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

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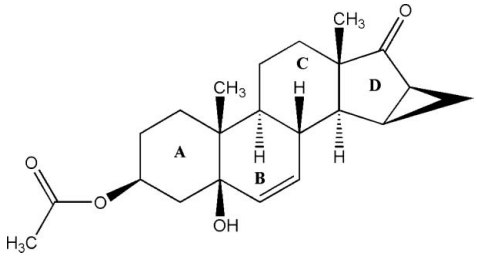
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.059; wR factor = 0.150; data-to-parameter ratio = 10.2.

In the crystal structure of the title compound, $C_{22}H_{30}O_4$, rings A and C have slightly distorted chair conformations, ring B shows an approximate symmetric half-chair conformation and ring D is in an envelope conformation. The molecules are connected by weak intermolecular C—H···O hydrogen bonding.

Related literature

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



Experimental

Crystal data

$C_{22}H_{30}O_4$
M_r = 358.46
Orthorhombic, $P2_12_12_1$
a = 7.160 (4) Å
b = 14.541 (8) Å
c = 18.733 (10) Å
V = 1950.4 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.40 × 0.35 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.957, T_{max} = 0.971
9446 measured reflections
2437 independent reflections
2036 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.150
S = 1.17
2437 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1	0.82	2.27	2.868 (3)	130
C18—H18B···O2^{i}	0.96	2.55	3.498 (4)	168
C9—H9···O4^{ii}	0.98	2.59	3.542 (3)	165

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

Data collection: *SMART* (Bruker, 1997); 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: *ORTEP-3* (Version 1.05; Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2066).

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supplementary materials

Acta Cryst. (2007). E**63**, o4653 [**doi:10.1107/S1600536807056000**]

3*β*-Acetoxy-5-hydroxy-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 antimineralocorticoid and antiandrogenic properties (Muhn *et al.*, 1995). In our attempts to synthesize this drug, the title compound, (I), was obtained as an intermediate by the reductive dechlorination of, 3β-acetoxy-7α-chloro-5,6β-epoxy-15β,16β-methylene-5β-androstan-17-one, (II). 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 (1.524 (4) *versus* 1.484 (3) 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) reported previously (Zhou *et al.*, 2007). The difference in these bond lengths might be caused by a hyperconjugation interacton between the C=O π-bonding and C16—C20 σ-bonding orbitals. However, in the analogous structure of 3 b-Acetoxy-17,17-ethylenedioxy-15*b*,16*b*-methylene-5-androsten-7 b-ol these bond lenghts are almost equal (1.499 (6) and 1.493 (6) Å) (Zhou *et al.*, 2006).

In the steroid skeleton, the ring A and ring C show slightly distorted chair conformations, ring B shows a closely symmetric half-chair conformation and ring D is in an an envelope conformation. In ring B, atom C9 and atom C10 deviate by −0.402 (4)Å and 0.410 (4)Å from the mean plane calculated through the ring C5—C8, respectively. In ring D, C13 deviates by 0.513 (4)Å from the mean plane of C14—C17, which makes a dihedral angle of 64.6 (2)° with the cyclopropyl ring C15/C16/C20. In the structure an intramolecular O—H···O hydrogen bond is found (Table 1). In addition, two weak intermolecular C—H···O hydrogen bonds are also observed (Table 1.).

Experimental

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

Refinement

Because no strong anomalous scattering atoms are present, the absolute structure cannot be determined. Therefore Friedel pairs were merged in the refinement. The absolute stereochemistry of the compound (I) is known from the synthetic route. The C—H H atoms were placed in calculated positions and were refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(C) [or 1.5Ueq(methyl C)] and C—H distances of 0.93–0.98 Å. The hydroxy H atom was positioned with idealized geometry allowed to rotate but not to tip and was refined using a riding model, with O—H = 0.82Å and U_{iso}(H) = 1.5 U_{eq}(O).

