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3-(3-Chloropropyl)-7,8-dimethoxy-2,3,4,5-tetrahydro-1*H*-3-benzazepin-2-one at 125 K

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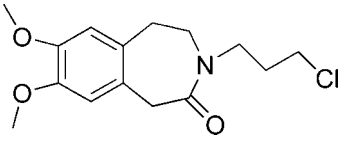
Key indicators: single-crystal X-ray study; T = 125 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.113; data-to-parameter ratio = 13.9.

In the title compound, $C_{15}H_{20}ClNO_3$, the seven-membered ring adopts a distorted boat–sofa conformation; the methylene C atoms of this ring are coplanar with the benzene ring. Both methoxy groups are almost coplanar with the attached benzene ring [C—C—O—C = 6.5 (2) and −13.5 (3)°]. An intramolecular C—H···O hydrogen bond is observed in the molecular structure. In the crystal structure, a C—H···π interaction involving the benzene ring is observed.

Related literature

For details of the synthesis, see: Reiffen *et al.* (1981). For general background, see: Ishihara *et al.* (1994). For a related structure, see: Reiffen *et al.* (1990).



Experimental

Crystal data

$C_{15}H_{20}ClNO_3$
M_r = 297.77
Triclinic, $P\overline{1}$
a = 8.134 (3) Å
b = 8.498 (3) Å
c = 11.701 (4) Å
α = 92.880 (12)°
β = 105.981 (12)°
γ = 106.440 (12)°
V = 738.5 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 125 K
0.29 × 0.28 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.927, T_{max} = 0.944
6809 measured reflections
2542 independent reflections
2137 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.112
S = 1.06
2542 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14A···O3	0.97	2.36	2.768 (2)	104
C1—H1A···Cg1	0.96	2.84	3.705 (3)	150

Cg1 is the centroid of the benzene ring.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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, 2008); 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: CI2575).

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supporting information

Acta Cryst. (2008). E**64**, o802 [doi:10.1107/S1600536808008787]

3-(3-Chloropropyl)-7,8-dimethoxy-2,3,4,5-tetrahydro-1*H*-3-benzazepin-2-one at 125 K

Xiang-Wei Cheng

S1. Comment

Benzazepine derivatives have been of considerable medicinal interest, partly because the skeleton is a component of amaryllydaceae alkaloids such as galanthamine as well as of ribasine alkaloids represented by ribasine (Ishihara *et al.*, 1994). Many benzazepine derivatives have been reported to possess interesting biological activities. The title compound is an important intermediate of ivabradine, which was listed in market in 2006 as the representative of a novel pharmacological class termed specific bradycardic agents. Here the crystal structure of the title compound is reported.

In the title molecule (Fig.1), the seven-membered ring adopts a distorted boat-sofa conformation with a pseudo mirror plane through C7 and the centre of the N1—C10 bond. Atoms C9, C12 and C13 of the seven-membered ring are coplanar with the benzene ring. The dihedral angle between the C3-C9/C12/C13 and C9/C10/N1/C12 planes is 60.61 (8)°. The chloropropyl substituent group is in a (-)-synclinal conformation, as evidenced by the torsion angle N1—C14—C15—C16 of -68.9 (2)°, similar to that in a related structure (-62.63 (2)°, Reiffen *et al.*, 1990). The methoxy groups are almost coplanar with the benzene ring [C8—C3—O1—C1 = 6.5 (2)° and C5—C4—O2—C2 = -13.5 (3)°].

An intramolecular C—H···O hydrogen bond is observed in the molecular structure (Fig.1). In the crystal structure, a C—H···π interaction involving the benzene ring is observed (Table 1).

S2. Experimental

The title compound was prepared according to the literature method (Reiffen *et al.*, 1981). Crystals suitable for X-ray analysis were obtained by slow evaporation of an isopropanol solution at 295 K.

S3. Refinement

H atoms were positioned geometrically (C-H = 0.93-0.97 Å) and refined using a riding model, with U_{iso}(H) = 1.2–1.5U_{eq}(C).

