IUCrData

ISSN 2414-3146

cis-[3-(2-Chloro-6-methylquinolin-3-yl)oxiran-2-yl]-(*p*-tolyl)methanone

Narasimhamurthy Preveena,[a]* Amar A. Hosamani,[b] Managutti Praveen,[b] Gopalpur Nagendrappa[c] and Tayur N. Guru Row[d]

[a]P.G. Department of Chemistry, Centre for Post Graduation Studies, Jain University, 3rd Block, Jayanagar, Bangalore 560 011, Karnataka, India, [b]Solid state and Structural Chemistry Unit, Indian Institute of Science, C.V. Raman Avenue, Bangalore 560 012, Karnataka, India, [c]P.G. Department of Chemistry, Centre for Post Graduation Studies, Jain, University, 3rd Block, Jayanagar, Bangalore 560 011, Karnataka, India, and [d]Solid state and Structural Chemistry Unit, Indian Institute of Science, C.V., Raman Avenue, Bangalore 560 012, Karnataka, India. *Correspondence e-mail: praveena.molecularconnection@gmail.com

Received 4 February 2017
Accepted 18 March 2017

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

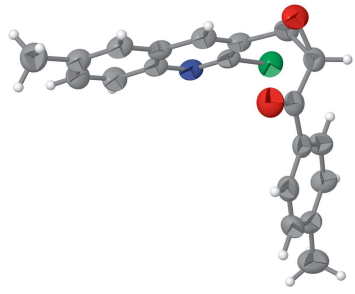
Keywords: crystal structure; synthesis; hydrogen bonding; supramolecular chemistry.

CCDC reference: 1477410

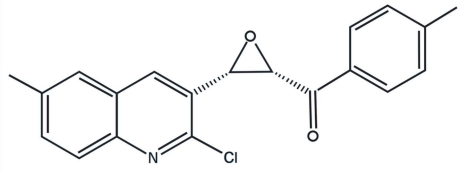
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{20}H_{16}ClNO_2$, the dihedral angle between the quinolyl ring system and the *p*-tolyl ring is 65.80 (7)°. The rings are bridged by a functionalized epoxide system, with the exocyclic bonds in a *cis* configuration. In the crystal, weak C—H···O and C—H···Cl interactions link the molecules into [100] chains.

3D view



Chemical scheme



Structure description

The synthesis and pharmacological properties of quinolinyl epoxy ketones have been reported by us recently (Preveena *et al.*, 2015). 2-Chloroquinoline-3-carbaldehydes have not been exploited for Darzens condensations, a powerful procedure for the formation of a carbon–carbon bond with a simultaneous generation of epoxy function group next to the keto or ester function, except for one report (Boulcina *et al.*, 2008) that describes the synthesis of a few quinolinyl epoxy ketones under stronger conditions and much longer duration, with modest yields of products. The structure of a related quinoline derivative was reported by the same workers (Boulcina *et al.*, 2007). Here we report the synthesis and crystal structure of the title compound (Fig. 1).

The dihedral angle between the quinoline ring system and the *p*-tolyl ring is 65.80 (7)°. The conformation about the epoxide group is *cis*. There are two types of weak intermolecular hydrogen-bonding interactions in the crystal: C5—H5···Cl1 and C12—H12···O2 (Table 1). Together, these lead to [100] chains in the (Fig. 2).

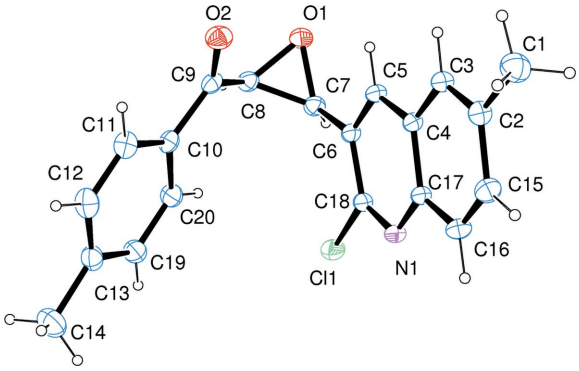


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

Synthesis and crystallization

1.0 g (4.86 mmol) of 2-chloro-6-methylquinoline-3-carbaldehyde was dissolved in DMF (4 ml); to this was added 1.25 g (5.86 mmol) of 2-bromo-1-*p*-tolylethanone and 0.20 g (1.45 mmol) of K_2CO_3 and was stirred at room temperature for about 6 h. The progress of reaction was periodically monitored by TLC. At the end of the reaction, the mixture was added to crushed ice and the precipitate obtained was filtered and purified by column chromatography on silica gel using a petroleum ether–ethyl acetate mixture (96:4) as eluting solvent to obtain the title compound (Preveena *et al.*, 2015) in 88% yield. Colourless blocks were recrystallized from ethyl acetate solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors are thankful to the authorities of Jain University for their encouragement and financial support for this work, and the Department of Solid State Chemistry, Indian Institute of Science, for the facility provided to record crystal data.

