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3β-Acetoxy-17-ethylenedioxyandrost-5-ene

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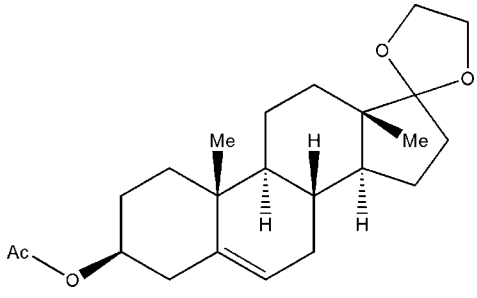
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.010 Å; R factor = 0.078; wR factor = 0.199; data-to-parameter ratio = 9.6.

In the molecule of the title compound, $C_{23}H_{34}O_4$, the six-membered rings A, B and C have chair, flattened boat and chair conformations, respectively, while the two five-membered rings D and E have envelope conformations. Intramolecular C—H···O hydrogen bonds result in the formation of three more five-membered rings.

Related literature

For related literature, see: Mohr & Nickisch (2005); Kelly & Sykes (1968). For general background, see: Cremer & Pople (1975). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{23}H_{34}O_4$
M_r = 374.50
Monoclinic, $C2$
a = 10.197 (2) Å
b = 7.5370 (15) Å
c = 27.143 (5) Å
β = 99.04 (3)°
V = 2060.2 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.40 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.976, T_{max} = 0.990
2324 measured reflections
2189 independent reflections
1420 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.199
S = 1.04
2189 reflections
227 parameters
62 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected torsion angles (°).

C19—C16—C17—C21	44.9 (6)	C16—C17—C21—C20	−35.8 (7)
C25—O3—C19—O4	14.6 (9)	C19—C20—C21—C17	12.0 (8)
C24—O4—C19—O3	−16.4 (8)	C19—O4—C24—C25	11.9 (9)
C17—C16—C19—C20	−35.9 (6)	C19—O3—C25—C24	−7.1 (11)
C16—C19—C20—C21	15.0 (8)	O4—C24—C25—O3	−3.0 (11)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O2	0.98	2.22	2.650 (10)	105
C15—H15A···O4	0.97	2.52	2.886 (8)	102
C23—H23B···O3	0.96	2.41	2.744 (9)	100

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); 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: HK2310).

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supplementary materials

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3β-Acetoxy-17-ethylenedioxyandrost-5-ene

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Comment

The title compound, (I), is an intermediate product of drospirenone, which is a new oral contraceptive (Mohr & Nickisch, 2005). We report herein its crystal structure.

In the molecule of (I) (Fig. 1), the ligand bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

Rings A (C4—C9), B (C7/C8/C10—C13) and C (C10/C11/C14—C17) are not planar, having total puckering amplitudes, Q_T, of 0.547 (3), 0.477 (2) and 0.676 (3) Å, respectively, and chair, flattened boat and chair conformations, respectively [φ = −173.98 (3)°, θ = 169.83 (3)°; φ = −16.01 (2)°, θ = 115.71 (3)° and φ = −147.03 (3)°, θ = 86.92 (3)°, respectively] (Cremer & Pople, 1975). Rings D (C16/C17/C19—C21) and E (O3/O4/C19/C24/C25) have envelope conformations with atoms C16 and C19 displaced by 0.646 (3) Å and 0.220 (2) Å from the planes of the other four ring atoms, respectively. Rings D and E have pseudo twofold axis and pseudo mirror plane, respectively, running through atom C20 and midpoint of C16—C17 bond (for ring D) and atom C19 and midpoint of C24—C25 bond (for ring E), as can be deduced from the torsion angles (Table 1).

The intramolecular C—H···O hydrogen bonds (Table 2, Fig. 1) result in the formation of three more five-membered rings F (O1/O2/C3/C4/H4A), G (C15/C16/C19/O4/H15A) and H (O3/C16/C19/C23/H23B), in which they may be effective in the stabilization of the structure.

