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2-Aminoanilinium 2-chloroacetate

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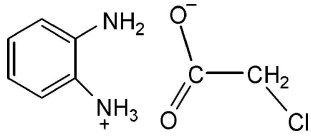
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.137; data-to-parameter ratio = 15.3.

In the crystal structure of the title compound, $C_6H_9N_2^+\cdot ClCH_2COO^-$, prepared by the reaction of OPDA (orthophenelynediamine) with chloroacetic acid, N—H···O hydrogen bonds generate ladder-like chains and very weak intermolecular C—H···Cl hydrogen-bonding interactions between the anions and cations lead to a supramolecular network. C—H···O interactions also occur.

Related literature

For hydrogen bonding with chlorine, see: Brammer *et al.* (2008); Metrangolo *et al.* (2006, 2009). For ladder-like networks, see: Kinbara, Hashimoto *et al.* (1996); Kinbara, Kai *et al.* (1996).



Experimental

Crystal data

$C_6H_9N_2^+\cdot C_2H_2ClO_2^-$
M_r = 202.64
Monoclinic, $P2_1/c$
a = 11.371 (3) Å
b = 4.4852 (11) Å
c = 20.115 (4) Å
β = 110.439 (12)°
V = 961.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 298 K
0.36 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.879, T_{max} = 0.944
9366 measured reflections
1922 independent reflections
1651 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.137
S = 1.09
1922 reflections
126 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O2^{i}	0.90	1.88	2.777 (2)	173
N1—H1B···O2^{ii}	0.94	1.82	2.763 (2)	173
N2—H2B···O1	0.87	2.16	3.004 (3)	163
C4—H4···O1^{iii}	0.93	2.66	3.527 (3)	156
C3—H3···Cl1^{iv}	0.93	3.24	3.985 (3)	138
N2—H2A···N2^{iii}	0.81	2.77	3.587 (4)	179
C8—H8A···Cl1^{v}	0.90 (3)	3.10 (3)	3.878 (3)	146 (3)
C8—H8B···O1^{vi}	0.99 (4)	2.71 (4)	3.491 (4)	136 (3)

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x+1, -y, -z+2$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $x-1, -y-\frac{1}{2}, z-\frac{1}{2}$; (v) $-x+2, -y, -z+2$; (vi) $x, y-1, z$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); 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: DS2035).

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supporting information

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2-Aminoanilinium 2-chloroacetate

A. Srinivasa Rao, Bharat Kumar Tripuramallu, Kishore Ravada and Samar K. Das

S1. Comment

We have reported here the synthesis and structural characterization of a hitherto unknown organic ion pair compound 1, consisting of orthophenylenediammonium cation and chloroacetate anion, that provides a good supramolecular information. The ladder-type one-dimensional chainlike arrangement has been generated because of N—H···O hydrogen bonding interaction in the crystal of compound 1, as shown in Fig. 3.

S2. Experimental

OPDA (Orthophenelynediamine)(0.108 g, 1 mmol) was dissolved in 20 ml of acetonitrile solution and which was added the solution of 25 ml of methanol containing chloroaceticacid (0.23 g, 1 mmol); this reaction mixture was stirred for 5 min and kept for crystalization at room temperature. Colorless needle-like crystals were formed after 3 days (yield: 0.145 g, 72% based on OPDA).

S3. Refinement

All H atoms were found on difference maps, with C—H=0.93 Å and included in the final cycles of refinement using a riding model, with U_{iso}(H)=1.2Ueq(C)

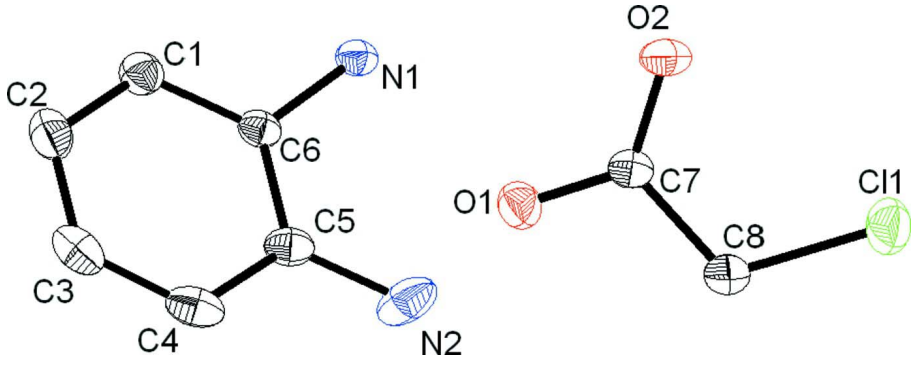


Figure 1

ORTEP diagram of the compound 1 (Thermal ellipsoids are at 50% probability level).

