Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5α-Acetamido-6β-hydroxy-17-oxoandrostan-3β-yl acetate

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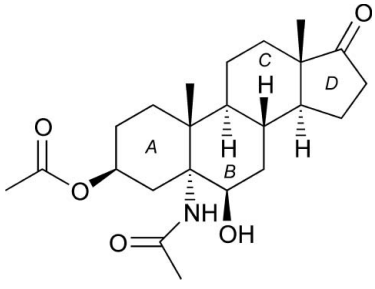
Received 18 June 2007; accepted 19 June 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.034; wR factor = 0.098; data-to-parameter ratio = 8.2.

Using a method developed by our group, we prepared, by a one-step reaction, the title *N*-acetylated hydroxyaminoandrostane, $C_{23}H_{35}NO_5$, from the corresponding 5β,6β-epoxy steroid. The stereoselective nucleophilic attack of acetonitrile to the α-face of the steroid nucleus at position 5 is unequivocally demonstrated by X-ray crystallographic analysis. Intermolecular O—H···O hydrogen bonds are present in the crystal structure.

Related literature

For vicinal amino alcohols see Bergmeier (2000). For androstanes with 2-amino-3-ol functionality see Tuba *et al.* (2002) and Gyermek (2005). For compounds inhibiting the proliferation of leukemia cells see He & Jiang (1999) and He & Na (2001), and for the preparation of *vic*-hydroxyacylamino steroids see Vincze *et al.* (1996). For related literature, see: Cremer & Pople (1975); Pinto *et al.* (2006); Salvador *et al.* (1996).



Experimental

Crystal data

$C_{23}H_{35}NO_5$
M_r = 405.52
Monoclinic, $P2_1$
a = 8.660 (3) Å
b = 9.423 (3) Å
c = 13.973 (2) Å
β = 104.00 (2)°
V = 1106.4 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.42 × 0.20 × 0.17 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
3483 measured reflections
2192 independent reflections
1883 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
3 standard reflections
frequency: 300 min
intensity decay: 9.2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.098
S = 1.03
2192 reflections
267 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6A···O17^{i}	0.82	2.03	2.823 (3)	164

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *PLATON* (Spek, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

This work was supported by Fundação para a Ciência e Tecnologia. RMAP thanks FCT for a grant (SFRH/BD/18013/2004).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2399).

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 doi:10.1107/S1600536807029893

supplementary materials

Acta Cryst. (2007). E**63**, o3321 [**doi:10.1107/S1600536807029893**]

5α-Acetamido-6β-hydroxy-17-oxoandrostan-3β-yl acetate

R. M. A. Pinto, M. Ramos Silva, A. Matos Beja, J. A. R. Salvador and J. A. Paixão

Comment

Using a method developed by our group (Pinto *et al.*, 2006) we prepared, by a one-step reaction, the *N*-acetylated-hydroxy-amino-androstane (I) from the corresponding 5β,6β-epoxysteroid. The steroselective nucleophilic attack of acetonitrile to the α-face of steroid nucleus at C5 is unequivocally demonstrated by X-ray crystallography.

The conformations of the six-membered rings are close to a chair form, as shown by the Cremer & Pople (1975) puckring parameters [ring A: Q= 0.586 (3) Å, θ = 7.9 (3)° and φ = 260 (2)°; ring B: Q= 0.562 (3) Å, θ = 2.7 (3)° and φ = 238 (6)°; ring C: Q= 0.580 (3) Å, θ = 5.4 (3)° and φ = 269 (3)°]. The D-ring has a C14 envelope conformation with puckering parameters q_2 = 0.580 (3)Å and φ_2 = 209.2 (4)°. All rings of the molecule are fused *trans*. The acetoxy group at C3 is equatorial to the A ring, and both substituents at the B ring are axial.

The molecules are hydrogen-bonded in infinite chains running parallel to the *b* axis through the hydroxyl group at C6, acting as donor towards the carbonyl O atom of the D ring.

The anistropic displacement tensor of the O3B atom is strongly anisotropic, suggesting a strong amplitude of vibration of this atom perpendicular to the mean plane of the acetoxy group.

Experimental

5β,6β-Epoxy-17-oxoandrostan-3β-yl acetate was easily prepared from commercially available dehydroepiandrosterone acetate by epoxidation with $KMnO_4/Fe_2(SO_4)_3nH_2O$ (Salvador *et al.*, 1996).