As can be seen from the packing diagram (Fig. 2), the molecules of (I) are elongated along the *c* axis, and stacked along the *a* axis. Dipole-dipole and van der Waals interactions may be effective in the molecular packing.

Experimental

The title compound, (I), was prepared by a literature method (Kelly & Sykes, 1968). The crystals were obtained by dissolving (I) (1.0 g, 2.5 mmol) in methanol (50 ml), and evaporating the solvent slowly at room temperature for about 15 d.

Refinement

H atoms were positioned geometrically, with C—H = 0.93, 0.98, 0.96 and 0.97 Å for aromatic, methine, methyl and methylene H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.5 methyl H, and x = 1.2 for all other H atoms.

Figures

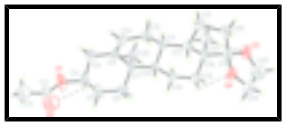

Fig. 1. A drawing of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Intramolecular hydrogen bonds are shown as dashed lines.

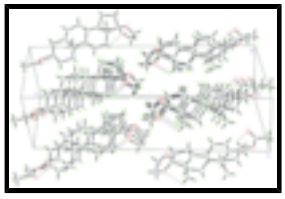

Fig. 2. A packing diagram for (I). Intramolecular hydrogen bonds are shown as dashed lines.

3β-Acetoxy-17-ethylenedioxyandrost-5-ene

Crystal data

$C_{23}H_{34}O_4$	F_{000} = 816
M_r = 374.50	D_x = 1.207 Mg m^{-3}
Monoclinic, *C*2	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C 2y	Cell parameters from 25 reflections
a = 10.197 (2) Å	θ = 9–12º
b = 7.5370 (15) Å	μ = 0.08 mm^{-1}
c = 27.143 (5) Å	*T* = 298 (2) K
β = 99.04 (3)º	Block, colorless
V = 2060.2 (7) Å^3	0.40 × 0.30 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.065
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 1.5º
T = 298(2) K	*h* = −12→12
ω/2θ scans	*k* = 0→9
Absorption correction: ψ scan (North *et al.*, 1968)	*l* = 0→33
T_{min} = 0.976, T_{max} = 0.990	3 standard reflections
2324 measured reflections	every 120 min
2189 independent reflections	intensity decay: none
1420 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.078$

$wR(F^2) = 0.199$

$S = 1.04$

2189 reflections

227 parameters

62 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 5P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.012$

$\Delta\rho_{max} = 0.48$ e Å^{-3}

$\Delta\rho_{min} = -0.33$ e Å^{-3}

Extinction correction: none

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	−0.1505 (5)	−0.0998 (10)	0.05652 (17)	0.095 (2)
O2	−0.3488 (7)	−0.0028 (10)	0.0652 (2)	0.109
O3	0.3844 (5)	0.0504 (10)	0.44585 (17)	0.0851 (18)
O4	0.1664 (4)	0.1086 (8)	0.42607 (16)	0.0675 (14)
C2	−0.3092 (10)	−0.0908 (17)	−0.0154 (3)	0.108
H2A	−0.2345	−0.0601	−0.0312	0.162*
H2B	−0.3834	−0.0169	−0.0282	0.162*
H2C	−0.3321	−0.2129	−0.0220	0.162*
C3	−0.2745 (8)	−0.0634 (13)	0.0394 (3)	0.078 (2)
C4	−0.1026 (7)	−0.0719 (15)	0.1084 (2)	0.078 (3)
H4A	−0.1720	−0.0156	0.1242	0.094*
C5	−0.0628 (8)	−0.2437 (12)	0.1339 (2)	0.069 (2)
H5A	0.0052	−0.2996	0.1179	0.083*
H5B	−0.1389	−0.3225	0.1304	0.083*
C6	−0.0101 (7)	−0.2169 (11)	0.1891 (2)	0.0611 (19)
H6A	0.0189	−0.3306	0.2036	0.073*
H6B	−0.0822	−0.1754	0.2055	0.073*
C7	0.1070 (6)	−0.0832 (10)	0.1997 (2)	0.0470 (16)
C8	0.0725 (6)	0.0808 (9)	0.1667 (2)	0.0465 (15)
C9	0.0181 (7)	0.0490 (11)	0.1131 (2)	0.0637 (19)
H9A	0.0859	−0.0052	0.0966	0.076*