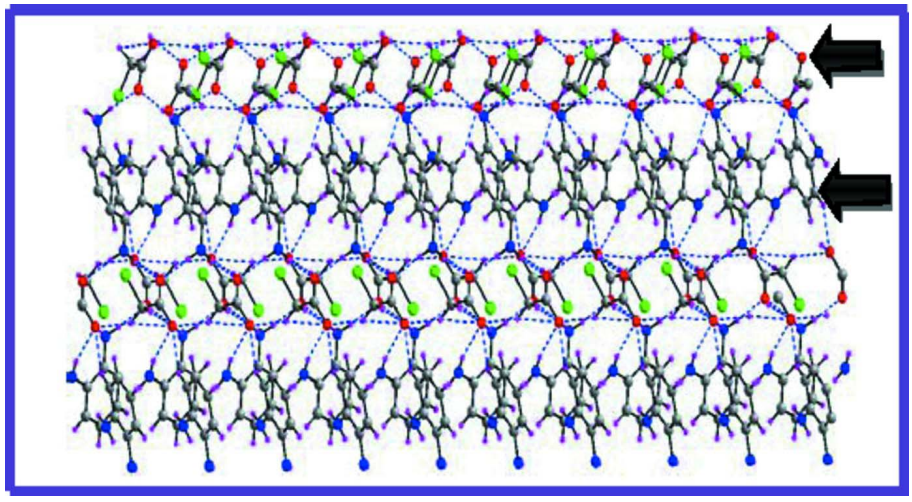


Figure 2

Interactions of C—H···cl in the compound 1 give rise to diverse supramolecular network and all the inetractions arround the cation and anion respectively with symetry codes All the symetry codes for hyderogen bonding were written in the Table 1

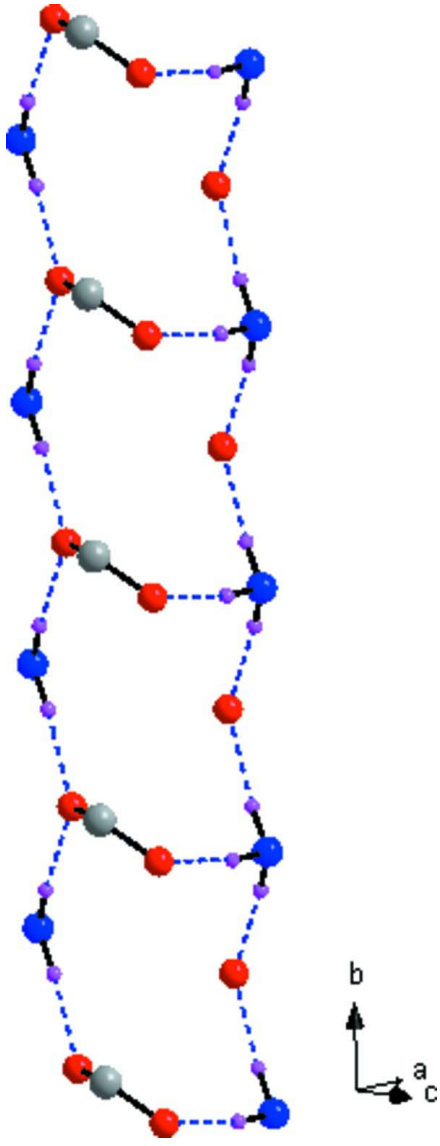


Figure 3

The ladder-type one-dimensional chainlike arrangement generated by N—H···O hydrogen bonding interactions.