Synthesis of 5α-acetamido-6β-hydroxy-17-oxoandrostan-3β-yl acetate (I) was efficiently accomplished by nucleophilic ring opening of the 5β,6β-epoxysteroid catalysed by $BiBr_3$ in acetonitrile (Pinto *et al.*, 2006). The product of this reaction was isolated in 86% yield and identified as the title compound (I) from IR, ^{1}H and ^{13}C NMR spectroscopy data (Pinto *et al.*, 2006). Recrystallization from ethyl acetate at room temperature gave colorless single crystals suitable for X-ray analysis.

Refinement

All hydrogen atoms were refined as riding on their parent atoms using *SHELXL97* defaults except for that of the hydroxyl group which had its coordinates freely refined with U_{iso}= 1.5 U_{eq} of the O atoms. In the absence of anomalous scatterers Friedel pairs had been merged. The absolute configuration was not determined from the X-ray data but was known from the synthetic route.

Figures

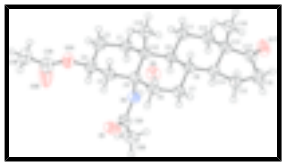


Fig. 1. *ORTEPII* (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 50% level.

5α-Acetamido-6β-hydroxy-17-oxoandrostan-3β-yl acetate

Crystal data

$C_{23}H_{35}NO_5$	F_{000} = 440
M_r = 405.52	D_x = 1.216 Mg m^{-3}
Monoclinic, $P2_1$	Melting point: 507 K
Hall symbol: P 2yb	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.660 (3) Å	Cell parameters from 25 reflections
b = 9.423 (3) Å	θ = 7.9–13.3º
c = 13.973 (2) Å	μ = 0.09 mm^{-1}
β = 104.00 (2)º	T = 293 (2) K
V = 1106.4 (5) Å^3	Prism, colourless
Z = 2	0.42 × 0.20 × 0.17 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.024
Radiation source: fine-focus sealed tube	θ_{max} = 25.5º
Monochromator: graphite	θ_{min} = 2.4º
T = 293(2) K	h = −10→10
profile data from ω–2θ scans	k = 0→11
Absorption correction: none	l = 0→16
3483 measured reflections	3 standard reflections
2192 independent reflections	every 300 min
1883 reflections with $I > 2\sigma(I)$	intensity decay: 9.2%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.034	H-atom parameters constrained
$wR(F^2)$ = 0.098	$w = 1/[\sigma^2(F_o^2) + (0.0652P)^2 + 0.0914P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2192 reflections	$\Delta\rho_{max}$ = 0.18 e Å^{-3}

267 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
1 restraint	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}
C1	−0.1925 (3)	0.6556 (3)	0.19050 (17)	0.0422 (5)
H1A	−0.2369	0.7358	0.2182	0.051*
H1B	−0.1785	0.6837	0.1263	0.051*
C2	−0.3100 (3)	0.5315 (3)	0.17750 (19)	0.0496 (6)
H2A	−0.3373	0.5133	0.2397	0.059*
H2B	−0.4068	0.5575	0.1295	0.059*
C3	−0.2429 (3)	0.3969 (3)	0.14364 (18)	0.0461 (6)
H3	−0.2379	0.4068	0.0746	0.055*
C4	−0.0795 (3)	0.3591 (3)	0.20714 (17)	0.0413 (5)
H4A	−0.0382	0.2771	0.1795	0.050*
H4B	−0.0880	0.3350	0.2732	0.050*
C5	0.0352 (3)	0.4848 (2)	0.21202 (16)	0.0369 (5)
C6	0.2071 (3)	0.4441 (2)	0.26681 (16)	0.0391 (5)
H6	0.2446	0.3703	0.2284	0.047*
C7	0.3188 (3)	0.5697 (3)	0.27595 (17)	0.0407 (5)
H7A	0.3306	0.5950	0.2108	0.049*
H7B	0.4227	0.5420	0.3153	0.049*
C8	0.2619 (3)	0.7002 (3)	0.32298 (17)	0.0364 (5)
H8	0.2591	0.6765	0.3908	0.044*
C9	0.0917 (3)	0.7437 (2)	0.26546 (16)	0.0342 (5)
H9	0.0982	0.7647	0.1978	0.041*
C10	−0.0284 (2)	0.6185 (3)	0.25829 (16)	0.0367 (5)
C11	0.0374 (3)	0.8822 (3)	0.30685 (18)	0.0412 (5)
H11A	−0.0640	0.9111	0.2648	0.049*
H11B	0.0210	0.8634	0.3719	0.049*
C12	0.1558 (3)	1.0047 (3)	0.31421 (18)	0.0442 (5)
H12A	0.1626	1.0332	0.2486	0.053*
H12B	0.1195	1.0856	0.3457	0.053*
C13	0.3196 (3)	0.9583 (3)	0.37413 (16)	0.0394 (5)