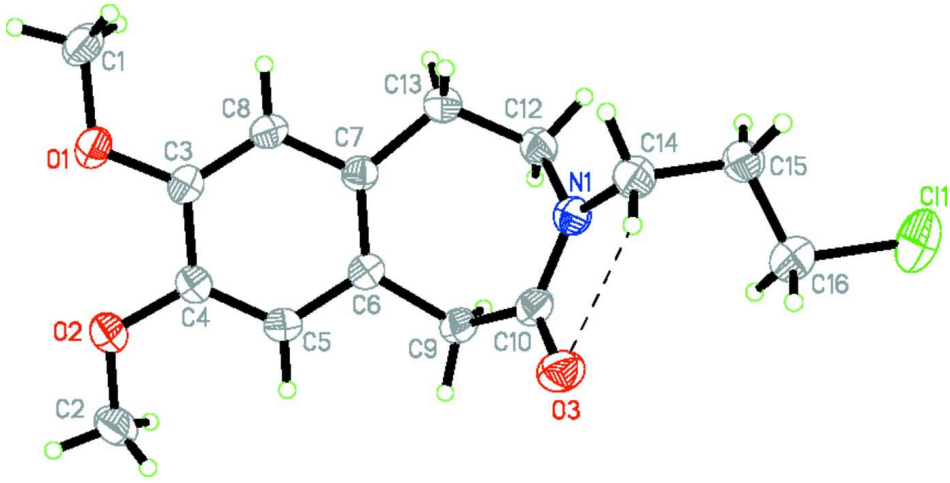

Figure 1

Molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atomic numbering.

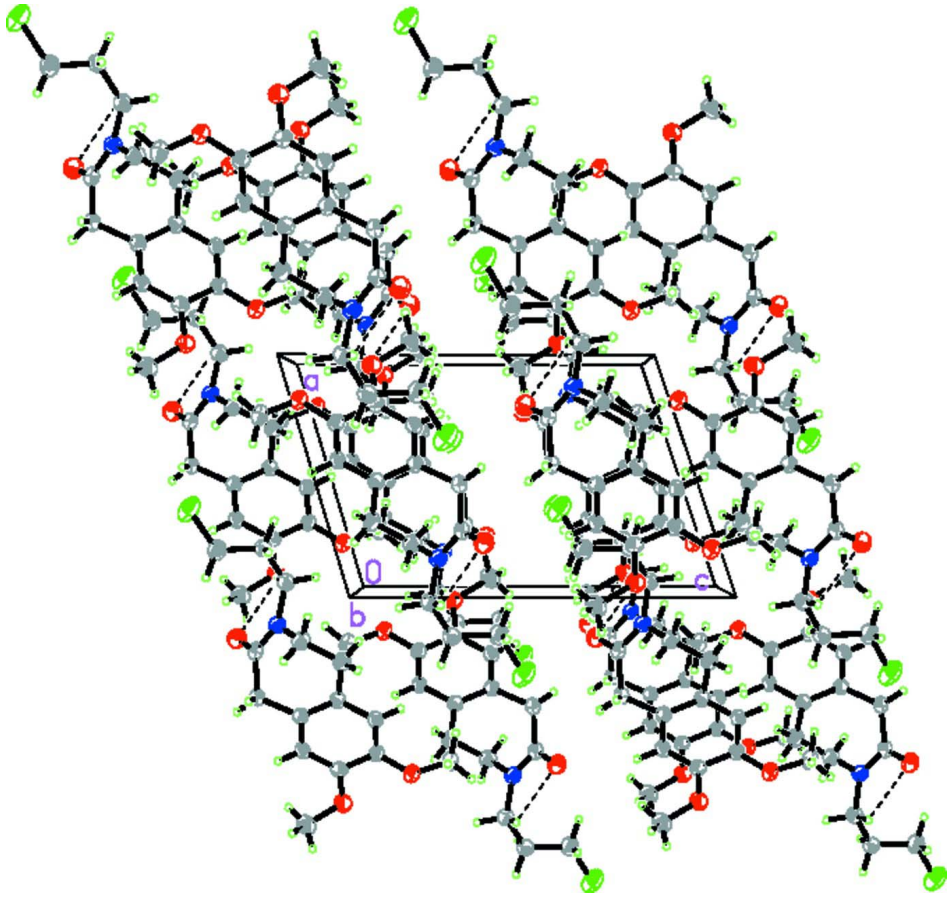

Figure 2

Crystal packing of the title compound, viewed approximately down the *b* axis. Dashed lines indicate intramolecular hydrogen bonds.