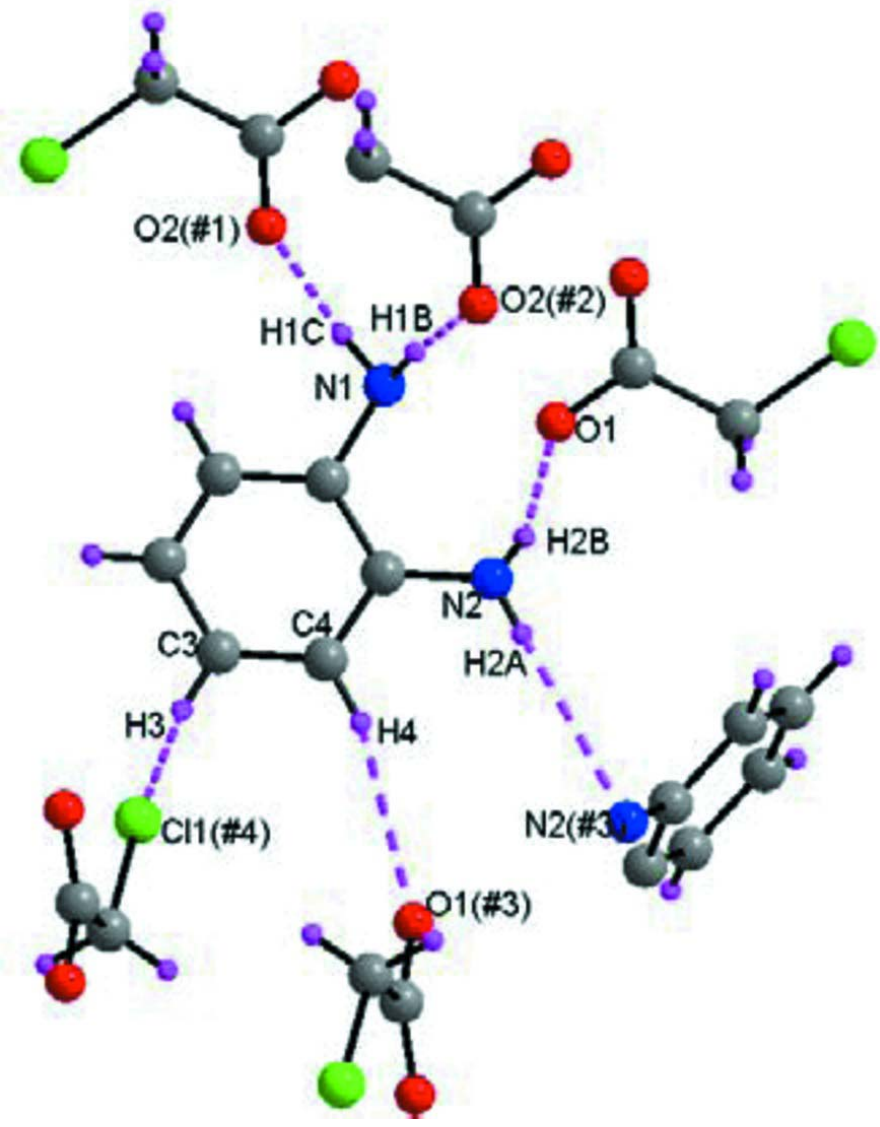


Figure 4
Hydrogen bonding situation around the cation.

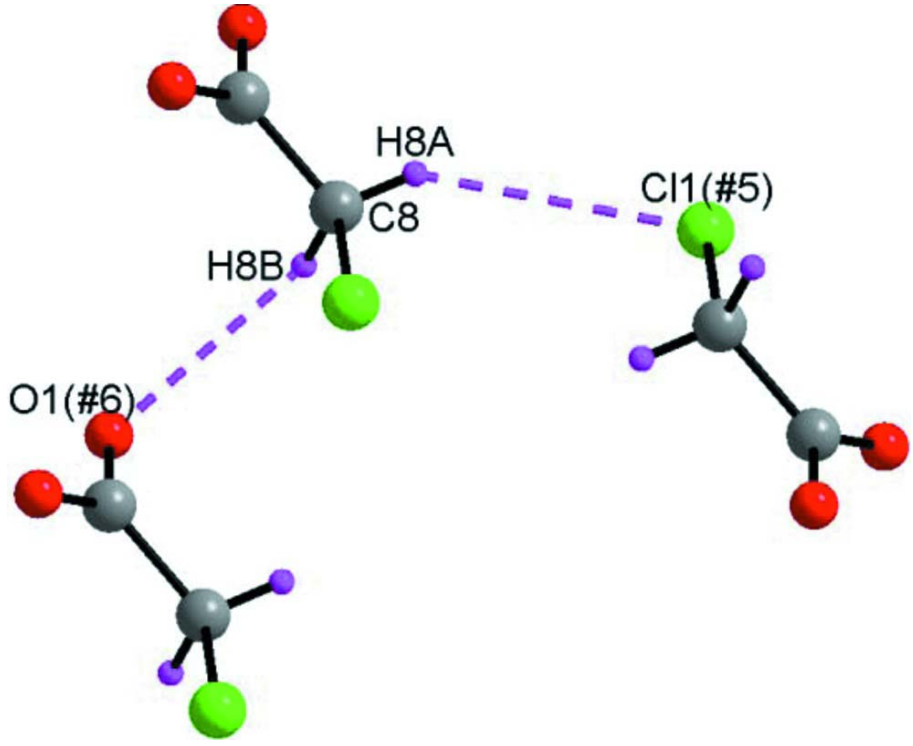
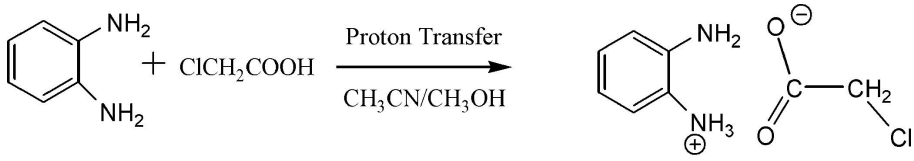