C14	0.3717 (2)	0.8260 (3)	0.32554 (16)	0.0373 (5)
H14	0.3651	0.8515	0.2567	0.045*
C15	0.5498 (3)	0.8126 (3)	0.37556 (19)	0.0470 (6)
H15A	0.5661	0.7693	0.4402	0.056*
H15B	0.6050	0.7572	0.3356	0.056*
C16	0.6055 (3)	0.9679 (3)	0.3833 (2)	0.0540 (7)
H16A	0.6812	0.9847	0.4458	0.065*
H16B	0.6558	0.9910	0.3303	0.065*
C17	0.4570 (3)	1.0565 (3)	0.37548 (16)	0.0442 (6)
C19	−0.0502 (3)	0.5862 (3)	0.36241 (17)	0.0450 (6)
H19A	−0.1069	0.6628	0.3838	0.068*
H19B	0.0522	0.5759	0.4074	0.068*
H19C	−0.1096	0.4999	0.3609	0.068*
C18	0.3203 (3)	0.9363 (3)	0.48361 (17)	0.0516 (6)
H18A	0.4266	0.9161	0.5206	0.077*
H18B	0.2518	0.8583	0.4895	0.077*
H18C	0.2827	1.0209	0.5089	0.077*
O17	0.4532 (2)	1.1856 (2)	0.37299 (15)	0.0592 (5)
O6	0.2078 (2)	0.3868 (2)	0.36148 (12)	0.0502 (4)
H6A	0.2764	0.3251	0.3757	0.075*
N5	0.0466 (2)	0.5232 (2)	0.11079 (12)	0.0375 (4)
H5	0.0289	0.6107	0.0943	0.045*
C5A	0.0811 (3)	0.4375 (3)	0.04173 (17)	0.0418 (5)
C5B	0.1138 (4)	0.5119 (3)	−0.04541 (19)	0.0613 (8)
H5B1	0.1887	0.4577	−0.0710	0.092*
H5B2	0.1574	0.6041	−0.0259	0.092*
H5B3	0.0166	0.5219	−0.0954	0.092*
O5	0.0876 (3)	0.3078 (2)	0.04790 (15)	0.0663 (6)
O3A	−0.3466 (2)	0.2780 (2)	0.15344 (13)	0.0534 (5)
O3B	−0.4874 (4)	0.3024 (3)	−0.0007 (2)	0.1098 (11)
C3A	−0.4650 (3)	0.2446 (3)	0.0767 (2)	0.0562 (7)
C3B	−0.5640 (3)	0.1271 (4)	0.1004 (2)	0.0636 (7)
H3B1	−0.6710	0.1366	0.0612	0.095*
H3B2	−0.5641	0.1311	0.1690	0.095*
H3B3	−0.5209	0.0379	0.0863	0.095*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0371 (11)	0.0423 (14)	0.0472 (12)	0.0075 (10)	0.0101 (9)	0.0009 (11)
C2	0.0364 (11)	0.0528 (16)	0.0584 (14)	0.0017 (12)	0.0093 (10)	0.0014 (13)
C3	0.0449 (13)	0.0456 (14)	0.0477 (13)	−0.0075 (11)	0.0111 (10)	0.0015 (11)
C4	0.0470 (12)	0.0346 (12)	0.0430 (12)	0.0013 (10)	0.0124 (10)	0.0009 (10)
C5	0.0404 (11)	0.0334 (12)	0.0369 (11)	0.0031 (9)	0.0094 (9)	0.0004 (9)
C6	0.0436 (12)	0.0317 (12)	0.0418 (12)	0.0110 (10)	0.0098 (9)	−0.0015 (10)
C7	0.0345 (10)	0.0403 (13)	0.0456 (12)	0.0092 (10)	0.0067 (9)	−0.0027 (10)
C8	0.0361 (11)	0.0353 (12)	0.0372 (11)	0.0084 (10)	0.0077 (9)	−0.0003 (9)
C9	0.0357 (10)	0.0325 (11)	0.0347 (10)	0.0062 (9)	0.0088 (8)	−0.0009 (9)

