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3β-Acetoxy-5α-chloro-6β-hydroxyandrostan-17-one

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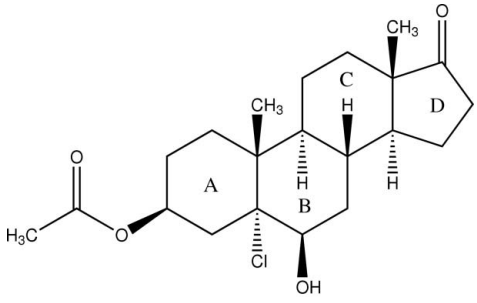
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.032; wR factor = 0.090; data-to-parameter ratio = 10.2.

The title compound, $C_{21}H_{31}ClO_4$, crystallizes with two molecules in the asymmetric unit. Rings *A*, *B* and *C* have chair conformations, while ring *D* has an envelope form. The crystal packing shows that the molecules are connected by intermolecular O—H···O hydrogen bonds.

Related literature

For related literature, see: Alvarez-Ginarte *et al.* (2005); Mousseron-Canet *et al.* (1968); Rendle & Trotter (1974); Stanley *et al.* (1997); Xia *et al.* (2007).



Experimental

Crystal data

$C_{21}H_{31}ClO_4$
M_r = 382.91
Triclinic, $P1$
a = 7.1025 (12) Å
b = 9.9840 (16) Å
c = 15.552 (3) Å
α = 100.521 (2)°
β = 99.211 (2)°
γ = 110.470 (2)°
V = 985.3 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 296 (2) K
0.30 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.938, T_{max} = 0.962
5701 measured reflections
4861 independent reflections
4372 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.090
S = 1.06
4861 reflections
476 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Absolute structure: Flack (1983), with 983 Friedel pairs
Flack parameter: 0.00 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3X···O4^{i}	0.82	1.98	2.745 (3)	155
O7—H7X···O8^{ii}	0.80	2.03	2.791 (3)	160

Symmetry codes: (i) $x, y+1, z$; (ii) $x-1, y-1, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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, 2005); 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: BT2381).

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supplementary materials

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3β-Acetoxy-5α-chloro-6β-hydroxyandrostan-17-one

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Comment

Many anabolic steroids have a reduced androgenic side effect (Stanley *et al.*, 1997). Androstane derivatives may be used for oral therapy aginst weight gain after extensive surgery, chronic infections and severe traumata (Alvarez-Ginarte *et al.*, 2005). In continuation of our work on the structure-activity relationship of androstanone derivatives, we present here the structure of the title compound (Fig. 1).

The title compound crystallizes with two molecules in the asymmetric unit. All bond lengths and angles correspond well to those observed in similar steriod structures (Rendle *et al.*, 1974; Xia *et al.*, 2007). Rings A, B and C adopt a chair conformation, while ring D shows an envelope conformation. The hydroxyl group is involved in an intramolecular O—H···O hydrogen bond (Table 1).

Experimental

3β-acetoxy-5α-chloro-6β-hydroxy-17-androstanone, in a form of a white powder, was synthesized according to Mousseron-Canet *et al.* (1968). Crystals suitable for structure analysis were obtained by slow evaporation from a mixture of tetrahydrofuran, acetone and water (volume proportion, 2:2:1) as colourless prisms (m.p. 518 K).

Refinement

The absolute stereochemistry of the title compound was known from the synthetic route (Mousseron-Canet *et al.*, 1968) and it was confirmed by the structure determination. The hydroxy atom H3X and H7X were located in a difference Fourier map but refined using a riding model with U_{iso}(H) = 1.2U_{eq}(O). Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and torsion angles were refined to fit the electron density, U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions with C—H = 0.97 or C—H = 0.98, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

Fig. 1. The molecular structure of one molecule in the asymmetric unit of the title compound, shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

3β-Acetoxy-5α-chloro-6β-hydroxyandrostan-17-one

Crystal data

$C_{21}H_{31}ClO_4$ $Z = 2$

M_r = 382.91	F_{000} = 412
Triclinic, $P\overline{1}$	D_x = 1.291 Mg m^{-3}
Hall symbol: P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.1025 (12) Å	Cell parameters from 2891 reflections
b = 9.9840 (16) Å	θ = 2.4–22.1º
c = 15.552 (3) Å	μ = 0.22 mm^{-1}
α = 100.521 (2)º	T = 296 (2) K
β = 99.211 (2)º	Prismatic, colourless
γ = 110.470 (2)º	0.30 × 0.20 × 0.15 mm
V = 985.3 (3) Å^3	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	4861 independent reflections
Radiation source: fine-focus sealed tube	4372 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.020
T = 296(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 1.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −8→8
T_{min} = 0.938, T_{max} = 0.962	k = −12→12
5701 measured reflections	l = −19→16

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.128P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.090	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.06	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
4861 reflections	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
476 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
3 restraints	Extinction coefficient: 0.007 (2)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with 983 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.00 (5)

