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2,4-Dichloro-*N*-*o*-tolylbenzamide

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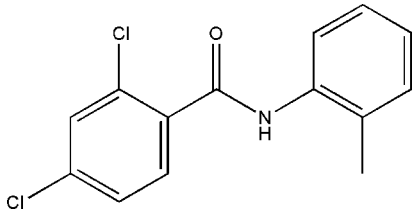
Key indicators: single-crystal X-ray study; T = 92 K; mean σ(C–C) = 0.005 Å; R factor = 0.057; wR factor = 0.158; data-to-parameter ratio = 21.2.

In the title compound, $C_{14}H_{11}Cl_2NO$, the central C—C(O)—N—C amide unit makes dihedral angles of 68.71 (11) and 54.92 (12)°, respectively, with the dichlorobenzene and tolyl rings. The two aromatic rings are inclined at 16.25 (17)°. In the crystal, N—H···O hydrogen bonds link molecules into zigzag chains propagating in [001]. C—H···Cl contacts link these chains and additional C—H···O contacts generate stacks down *b*. Weak C—H···π and C—Cl···π interactions [Cl···centroid distance = 3.5422 (15) Å] may also stabilize the structure.

Related literature

For the biological activity of benzamide derivatives, see: Saeed *et al.* (2008*a*). For related structures, see: Gowda *et al.* (2008); Saeed *et al.* (2008*b*); Zhou & Zheng (2007). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{14}H_{11}Cl_2NO$
M_r = 280.14
Monoclinic, *Cc*
a = 22.517 (4) Å
b = 6.0405 (9) Å
c = 9.6332 (17) Å
β = 104.838 (9)°
V = 1266.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.50 mm^{-1}
T = 92 K
0.46 × 0.27 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
T_{min} = 0.615, T_{max} = 0.91
9889 measured reflections
3475 independent reflections
3195 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.158
S = 1.15
3475 reflections
164 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}
Absolute structure: Flack (1983), 1248 Friedel pairs
Flack parameter: 0.05 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.88	2.04	2.865 (4)	156
C6—H6···O1^{ii}	0.95	2.55	3.415 (4)	151
C11—H11···Cl2^{iii}	0.95	2.83	3.680 (3)	150
C14—H14*C*···*Cg*2^{i}	0.98	2.87	3.528 (4)	125

Symmetry codes: (i) $x, -y+1, z-\frac{1}{2}$; (ii) $x, -y+2, z-\frac{1}{2}$; (iii) $x-\frac{1}{2}, y-\frac{3}{2}, z$. *Cg*2 is the centroid of the C8–C13 ring.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2* and *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) and *TITAN2000* (Hunter & Simpson, 1999); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*, *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2009).

We thank the University of Otago for purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2478).

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supporting information

Acta Cryst. (2009). E**65**, o1642 [doi:10.1107/S1600536809022752]

2,4-Dichloro-*N*-*o*-tolylbenzamide

Aamer Saeed, Rasheed Ahmad Khera, Jim Simpson and Roderick G. Stanley

S1. Comment

The biological activity and applications of benzamide derivatives have been described in an earlier paper (Saeed *et al.* 2008*a*). In the title compound, (I) & Fig. 1, the central C2–C1(O1)–N1–C8 amide unit makes dihedral angles of 68.71 (11) ° and 54.92 (12) ° with the C2···C7 and C8···C13 rings, respectively. The two aromatic rings are inclined at 16.25 (17)°. Bond distances within the molecule are normal (Allen *et al.* 1987) and similar to those observed in comparable structures (Gowda *et al.* 2008; Saeed *et al.* 2008*b*; Zhou & Zheng 2007). In the crystal, N1—H1···O1 hydrogen bonds link molecules into zig-zag chains down the *c* axis; Table 1 & Fig. 2. C11—H11···Cl2 contacts link these chains and additional C6—H6···O1 contacts generate three-dimensional stacks down *b*, Fig. 3. C14—H14···π and Cl1···π interactions (Cl···*Cg*1 distance 3.5422 (15) Å; *Cg*1 is the centroid of the C2···C7 ring) may also stabilize the structure.