Figures

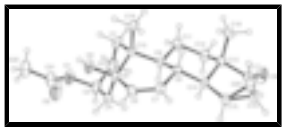

Fig. 1. Crystal structure of (I) with labelling and displacement ellipsoids drawn at the 30% probability level.

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

Crystal data

$C_{22}H_{30}O_4$	F_{000} = 776
M_r = 358.46	D_x = 1.221 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 833 reflections
a = 7.160 (4) Å	θ = 2.8–26.8º
b = 14.541 (8) Å	μ = 0.08 mm^{-1}
c = 18.733 (10) Å	T = 293 (2) K
V = 1950.4 (18) Å^3	Block, colorless
Z = 4	0.40 × 0.35 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2437 independent reflections
Radiation source: fine-focus sealed tube	2036 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.059
T = 293(2) K	θ_{max} = 27.0º
phi and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	h = −9→6
T_{min} = 0.957, T_{max} = 0.971	k = −18→13
9446 measured reflections	l = −23→23

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.059	$w = 1/[\sigma^2(F_o^2) + (0.0893P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.150	$(\Delta/\sigma)_{max} < 0.001$
S = 1.17	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
2437 reflections	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
239 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Extinction coefficient: 0.032 (4)

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	0.3040 (4)	0.30323 (16)	0.11757 (11)	0.0630 (7)
O2	0.2403 (7)	0.1667 (2)	0.07470 (17)	0.1242 (17)
O3	0.3461 (4)	0.48147 (15)	0.18108 (10)	0.0544 (6)
H3	0.3806	0.4496	0.1475	0.082*
O4	0.7636 (5)	0.29845 (19)	0.60324 (13)	0.0794 (8)
C1	0.6368 (5)	0.3314 (2)	0.20925 (15)	0.0481 (7)
H1A	0.6389	0.3573	0.1615	0.058*
H1B	0.7636	0.3138	0.2213	0.058*
C2	0.5145 (5)	0.2454 (2)	0.20878 (16)	0.0548 (8)
H2A	0.5619	0.2023	0.1736	0.066*
H2B	0.5204	0.2159	0.2552	0.066*
C3	0.3140 (5)	0.2686 (2)	0.19154 (16)	0.0531 (8)
H3A	0.2365	0.2134	0.1962	0.064*
C4	0.2399 (4)	0.3433 (2)	0.23997 (15)	0.0473 (7)
H4A	0.2227	0.3175	0.2873	0.057*
H4B	0.1180	0.3618	0.2226	0.057*
C5	0.3625 (4)	0.42965 (19)	0.24669 (13)	0.0385 (6)
C6	0.2822 (4)	0.4891 (2)	0.30472 (15)	0.0438 (6)
H6	0.1752	0.5226	0.2940	0.053*
C7	0.3512 (4)	0.49757 (18)	0.36930 (14)	0.0396 (6)
H7	0.2857	0.5326	0.4025	0.048*
C8	0.5309 (3)	0.45324 (17)	0.39208 (12)	0.0316 (5)
H8	0.6304	0.4995	0.3902	0.038*
C9	0.5828 (4)	0.37321 (18)	0.34103 (13)	0.0334 (5)
H9	0.4852	0.3266	0.3469	0.040*
C10	0.5714 (4)	0.40595 (18)	0.26191 (13)	0.0358 (6)
C11	0.7666 (4)	0.3270 (2)	0.36250 (15)	0.0477 (7)
H11A	0.8686	0.3696	0.3544	0.057*
H11B	0.7869	0.2738	0.3322	0.057*
C12	0.7699 (5)	0.2960 (2)	0.44137 (15)	0.0495 (8)