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}
Cl1	0.17673 (11)	0.05164 (8)	0.62739 (5)	0.0546 (2)
O1	0.7135 (4)	0.4524 (2)	0.63658 (16)	0.0629 (6)
O2	0.6585 (6)	0.3949 (3)	0.4876 (2)	0.1031 (11)
O3	0.3879 (4)	0.2852 (2)	0.88199 (15)	0.0569 (6)
H3X	0.3583	0.3580	0.8939	0.085*
O4	0.1729 (4)	−0.5336 (2)	0.89166 (19)	0.0684 (7)
C1	0.6278 (5)	0.0694 (3)	0.6709 (2)	0.0507 (7)
H1A	0.7268	0.0246	0.6836	0.061*
H1B	0.5207	0.0035	0.6180	0.061*
C2	0.7389 (6)	0.2187 (4)	0.6503 (3)	0.0610 (9)
H2A	0.8576	0.2803	0.7000	0.073*
H2B	0.7889	0.2015	0.5965	0.073*
C3	0.5950 (5)	0.2993 (3)	0.6362 (2)	0.0540 (8)
H3	0.4917	0.2486	0.5786	0.065*
C4	0.4876 (5)	0.3124 (3)	0.7115 (2)	0.0475 (7)
H4A	0.3867	0.3543	0.6957	0.057*
H4B	0.5890	0.3798	0.7657	0.057*
C5	0.3783 (4)	0.1642 (3)	0.73129 (18)	0.0401 (6)
C6	0.2540 (5)	0.1776 (3)	0.8018 (2)	0.0435 (7)
H6	0.1471	0.2115	0.7782	0.052*
C7	0.1487 (5)	0.0306 (3)	0.8224 (2)	0.0446 (7)
H7A	0.0354	−0.0335	0.7711	0.054*
H7B	0.0901	0.0470	0.8736	0.054*
C8	0.2942 (4)	−0.0476 (3)	0.84321 (18)	0.0356 (6)
H8	0.3996	0.0129	0.8989	0.043*
C9	0.4043 (4)	−0.0669 (3)	0.76705 (18)	0.0371 (6)
H9	0.2953	−0.1227	0.7117	0.044*
C10	0.5290 (4)	0.0854 (3)	0.75172 (19)	0.0392 (6)
C11	0.5344 (5)	−0.1600 (3)	0.7817 (2)	0.0470 (7)
H11A	0.6544	−0.1026	0.8316	0.056*
H11B	0.5839	−0.1807	0.7282	0.056*
C12	0.4125 (5)	−0.3077 (3)	0.8018 (2)	0.0461 (7)
H12A	0.3041	−0.3719	0.7489	0.055*
H12B	0.5049	−0.3569	0.8162	0.055*
C13	0.3171 (4)	−0.2796 (3)	0.88046 (19)	0.0378 (6)
C14	0.1758 (4)	−0.1985 (3)	0.85734 (18)	0.0373 (6)
H14	0.0798	−0.2584	0.7992	0.045*
C15	0.0463 (5)	−0.2124 (3)	0.9272 (2)	0.0482 (7)
H15A	−0.0805	−0.1989	0.9066	0.058*
H15B	0.1236	−0.1409	0.9844	0.058*

C16	0.0003 (5)	−0.3715 (3)	0.9351 (3)	0.0539 (8)
H16A	0.0079	−0.3760	0.9973	0.065*
H16B	−0.1365	−0.4381	0.8987	0.065*
C17	0.1666 (5)	−0.4119 (3)	0.9006 (2)	0.0462 (7)
C18	0.4835 (5)	−0.1995 (3)	0.9694 (2)	0.0494 (7)
H18A	0.5567	−0.2607	0.9828	0.074*
H18B	0.4186	−0.1801	1.0172	0.074*
H18C	0.5790	−0.1078	0.9637	0.074*
C19	0.7040 (4)	0.1796 (3)	0.8360 (2)	0.0489 (7)
H19A	0.7732	0.2766	0.8283	0.073*
H19B	0.8013	0.1336	0.8447	0.073*
H19C	0.6468	0.1875	0.8877	0.073*
C20	0.7226 (6)	0.4851 (4)	0.5575 (3)	0.0629 (9)
C21	0.8229 (7)	0.6486 (4)	0.5700 (3)	0.0785 (12)
H21A	0.8258	0.6694	0.5123	0.118*
H21B	0.9619	0.6852	0.6064	0.118*
H21C	0.7453	0.6961	0.5994	0.118*
Cl2	0.02987 (12)	0.94245 (8)	0.40404 (5)	0.0557 (2)
O5	0.1815 (4)	0.5505 (2)	0.40842 (17)	0.0656 (6)
O6	0.2826 (6)	0.6093 (4)	0.5582 (2)	0.0982 (10)
O7	−0.1161 (3)	0.7115 (2)	0.14985 (15)	0.0588 (6)
H7X	−0.2290	0.6472	0.1372	0.088*
O8	0.4767 (4)	1.5323 (2)	0.1432 (2)	0.0817 (8)
C31	0.4535 (5)	0.9362 (3)	0.3801 (2)	0.0540 (8)
H31A	0.5922	0.9836	0.3725	0.065*
H31B	0.4369	1.0006	0.4306	0.065*
C32	0.4305 (6)	0.7887 (4)	0.4023 (3)	0.0614 (9)
H32A	0.4676	0.7298	0.3564	0.074*
H32B	0.5256	0.8077	0.4596	0.074*
C33	0.2126 (5)	0.7024 (4)	0.4075 (2)	0.0554 (8)
H33	0.1857	0.7506	0.4623	0.067*
C34	0.0500 (5)	0.6868 (3)	0.3261 (2)	0.0491 (7)
H34A	−0.0863	0.6421	0.3368	0.059*
H34B	0.0589	0.6210	0.2741	0.059*
C35	0.0747 (4)	0.8347 (3)	0.30524 (19)	0.0403 (6)
C36	−0.1024 (4)	0.8177 (3)	0.2280 (2)	0.0451 (7)
H36	−0.2326	0.7820	0.2468	0.054*
C37	−0.0746 (4)	0.9647 (3)	0.2056 (2)	0.0464 (7)
H37A	−0.1756	0.9464	0.1503	0.056*
H37B	−0.1027	1.0267	0.2534	0.056*
C38	0.1410 (4)	1.0482 (3)	0.19419 (19)	0.0385 (6)
H38	0.1624	0.9897	0.1416	0.046*
C39	0.3107 (4)	1.0710 (3)	0.27751 (19)	0.0389 (6)
H39	0.2798	1.1235	0.3295	0.047*
C40	0.2954 (4)	0.9187 (3)	0.29435 (19)	0.0401 (6)
C41	0.5284 (4)	1.1703 (3)	0.2727 (2)	0.0505 (7)
H41A	0.6244	1.1933	0.3303	0.061*
H41B	0.5726	1.1159	0.2274	0.061*
C42	0.5396 (5)	1.3155 (3)	0.2498 (2)	0.0526 (7)