S2. Experimental

2,4-Dichlorobenzoyl chloride (5.4 mmol) in $CHCl_3$ was treated with 2-methylaniline (21.6 mmol) under a nitrogen atmosphere at reflux for 3 h. Upon cooling, the reaction mixture was diluted with $CHCl_3$ and washed consecutively with aq. 1 *M* HCl and saturated aq. $NaHCO_3$. The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. Crystallization of the residue in $CHCl_3$ afforded (I) (81%) as colourless crystals: Anal. calcd. for $C_{14}H_{11}Cl_2NO$: C 60.02, H 3.96, N 5.00%; found: C 60.05, H 3.97, N 4.97%.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.95 Å, U_{iso}=1.2U_{eq} (C) for aromatic 0.98 Å, U_{iso} = 1.5U_{eq} (C) for CH_3 atoms and 0.88 Å, U_{iso} = 1.2U_{eq} (N) for the NH group. In the final electron density maps, peaks in excess of 1.0 e Å^{-3} were found approximately 0.8 Å from both Cl atoms but no obvious chemical significance could be attached to them; there was no obvious evidence for disorder involving the Cl atoms.

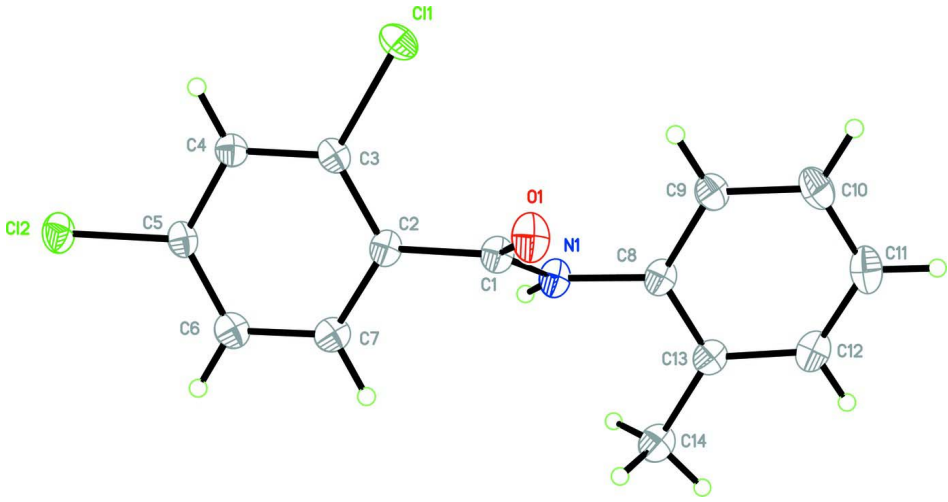

Figure 1

The structure of (I) with displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level.

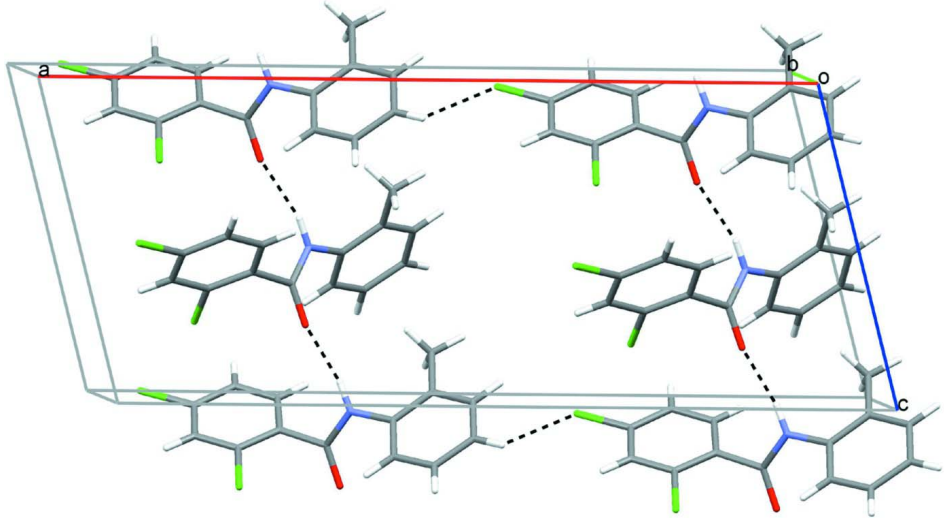

Figure 2

Zig-zag chains of (I) down the *c* axis. Dashed lines indicate N—H···O hydrogen bonds and C—H···Cl contacts.

