	178.2 (2)
C10—C9—C11—O2	48.8 (3)	C18—C13—C17A—C17	63.1 (3)
C8—C9—C11—C12	45.8 (3)	C12—C13—C17A—C17	-176.0 (2)
C10—C9—C11—C12	176.5 (2)	C14—C13—C17A—C17	-58.6 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2X \cdots O5	0.821 (19)	2.00 (2)	2.799 (3)	166 (3)
C1—H1 \cdots O2	0.93	2.72	3.191 (3)	112
C12—H12A \cdots O5	0.97	2.52	3.315 (4)	139
C18—H18A \cdots O2	0.96	2.53	3.028 (4)	112
C19—H19B \cdots O2	0.96	2.34	2.915 (4)	118
O4—H4X \cdots O1 ⁱ	0.810 (18)	1.97 (2)	2.758 (3)	164 (3)
O5—H5Y \cdots O3 ⁱⁱ	0.86 (2)	1.99 (2)	2.851 (4)	171 (4)
O5—H5X \cdots O4 ⁱⁱⁱ	0.85 (2)	1.97 (2)	2.811 (3)	172 (4)
C16—H16A \cdots O5 ^{iv}	0.97	2.64	3.427 (4)	138
C6—H6A \cdots O3 ^v	0.97	2.70	3.297 (3)	120

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

Fig. 1

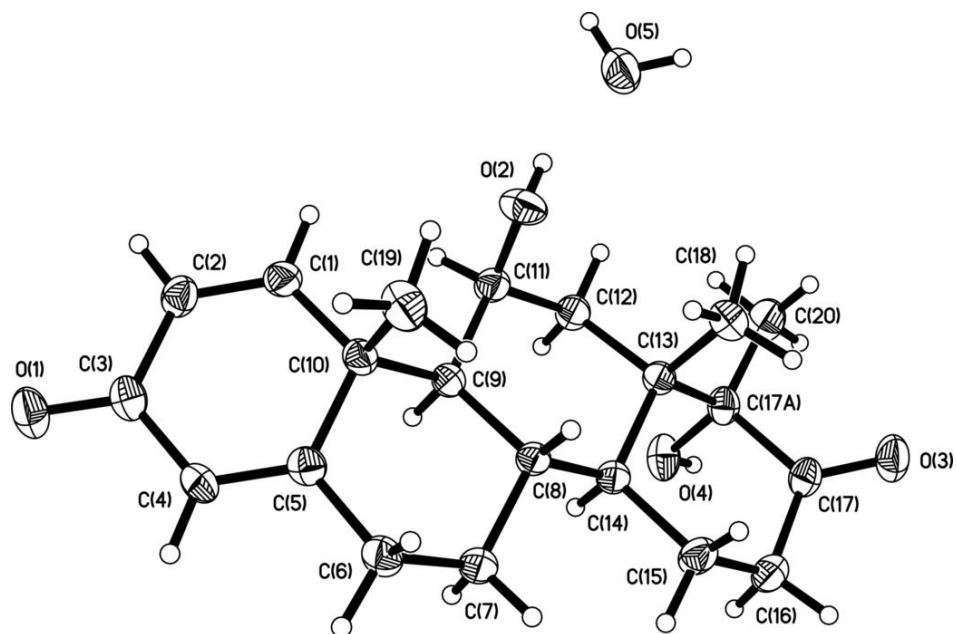


Fig. 2