C10	0.0357 (10)	0.0355 (11)	0.0391 (11)	0.0055 (10)	0.0095 (8)	−0.0004 (9)
C11	0.0360 (11)	0.0379 (12)	0.0485 (12)	0.0101 (10)	0.0078 (9)	−0.0065 (11)
C12	0.0418 (12)	0.0369 (13)	0.0507 (13)	0.0089 (10)	0.0053 (10)	−0.0062 (11)
C13	0.0407 (12)	0.0363 (12)	0.0380 (11)	0.0094 (10)	0.0035 (9)	−0.0046 (10)
C14	0.0381 (11)	0.0382 (12)	0.0343 (11)	0.0046 (10)	0.0062 (9)	−0.0034 (10)
C15	0.0370 (11)	0.0471 (14)	0.0542 (14)	0.0098 (11)	0.0056 (10)	−0.0055 (12)
C16	0.0397 (12)	0.0499 (15)	0.0676 (16)	0.0000 (12)	0.0036 (11)	−0.0063 (13)
C17	0.0467 (13)	0.0432 (14)	0.0382 (11)	0.0015 (11)	0.0015 (10)	−0.0061 (10)
C19	0.0480 (12)	0.0454 (14)	0.0457 (12)	0.0032 (11)	0.0191 (10)	0.0005 (11)
C18	0.0584 (14)	0.0527 (15)	0.0427 (13)	0.0046 (13)	0.0104 (10)	−0.0105 (12)
O17	0.0611 (11)	0.0375 (10)	0.0738 (12)	0.0001 (9)	0.0063 (9)	−0.0050 (9)
O6	0.0572 (10)	0.0440 (10)	0.0459 (9)	0.0116 (8)	0.0057 (7)	0.0097 (8)
N5	0.0450 (10)	0.0308 (10)	0.0371 (9)	0.0045 (8)	0.0109 (7)	0.0009 (8)
C5A	0.0461 (13)	0.0361 (14)	0.0447 (12)	0.0014 (10)	0.0134 (10)	−0.0055 (10)
C5B	0.093 (2)	0.0482 (16)	0.0485 (14)	0.0068 (15)	0.0286 (14)	−0.0020 (13)
O5	0.1023 (15)	0.0348 (11)	0.0745 (13)	0.0041 (10)	0.0460 (11)	−0.0049 (9)
O3A	0.0525 (9)	0.0529 (11)	0.0524 (10)	−0.0122 (9)	0.0081 (8)	0.0028 (9)
O3B	0.1223 (18)	0.111 (2)	0.0716 (13)	−0.0607 (19)	−0.0249 (13)	0.0291 (16)
C3A	0.0540 (14)	0.0500 (16)	0.0595 (16)	−0.0062 (13)	0.0038 (12)	0.0006 (14)
C3B	0.0577 (15)	0.0591 (18)	0.0717 (18)	−0.0146 (15)	0.0112 (13)	−0.0009 (15)

Geometric parameters (Å, °)