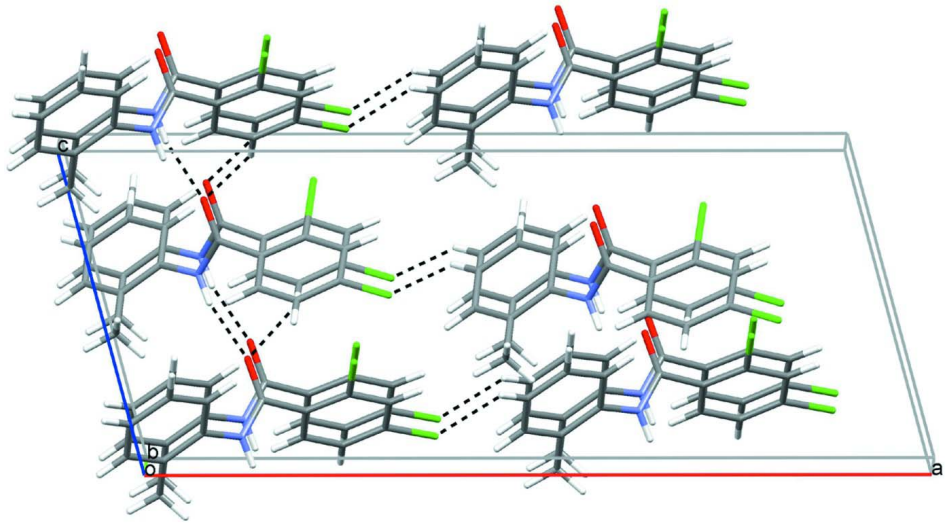

Figure 3

Crystal packing of (I) viewed down the *b* axis. Dashed lines indicate N—H···O hydrogen bonds as well as C—H···O and C—H···Cl contacts.

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Crystal data

$C_{14}H_{11}Cl_2NO$
$M_r = 280.14$
Monoclinic, *Cc*
Hall symbol: C -2yc
$a = 22.517\ (4)$ Å
$b = 6.0405\ (9)$ Å
$c = 9.6332\ (17)$ Å
$\beta = 104.838\ (9)°$
$V = 1266.6\ (4)$ Å^3
$Z = 4$
$F(000) = 576$
$D_x = 1.469$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5253 reflections
$\theta = 2.2–32.4°$
$\mu = 0.50$ mm^{-1}
$T = 92$ K
Block, colourless
0.46 × 0.27 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
$T_{min} = 0.615$, $T_{max} = 0.91$
9889 measured reflections
3475 independent reflections
3195 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 33.1°$, $\theta_{min} = 1.9°$
$h = -34\rightarrow33$
$k = -9\rightarrow8$
$l = -12\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.158$
$S = 1.15$
3475 reflections
164 parameters
2 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

$w = 1/[\sigma^2(F_o^2) + (0.0952P)^2 + 1.0992P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.36$ e Å^{-3}
$\Delta\rho_{min} = -0.62$ e Å^{-3}
Absolute structure: Flack (1983), 1248 Friedel pairs
Absolute structure parameter: 0.05 (8)