H42A	0.5169	1.3780	0.2993	0.063*
H42B	0.6760	1.3679	0.2414	0.063*
C43	0.3770 (4)	1.2835 (3)	0.1647 (2)	0.0437 (7)
C44	0.1637 (4)	1.1982 (3)	0.1781 (2)	0.0424 (6)
H44	0.1516	1.2563	0.2334	0.051*
C45	0.0106 (5)	1.2077 (4)	0.1005 (3)	0.0588 (8)
H45A	−0.1234	1.1900	0.1139	0.071*
H45B	−0.0068	1.1376	0.0447	0.071*
C46	0.1169 (6)	1.3678 (4)	0.0952 (3)	0.0687 (10)
H46A	0.0955	1.3735	0.0330	0.082*
H46B	0.0622	1.4316	0.1284	0.082*
C47	0.3440 (5)	1.4129 (3)	0.1368 (2)	0.0566 (8)
C48	0.4288 (5)	1.2055 (3)	0.0830 (2)	0.0549 (8)
H48A	0.3259	1.1881	0.0295	0.082*
H48B	0.4306	1.1128	0.0909	0.082*
H48C	0.5624	1.2668	0.0775	0.082*
C49	0.3373 (5)	0.8272 (3)	0.2138 (2)	0.0502 (7)
H49A	0.4703	0.8825	0.2049	0.075*
H49B	0.2318	0.8060	0.1605	0.075*
H49C	0.3360	0.7361	0.2261	0.075*
C50	0.2136 (6)	0.5192 (4)	0.4881 (3)	0.0652 (9)
C51	0.1468 (7)	0.3559 (4)	0.4753 (3)	0.0844 (13)
H51A	0.0008	0.3079	0.4482	0.127*
H51B	0.1760	0.3351	0.5327	0.127*
H51C	0.2209	0.3197	0.4367	0.127*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0556 (5)	0.0553 (4)	0.0446 (4)	0.0200 (4)	−0.0035 (3)	0.0104 (3)
O1	0.0739 (16)	0.0501 (12)	0.0605 (15)	0.0153 (11)	0.0133 (12)	0.0248 (11)
O2	0.144 (3)	0.088 (2)	0.0557 (19)	0.016 (2)	0.0311 (19)	0.0215 (16)
O3	0.0799 (16)	0.0516 (12)	0.0463 (13)	0.0407 (12)	0.0107 (11)	0.0035 (10)
O4	0.0709 (16)	0.0387 (11)	0.107 (2)	0.0280 (11)	0.0326 (15)	0.0214 (11)
C1	0.0559 (19)	0.0529 (16)	0.0543 (19)	0.0288 (15)	0.0211 (15)	0.0182 (14)
C2	0.062 (2)	0.066 (2)	0.070 (2)	0.0292 (18)	0.0318 (18)	0.0309 (18)
C3	0.063 (2)	0.0438 (16)	0.0555 (19)	0.0185 (15)	0.0133 (16)	0.0192 (14)
C4	0.0541 (18)	0.0419 (15)	0.0464 (17)	0.0210 (14)	0.0052 (14)	0.0129 (13)
C5	0.0436 (16)	0.0405 (13)	0.0343 (15)	0.0193 (12)	0.0016 (12)	0.0065 (11)
C6	0.0477 (17)	0.0481 (16)	0.0485 (18)	0.0330 (14)	0.0120 (14)	0.0161 (13)
C7	0.0439 (17)	0.0504 (16)	0.0531 (18)	0.0307 (14)	0.0165 (14)	0.0162 (13)
C8	0.0339 (14)	0.0362 (12)	0.0397 (15)	0.0192 (11)	0.0066 (11)	0.0076 (11)
C9	0.0361 (14)	0.0396 (13)	0.0364 (14)	0.0191 (11)	0.0064 (11)	0.0055 (11)
C10	0.0395 (15)	0.0391 (13)	0.0432 (15)	0.0199 (12)	0.0109 (12)	0.0102 (11)
C11	0.0508 (18)	0.0477 (15)	0.0592 (19)	0.0322 (14)	0.0253 (15)	0.0167 (13)
C12	0.0501 (17)	0.0405 (14)	0.0544 (18)	0.0271 (13)	0.0148 (14)	0.0074 (12)
C13	0.0363 (15)	0.0336 (12)	0.0442 (15)	0.0169 (11)	0.0069 (12)	0.0073 (11)
C14	0.0346 (14)	0.0368 (13)	0.0401 (15)	0.0176 (11)	0.0060 (11)	0.0047 (11)