C1—C2	1.532 (4)	C12—H12B	0.9700
C1—C10	1.545 (3)	C13—C17	1.504 (4)
C1—H1A	0.9700	C13—C14	1.538 (3)
C1—H1B	0.9700	C13—C18	1.542 (3)
C2—C3	1.517 (4)	C14—C15	1.537 (3)
C2—H2A	0.9700	C14—H14	0.9800
C2—H2B	0.9700	C15—C16	1.536 (4)
C3—O3A	1.463 (3)	C15—H15A	0.9700
C3—C4	1.520 (3)	C15—H15B	0.9700
C3—H3	0.9800	C16—C17	1.515 (4)
C4—C5	1.537 (3)	C16—H16A	0.9700
C4—H4A	0.9700	C16—H16B	0.9700
C4—H4B	0.9700	C17—O17	1.217 (3)
C5—N5	1.486 (3)	C19—H19A	0.9600
C5—C6	1.548 (3)	C19—H19B	0.9600
C5—C10	1.575 (3)	C19—H19C	0.9600
C6—O6	1.427 (3)	C18—H18A	0.9600
C6—C7	1.515 (3)	C18—H18B	0.9600
C6—H6	0.9800	C18—H18C	0.9600
C7—C8	1.531 (3)	O6—H6A	0.8200
C7—H7A	0.9700	N5—C5A	1.346 (3)
C7—H7B	0.9700	N5—H5	0.8600
C8—C14	1.515 (3)	C5A—O5	1.226 (3)
C8—C9	1.554 (3)	C5A—C5B	1.490 (4)
C8—H8	0.9800	C5B—H5B1	0.9600
C9—C11	1.546 (3)	C5B—H5B2	0.9600

C9—C10	1.560 (3)	C5B—H5B3	0.9600
C9—H9	0.9800	O3A—C3A	1.330 (3)
C10—C19	1.542 (3)	O3B—C3A	1.184 (4)
C11—C12	1.531 (3)	C3A—C3B	1.486 (4)
C11—H11A	0.9700	C3B—H3B1	0.9600
C11—H11B	0.9700	C3B—H3B2	0.9600
C12—C13	1.526 (3)	C3B—H3B3	0.9600
C12—H12A	0.9700		
C2—C1—C10	112.70 (19)	C13—C12—H12A	109.7
C2—C1—H1A	109.1	C11—C12—H12A	109.7
C10—C1—H1A	109.1	C13—C12—H12B	109.7
C2—C1—H1B	109.1	C11—C12—H12B	109.7
C10—C1—H1B	109.1	H12A—C12—H12B	108.2
H1A—C1—H1B	107.8	C17—C13—C12	117.2 (2)
C3—C2—C1	112.46 (18)	C17—C13—C14	100.86 (18)
C3—C2—H2A	109.1	C12—C13—C14	108.86 (18)
C1—C2—H2A	109.1	C17—C13—C18	104.90 (19)
C3—C2—H2B	109.1	C12—C13—C18	111.24 (19)
C1—C2—H2B	109.1	C14—C13—C18	113.5 (2)
H2A—C2—H2B	107.8	C8—C14—C15	120.1 (2)
O3A—C3—C2	108.76 (18)	C8—C14—C13	113.26 (17)
O3A—C3—C4	105.8 (2)	C15—C14—C13	103.60 (18)
C2—C3—C4	112.7 (2)	C8—C14—H14	106.3
O3A—C3—H3	109.8	C15—C14—H14	106.3
C2—C3—H3	109.8	C13—C14—H14	106.3
C4—C3—H3	109.8	C16—C15—C14	102.54 (19)
C3—C4—C5	110.2 (2)	C16—C15—H15A	111.3
C3—C4—H4A	109.6	C14—C15—H15A	111.3
C5—C4—H4A	109.6	C16—C15—H15B	111.3
C3—C4—H4B	109.6	C14—C15—H15B	111.3
C5—C4—H4B	109.6	H15A—C15—H15B	109.2
H4A—C4—H4B	108.1	C17—C16—C15	105.8 (2)
N5—C5—C4	109.63 (18)	C17—C16—H16A	110.6
N5—C5—C6	104.71 (17)	C15—C16—H16A	110.6
C4—C5—C6	111.37 (18)	C17—C16—H16B	110.6
N5—C5—C10	107.85 (17)	C15—C16—H16B	110.6
C4—C5—C10	110.37 (17)	H16A—C16—H16B	108.7
C6—C5—C10	112.67 (17)	O17—C17—C13	126.8 (2)
O6—C6—C7	110.97 (18)	O17—C17—C16	124.7 (3)
O6—C6—C5	109.90 (17)	C13—C17—C16	108.5 (2)
C7—C6—C5	111.45 (18)	C10—C19—H19A	109.5
O6—C6—H6	108.1	C10—C19—H19B	109.5
C7—C6—H6	108.1	H19A—C19—H19B	109.5
C5—C6—H6	108.1	C10—C19—H19C	109.5
C6—C7—C8	113.49 (17)	H19A—C19—H19C	109.5
C6—C7—H7A	108.9	H19B—C19—H19C	109.5
C8—C7—H7A	108.9	C13—C18—H18A	109.5
C6—C7—H7B	108.9	C13—C18—H18B	109.5
C8—C7—H7B	108.9	H18A—C18—H18B	109.5