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	0.16543 (13)	0.5847 (4)	0.3277 (3)	0.0250 (5)
C1	0.17020 (14)	0.5816 (5)	0.2022 (3)	0.0185 (5)
C2	0.22063 (13)	0.7094 (5)	0.1617 (3)	0.0172 (5)
C3	0.28213 (14)	0.6506 (5)	0.2168 (3)	0.0173 (5)
Cl1	0.30103 (4)	0.41955 (12)	0.32639 (7)	0.02356 (18)
C4	0.32950 (13)	0.7736 (5)	0.1848 (3)	0.0178 (5)
H4	0.3712	0.7311	0.2212	0.021*
C5	0.31386 (14)	0.9607 (5)	0.0978 (3)	0.0184 (5)
Cl2	0.37258 (4)	1.11763 (13)	0.06163 (7)	0.02513 (18)
C6	0.25310 (15)	1.0223 (5)	0.0405 (3)	0.0211 (5)
H6	0.2433	1.1492	−0.0192	0.025*
C7	0.20711 (15)	0.8956 (5)	0.0719 (3)	0.0202 (6)
H7	0.1654	0.9355	0.0319	0.024*
N1	0.13248 (12)	0.4699 (5)	0.0939 (3)	0.0197 (5)
H1	0.1365	0.4894	0.0063	0.024*
C8	0.08611 (13)	0.3210 (5)	0.1147 (3)	0.0191 (5)
C9	0.10239 (14)	0.1560 (5)	0.2194 (3)	0.0218 (6)
H9	0.1435	0.1458	0.2762	0.026*
C10	0.05858 (17)	0.0073 (6)	0.2407 (4)	0.0272 (7)
H10	0.0696	−0.1041	0.3123	0.033*
C11	−0.00132 (17)	0.0223 (6)	0.1568 (4)	0.0291 (7)
H11	−0.0316	−0.0780	0.1719	0.035*
C12	−0.01736 (15)	0.1832 (6)	0.0507 (4)	0.0261 (6)
H12	−0.0584	0.1889	−0.0075	0.031*
C13	0.02600 (14)	0.3385 (5)	0.0276 (3)	0.0203 (5)
C14	0.00831 (15)	0.5163 (6)	−0.0857 (4)	0.0261 (6)
H14A	0.0153	0.6622	−0.0401	0.039*
H14B	−0.0352	0.5007	−0.1358	0.039*
H14C	0.0333	0.5012	−0.1548	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0322 (12)	0.0350 (13)	0.0103 (10)	−0.0082 (9)	0.0100 (9)	−0.0035 (8)
C1	0.0219 (12)	0.0242 (13)	0.0107 (12)	−0.0017 (9)	0.0067 (10)	−0.0010 (9)
C2	0.0202 (11)	0.0246 (13)	0.0082 (10)	−0.0028 (9)	0.0060 (9)	−0.0016 (9)
C3	0.0242 (12)	0.0183 (11)	0.0107 (11)	−0.0014 (9)	0.0067 (9)	−0.0010 (9)
Cl1	0.0313 (4)	0.0213 (3)	0.0185 (3)	0.0008 (2)	0.0070 (3)	0.0049 (2)
C4	0.0207 (13)	0.0207 (12)	0.0125 (12)	−0.0008 (9)	0.0048 (10)	0.0008 (9)
C5	0.0230 (12)	0.0197 (11)	0.0139 (13)	−0.0038 (9)	0.0074 (10)	−0.0004 (9)
Cl2	0.0260 (3)	0.0289 (3)	0.0217 (4)	−0.0064 (3)	0.0084 (3)	0.0042 (3)
C6	0.0262 (13)	0.0243 (13)	0.0137 (12)	−0.0005 (10)	0.0068 (11)	0.0018 (10)
C7	0.0225 (13)	0.0270 (14)	0.0115 (13)	0.0012 (10)	0.0053 (10)	0.0014 (10)
N1	0.0225 (11)	0.0289 (12)	0.0092 (10)	−0.0043 (9)	0.0069 (9)	−0.0008 (9)
C8	0.0207 (12)	0.0266 (13)	0.0114 (12)	−0.0019 (10)	0.0067 (10)	−0.0032 (9)
C9	0.0256 (13)	0.0275 (14)	0.0141 (13)	0.0000 (11)	0.0085 (11)	0.0030 (11)
C10	0.0376 (17)	0.0237 (14)	0.0233 (16)	−0.0026 (12)	0.0134 (14)	0.0027 (11)
C11	0.0348 (17)	0.0288 (15)	0.0274 (18)	−0.0101 (13)	0.0149 (14)	−0.0045 (13)
C12	0.0236 (13)	0.0322 (16)	0.0231 (16)	−0.0039 (11)	0.0070 (12)	−0.0050 (13)
C13	0.0224 (13)	0.0261 (13)	0.0139 (13)	0.0008 (10)	0.0073 (10)	−0.0032 (10)
C14	0.0252 (14)	0.0351 (16)	0.0181 (15)	−0.0001 (12)	0.0060 (12)	0.0004 (12)

Geometric parameters (Å, º)