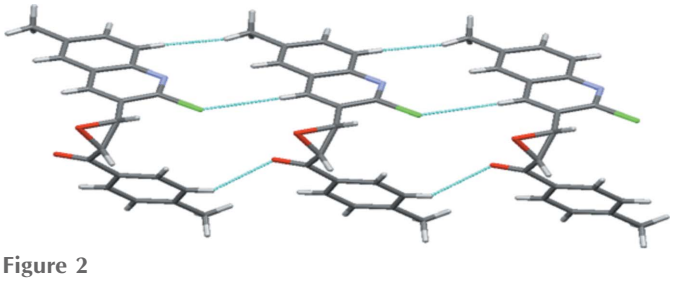


Figure 2
Fragment of the crystal structure of the title compound, showing the formation of the [100] chains.

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···Cl1^{i}	0.93	2.87	3.788 (2)	168
C12—H12···O2^{ii}	0.93	2.63	3.426 (3)	144

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{16}ClNO_2$
M_r	337.79
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	298
a, b, c (Å)	7.412 (2), 10.963 (4), 11.070 (4)
α, β, γ (°)	105.124 (18), 95.633 (18), 104.398 (17)
V (Å^3)	828.2 (5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.24
Crystal size (mm)	0.25 × 0.24 × 0.23
Data collection	
Diffractometer	Bruker *SMART* APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.686, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11538, 2880, 2411
R_{int}	0.045
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.124, 1.13
No. of reflections	2880
No. of parameters	219
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.18

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

References

Boulcina, R., Belfaitah, A., Rhouati, S. & Debache, A. (2008). *J. Soc. Alger. Chim*, **18**, 61–70.
Boulcina, R., Bouacida, S., Roisnel, T. & Debache, A. (2007). *Acta Cryst.* E**63**, o3795–o3796.
Bruker (2012). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Preveena, N., Nagendrappa, G., Suresha Kumara, T. H., Tiwari, A. K., Chaithanya, M. S., Nagananda, G. S., Sujan Ganapathy, P. S., Guru Row, T. N., Amar, A. H. & Chethana, P. R. (2015). *Int. J. Phar. Sci. Inv.* **4**, 53–76.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2017). **2**, x170434 [https://doi.org/10.1107/S2414314617004345]

cis-[3-(2-Chloro-6-methylquinolin-3-yl)oxiran-2-yl](*p*-tolyl)methanone

Narasimhamurthy Preveena, Amar A. Hosamani, Managutti Praveen, Gopalpur Nagendrappa and Tayur N. Guru Row