H12A	0.6788	0.2475	0.4487	0.059*
H12B	0.8923	0.2719	0.4533	0.059*
C13	0.7241 (4)	0.37802 (19)	0.48968 (13)	0.0393 (6)
C14	0.5262 (4)	0.41171 (17)	0.46729 (13)	0.0334 (6)
H14	0.4524	0.3553	0.4623	0.040*
C15	0.4424 (4)	0.4606 (2)	0.53111 (13)	0.0420 (6)
H15	0.3062	0.4661	0.5337	0.050*
C16	0.5460 (4)	0.4216 (2)	0.59609 (14)	0.0479 (7)
H16	0.4739	0.4039	0.6384	0.057*
C17	0.6908 (5)	0.3584 (2)	0.56908 (15)	0.0482 (7)
C18	0.8868 (4)	0.4478 (3)	0.48569 (17)	0.0540 (8)
H18A	0.9279	0.4626	0.5331	0.081*
H18B	0.8449	0.5028	0.4622	0.081*
H18C	0.9884	0.4214	0.4593	0.081*
C19	0.6941 (4)	0.4917 (2)	0.25053 (15)	0.0486 (7)
H19A	0.6896	0.5095	0.2012	0.073*
H19B	0.8206	0.4778	0.2636	0.073*
H19C	0.6485	0.5411	0.2797	0.073*
C20	0.5571 (5)	0.5230 (2)	0.57580 (15)	0.0520 (8)
H20A	0.4920	0.5667	0.6059	0.062*
H20B	0.6737	0.5453	0.5559	0.062*
C21	0.2665 (6)	0.2466 (2)	0.06563 (18)	0.0650 (10)
C22	0.2613 (7)	0.2936 (3)	−0.00473 (18)	0.0801 (13)
H22A	0.2474	0.2487	−0.0419	0.120*
H22B	0.3755	0.3270	−0.0117	0.120*
H22C	0.1578	0.3355	−0.0060	0.120*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0952 (18)	0.0565 (12)	0.0374 (10)	−0.0129 (14)	−0.0124 (12)	−0.0032 (9)
O2	0.236 (5)	0.0564 (17)	0.0799 (19)	−0.005 (2)	−0.055 (3)	−0.0110 (15)
O3	0.0732 (15)	0.0544 (12)	0.0357 (10)	0.0020 (11)	−0.0109 (10)	0.0095 (9)
O4	0.104 (2)	0.0818 (17)	0.0527 (14)	0.0325 (16)	−0.0088 (14)	0.0228 (13)
C1	0.0591 (18)	0.0529 (17)	0.0323 (13)	0.0073 (15)	0.0072 (13)	−0.0005 (12)
C2	0.085 (2)	0.0410 (16)	0.0381 (14)	0.0076 (17)	0.0014 (15)	−0.0057 (12)
C3	0.075 (2)	0.0442 (15)	0.0402 (15)	−0.0139 (16)	−0.0062 (15)	−0.0001 (12)
C4	0.0478 (17)	0.0592 (18)	0.0350 (13)	−0.0089 (14)	−0.0037 (12)	0.0012 (12)
C5	0.0430 (15)	0.0430 (14)	0.0295 (12)	0.0001 (12)	−0.0034 (11)	0.0060 (10)
C6	0.0413 (14)	0.0465 (15)	0.0437 (14)	0.0107 (13)	−0.0089 (12)	0.0013 (12)
C7	0.0418 (14)	0.0388 (14)	0.0382 (13)	0.0113 (12)	0.0003 (12)	−0.0013 (11)
C8	0.0330 (12)	0.0321 (12)	0.0297 (11)	−0.0001 (10)	−0.0005 (10)	0.0000 (10)
C9	0.0365 (13)	0.0337 (12)	0.0299 (11)	0.0000 (11)	0.0019 (10)	0.0019 (10)
C10	0.0414 (14)	0.0370 (13)	0.0291 (12)	−0.0007 (11)	0.0036 (11)	0.0025 (10)
C11	0.0537 (17)	0.0495 (16)	0.0399 (14)	0.0154 (14)	0.0015 (14)	−0.0004 (12)
C12	0.0561 (19)	0.0487 (16)	0.0437 (15)	0.0211 (15)	−0.0045 (14)	0.0039 (13)
C13	0.0400 (13)	0.0415 (14)	0.0363 (13)	0.0065 (12)	−0.0066 (11)	0.0032 (11)
C14	0.0329 (12)	0.0369 (13)	0.0302 (12)	−0.0014 (10)	−0.0020 (10)	−0.0004 (10)