C15	0.0456 (17)	0.0446 (15)	0.061 (2)	0.0219 (14)	0.0208 (15)	0.0145 (14)
C16	0.0527 (19)	0.0415 (16)	0.071 (2)	0.0178 (14)	0.0224 (17)	0.0169 (15)
C17	0.0459 (17)	0.0362 (14)	0.0548 (18)	0.0181 (13)	0.0076 (13)	0.0081 (12)
C18	0.0430 (17)	0.0507 (16)	0.0519 (18)	0.0190 (13)	0.0021 (14)	0.0139 (13)
C19	0.0401 (16)	0.0450 (15)	0.061 (2)	0.0175 (13)	0.0069 (14)	0.0138 (14)
C20	0.065 (2)	0.074 (2)	0.058 (2)	0.0274 (19)	0.0231 (18)	0.0299 (19)
C21	0.081 (3)	0.069 (2)	0.098 (3)	0.025 (2)	0.034 (2)	0.049 (2)
Cl2	0.0674 (5)	0.0598 (4)	0.0461 (4)	0.0281 (4)	0.0242 (4)	0.0128 (3)
O5	0.0881 (18)	0.0547 (12)	0.0625 (15)	0.0336 (13)	0.0169 (13)	0.0250 (11)
O6	0.140 (3)	0.096 (2)	0.065 (2)	0.056 (2)	0.0091 (19)	0.0288 (17)
O7	0.0478 (13)	0.0588 (13)	0.0467 (13)	0.0033 (10)	0.0051 (10)	0.0000 (10)
O8	0.0619 (15)	0.0400 (12)	0.133 (3)	0.0076 (11)	0.0193 (16)	0.0271 (14)
C31	0.0447 (18)	0.0548 (17)	0.057 (2)	0.0176 (15)	0.0009 (15)	0.0167 (15)
C32	0.058 (2)	0.066 (2)	0.064 (2)	0.0299 (17)	0.0041 (17)	0.0252 (17)
C33	0.065 (2)	0.0505 (16)	0.0541 (19)	0.0242 (16)	0.0120 (16)	0.0206 (15)
C34	0.0514 (18)	0.0429 (15)	0.0513 (18)	0.0154 (14)	0.0143 (14)	0.0122 (13)
C35	0.0385 (15)	0.0428 (14)	0.0388 (15)	0.0149 (12)	0.0120 (12)	0.0084 (11)
C36	0.0277 (14)	0.0485 (16)	0.0483 (18)	0.0035 (12)	0.0060 (12)	0.0124 (13)
C37	0.0280 (14)	0.0536 (16)	0.0548 (18)	0.0117 (13)	0.0057 (13)	0.0200 (14)
C38	0.0295 (14)	0.0427 (14)	0.0374 (14)	0.0093 (11)	0.0066 (11)	0.0071 (11)
C39	0.0322 (14)	0.0418 (14)	0.0380 (15)	0.0118 (11)	0.0055 (11)	0.0071 (11)
C40	0.0338 (15)	0.0411 (13)	0.0403 (15)	0.0113 (12)	0.0068 (12)	0.0076 (11)
C41	0.0312 (15)	0.0515 (16)	0.0556 (19)	0.0054 (13)	0.0001 (13)	0.0133 (14)
C42	0.0377 (16)	0.0437 (15)	0.059 (2)	0.0017 (13)	0.0034 (14)	0.0070 (14)
C43	0.0372 (15)	0.0356 (13)	0.0489 (17)	0.0068 (12)	0.0091 (13)	0.0050 (12)
C44	0.0376 (15)	0.0417 (14)	0.0462 (17)	0.0141 (12)	0.0106 (13)	0.0098 (12)
C45	0.0423 (18)	0.0583 (19)	0.075 (2)	0.0151 (15)	0.0072 (16)	0.0311 (17)
C46	0.059 (2)	0.058 (2)	0.096 (3)	0.0241 (17)	0.018 (2)	0.037 (2)
C47	0.0534 (19)	0.0433 (16)	0.069 (2)	0.0134 (15)	0.0171 (17)	0.0135 (15)
C48	0.058 (2)	0.0531 (17)	0.053 (2)	0.0170 (15)	0.0225 (16)	0.0138 (14)
C49	0.0431 (17)	0.0518 (16)	0.0599 (19)	0.0208 (14)	0.0205 (15)	0.0127 (14)
C50	0.069 (2)	0.076 (2)	0.069 (3)	0.038 (2)	0.023 (2)	0.036 (2)
C51	0.096 (3)	0.078 (3)	0.104 (3)	0.046 (3)	0.030 (3)	0.052 (2)