H9B	−0.0067	0.1613	0.0968	0.076*
C10	0.1242 (5)	−0.0273 (8)	0.2554 (2)	0.0393 (13)
H10A	0.0351	−0.0008	0.2627	0.047*
C11	0.2064 (6)	0.1409 (9)	0.2669 (2)	0.0449 (15)
H11A	0.2946	0.1220	0.2579	0.054*
C12	0.1398 (6)	0.2947 (10)	0.2366 (2)	0.0528 (16)
H12A	0.0666	0.3378	0.2521	0.063*
H12B	0.2031	0.3907	0.2366	0.063*
C13	0.0893 (6)	0.2431 (10)	0.1843 (2)	0.0538 (17)
H13A	0.0671	0.3350	0.1616	0.065*
C14	0.1793 (7)	−0.1834 (10)	0.2906 (2)	0.0566 (18)
H14A	0.1186	−0.2830	0.2848	0.068*
H14B	0.2640	−0.2211	0.2822	0.068*
C15	0.1978 (7)	−0.1332 (10)	0.3463 (2)	0.0550 (17)
H15A	0.1117	−0.1110	0.3560	0.066*
H15B	0.2387	−0.2314	0.3661	0.066*
C16	0.2849 (6)	0.0321 (9)	0.3568 (2)	0.0473 (15)
C17	0.2215 (5)	0.1801 (8)	0.3228 (2)	0.0434 (14)
H17A	0.1308	0.1918	0.3301	0.052*
C19	0.2886 (6)	0.1226 (11)	0.4089 (3)	0.0588 (17)
C20	0.3178 (8)	0.3188 (13)	0.3994 (3)	0.076 (2)
H20A	0.4092	0.3464	0.4131	0.092*
H20B	0.2602	0.3943	0.4154	0.092*
C21	0.2937 (7)	0.3518 (11)	0.3427 (3)	0.0630 (19)
H21A	0.2385	0.4555	0.3342	0.076*
H21B	0.3767	0.3664	0.3299	0.076*
C22	0.2318 (6)	−0.1720 (12)	0.1853 (2)	0.066 (2)
H22A	0.2554	−0.2731	0.2063	0.099*
H22B	0.3039	−0.0886	0.1894	0.099*
H22C	0.2137	−0.2094	0.1511	0.099*
C23	0.4290 (6)	−0.0059 (12)	0.3500 (3)	0.068 (2)
H23A	0.4298	−0.0641	0.3186	0.102*
H23B	0.4702	−0.0810	0.3766	0.102*
H23C	0.4771	0.1038	0.3506	0.102*
C24	0.1802 (8)	−0.0145 (15)	0.4659 (3)	0.088 (3)
H24A	0.1344	−0.1244	0.4556	0.105*
H24B	0.1445	0.0336	0.4942	0.105*
C25	0.3240 (9)	−0.0442 (17)	0.4786 (4)	0.103
H25B	0.3553	−0.0051	0.5125	0.123*
H25A	0.3440	−0.1694	0.4763	0.123*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.080 (4)	0.156 (6)	0.043 (3)	0.023 (4)	−0.014 (2)	−0.027 (4)
O2	0.109	0.109	0.109	0.000	0.017	0.000
O3	0.056 (2)	0.141 (5)	0.052 (3)	0.003 (3)	−0.012 (2)	0.003 (3)
O4	0.057 (2)	0.104 (4)	0.044 (2)	0.006 (3)	0.0146 (19)	−0.004 (3)