***cis*-[3-(2-Chloro-6-methylquinolin-3-yl)oxiran-2-yl](*p*-tolyl)methanone**

Crystal data

$C_{20}H_{16}ClNO_2$
M_r = 337.79
Triclinic, $P\overline{1}$
a = 7.412 (2) Å
b = 10.963 (4) Å
c = 11.070 (4) Å
α = 105.124 (18)°
β = 95.633 (18)°
γ = 104.398 (17)°
V = 828.2 (5) Å^3
Z = 2
$F(000)$ = 352
D_x = 1.355 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5696 reflections
θ = 2.3–27.1°
μ = 0.24 mm^{-1}
T = 298 K
Block, clear light colourless
0.25 × 0.24 × 0.23 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: microfocus sealed X-ray tube, Incoatec Iμs
Mirror optics monochromator
Detector resolution: 7.9 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.686, T_{max} = 0.746
11538 measured reflections
2880 independent reflections
2411 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 25.0°, θ_{min} = 1.9°
h = −8→8
k = −13→13
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.124
S = 1.13
2880 reflections
219 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 0.1667P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	−0.14999 (6)	0.54946 (5)	0.24404 (5)	0.0599 (2)
O1	0.38544 (19)	0.45396 (13)	0.34241 (13)	0.0574 (4)
O2	0.58876 (19)	0.71396 (15)	0.45887 (14)	0.0631 (4)
N1	0.0398 (2)	0.66501 (15)	0.10097 (14)	0.0472 (4)
C1	0.6831 (3)	0.8368 (3)	−0.1198 (2)	0.0716 (6)
H1A	0.7900	0.8139	−0.0859	0.107*
H1B	0.7136	0.9308	−0.1020	0.107*
H1C	0.6520	0.7961	−0.2101	0.107*
C2	0.5166 (3)	0.7893 (2)	−0.05940 (18)	0.0526 (5)
C3	0.5226 (3)	0.7133 (2)	0.01956 (18)	0.0499 (5)
H3	0.6322	0.6895	0.0365	0.060*
C4	0.3657 (2)	0.66963 (18)	0.07658 (16)	0.0435 (4)
C5	0.3670 (3)	0.59374 (18)	0.16096 (17)	0.0463 (5)
H5	0.4748	0.5690	0.1809	0.056*
C6	0.2119 (2)	0.55591 (17)	0.21394 (16)	0.0433 (4)
C7	0.2094 (3)	0.47792 (18)	0.30524 (18)	0.0488 (5)
H7	0.0948	0.4052	0.2931	0.059*
C8	0.3164 (3)	0.53792 (19)	0.43619 (18)	0.0478 (4)
H8	0.2644	0.5009	0.5012	0.057*
C9	0.4290 (2)	0.68091 (19)	0.48030 (16)	0.0456 (4)
C10	0.3345 (2)	0.77642 (18)	0.54584 (16)	0.0443 (4)
C11	0.1419 (3)	0.7400 (2)	0.54987 (19)	0.0531 (5)
H11	0.0717	0.6520	0.5161	0.064*
C12	0.0542 (3)	0.8326 (2)	0.6033 (2)	0.0584 (5)
H12	−0.0749	0.8064	0.6043	0.070*
C13	0.1545 (3)	0.9635 (2)	0.65515 (19)	0.0571 (5)
C14	0.0577 (4)	1.0655 (3)	0.7101 (3)	0.0872 (8)
H14A	−0.0707	1.0386	0.6664	0.131*
H14B	0.1231	1.1486	0.7000	0.131*
H14C	0.0588	1.0745	0.7988	0.131*
C15	0.3474 (3)	0.8234 (2)	−0.0848 (2)	0.0593 (5)
H15	0.3415	0.8753	−0.1387	0.071*
C16	0.1942 (3)	0.7824 (2)	−0.0326 (2)	0.0559 (5)
H16	0.0852	0.8062	−0.0516	0.067*
C17	0.1976 (2)	0.70482 (18)	0.04924 (16)	0.0446 (4)
C18	0.0518 (2)	0.59602 (18)	0.17794 (17)	0.0434 (4)
C19	0.3476 (3)	0.9993 (2)	0.6530 (2)	0.0618 (6)
H19	0.4181	1.0870	0.6888	0.074*
C20	0.4360 (3)	0.9079 (2)	0.59925 (18)	0.0545 (5)