Geometric parameters (Å, °)

Cl1—C5	1.842 (3)	Cl2—C35	1.848 (3)
O1—C20	1.334 (4)	O5—C50	1.337 (4)
O1—C3	1.463 (4)	O5—C33	1.456 (4)
O2—C20	1.183 (5)	O6—C50	1.183 (5)
O3—C6	1.425 (4)	O7—C36	1.425 (4)
O3—H3X	0.8229	O7—H7X	0.7983
O4—C17	1.215 (3)	O8—C47	1.209 (4)
C1—C2	1.543 (4)	C31—C32	1.534 (4)
C1—C10	1.544 (4)	C31—C40	1.539 (4)
C1—H1A	0.9700	C31—H31A	0.9700
C1—H1B	0.9700	C31—H31B	0.9700
C2—C3	1.519 (5)	C32—C33	1.509 (5)
C2—H2A	0.9700	C32—H32A	0.9700

C2—H2B	0.9700	C32—H32B	0.9700
C3—C4	1.507 (4)	C33—C34	1.515 (5)
C3—H3	0.9800	C33—H33	0.9800
C4—C5	1.525 (4)	C34—C35	1.525 (4)
C4—H4A	0.9700	C34—H34A	0.9700
C4—H4B	0.9700	C34—H34B	0.9700
C5—C6	1.530 (4)	C35—C36	1.532 (4)
C5—C10	1.561 (3)	C35—C40	1.551 (4)
C6—C7	1.515 (4)	C36—C37	1.523 (4)
C6—H6	0.9800	C36—H36	0.9800
C7—C8	1.528 (3)	C37—C38	1.523 (4)
C7—H7A	0.9700	C37—H37A	0.9700
C7—H7B	0.9700	C37—H37B	0.9700
C8—C14	1.523 (4)	C38—C44	1.520 (4)
C8—C9	1.540 (3)	C38—C39	1.544 (4)
C8—H8	0.9800	C38—H38	0.9800
C9—C11	1.542 (3)	C39—C41	1.540 (4)
C9—C10	1.553 (4)	C39—C40	1.559 (4)
C9—H9	0.9800	C39—H39	0.9800
C10—C19	1.536 (4)	C40—C49	1.544 (4)
C11—C12	1.544 (4)	C41—C42	1.533 (4)
C11—H11A	0.9700	C41—H41A	0.9700
C11—H11B	0.9700	C41—H41B	0.9700
C12—C13	1.519 (4)	C42—C43	1.512 (4)
C12—H12A	0.9700	C42—H42A	0.9700
C12—H12B	0.9700	C42—H42B	0.9700
C13—C17	1.503 (4)	C43—C47	1.516 (4)
C13—C14	1.533 (3)	C43—C44	1.526 (4)
C13—C18	1.536 (4)	C43—C48	1.531 (4)
C14—C15	1.527 (4)	C44—C45	1.530 (4)
C14—H14	0.9800	C44—H44	0.9800
C15—C16	1.538 (4)	C45—C46	1.534 (4)
C15—H15A	0.9700	C45—H45A	0.9700
C15—H15B	0.9700	C45—H45B	0.9700
C16—C17	1.517 (4)	C46—C47	1.508 (5)
C16—H16A	0.9700	C46—H46A	0.9700
C16—H16B	0.9700	C46—H46B	0.9700
C18—H18A	0.9600	C48—H48A	0.9600
C18—H18B	0.9600	C48—H48B	0.9600
C18—H18C	0.9600	C48—H48C	0.9600
C19—H19A	0.9600	C49—H49A	0.9600
C19—H19B	0.9600	C49—H49B	0.9600
C19—H19C	0.9600	C49—H49C	0.9600
C20—C21	1.495 (5)	C50—C51	1.494 (5)
C21—H21A	0.9600	C51—H51A	0.9600
C21—H21B	0.9600	C51—H51B	0.9600
C21—H21C	0.9600	C51—H51C	0.9600
C20—O1—C3	118.3 (3)	C50—O5—C33	118.4 (3)
C6—O3—H3X	113.1	C36—O7—H7X	107.3