Figure 5

Hydrogen bonding situation around the anion.



Organic Ion-Pair Compound

Figure 6

The formation of the title compound.

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Crystal data

$C_6H_9N_2^+ \cdot C_2H_2ClO_2^-$
$M_r = 202.64$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.371\ (3)$ Å
$b = 4.4852\ (11)$ Å
$c = 20.115\ (4)$ Å
$\beta = 110.439\ (12)°$
$V = 961.3\ (4)$ Å^3
$Z = 4$
$F(000) = 424$
$D_x = 1.400$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5050 reflections
$\theta = 2.3–26.1°$
$\mu = 0.37$ mm^{-1}
$T = 298$ K
Needle, colorless
0.36 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.879, T_{max} = 0.944
9366 measured reflections
1922 independent reflections
1651 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 26.2°, θ_{min} = 1.9°
h = −14→14
k = −5→5
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.137
S = 1.09
1922 reflections
126 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0676P)^2 + 0.3616P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
N1	0.38338 (15)	0.3513 (3)	0.89834 (8)	0.0425 (4)
H1A	0.4696	0.3585	0.9056	0.051*
H1B	0.3687	0.1914	0.9249	0.051*
H1C	0.3625	0.5114	0.9189	0.051*
N2	0.4570 (2)	−0.0303 (6)	0.80725 (13)	0.0819 (7)
H2A	0.4758	−0.1428	0.7809	0.098*
H2B	0.5201	0.0394	0.8428	0.098*
C1	0.1923 (2)	0.4640 (5)	0.79778 (11)	0.0539 (5)
H1	0.1695	0.5954	0.8269	0.065*
C2	0.1128 (2)	0.4187 (6)	0.72902 (12)	0.0641 (6)
H2	0.0360	0.5167	0.7117	0.077*
C3	0.1485 (3)	0.2267 (6)	0.68631 (12)	0.0655 (7)
H3	0.0950	0.1932	0.6400	0.079*
C4	0.2628 (2)	0.0833 (6)	0.71141 (12)	0.0630 (6)
H4	0.2861	−0.0422	0.6813	0.076*

C5	0.3443 (2)	0.1229 (5)	0.78112 (11)	0.0499 (5)
C6	0.30556 (18)	0.3157 (4)	0.82373 (10)	0.0422 (4)
Cl1	0.88856 (6)	−0.20415 (18)	1.05309 (4)	0.0787 (3)
O1	0.63853 (15)	0.3221 (4)	0.92605 (9)	0.0630 (5)
O2	0.65904 (16)	0.1467 (3)	1.03239 (8)	0.0563 (4)
C7	0.69161 (19)	0.1690 (4)	0.97966 (10)	0.0453 (5)
C8	0.8017 (2)	−0.0074 (7)	0.97533 (13)	0.0612 (6)
H8A	0.855 (3)	0.122 (8)	0.9661 (16)	0.090 (10)*
H8B	0.767 (3)	−0.161 (9)	0.938 (2)	0.118 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0520 (9)	0.0381 (8)	0.0424 (8)	−0.0007 (7)	0.0229 (7)	−0.0024 (6)
N2	0.0660 (13)	0.0873 (16)	0.0926 (16)	0.0075 (12)	0.0278 (12)	−0.0425 (13)
C1	0.0615 (13)	0.0508 (12)	0.0534 (12)	0.0014 (10)	0.0250 (10)	0.0040 (9)
C2	0.0615 (13)	0.0702 (15)	0.0563 (13)	0.0000 (12)	0.0150 (11)	0.0151 (12)
C3	0.0715 (15)	0.0782 (16)	0.0434 (12)	−0.0223 (13)	0.0160 (11)	0.0026 (11)
C4	0.0819 (17)	0.0650 (14)	0.0515 (12)	−0.0217 (13)	0.0351 (12)	−0.0159 (11)
C5	0.0571 (12)	0.0476 (11)	0.0528 (11)	−0.0117 (9)	0.0290 (10)	−0.0088 (9)
C6	0.0515 (11)	0.0379 (9)	0.0421 (10)	−0.0073 (8)	0.0228 (8)	0.0004 (7)
Cl1	0.0550 (4)	0.0965 (6)	0.0777 (5)	0.0095 (3)	0.0146 (3)	0.0200 (4)
O1	0.0545 (9)	0.0749 (11)	0.0602 (10)	0.0028 (8)	0.0207 (8)	0.0171 (8)
O2	0.0803 (10)	0.0421 (8)	0.0640 (9)	−0.0001 (7)	0.0472 (8)	−0.0018 (6)
C7	0.0480 (11)	0.0445 (10)	0.0471 (11)	−0.0094 (8)	0.0214 (9)	−0.0046 (8)
C8	0.0584 (13)	0.0771 (17)	0.0549 (13)	0.0100 (12)	0.0285 (11)	0.0064 (12)