H7A—C7—H7B	107.7	C13—C18—H18C	109.5
C14—C8—C7	111.91 (16)	H18A—C18—H18C	109.5
C14—C8—C9	108.50 (18)	H18B—C18—H18C	109.5
C7—C8—C9	110.71 (18)	C6—O6—H6A	109.5
C14—C8—H8	108.5	C5A—N5—C5	127.9 (2)
C7—C8—H8	108.5	C5A—N5—H5	116.1
C9—C8—H8	108.5	C5—N5—H5	116.1
C11—C9—C8	111.45 (17)	O5—C5A—N5	124.2 (2)
C11—C9—C10	113.83 (17)	O5—C5A—C5B	120.8 (2)
C8—C9—C10	111.59 (17)	N5—C5A—C5B	115.0 (2)
C11—C9—H9	106.5	C5A—C5B—H5B1	109.5
C8—C9—H9	106.5	C5A—C5B—H5B2	109.5
C10—C9—H9	106.5	H5B1—C5B—H5B2	109.5
C19—C10—C1	108.57 (18)	C5A—C5B—H5B3	109.5
C19—C10—C9	108.71 (18)	H5B1—C5B—H5B3	109.5
C1—C10—C9	111.61 (18)	H5B2—C5B—H5B3	109.5
C19—C10—C5	111.37 (19)	C3A—O3A—C3	118.5 (2)
C1—C10—C5	106.89 (17)	O3B—C3A—O3A	123.5 (3)
C9—C10—C5	109.71 (16)	O3B—C3A—C3B	124.8 (3)
C12—C11—C9	113.71 (17)	O3A—C3A—C3B	111.7 (2)
C12—C11—H11A	108.8	C3A—C3B—H3B1	109.5
C9—C11—H11A	108.8	C3A—C3B—H3B2	109.5
C12—C11—H11B	108.8	H3B1—C3B—H3B2	109.5
C9—C11—H11B	108.8	C3A—C3B—H3B3	109.5
H11A—C11—H11B	107.7	H3B1—C3B—H3B3	109.5
C13—C12—C11	109.83 (19)	H3B2—C3B—H3B3	109.5
C10—C1—C2—C3	−53.3 (3)	C4—C5—C10—C9	178.40 (16)
C1—C2—C3—O3A	168.00 (18)	C6—C5—C10—C9	53.2 (2)
C1—C2—C3—C4	51.0 (3)	C8—C9—C11—C12	52.5 (3)
O3A—C3—C4—C5	−173.44 (17)	C10—C9—C11—C12	179.75 (18)
C2—C3—C4—C5	−54.7 (3)	C9—C11—C12—C13	−54.5 (3)
C3—C4—C5—N5	−58.5 (2)	C11—C12—C13—C17	170.42 (19)
C3—C4—C5—C6	−173.89 (19)	C11—C12—C13—C14	56.8 (2)
C3—C4—C5—C10	60.2 (2)	C11—C12—C13—C18	−68.9 (3)
N5—C5—C6—O6	−172.27 (18)	C7—C8—C14—C15	−55.9 (3)
C4—C5—C6—O6	−53.9 (2)	C9—C8—C14—C15	−178.35 (19)
C10—C5—C6—O6	70.8 (2)	C7—C8—C14—C13	−178.94 (17)
N5—C5—C6—C7	64.3 (2)	C9—C8—C14—C13	58.6 (2)
C4—C5—C6—C7	−177.31 (18)	C17—C13—C14—C8	174.28 (17)
C10—C5—C6—C7	−52.7 (2)	C12—C13—C14—C8	−61.8 (2)
O6—C6—C7—C8	−69.0 (2)	C18—C13—C14—C8	62.7 (3)
C5—C6—C7—C8	53.8 (3)	C17—C13—C14—C15	42.5 (2)
C6—C7—C8—C14	−176.75 (17)	C12—C13—C14—C15	166.5 (2)
C6—C7—C8—C9	−55.6 (3)	C18—C13—C14—C15	−69.1 (2)
C14—C8—C9—C11	−52.3 (2)	C8—C14—C15—C16	−167.0 (2)
C7—C8—C9—C11	−175.51 (18)	C13—C14—C15—C16	−39.4 (2)
C14—C8—C9—C10	179.18 (17)	C14—C15—C16—C17	20.7 (3)
C7—C8—C9—C10	56.0 (2)	C12—C13—C17—O17	33.8 (4)
C2—C1—C10—C19	−63.4 (3)	C14—C13—C17—O17	151.8 (3)