3-(3-Chloropropyl)-7,8-dimethoxy-2,3,4,5-tetrahydro-1*H*- 3-benzazepin-2-one

Crystal data

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Triclinic, $P\overline{1}$
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$c = 11.701$ (4) Å
$\alpha = 92.880$ (12)°
$\beta = 105.981$ (12)°
$\gamma = 106.440$ (12)°
$V = 738.5$ (5) Å^3
$Z = 2$
$F(000) = 316$
$D_x = 1.339$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2542 reflections
$\theta = 1.8$–25.0°
$\mu = 0.27$ mm^{-1}
$T = 125$ K
Block, colourless
0.29 × 0.28 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.927$, $T_{max} = 0.944$
6809 measured reflections
2542 independent reflections
2137 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -13 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.112$
$S = 1.06$
2542 reflections
183 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.1232P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

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	0.2530 (3)	0.0704 (3)	1.09762 (17)	0.0633 (5)
H1A	0.3722	0.0674	1.1371	0.095*
H1B	0.1732	0.0182	1.1413	0.095*

H1C	0.2544	0.1834	1.0945	0.095*
C2	−0.0455 (3)	−0.2072 (3)	0.63151 (18)	0.0689 (6)
H2A	−0.0677	−0.1141	0.5942	0.103*
H2B	−0.1580	−0.2894	0.6243	0.103*
H2C	0.0232	−0.2539	0.5927	0.103*
C3	0.2849 (2)	0.0480 (2)	0.90096 (14)	0.0445 (4)
C4	0.2103 (2)	−0.02661 (19)	0.78048 (15)	0.0445 (4)
C5	0.2973 (2)	0.0306 (2)	0.69805 (14)	0.0450 (4)
H5	0.2487	−0.0210	0.6189	0.054*
C6	0.4565 (2)	0.1640 (2)	0.72932 (14)	0.0430 (4)
C7	0.5290 (2)	0.2400 (2)	0.84825 (14)	0.0439 (4)
C8	0.4413 (2)	0.1789 (2)	0.93214 (14)	0.0451 (4)
H8	0.4907	0.2287	1.0117	0.054*
C9	0.5404 (2)	0.2131 (2)	0.62888 (15)	0.0501 (4)
H9A	0.4721	0.1345	0.5568	0.060*
H9B	0.5321	0.3214	0.6114	0.060*
C10	0.7353 (2)	0.2180 (2)	0.66095 (15)	0.0493 (4)
N1	0.85388 (18)	0.33947 (17)	0.74910 (12)	0.0489 (4)
C12	0.7942 (2)	0.4582 (2)	0.80910 (17)	0.0539 (5)
H12A	0.7125	0.4977	0.7491	0.065*
H12B	0.8975	0.5526	0.8506	0.065*
C13	0.7006 (2)	0.3845 (2)	0.89834 (16)	0.0556 (5)
H13A	0.6725	0.4718	0.9383	0.067*
H13B	0.7848	0.3482	0.9589	0.067*
C14	1.0354 (2)	0.3319 (2)	0.80602 (16)	0.0544 (4)
H14A	1.0420	0.2247	0.7789	0.065*
H14B	1.0568	0.3403	0.8922	0.065*
C15	1.1839 (2)	0.4668 (2)	0.77991 (16)	0.0556 (5)
H15A	1.1704	0.5743	0.7980	0.067*
H15B	1.3001	0.4661	0.8307	0.067*
C16	1.1757 (3)	0.4392 (3)	0.65120 (18)	0.0636 (5)
H16A	1.0595	0.4403	0.6005	0.076*
H16B	1.1884	0.3314	0.6332	0.076*
O1	0.19242 (16)	−0.01575 (16)	0.97846 (11)	0.0577 (3)
O2	0.05252 (16)	−0.15427 (16)	0.75527 (11)	0.0600 (4)
O3	0.78269 (18)	0.11586 (17)	0.61105 (12)	0.0660 (4)
Cl1	1.35151 (9)	0.59760 (9)	0.61897 (6)	0.0942 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0704 (12)	0.0768 (13)	0.0502 (11)	0.0225 (10)	0.0293 (9)	0.0132 (9)
C2	0.0645 (12)	0.0601 (11)	0.0607 (12)	0.0006 (9)	0.0060 (9)	−0.0011 (9)
C3	0.0455 (9)	0.0489 (9)	0.0460 (9)	0.0206 (7)	0.0177 (7)	0.0124 (7)
C4	0.0421 (8)	0.0410 (8)	0.0499 (9)	0.0140 (7)	0.0120 (7)	0.0066 (7)
C5	0.0470 (9)	0.0471 (9)	0.0397 (9)	0.0171 (7)	0.0095 (7)	0.0026 (7)
C6	0.0453 (9)	0.0453 (9)	0.0416 (9)	0.0180 (7)	0.0137 (7)	0.0076 (7)
C7	0.0451 (9)	0.0439 (8)	0.0442 (9)	0.0160 (7)	0.0141 (7)	0.0041 (7)