C15	0.0395 (14)	0.0560 (16)	0.0305 (12)	0.0006 (13)	−0.0016 (12)	−0.0008 (12)
C16	0.0522 (17)	0.0620 (18)	0.0294 (13)	−0.0018 (15)	−0.0038 (12)	0.0013 (12)
C17	0.0529 (16)	0.0534 (17)	0.0383 (14)	−0.0017 (15)	−0.0109 (13)	0.0060 (13)
C18	0.0366 (15)	0.072 (2)	0.0537 (17)	−0.0035 (14)	−0.0084 (13)	0.0048 (16)
C19	0.0515 (17)	0.0524 (16)	0.0418 (14)	−0.0116 (14)	0.0065 (13)	0.0079 (13)
C20	0.0631 (18)	0.0525 (17)	0.0403 (15)	0.0057 (16)	−0.0057 (14)	−0.0125 (13)
C21	0.090 (3)	0.0481 (18)	0.0573 (19)	0.0094 (19)	−0.0220 (19)	−0.0121 (15)
C22	0.114 (3)	0.083 (3)	0.0432 (17)	0.017 (3)	−0.018 (2)	−0.0103 (18)

Geometric parameters (Å, °)

O1—C21	1.303 (4)	C10—C19	1.540 (4)
O1—C3	1.476 (4)	C11—C12	1.545 (4)
O2—C21	1.188 (4)	C11—H11A	0.9700
O3—C5	1.446 (3)	C11—H11B	0.9700
O3—H3	0.8200	C12—C13	1.533 (4)
O4—C17	1.200 (4)	C12—H12A	0.9700
C1—C2	1.527 (5)	C12—H12B	0.9700
C1—C10	1.539 (4)	C13—C17	1.533 (4)
C1—H1A	0.9700	C13—C18	1.546 (4)
C1—H1B	0.9700	C13—C14	1.557 (4)
C2—C3	1.510 (5)	C14—C15	1.515 (4)
C2—H2A	0.9700	C14—H14	0.9800
C2—H2B	0.9700	C15—C20	1.483 (4)
C3—C4	1.511 (4)	C15—C16	1.534 (4)
C3—H3A	0.9800	C15—H15	0.9800
C4—C5	1.537 (4)	C16—C17	1.475 (5)
C4—H4A	0.9700	C16—C20	1.524 (4)
C4—H4B	0.9700	C16—H16	0.9800
C5—C6	1.503 (4)	C18—H18A	0.9599
C5—C10	1.561 (4)	C18—H18B	0.9599
C6—C7	1.313 (4)	C18—H18C	0.9599
C6—H6	0.9300	C19—H19A	0.9599
C7—C8	1.501 (3)	C19—H19B	0.9599
C7—H7	0.9300	C19—H19C	0.9599
C8—C14	1.533 (3)	C20—H20A	0.9700
C8—C9	1.551 (3)	C20—H20B	0.9700
C8—H8	0.9800	C21—C22	1.485 (5)
C9—C11	1.532 (4)	C22—H22A	0.9599
C9—C10	1.559 (3)	C22—H22B	0.9599
C9—H9	0.9800	C22—H22C	0.9599
C21—O1—C3	119.7 (3)	H11A—C11—H11B	107.8
C5—O3—H3	109.5	C13—C12—C11	109.5 (2)
C2—C1—C10	114.0 (2)	C13—C12—H12A	109.8
C2—C1—H1A	108.8	C11—C12—H12A	109.8
C10—C1—H1A	108.8	C13—C12—H12B	109.8
C2—C1—H1B	108.8	C11—C12—H12B	109.8
C10—C1—H1B	108.8	H12A—C12—H12B	108.2
H1A—C1—H1B	107.7	C12—C13—C17	117.4 (2)