C2—C1—C10	112.8 (2)	C32—C31—C40	113.2 (3)
C2—C1—H1A	109.0	C32—C31—H31A	108.9
C10—C1—H1A	109.0	C40—C31—H31A	108.9
C2—C1—H1B	109.0	C32—C31—H31B	108.9
C10—C1—H1B	109.0	C40—C31—H31B	108.9
H1A—C1—H1B	107.8	H31A—C31—H31B	107.8
C3—C2—C1	111.4 (3)	C33—C32—C31	112.0 (3)
C3—C2—H2A	109.3	C33—C32—H32A	109.2
C1—C2—H2A	109.3	C31—C32—H32A	109.2
C3—C2—H2B	109.3	C33—C32—H32B	109.2
C1—C2—H2B	109.3	C31—C32—H32B	109.2
H2A—C2—H2B	108.0	H32A—C32—H32B	107.9
O1—C3—C4	104.5 (2)	O5—C33—C32	111.1 (3)
O1—C3—C2	110.2 (3)	O5—C33—C34	104.3 (3)
C4—C3—C2	112.8 (3)	C32—C33—C34	112.9 (3)
O1—C3—H3	109.7	O5—C33—H33	109.5
C4—C3—H3	109.7	C32—C33—H33	109.5
C2—C3—H3	109.7	C34—C33—H33	109.5
C3—C4—C5	112.8 (2)	C33—C34—C35	112.7 (2)
C3—C4—H4A	109.0	C33—C34—H34A	109.0
C5—C4—H4A	109.0	C35—C34—H34A	109.0
C3—C4—H4B	109.0	C33—C34—H34B	109.0
C5—C4—H4B	109.0	C35—C34—H34B	109.0
H4A—C4—H4B	107.8	H34A—C34—H34B	107.8
C4—C5—C6	111.9 (2)	C34—C35—C36	111.6 (2)
C4—C5—C10	111.9 (2)	C34—C35—C40	112.1 (2)
C6—C5—C10	114.5 (2)	C36—C35—C40	115.0 (2)
C4—C5—Cl1	105.02 (18)	C34—C35—Cl2	104.84 (19)
C6—C5—Cl1	103.4 (2)	C36—C35—Cl2	103.07 (19)
C10—C5—Cl1	109.34 (17)	C40—C35—Cl2	109.35 (18)
O3—C6—C7	110.3 (2)	O7—C36—C37	109.9 (2)
O3—C6—C5	109.4 (2)	O7—C36—C35	110.2 (2)
C7—C6—C5	111.7 (2)	C37—C36—C35	111.6 (2)
O3—C6—H6	108.5	O7—C36—H36	108.4
C7—C6—H6	108.5	C37—C36—H36	108.4
C5—C6—H6	108.5	C35—C36—H36	108.4
C6—C7—C8	113.5 (2)	C38—C37—C36	113.8 (2)
C6—C7—H7A	108.9	C38—C37—H37A	108.8
C8—C7—H7A	108.9	C36—C37—H37A	108.8
C6—C7—H7B	108.9	C38—C37—H37B	108.8
C8—C7—H7B	108.9	C36—C37—H37B	108.8
H7A—C7—H7B	107.7	H37A—C37—H37B	107.7
C14—C8—C7	110.7 (2)	C44—C38—C37	111.3 (2)
C14—C8—C9	109.35 (19)	C44—C38—C39	108.9 (2)
C7—C8—C9	111.4 (2)	C37—C38—C39	111.4 (2)
C14—C8—H8	108.5	C44—C38—H38	108.3
C7—C8—H8	108.5	C37—C38—H38	108.3
C9—C8—H8	108.5	C39—C38—H38	108.3
C8—C9—C11	112.3 (2)	C41—C39—C38	112.6 (2)

C8—C9—C10	110.65 (19)	C41—C39—C40	113.8 (2)
C11—C9—C10	113.5 (2)	C38—C39—C40	110.2 (2)
C8—C9—H9	106.6	C41—C39—H39	106.5
C11—C9—H9	106.6	C38—C39—H39	106.5
C10—C9—H9	106.6	C40—C39—H39	106.5
C19—C10—C1	107.9 (2)	C31—C40—C49	107.9 (2)
C19—C10—C9	109.7 (2)	C31—C40—C35	108.4 (2)
C1—C10—C9	112.1 (2)	C49—C40—C35	109.8 (2)
C19—C10—C5	110.6 (2)	C31—C40—C39	112.1 (2)
C1—C10—C5	107.9 (2)	C49—C40—C39	109.8 (2)
C9—C10—C5	108.7 (2)	C35—C40—C39	108.8 (2)
C9—C11—C12	113.2 (2)	C42—C41—C39	113.7 (2)
C9—C11—H11A	108.9	C42—C41—H41A	108.8
C12—C11—H11A	108.9	C39—C41—H41A	108.8
C9—C11—H11B	108.9	C42—C41—H41B	108.8
C12—C11—H11B	108.9	C39—C41—H41B	108.8
H11A—C11—H11B	107.8	H41A—C41—H41B	107.7
C13—C12—C11	110.1 (2)	C43—C42—C41	109.9 (2)
C13—C12—H12A	109.6	C43—C42—H42A	109.7
C11—C12—H12A	109.6	C41—C42—H42A	109.7
C13—C12—H12B	109.6	C43—C42—H42B	109.7
C11—C12—H12B	109.6	C41—C42—H42B	109.7
H12A—C12—H12B	108.1	H42A—C42—H42B	108.2
C17—C13—C12	117.4 (2)	C42—C43—C47	118.3 (2)
C17—C13—C14	100.4 (2)	C42—C43—C44	109.2 (2)
C12—C13—C14	108.9 (2)	C47—C43—C44	100.8 (2)
C17—C13—C18	104.4 (2)	C42—C43—C48	110.6 (3)
C12—C13—C18	111.6 (2)	C47—C43—C48	103.8 (2)
C14—C13—C18	113.8 (2)	C44—C43—C48	114.0 (2)
C8—C14—C15	120.5 (2)	C38—C44—C43	112.6 (2)
C8—C14—C13	112.4 (2)	C38—C44—C45	119.7 (2)
C15—C14—C13	104.5 (2)	C43—C44—C45	104.7 (2)
C8—C14—H14	106.2	C38—C44—H44	106.3
C15—C14—H14	106.2	C43—C44—H44	106.3
C13—C14—H14	106.2	C45—C44—H44	106.3
C14—C15—C16	102.8 (2)	C44—C45—C46	102.2 (3)
C14—C15—H15A	111.2	C44—C45—H45A	111.3
C16—C15—H15A	111.2	C46—C45—H45A	111.3
C14—C15—H15B	111.2	C44—C45—H45B	111.3
C16—C15—H15B	111.2	C46—C45—H45B	111.3
H15A—C15—H15B	109.1	H45A—C45—H45B	109.2
C17—C16—C15	104.9 (2)	C47—C46—C45	105.7 (3)
C17—C16—H16A	110.8	C47—C46—H46A	110.6
C15—C16—H16A	110.8	C45—C46—H46A	110.6
C17—C16—H16B	110.8	C47—C46—H46B	110.6
C15—C16—H16B	110.8	C45—C46—H46B	110.6
H16A—C16—H16B	108.8	H46A—C46—H46B	108.7
O4—C17—C13	126.7 (3)	O8—C47—C46	124.4 (3)
O4—C17—C16	123.9 (3)	O8—C47—C43	126.5 (3)