Geometric parameters (Å, º)

N1—C6	1.461 (2)	C3—C4	1.378 (4)
N1—H1A	0.9402	C3—H3	0.9300
N1—H1B	0.9425	C4—C5	1.396 (3)
N1—H1C	0.9015	C4—H4	0.9300
N2—C5	1.385 (3)	C5—C6	1.393 (3)
N2—H2A	0.8138	Cl1—C8	1.767 (3)
N2—H2B	0.8747	O1—C7	1.243 (3)
C1—C2	1.378 (3)	O2—C7	1.244 (2)
C1—C6	1.380 (3)	C7—C8	1.509 (3)
C1—H1	0.9300	C8—H8A	0.90 (3)
C2—C3	1.374 (4)	C8—H8B	0.99 (4)
C2—H2	0.9300		
C6—N1—H1A	112.9	C3—C4—C5	121.3 (2)
C6—N1—H1B	109.6	C3—C4—H4	119.4
H1A—N1—H1B	108.6	C5—C4—H4	119.4
C6—N1—H1C	113.4	N2—C5—C6	121.6 (2)
H1A—N1—H1C	109.2	N2—C5—C4	121.3 (2)
H1B—N1—H1C	102.6	C6—C5—C4	117.1 (2)

C5—N2—H2A	118.7	C1—C6—C5	121.37 (19)
C5—N2—H2B	121.3	C1—C6—N1	119.11 (17)
H2A—N2—H2B	115.2	C5—C6—N1	119.45 (18)
C2—C1—C6	120.5 (2)	O1—C7—O2	125.8 (2)
C2—C1—H1	119.8	O1—C7—C8	113.63 (18)
C6—C1—H1	119.8	O2—C7—C8	120.5 (2)
C3—C2—C1	119.1 (2)	C7—C8—Cl1	115.41 (16)
C3—C2—H2	120.4	C7—C8—H8A	107 (2)
C1—C2—H2	120.4	Cl1—C8—H8A	107 (2)
C2—C3—C4	120.7 (2)	C7—C8—H8B	107 (2)
C2—C3—H3	119.7	Cl1—C8—H8B	106 (2)
C4—C3—H3	119.7	H8A—C8—H8B	114 (3)
C6—C1—C2—C3	−0.8 (3)	N2—C5—C6—C1	−178.9 (2)
C1—C2—C3—C4	−0.7 (4)	C4—C5—C6—C1	−0.9 (3)
C2—C3—C4—C5	1.5 (4)	N2—C5—C6—N1	−1.8 (3)
C3—C4—C5—N2	177.4 (2)	C4—C5—C6—N1	176.18 (18)
C3—C4—C5—C6	−0.7 (3)	O1—C7—C8—Cl1	174.88 (19)
C2—C1—C6—C5	1.6 (3)	O2—C7—C8—Cl1	−7.3 (3)
C2—C1—C6—N1	−175.42 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O2^{i}	0.90	1.88	2.777 (2)	173
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C8—H8*B*···O1^{vi}	0.99 (4)	2.71 (4)	3.491 (4)	136 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) −*x*+1, −*y*, −*z*+2; (iii) −*x*+1, *y*−1/2, −*z*+3/2; (iv) *x*−1, −*y*−1/2, *z*−1/2; (v) −*x*+2, −*y*, −*z*+2; (vi) *x*, *y*−1, *z*.