C3—C2—C1	111.3 (3)	C12—C13—C18	108.7 (3)
C3—C2—H2A	109.4	C17—C13—C18	106.6 (2)
C1—C2—H2A	109.4	C12—C13—C14	106.3 (2)
C3—C2—H2B	109.4	C17—C13—C14	100.3 (2)
C1—C2—H2B	109.4	C18—C13—C14	117.8 (2)
H2A—C2—H2B	108.0	C15—C14—C8	123.3 (2)
O1—C3—C2	108.9 (3)	C15—C14—C13	107.2 (2)
O1—C3—C4	107.5 (3)	C8—C14—C13	110.6 (2)
C2—C3—C4	111.5 (3)	C15—C14—H14	104.7
O1—C3—H3A	109.6	C8—C14—H14	104.7
C2—C3—H3A	109.6	C13—C14—H14	104.7
C4—C3—H3A	109.6	C20—C15—C14	120.9 (3)
C3—C4—C5	115.8 (3)	C20—C15—C16	60.7 (2)
C3—C4—H4A	108.3	C14—C15—C16	105.2 (2)
C5—C4—H4A	108.3	C20—C15—H15	118.2
C3—C4—H4B	108.3	C14—C15—H15	118.2
C5—C4—H4B	108.3	C16—C15—H15	118.2
H4A—C4—H4B	107.4	C17—C16—C20	118.7 (3)
O3—C5—C6	106.5 (2)	C17—C16—C15	107.3 (2)
O3—C5—C4	108.0 (2)	C20—C16—C15	58.02 (19)
C6—C5—C4	108.1 (2)	C17—C16—H16	118.9
O3—C5—C10	110.3 (2)	C20—C16—H16	118.9
C6—C5—C10	111.2 (2)	C15—C16—H16	118.9
C4—C5—C10	112.4 (2)	O4—C17—C16	125.1 (3)
C7—C6—C5	125.2 (3)	O4—C17—C13	125.8 (3)
C7—C6—H6	117.4	C16—C17—C13	109.0 (2)
C5—C6—H6	117.4	C13—C18—H18A	109.5
C6—C7—C8	123.0 (2)	C13—C18—H18B	109.5
C6—C7—H7	118.5	H18A—C18—H18B	109.5
C8—C7—H7	118.5	C13—C18—H18C	109.5
C7—C8—C14	114.3 (2)	H18A—C18—H18C	109.5
C7—C8—C9	110.6 (2)	H18B—C18—H18C	109.5
C14—C8—C9	106.03 (19)	C10—C19—H19A	109.5
C7—C8—H8	108.6	C10—C19—H19B	109.5
C14—C8—H8	108.6	H19A—C19—H19B	109.5
C9—C8—H8	108.6	C10—C19—H19C	109.5
C11—C9—C8	111.9 (2)	H19A—C19—H19C	109.5
C11—C9—C10	115.4 (2)	H19B—C19—H19C	109.5
C8—C9—C10	110.1 (2)	C15—C20—C16	61.3 (2)
C11—C9—H9	106.2	C15—C20—H20A	117.6
C8—C9—H9	106.2	C16—C20—H20A	117.6
C10—C9—H9	106.2	C15—C20—H20B	117.6
C1—C10—C19	107.9 (2)	C16—C20—H20B	117.6
C1—C10—C9	112.2 (2)	H20A—C20—H20B	114.7
C19—C10—C9	110.4 (2)	O2—C21—O1	122.9 (3)
C1—C10—C5	109.3 (2)	O2—C21—C22	124.9 (3)
C19—C10—C5	110.0 (2)	O1—C21—C22	112.1 (3)
C9—C10—C5	106.9 (2)	C21—C22—H22A	109.5
C9—C11—C12	113.1 (2)	C21—C22—H22B	109.5