C13—C17—C16	109.5 (2)	C46—C47—C43	109.1 (3)
C13—C18—H18A	109.5	C43—C48—H48A	109.5
C13—C18—H18B	109.5	C43—C48—H48B	109.5
H18A—C18—H18B	109.5	H48A—C48—H48B	109.5
C13—C18—H18C	109.5	C43—C48—H48C	109.5
H18A—C18—H18C	109.5	H48A—C48—H48C	109.5
H18B—C18—H18C	109.5	H48B—C48—H48C	109.5
C10—C19—H19A	109.5	C40—C49—H49A	109.5
C10—C19—H19B	109.5	C40—C49—H49B	109.5
H19A—C19—H19B	109.5	H49A—C49—H49B	109.5
C10—C19—H19C	109.5	C40—C49—H49C	109.5
H19A—C19—H19C	109.5	H49A—C49—H49C	109.5
H19B—C19—H19C	109.5	H49B—C49—H49C	109.5
O2—C20—O1	123.5 (3)	O6—C50—O5	124.0 (3)
O2—C20—C21	125.5 (4)	O6—C50—C51	125.7 (4)
O1—C20—C21	111.0 (3)	O5—C50—C51	110.3 (4)
C20—C21—H21A	109.5	C50—C51—H51A	109.5
C20—C21—H21B	109.5	C50—C51—H51B	109.5
H21A—C21—H21B	109.5	H51A—C51—H51B	109.5
C20—C21—H21C	109.5	C50—C51—H51C	109.5
H21A—C21—H21C	109.5	H51A—C51—H51C	109.5
H21B—C21—H21C	109.5	H51B—C51—H51C	109.5
C10—C1—C2—C3	−54.8 (4)	C40—C31—C32—C33	−53.9 (4)
C20—O1—C3—C4	−140.8 (3)	C50—O5—C33—C32	92.6 (4)
C20—O1—C3—C2	97.8 (3)	C50—O5—C33—C34	−145.5 (3)
C1—C2—C3—O1	167.6 (3)	C31—C32—C33—O5	167.2 (3)
C1—C2—C3—C4	51.3 (4)	C31—C32—C33—C34	50.4 (4)
O1—C3—C4—C5	−172.0 (2)	O5—C33—C34—C35	−172.2 (2)
C2—C3—C4—C5	−52.3 (4)	C32—C33—C34—C35	−51.4 (4)
C3—C4—C5—C6	−174.7 (3)	C33—C34—C35—C36	−174.9 (3)
C3—C4—C5—C10	55.3 (3)	C33—C34—C35—C40	54.5 (3)
C3—C4—C5—Cl1	−63.3 (3)	C33—C34—C35—Cl2	−64.0 (3)
C4—C5—C6—O3	−56.6 (3)	C34—C35—C36—O7	−56.2 (3)
C10—C5—C6—O3	72.0 (3)	C40—C35—C36—O7	72.9 (3)
Cl1—C5—C6—O3	−169.16 (17)	Cl2—C35—C36—O7	−168.19 (18)
C4—C5—C6—C7	−179.0 (2)	C34—C35—C36—C37	−178.5 (2)
C10—C5—C6—C7	−50.3 (3)	C40—C35—C36—C37	−49.4 (3)
Cl1—C5—C6—C7	68.5 (3)	Cl2—C35—C36—C37	69.5 (2)
O3—C6—C7—C8	−72.0 (3)	O7—C36—C37—C38	−73.7 (3)
C5—C6—C7—C8	49.8 (3)	C35—C36—C37—C38	48.8 (3)
C6—C7—C8—C14	−176.4 (2)	C36—C37—C38—C44	−176.2 (2)
C6—C7—C8—C9	−54.6 (3)	C36—C37—C38—C39	−54.4 (3)
C14—C8—C9—C11	−51.4 (3)	C44—C38—C39—C41	−50.2 (3)
C7—C8—C9—C11	−174.0 (2)	C37—C38—C39—C41	−173.4 (2)
C14—C8—C9—C10	−179.3 (2)	C44—C38—C39—C40	−178.5 (2)
C7—C8—C9—C10	58.1 (3)	C37—C38—C39—C40	58.3 (3)
C2—C1—C10—C19	−63.3 (3)	C32—C31—C40—C49	−63.4 (3)
C2—C1—C10—C9	175.8 (3)	C32—C31—C40—C35	55.4 (3)
C2—C1—C10—C5	56.2 (3)	C32—C31—C40—C39	175.5 (2)