C2	0.108	0.108	0.108	0.000	0.017	0.000
C3	0.069 (4)	0.100 (6)	0.056 (4)	0.010 (4)	−0.020 (3)	0.000 (4)
C4	0.063 (4)	0.132 (8)	0.037 (4)	0.015 (5)	0.000 (3)	−0.015 (5)
C5	0.075 (5)	0.087 (6)	0.047 (4)	−0.024 (5)	0.012 (3)	−0.026 (4)
C6	0.064 (4)	0.067 (5)	0.052 (4)	−0.010 (4)	0.009 (3)	−0.018 (4)
C7	0.043 (3)	0.069 (4)	0.030 (3)	0.003 (3)	0.009 (2)	0.002 (3)
C8	0.047 (3)	0.058 (4)	0.035 (3)	0.000 (3)	0.008 (2)	−0.003 (3)
C9	0.074 (5)	0.072 (5)	0.045 (4)	−0.003 (4)	0.007 (3)	−0.004 (4)
C10	0.038 (3)	0.047 (3)	0.035 (3)	0.002 (3)	0.009 (2)	−0.004 (3)
C11	0.037 (3)	0.066 (4)	0.033 (3)	0.001 (3)	0.010 (2)	−0.004 (3)
C12	0.054 (4)	0.060 (4)	0.045 (4)	0.001 (3)	0.011 (3)	−0.001 (3)
C13	0.054 (4)	0.066 (5)	0.041 (4)	0.006 (4)	0.005 (3)	0.011 (3)
C14	0.064 (4)	0.058 (4)	0.046 (4)	−0.008 (4)	0.004 (3)	0.006 (3)
C15	0.062 (4)	0.052 (4)	0.051 (4)	−0.004 (3)	0.007 (3)	0.010 (3)
C16	0.046 (3)	0.057 (4)	0.038 (3)	0.001 (3)	0.005 (3)	−0.003 (3)
C17	0.033 (3)	0.050 (4)	0.047 (3)	0.003 (3)	0.008 (2)	0.000 (3)
C19	0.045 (3)	0.076 (5)	0.056 (4)	0.001 (3)	0.007 (3)	−0.003 (3)
C20	0.075 (5)	0.094 (7)	0.058 (4)	−0.014 (5)	0.002 (4)	−0.027 (5)
C21	0.060 (4)	0.065 (5)	0.062 (4)	−0.009 (4)	0.005 (3)	−0.007 (4)
C22	0.065 (4)	0.090 (6)	0.045 (4)	0.022 (4)	0.013 (3)	−0.001 (4)
C23	0.044 (3)	0.095 (6)	0.065 (4)	0.005 (4)	0.011 (3)	0.001 (4)
C24	0.080 (4)	0.117 (6)	0.067 (4)	−0.015 (5)	0.014 (4)	0.008 (4)
C25	0.103	0.103	0.103	0.000	0.016	0.000

Geometric parameters (Å, °)

O1—C3	1.307 (8)	C12—C13	1.483 (9)
O1—C4	1.432 (7)	C12—H12A	0.9700
O4—C19	1.402 (8)	C12—H12B	0.9700
O4—C24	1.414 (10)	C13—H13A	0.9300
O3—C25	1.361 (11)	C14—C15	1.540 (8)
O3—C19	1.396 (8)	C14—H14A	0.9700
O2—C3	1.201 (10)	C14—H14B	0.9700
C2—C3	1.487 (11)	C15—C16	1.530 (9)
C2—H2A	0.9600	C15—H15A	0.9700
C2—H2B	0.9600	C15—H15B	0.9700
C2—H2C	0.9600	C16—C17	1.526 (9)
C4—C5	1.494 (13)	C16—C23	1.537 (8)
C4—C9	1.521 (11)	C16—C19	1.564 (9)
C4—H4A	0.9800	C17—C21	1.544 (10)
C5—C6	1.523 (9)	C17—H17A	0.9800
C5—H5A	0.9700	C19—C20	1.538 (12)
C5—H5B	0.9700	C20—C21	1.539 (10)
C6—C7	1.555 (9)	C20—H20A	0.9700
C6—H6A	0.9700	C20—H20B	0.9700
C6—H6B	0.9700	C21—H21A	0.9700
C7—C8	1.535 (9)	C21—H21B	0.9700
C7—C22	1.542 (8)	C22—H22A	0.9600
C7—C10	1.552 (7)	C22—H22B	0.9600