C8	0.0472 (9)	0.0512 (9)	0.0378 (8)	0.0180 (7)	0.0121 (7)	0.0028 (7)
C9	0.0516 (10)	0.0584 (10)	0.0392 (9)	0.0150 (8)	0.0142 (7)	0.0059 (7)
C10	0.0556 (10)	0.0540 (10)	0.0420 (9)	0.0171 (8)	0.0199 (8)	0.0094 (8)
N1	0.0456 (8)	0.0517 (8)	0.0505 (8)	0.0153 (6)	0.0166 (6)	0.0040 (6)
C12	0.0520 (10)	0.0473 (9)	0.0595 (11)	0.0093 (8)	0.0198 (8)	0.0011 (8)
C13	0.0565 (10)	0.0529 (10)	0.0523 (10)	0.0074 (8)	0.0200 (8)	−0.0039 (8)
C14	0.0516 (10)	0.0603 (11)	0.0518 (10)	0.0201 (8)	0.0133 (8)	0.0098 (8)
C15	0.0487 (9)	0.0630 (11)	0.0538 (11)	0.0186 (8)	0.0126 (8)	0.0048 (8)
C16	0.0696 (12)	0.0699 (12)	0.0594 (12)	0.0288 (10)	0.0245 (10)	0.0111 (9)
O1	0.0544 (7)	0.0688 (8)	0.0494 (7)	0.0114 (6)	0.0225 (6)	0.0116 (6)
O2	0.0532 (7)	0.0581 (7)	0.0558 (7)	0.0010 (6)	0.0136 (6)	0.0044 (6)
O3	0.0664 (8)	0.0721 (9)	0.0619 (8)	0.0255 (7)	0.0217 (7)	−0.0058 (7)
Cl1	0.1040 (5)	0.0999 (5)	0.1101 (5)	0.0398 (4)	0.0683 (4)	0.0438 (4)

Geometric parameters (Å, º)

C1—O1	1.428 (2)	C9—H9A	0.97
C1—H1A	0.96	C9—H9B	0.97
C1—H1B	0.96	C10—O3	1.227 (2)
C1—H1C	0.96	C10—N1	1.357 (2)
C2—O2	1.425 (2)	N1—C12	1.463 (2)
C2—H2A	0.96	N1—C14	1.465 (2)
C2—H2B	0.96	C12—C13	1.515 (2)
C2—H2C	0.96	C12—H12A	0.97
C3—O1	1.3697 (19)	C12—H12B	0.97
C3—C8	1.375 (2)	C13—H13A	0.97
C3—C4	1.407 (2)	C13—H13B	0.97
C4—O2	1.372 (2)	C14—C15	1.524 (3)
C4—C5	1.377 (2)	C14—H14A	0.97
C5—C6	1.401 (2)	C14—H14B	0.97
C5—H5	0.93	C15—C16	1.492 (3)
C6—C7	1.394 (2)	C15—H15A	0.97
C6—C9	1.533 (2)	C15—H15B	0.97
C7—C8	1.403 (2)	C16—Cl1	1.805 (2)
C7—C13	1.519 (2)	C16—H16A	0.97
C8—H8	0.93	C16—H16B	0.97
C9—C10	1.513 (2)		

O1—C1—H1A	109.5	O3—C10—C9	121.63 (16)
O1—C1—H1B	109.5	N1—C10—C9	116.40 (15)
H1A—C1—H1B	109.5	C10—N1—C12	121.13 (14)
O1—C1—H1C	109.5	C10—N1—C14	120.32 (15)
H1A—C1—H1C	109.5	C12—N1—C14	117.01 (14)
H1B—C1—H1C	109.5	N1—C12—C13	112.78 (15)
O2—C2—H2A	109.5	N1—C12—H12A	109.0
O2—C2—H2B	109.5	C13—C12—H12A	109.0
H2A—C2—H2B	109.5	N1—C12—H12B	109.0
O2—C2—H2C	109.5	C13—C12—H12B	109.0