C8—C9—C10—C19	64.5 (3)	C34—C35—C40—C31	−55.3 (3)
C11—C9—C10—C19	−62.8 (3)	C36—C35—C40—C31	175.8 (2)
C8—C9—C10—C1	−175.6 (2)	Cl2—C35—C40—C31	60.5 (2)
C11—C9—C10—C1	57.1 (3)	C34—C35—C40—C49	62.3 (3)
C8—C9—C10—C5	−56.5 (3)	C36—C35—C40—C49	−66.5 (3)
C11—C9—C10—C5	176.2 (2)	Cl2—C35—C40—C49	178.1 (2)
C4—C5—C10—C19	61.9 (3)	C34—C35—C40—C39	−177.5 (2)
C6—C5—C10—C19	−66.8 (3)	C36—C35—C40—C39	53.6 (3)
Cl1—C5—C10—C19	177.8 (2)	Cl2—C35—C40—C39	−61.7 (2)
C4—C5—C10—C1	−55.9 (3)	C41—C39—C40—C31	55.8 (3)
C6—C5—C10—C1	175.4 (2)	C38—C39—C40—C31	−176.6 (2)
Cl1—C5—C10—C1	60.0 (2)	C41—C39—C40—C49	−64.2 (3)
C4—C5—C10—C9	−177.6 (2)	C38—C39—C40—C49	63.4 (3)
C6—C5—C10—C9	53.7 (3)	C41—C39—C40—C35	175.6 (2)
Cl1—C5—C10—C9	−61.7 (2)	C38—C39—C40—C35	−56.8 (3)
C8—C9—C11—C12	50.6 (3)	C38—C39—C41—C42	49.7 (3)
C10—C9—C11—C12	177.0 (2)	C40—C39—C41—C42	176.1 (2)
C9—C11—C12—C13	−53.7 (3)	C39—C41—C42—C43	−53.2 (3)
C11—C12—C13—C17	171.0 (2)	C41—C42—C43—C47	172.5 (3)
C11—C12—C13—C14	58.0 (3)	C41—C42—C43—C44	58.1 (3)
C11—C12—C13—C18	−68.5 (3)	C41—C42—C43—C48	−68.1 (3)
C7—C8—C14—C15	−55.0 (3)	C37—C38—C44—C43	−179.0 (2)
C9—C8—C14—C15	−178.1 (2)	C39—C38—C44—C43	57.7 (3)
C7—C8—C14—C13	−178.8 (2)	C37—C38—C44—C45	−55.3 (3)
C9—C8—C14—C13	58.2 (3)	C39—C38—C44—C45	−178.6 (2)
C17—C13—C14—C8	173.8 (2)	C42—C43—C44—C38	−62.9 (3)
C12—C13—C14—C8	−62.3 (3)	C47—C43—C44—C38	171.9 (2)
C18—C13—C14—C8	62.8 (3)	C48—C43—C44—C38	61.3 (3)
C17—C13—C14—C15	41.6 (3)	C42—C43—C44—C45	165.4 (2)
C12—C13—C14—C15	165.4 (2)	C47—C43—C44—C45	40.2 (3)
C18—C13—C14—C15	−69.4 (3)	C48—C43—C44—C45	−70.4 (3)
C8—C14—C15—C16	−166.9 (3)	C38—C44—C45—C46	−167.4 (3)
C13—C14—C15—C16	−39.5 (3)	C43—C44—C45—C46	−40.0 (3)
C14—C15—C16—C17	21.2 (3)	C44—C45—C46—C47	23.4 (4)
C12—C13—C17—O4	34.2 (4)	C45—C46—C47—O8	178.9 (4)
C14—C13—C17—O4	151.9 (3)	C45—C46—C47—C43	1.3 (4)
C18—C13—C17—O4	−90.0 (4)	C42—C43—C47—O8	38.3 (5)
C12—C13—C17—C16	−146.0 (3)	C44—C43—C47—O8	157.1 (4)
C14—C13—C17—C16	−28.3 (3)	C48—C43—C47—O8	−84.6 (4)
C18—C13—C17—C16	89.9 (3)	C42—C43—C47—C46	−144.2 (3)
C15—C16—C17—O4	−175.4 (3)	C44—C43—C47—C46	−25.4 (3)
C15—C16—C17—C13	4.7 (4)	C48—C43—C47—C46	92.9 (3)
C3—O1—C20—O2	−8.1 (6)	C33—O5—C50—O6	−6.6 (6)
C3—O1—C20—C21	171.7 (3)	C33—O5—C50—C51	172.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3X···O4^{i}	0.82	1.98	2.745 (3)	155

O7—H7X···O8^{ii}	0.80	2.03	2.791 (3)	160

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

Fig. 1