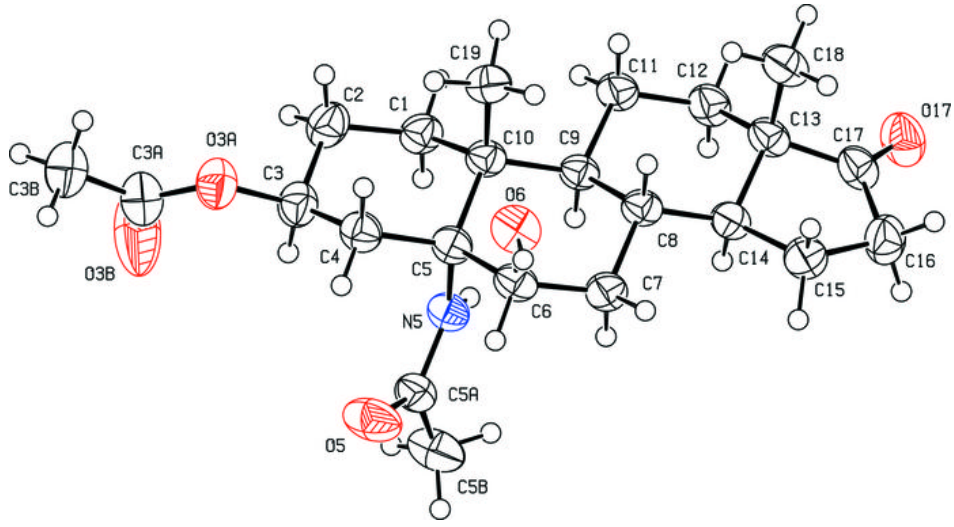
C2—C1—C10—C9	176.77 (18)	C18—C13—C17—O17	−90.1 (3)
C2—C1—C10—C5	56.8 (2)	C12—C13—C17—C16	−147.7 (2)
C11—C9—C10—C19	−60.0 (2)	C14—C13—C17—C16	−29.7 (2)
C8—C9—C10—C19	67.3 (2)	C18—C13—C17—C16	88.4 (2)
C11—C9—C10—C1	59.8 (2)	C15—C16—C17—O17	−175.6 (3)
C8—C9—C10—C1	−173.03 (17)	C15—C16—C17—C13	5.8 (3)
C11—C9—C10—C5	178.04 (16)	C4—C5—N5—C5A	−53.0 (3)
C8—C9—C10—C5	−54.8 (2)	C6—C5—N5—C5A	66.5 (3)
N5—C5—C10—C19	177.75 (18)	C10—C5—N5—C5A	−173.3 (2)
C4—C5—C10—C19	58.0 (2)	C5—N5—C5A—O5	10.5 (4)
C6—C5—C10—C19	−67.2 (2)	C5—N5—C5A—C5B	−168.9 (2)
N5—C5—C10—C1	59.3 (2)	C2—C3—O3A—C3A	91.2 (3)
C4—C5—C10—C1	−60.4 (2)	C4—C3—O3A—C3A	−147.5 (2)
C6—C5—C10—C1	174.38 (18)	C3—O3A—C3A—O3B	3.2 (5)
N5—C5—C10—C9	−61.9 (2)	C3—O3A—C3A—C3B	−176.8 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6A···O17^{i}	0.82	2.03	2.823 (3)	164

Symmetry codes: (i) *x*, *y*−1, *z*.

Fig. 1


C19
C11
C12
C18
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C13
C17
O17
O3A
C3A
C3B
C3
O6
C8
C14
C16
O3B
C4
C5
C7
C15
N5
C6
C5A
O5
C5B