H2A—C2—H2C	109.5	H12A—C12—H12B	107.8
H2B—C2—H2C	109.5	C12—C13—C7	116.64 (15)
O1—C3—C8	124.96 (15)	C12—C13—H13A	108.1
O1—C3—C4	116.39 (14)	C7—C13—H13A	108.1
C8—C3—C4	118.64 (15)	C12—C13—H13B	108.1
O2—C4—C5	125.10 (15)	C7—C13—H13B	108.1
O2—C4—C3	115.54 (15)	H13A—C13—H13B	107.3
C5—C4—C3	119.36 (14)	N1—C14—C15	114.14 (15)
C4—C5—C6	122.21 (15)	N1—C14—H14A	108.7
C4—C5—H5	118.9	C15—C14—H14A	108.7
C6—C5—H5	118.9	N1—C14—H14B	108.7
C7—C6—C5	118.52 (15)	C15—C14—H14B	108.7
C7—C6—C9	124.80 (14)	H14A—C14—H14B	107.6
C5—C6—C9	116.68 (14)	C16—C15—C14	110.34 (15)
C6—C7—C8	118.89 (15)	C16—C15—H15A	109.6
C6—C7—C13	125.75 (15)	C14—C15—H15A	109.6
C8—C7—C13	115.34 (14)	C16—C15—H15B	109.6
C3—C8—C7	122.35 (15)	C14—C15—H15B	109.6
C3—C8—H8	118.8	H15A—C15—H15B	108.1
C7—C8—H8	118.8	C15—C16—Cl1	110.88 (14)
C10—C9—C6	112.81 (14)	C15—C16—H16A	109.5
C10—C9—H9A	109.0	Cl1—C16—H16A	109.5
C6—C9—H9A	109.0	C15—C16—H16B	109.5
C10—C9—H9B	109.0	Cl1—C16—H16B	109.5
C6—C9—H9B	109.0	H16A—C16—H16B	108.1
H9A—C9—H9B	107.8	C3—O1—C1	117.37 (14)
O3—C10—N1	121.96 (16)	C4—O2—C2	116.36 (14)

O1—C3—C4—O2	0.3 (2)	C6—C9—C10—N1	−68.2 (2)
C8—C3—C4—O2	−178.75 (15)	O3—C10—N1—C12	−178.83 (16)
O1—C3—C4—C5	−179.49 (14)	C9—C10—N1—C12	0.1 (2)
C8—C3—C4—C5	1.4 (2)	O3—C10—N1—C14	−13.4 (3)
O2—C4—C5—C6	178.70 (15)	C9—C10—N1—C14	165.52 (15)
C3—C4—C5—C6	−1.5 (3)	C10—N1—C12—C13	75.6 (2)
C4—C5—C6—C7	0.3 (3)	C14—N1—C12—C13	−90.34 (18)
C4—C5—C6—C9	179.18 (15)	N1—C12—C13—C7	−61.8 (2)
C5—C6—C7—C8	0.9 (2)	C6—C7—C13—C12	3.9 (3)
C9—C6—C7—C8	−177.88 (15)	C8—C7—C13—C12	−177.43 (15)
C5—C6—C7—C13	179.47 (16)	C10—N1—C14—C15	112.96 (18)
C9—C6—C7—C13	0.7 (3)	C12—N1—C14—C15	−81.02 (19)
O1—C3—C8—C7	−179.23 (15)	N1—C14—C15—C16	−68.9 (2)
C4—C3—C8—C7	−0.3 (2)	C14—C15—C16—Cl1	−179.73 (13)
C6—C7—C8—C3	−0.9 (3)	C8—C3—O1—C1	6.5 (2)
C13—C7—C8—C3	−179.66 (16)	C4—C3—O1—C1	−172.49 (15)
C7—C6—C9—C10	52.6 (2)	C5—C4—O2—C2	−13.5 (3)
C5—C6—C9—C10	−126.15 (16)	C3—C4—O2—C2	166.68 (16)
C6—C9—C10—O3	110.71 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···O3	0.97	2.36	2.768 (2)	104
C1—H1*A*···*Cg*1	0.96	2.84	3.705 (3)	150