H20	0.5652	0.9343	0.5986	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0448 (3)	0.0745 (4)	0.0624 (4)	0.0206 (2)	0.0093 (2)	0.0201 (3)
O1	0.0665 (9)	0.0583 (8)	0.0578 (8)	0.0356 (7)	0.0060 (7)	0.0199 (7)
O2	0.0465 (8)	0.0728 (10)	0.0708 (10)	0.0140 (7)	0.0126 (7)	0.0245 (8)
N1	0.0433 (8)	0.0526 (9)	0.0473 (9)	0.0246 (7)	0.0020 (7)	0.0091 (7)
C1	0.0675 (14)	0.0770 (16)	0.0745 (16)	0.0218 (12)	0.0203 (12)	0.0256 (13)
C2	0.0541 (11)	0.0539 (11)	0.0479 (11)	0.0185 (9)	0.0065 (9)	0.0098 (9)
C3	0.0438 (10)	0.0571 (12)	0.0470 (11)	0.0224 (9)	0.0017 (8)	0.0072 (9)
C4	0.0433 (9)	0.0456 (10)	0.0391 (9)	0.0192 (8)	−0.0001 (7)	0.0046 (8)
C5	0.0452 (10)	0.0510 (11)	0.0445 (10)	0.0253 (8)	−0.0016 (8)	0.0095 (8)
C6	0.0443 (9)	0.0426 (10)	0.0402 (9)	0.0181 (8)	−0.0003 (7)	0.0049 (8)
C7	0.0509 (10)	0.0428 (10)	0.0537 (11)	0.0189 (8)	0.0046 (9)	0.0126 (9)
C8	0.0512 (10)	0.0514 (11)	0.0486 (10)	0.0215 (8)	0.0091 (8)	0.0215 (9)
C9	0.0452 (10)	0.0540 (11)	0.0400 (10)	0.0135 (8)	0.0014 (8)	0.0207 (8)
C10	0.0472 (10)	0.0479 (10)	0.0369 (9)	0.0085 (8)	0.0037 (7)	0.0172 (8)
C11	0.0467 (10)	0.0474 (11)	0.0558 (11)	0.0057 (8)	0.0043 (9)	0.0083 (9)
C12	0.0478 (11)	0.0585 (12)	0.0612 (12)	0.0131 (9)	0.0070 (9)	0.0078 (10)
C13	0.0729 (13)	0.0545 (12)	0.0457 (11)	0.0226 (10)	0.0093 (10)	0.0141 (9)
C14	0.104 (2)	0.0712 (16)	0.0887 (19)	0.0398 (15)	0.0215 (16)	0.0124 (14)
C15	0.0676 (13)	0.0622 (13)	0.0564 (12)	0.0282 (11)	0.0072 (10)	0.0236 (10)
C16	0.0566 (11)	0.0626 (13)	0.0586 (12)	0.0331 (10)	0.0047 (9)	0.0215 (10)
C17	0.0455 (10)	0.0466 (10)	0.0407 (10)	0.0226 (8)	−0.0006 (8)	0.0051 (8)
C18	0.0411 (9)	0.0439 (10)	0.0407 (9)	0.0162 (8)	0.0006 (7)	0.0035 (8)
C19	0.0773 (15)	0.0419 (11)	0.0585 (13)	0.0035 (10)	0.0129 (11)	0.0142 (9)
C20	0.0550 (11)	0.0528 (12)	0.0512 (11)	0.0027 (9)	0.0109 (9)	0.0193 (9)

Geometric parameters (Å, º)

Cl1—C18	1.750 (2)	C8—H8	0.9800
O1—C7	1.436 (2)	C8—C9	1.504 (3)
O1—C8	1.424 (2)	C9—C10	1.477 (3)
O2—C9	1.215 (2)	C10—C11	1.392 (3)
N1—C17	1.376 (2)	C10—C20	1.386 (3)
N1—C18	1.289 (2)	C11—H11	0.9300
C1—H1A	0.9600	C11—C12	1.376 (3)
C1—H1B	0.9600	C12—H12	0.9300
C1—H1C	0.9600	C12—C13	1.377 (3)
C1—C2	1.506 (3)	C13—C14	1.506 (3)
C2—C3	1.361 (3)	C13—C19	1.391 (3)
C2—C15	1.417 (3)	C14—H14A	0.9600
C3—H3	0.9300	C14—H14B	0.9600
C3—C4	1.416 (3)	C14—H14C	0.9600
C4—C5	1.405 (3)	C15—H15	0.9300
C4—C17	1.420 (2)	C15—C16	1.355 (3)

C5—H5	0.9300	C16—H16	0.9300
C5—C6	1.364 (3)	C16—C17	1.398 (3)
C6—C7	1.483 (3)	C19—H19	0.9300
C6—C18	1.421 (2)	C19—C20	1.371 (3)
C7—H7	0.9800	C20—H20	0.9300
C7—C8	1.476 (3)		