C8—C13	1.314 (10)	C22—H22C	0.9600
C8—C9	1.494 (8)	C23—H23A	0.9600
C9—H9A	0.9700	C23—H23B	0.9600
C9—H9B	0.9700	C23—H23C	0.9600
C10—C11	1.525 (8)	C24—C25	1.470 (11)
C10—C14	1.563 (9)	C24—H24A	0.9700
C10—H10A	0.9800	C24—H24B	0.9700
C11—C12	1.519 (9)	C25—H25B	0.9700
C11—C17	1.528 (8)	C25—H25A	0.9700
C11—H11A	0.9800		
C3—O1—C4	118.7 (6)	C15—C14—C10	113.0 (6)
C19—O4—C24	108.5 (6)	C15—C14—H14A	109.0
C25—O3—C19	109.7 (6)	C10—C14—H14A	109.0
C3—C2—H2A	109.5	C15—C14—H14B	109.0
C3—C2—H2B	109.5	C10—C14—H14B	109.0
H2A—C2—H2B	109.5	H14A—C14—H14B	107.8
C3—C2—H2C	109.5	C16—C15—C14	111.2 (5)
H2A—C2—H2C	109.5	C16—C15—H15A	109.4
H2B—C2—H2C	109.5	C14—C15—H15A	109.4
O2—C3—O1	122.4 (7)	C16—C15—H15B	109.4
O2—C3—C2	124.4 (8)	C14—C15—H15B	109.4
O1—C3—C2	113.1 (8)	H15A—C15—H15B	108.0
O1—C4—C5	110.7 (8)	C15—C16—C17	107.7 (5)
O1—C4—C9	108.5 (7)	C15—C16—C23	111.3 (6)
C5—C4—C9	108.9 (6)	C17—C16—C23	112.7 (5)
O1—C4—H4A	109.6	C15—C16—C19	117.1 (5)
C5—C4—H4A	109.6	C17—C16—C19	100.2 (5)
C9—C4—H4A	109.6	C23—C16—C19	107.6 (5)
C4—C5—C6	111.7 (7)	C16—C17—C11	115.3 (5)
C4—C5—H5A	109.3	C16—C17—C21	105.5 (5)
C6—C5—H5A	109.3	C11—C17—C21	118.6 (5)
C4—C5—H5B	109.3	C16—C17—H17A	105.5
C6—C5—H5B	109.3	C11—C17—H17A	105.5
H5A—C5—H5B	108.0	C21—C17—H17A	105.5
C5—C6—C7	114.3 (6)	O3—C19—O4	106.8 (6)
C5—C6—H6A	108.7	O3—C19—C20	111.3 (7)
C7—C6—H6A	108.7	O4—C19—C20	109.5 (6)
C5—C6—H6B	108.7	O3—C19—C16	113.2 (6)
C7—C6—H6B	108.7	O4—C19—C16	111.8 (5)
H6A—C6—H6B	107.6	C20—C19—C16	104.2 (6)
C8—C7—C22	108.9 (5)	C19—C20—C21	108.6 (6)
C8—C7—C10	109.3 (5)	C19—C20—H20A	110.0
C22—C7—C10	113.2 (5)	C21—C20—H20A	110.0
C8—C7—C6	108.4 (5)	C19—C20—H20B	110.0
C22—C7—C6	108.2 (6)	C21—C20—H20B	110.0
C10—C7—C6	108.9 (5)	H20A—C20—H20B	108.4
C13—C8—C9	120.7 (6)	C17—C21—C20	102.0 (6)
C13—C8—C7	122.2 (5)	C17—C21—H21A	111.4
C9—C8—C7	117.1 (6)	C20—C21—H21A	111.4