C9—C11—H11A	109.0	H22A—C22—H22B	109.5
C12—C11—H11A	109.0	C21—C22—H22C	109.5
C9—C11—H11B	109.0	H22A—C22—H22C	109.5
C12—C11—H11B	109.0	H22B—C22—H22C	109.5
C10—C1—C2—C3	57.5 (3)	C8—C9—C11—C12	53.0 (3)
C21—O1—C3—C2	93.8 (4)	C10—C9—C11—C12	−180.0 (2)
C21—O1—C3—C4	−145.3 (3)	C9—C11—C12—C13	−54.7 (3)
C1—C2—C3—O1	65.6 (3)	C11—C12—C13—C17	170.8 (3)
C1—C2—C3—C4	−52.8 (3)	C11—C12—C13—C18	−68.0 (3)
O1—C3—C4—C5	−69.1 (3)	C11—C12—C13—C14	59.7 (3)
C2—C3—C4—C5	50.1 (3)	C7—C8—C14—C15	−44.5 (3)
C3—C4—C5—O3	73.5 (3)	C9—C8—C14—C15	−166.7 (2)
C3—C4—C5—C6	−171.6 (2)	C7—C8—C14—C13	−173.2 (2)
C3—C4—C5—C10	−48.5 (3)	C9—C8—C14—C13	64.7 (2)
O3—C5—C6—C7	−140.1 (3)	C12—C13—C14—C15	155.2 (2)
C4—C5—C6—C7	104.0 (3)	C17—C13—C14—C15	32.5 (3)
C10—C5—C6—C7	−19.9 (4)	C18—C13—C14—C15	−82.6 (3)
C5—C6—C7—C8	4.7 (5)	C12—C13—C14—C8	−67.8 (3)
C6—C7—C8—C14	−139.1 (3)	C17—C13—C14—C8	169.4 (2)
C6—C7—C8—C9	−19.5 (4)	C18—C13—C14—C8	54.3 (3)
C7—C8—C9—C11	179.5 (2)	C8—C14—C15—C20	−88.7 (3)
C14—C8—C9—C11	−56.0 (3)	C13—C14—C15—C20	41.4 (3)
C7—C8—C9—C10	49.7 (3)	C8—C14—C15—C16	−153.3 (2)
C14—C8—C9—C10	174.2 (2)	C13—C14—C15—C16	−23.2 (3)
C2—C1—C10—C19	−173.9 (2)	C20—C15—C16—C17	−113.5 (3)
C2—C1—C10—C9	64.2 (3)	C14—C15—C16—C17	3.7 (3)
C2—C1—C10—C5	−54.3 (3)	C14—C15—C16—C20	117.2 (3)
C11—C9—C10—C1	47.5 (3)	C20—C16—C17—O4	138.3 (3)
C8—C9—C10—C1	175.4 (2)	C15—C16—C17—O4	−159.2 (3)
C11—C9—C10—C19	−73.0 (3)	C20—C16—C17—C13	−44.9 (3)
C8—C9—C10—C19	55.0 (3)	C15—C16—C17—C13	17.6 (3)
C11—C9—C10—C5	167.3 (2)	C12—C13—C17—O4	31.7 (5)
C8—C9—C10—C5	−64.8 (3)	C18—C13—C17—O4	−90.5 (4)
O3—C5—C10—C1	−72.4 (3)	C14—C13—C17—O4	146.2 (3)
C6—C5—C10—C1	169.7 (2)	C12—C13—C17—C16	−145.0 (3)
C4—C5—C10—C1	48.3 (3)	C18—C13—C17—C16	92.7 (3)
O3—C5—C10—C19	46.0 (3)	C14—C13—C17—C16	−30.5 (3)
C6—C5—C10—C19	−72.0 (3)	C14—C15—C20—C16	−90.8 (3)
C4—C5—C10—C19	166.7 (2)	C17—C16—C20—C15	93.3 (3)
O3—C5—C10—C9	165.9 (2)	C3—O1—C21—O2	−0.4 (7)
C6—C5—C10—C9	48.0 (3)	C3—O1—C21—C22	179.7 (3)
C4—C5—C10—C9	−73.4 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1	0.82	2.27	2.868 (3)	130
C18—H18B···O2^{i}	0.96	2.55	3.498 (4)	168

C9—H9···O4^{ii}	0.98	2.59	3.542 (3)	165

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

Fig. 1