C8—O1—C7	62.16 (12)	C10—C9—C8	117.05 (16)
C18—N1—C17	117.07 (15)	C11—C10—C9	121.83 (16)
H1A—C1—H1B	109.5	C20—C10—C9	119.83 (17)
H1A—C1—H1C	109.5	C20—C10—C11	118.24 (18)
H1B—C1—H1C	109.5	C10—C11—H11	119.6
C2—C1—H1A	109.5	C12—C11—C10	120.79 (18)
C2—C1—H1B	109.5	C12—C11—H11	119.6
C2—C1—H1C	109.5	C11—C12—H12	119.5
C3—C2—C1	121.85 (19)	C11—C12—C13	121.02 (19)
C3—C2—C15	118.32 (19)	C13—C12—H12	119.5
C15—C2—C1	119.8 (2)	C12—C13—C14	121.1 (2)
C2—C3—H3	119.2	C12—C13—C19	118.09 (19)
C2—C3—C4	121.51 (17)	C19—C13—C14	120.8 (2)
C4—C3—H3	119.2	C13—C14—H14A	109.5
C3—C4—C17	119.07 (17)	C13—C14—H14B	109.5
C5—C4—C3	123.48 (16)	C13—C14—H14C	109.5
C5—C4—C17	117.45 (17)	H14A—C14—H14B	109.5
C4—C5—H5	119.6	H14A—C14—H14C	109.5
C6—C5—C4	120.71 (16)	H14B—C14—H14C	109.5
C6—C5—H5	119.6	C2—C15—H15	119.2
C5—C6—C7	122.20 (16)	C16—C15—C2	121.7 (2)
C5—C6—C18	116.50 (17)	C16—C15—H15	119.2
C18—C6—C7	121.30 (17)	C15—C16—H16	119.5
O1—C7—C6	115.74 (16)	C15—C16—C17	120.95 (18)
O1—C7—H7	116.2	C17—C16—H16	119.5
O1—C7—C8	58.52 (12)	N1—C17—C4	121.97 (17)
C6—C7—H7	116.2	N1—C17—C16	119.55 (16)
C8—C7—C6	121.17 (16)	C16—C17—C4	118.47 (18)
C8—C7—H7	116.2	N1—C18—Cl1	115.87 (13)
O1—C8—C7	59.32 (12)	N1—C18—C6	126.28 (18)
O1—C8—H8	116.4	C6—C18—Cl1	117.85 (15)
O1—C8—C9	115.69 (16)	C13—C19—H19	119.3
C7—C8—H8	116.4	C20—C19—C13	121.31 (19)
C7—C8—C9	120.34 (16)	C20—C19—H19	119.3
C9—C8—H8	116.4	C10—C20—H20	119.7
O2—C9—C8	120.14 (17)	C19—C20—C10	120.54 (19)
O2—C9—C10	122.79 (17)	C19—C20—H20	119.7

O1—C7—C8—C9	103.70 (19)	C7—C6—C18—N1	179.09 (17)
O1—C8—C9—O2	−16.3 (2)	C7—C8—C9—O2	−84.3 (2)
O1—C8—C9—C10	161.95 (14)	C7—C8—C9—C10	93.9 (2)

O2—C9—C10—C11	168.50 (18)	C8—O1—C7—C6	112.25 (18)
O2—C9—C10—C20	−7.9 (3)	C8—C9—C10—C11	−9.7 (2)
C1—C2—C3—C4	179.65 (18)	C8—C9—C10—C20	173.97 (16)
C1—C2—C15—C16	179.8 (2)	C9—C10—C11—C12	−175.15 (17)
C2—C3—C4—C5	−178.43 (17)	C9—C10—C20—C19	175.67 (17)
C2—C3—C4—C17	0.8 (3)	C10—C11—C12—C13	−0.6 (3)
C2—C15—C16—C17	0.3 (3)	C11—C10—C20—C19	−0.8 (3)
C3—C2—C15—C16	0.0 (3)	C11—C12—C13—C14	178.1 (2)
C3—C4—C5—C6	179.25 (16)	C11—C12—C13—C19	−0.5 (3)
C3—C4—C17—N1	179.65 (16)	C12—C13—C19—C20	1.0 (3)
C3—C4—C17—C16	−0.4 (3)	C13—C19—C20—C10	−0.3 (3)
C4—C5—C6—C7	−178.83 (16)	C14—C13—C19—C20	−177.7 (2)
C4—C5—C6—C18	0.7 (3)	C15—C2—C3—C4	−0.6 (3)
C5—C4—C17—N1	−1.1 (3)	C15—C16—C17—N1	179.83 (18)
C5—C4—C17—C16	178.81 (16)	C15—C16—C17—C4	−0.1 (3)
C5—C6—C7—O1	4.9 (2)	C17—N1—C18—Cl1	178.95 (12)
C5—C6—C7—C8	72.2 (2)	C17—N1—C18—C6	−0.6 (3)
C5—C6—C18—Cl1	−179.95 (13)	C17—C4—C5—C6	0.0 (3)
C5—C6—C18—N1	−0.4 (3)	C18—N1—C17—C4	1.3 (3)
C6—C7—C8—O1	−102.98 (19)	C18—N1—C17—C16	−178.55 (16)
C6—C7—C8—C9	0.7 (3)	C18—C6—C7—O1	−174.58 (15)
C7—O1—C8—C9	−111.49 (18)	C18—C6—C7—C8	−107.3 (2)
C7—C6—C18—Cl1	−0.4 (2)	C20—C10—C11—C12	1.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···Cl1^{i}	0.93	2.87	3.788 (2)	168
C12—H12···O2^{ii}	0.93	2.63	3.426 (3)	144

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