C8—C9—C4	110.4 (5)	C17—C21—H21B	111.4
C8—C9—H9A	109.6	C20—C21—H21B	111.4
C4—C9—H9A	109.6	H21A—C21—H21B	109.2
C8—C9—H9B	109.6	C7—C22—H22A	109.5
C4—C9—H9B	109.6	C7—C22—H22B	109.5
H9A—C9—H9B	108.1	H22A—C22—H22B	109.5
C11—C10—C7	113.6 (5)	C7—C22—H22C	109.5
C11—C10—C14	111.5 (4)	H22A—C22—H22C	109.5
C7—C10—C14	111.4 (5)	H22B—C22—H22C	109.5
C11—C10—H10A	106.6	C16—C23—H23A	109.5
C7—C10—H10A	106.6	C16—C23—H23B	109.5
C14—C10—H10A	106.6	H23A—C23—H23B	109.5
C12—C11—C10	109.8 (5)	C16—C23—H23C	109.5
C12—C11—C17	111.0 (5)	H23A—C23—H23C	109.5
C10—C11—C17	109.3 (5)	H23B—C23—H23C	109.5
C12—C11—H11A	108.9	O4—C24—C25	104.8 (7)
C10—C11—H11A	108.9	O4—C24—H24A	110.8
C17—C11—H11A	108.9	C25—C24—H24A	110.8
C13—C12—C11	112.2 (6)	O4—C24—H24B	110.8
C13—C12—H12A	109.2	C25—C24—H24B	110.8
C11—C12—H12A	109.2	H24A—C24—H24B	108.9
C13—C12—H12B	109.2	O3—C25—C24	107.7 (8)
C11—C12—H12B	109.2	O3—C25—H25B	110.2
H12A—C12—H12B	107.9	C24—C25—H25B	110.2
C8—C13—C12	126.6 (6)	O3—C25—H25A	110.2
C8—C13—H13A	116.7	C24—C25—H25A	110.2
C12—C13—H13A	116.7	H25B—C25—H25A	108.5
C4—O1—C3—O2	2.1 (15)	C14—C15—C16—C17	55.8 (7)
C4—O1—C3—C2	177.4 (10)	C14—C15—C16—C23	−68.2 (7)
C3—O1—C4—C5	115.6 (9)	C14—C15—C16—C19	167.5 (6)
C3—O1—C4—C9	−125.0 (9)	C15—C16—C17—C11	−59.4 (7)
O1—C4—C5—C6	179.4 (5)	C23—C16—C17—C11	63.7 (7)
C9—C4—C5—C6	60.2 (8)	C19—C16—C17—C11	177.7 (5)
C4—C5—C6—C7	−55.3 (9)	C15—C16—C17—C21	167.8 (5)
C5—C6—C7—C8	45.2 (8)	C23—C16—C17—C21	−69.1 (7)
C5—C6—C7—C22	−72.6 (8)	C19—C16—C17—C21	44.9 (6)
C5—C6—C7—C10	163.9 (6)	C12—C11—C17—C16	178.8 (5)
C22—C7—C8—C13	−108.8 (7)	C10—C11—C17—C16	57.6 (6)
C10—C7—C8—C13	15.3 (8)	C12—C11—C17—C21	−54.8 (7)
C6—C7—C8—C13	133.7 (7)	C10—C11—C17—C21	−176.1 (5)
C22—C7—C8—C9	71.3 (7)	C25—O3—C19—O4	14.6 (9)
C10—C7—C8—C9	−164.6 (5)	C25—O3—C19—C20	134.1 (8)
C6—C7—C8—C9	−46.1 (7)	C25—O3—C19—C16	−108.9 (8)
C13—C8—C9—C4	−125.4 (8)	C24—O4—C19—O3	−16.4 (8)
C7—C8—C9—C4	54.4 (8)	C24—O4—C19—C20	−137.0 (7)
O1—C4—C9—C8	−179.5 (6)	C24—O4—C19—C16	108.0 (7)
C5—C4—C9—C8	−58.9 (8)	C15—C16—C19—O3	87.0 (7)
C8—C7—C10—C11	−44.8 (6)	C17—C16—C19—O3	−157.0 (6)
C22—C7—C10—C11	76.7 (7)	C23—C16—C19—O3	−39.1 (8)

C6—C7—C10—C11	−163.0 (5)	C15—C16—C19—O4	−33.7 (8)
C8—C7—C10—C14	−171.8 (5)	C17—C16—C19—O4	82.3 (7)
C22—C7—C10—C14	−50.3 (7)	C23—C16—C19—O4	−159.9 (6)
C6—C7—C10—C14	70.1 (7)	C15—C16—C19—C20	−151.9 (6)
C7—C10—C11—C12	60.1 (6)	C17—C16—C19—C20	−35.9 (6)
C14—C10—C11—C12	−172.9 (5)	C23—C16—C19—C20	82.0 (7)
C7—C10—C11—C17	−177.9 (5)	O3—C19—C20—C21	137.3 (6)
C14—C10—C11—C17	−51.0 (6)	O4—C19—C20—C21	−104.8 (6)
C10—C11—C12—C13	−42.8 (7)	C16—C19—C20—C21	15.0 (8)
C17—C11—C12—C13	−163.8 (5)	C16—C17—C21—C20	−35.8 (7)
C9—C8—C13—C12	178.9 (6)	C11—C17—C21—C20	−166.7 (6)
C7—C8—C13—C12	−0.9 (10)	C19—C20—C21—C17	12.0 (8)
C11—C12—C13—C8	15.1 (10)	C19—O4—C24—C25	11.9 (9)
C11—C10—C14—C15	51.9 (7)	C19—O3—C25—C24	−7.1 (11)
C7—C10—C14—C15	180.0 (5)	O4—C24—C25—O3	−3.0 (11)
C10—C14—C15—C16	−54.5 (7)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4A···O2	0.98	2.22	2.650 (10)	105
C15—H15A···O4	0.97	2.52	2.886 (8)	102
C23—H23B···O3	0.96	2.41	2.744 (9)	100

Fig. 1

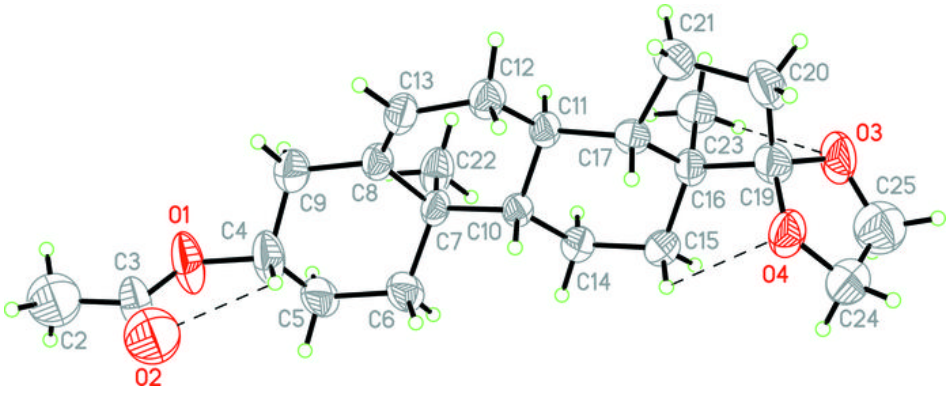
C21
C20
C13
C12
C11
O3
C23
C22
C17
C25
C9
C8
C16
C19
O1
C4
C7
C10
C15
C3
O4
C14
C5
C6
C24
C2
O2

Fig. 2