O1—C1	1.241 (4)	N1—H1	0.8800
C1—N1	1.346 (4)	C8—C9	1.399 (4)
C1—C2	1.505 (4)	C8—C13	1.402 (4)
C2—C3	1.396 (4)	C9—C10	1.388 (5)
C2—C7	1.404 (4)	C9—H9	0.9500
C3—C4	1.397 (4)	C10—C11	1.386 (5)
C3—Cl1	1.736 (3)	C10—H10	0.9500
C4—C5	1.398 (4)	C11—C12	1.389 (5)
C4—H4	0.9500	C11—H11	0.9500
C5—C6	1.388 (4)	C12—C13	1.412 (5)
C5—Cl2	1.733 (3)	C12—H12	0.9500
C6—C7	1.382 (5)	C13—C14	1.510 (5)
C6—H6	0.9500	C14—H14A	0.9800
C7—H7	0.9500	C14—H14B	0.9800
N1—C8	1.431 (4)	C14—H14C	0.9800
O1—C1—N1	124.6 (3)	C9—C8—C13	121.3 (3)
O1—C1—C2	120.3 (3)	C9—C8—N1	118.9 (3)
N1—C1—C2	115.1 (3)	C13—C8—N1	119.7 (3)
C3—C2—C7	118.4 (3)	C10—C9—C8	120.2 (3)
C3—C2—C1	120.8 (3)	C10—C9—H9	119.9
C7—C2—C1	120.8 (3)	C8—C9—H9	119.9
C2—C3—C4	121.3 (3)	C11—C10—C9	119.6 (3)
C2—C3—Cl1	120.0 (2)	C11—C10—H10	120.2

C4—C3—Cl1	118.7 (2)	C9—C10—H10	120.2
C3—C4—C5	118.2 (3)	C10—C11—C12	120.3 (3)
C3—C4—H4	120.9	C10—C11—H11	119.8
C5—C4—H4	120.9	C12—C11—H11	119.8
C6—C5—C4	121.8 (3)	C11—C12—C13	121.4 (3)
C6—C5—Cl2	119.9 (2)	C11—C12—H12	119.3
C4—C5—Cl2	118.3 (2)	C13—C12—H12	119.3
C7—C6—C5	118.8 (3)	C8—C13—C12	117.1 (3)
C7—C6—H6	120.6	C8—C13—C14	121.5 (3)
C5—C6—H6	120.6	C12—C13—C14	121.4 (3)
C6—C7—C2	121.5 (3)	C13—C14—H14A	109.5
C6—C7—H7	119.3	C13—C14—H14B	109.5
C2—C7—H7	119.3	H14A—C14—H14B	109.5
C1—N1—C8	123.1 (3)	C13—C14—H14C	109.5
C1—N1—H1	118.5	H14A—C14—H14C	109.5
C8—N1—H1	118.5	H14B—C14—H14C	109.5

O1—C1—C2—C3	66.1 (4)	C1—C2—C7—C6	176.2 (3)
N1—C1—C2—C3	−114.1 (3)	O1—C1—N1—C8	−7.1 (5)
O1—C1—C2—C7	−111.4 (4)	C2—C1—N1—C8	173.2 (3)
N1—C1—C2—C7	68.4 (4)	C1—N1—C8—C9	−50.9 (4)
C7—C2—C3—C4	0.3 (4)	C1—N1—C8—C13	130.9 (3)
C1—C2—C3—C4	−177.3 (3)	C13—C8—C9—C10	−1.0 (5)
C7—C2—C3—Cl1	−179.2 (2)	N1—C8—C9—C10	−179.1 (3)
C1—C2—C3—Cl1	3.2 (4)	C8—C9—C10—C11	0.4 (5)
C2—C3—C4—C5	1.2 (4)	C9—C10—C11—C12	0.8 (6)
Cl1—C3—C4—C5	−179.3 (2)	C10—C11—C12—C13	−1.6 (6)
C3—C4—C5—C6	−1.7 (5)	C9—C8—C13—C12	0.3 (5)
C3—C4—C5—Cl2	178.4 (2)	N1—C8—C13—C12	178.4 (3)
C4—C5—C6—C7	0.6 (5)	C9—C8—C13—C14	−180.0 (3)
Cl2—C5—C6—C7	−179.4 (2)	N1—C8—C13—C14	−1.8 (4)
C5—C6—C7—C2	1.0 (5)	C11—C12—C13—C8	1.0 (5)
C3—C2—C7—C6	−1.4 (5)	C11—C12—C13—C14	−178.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.88	2.04	2.865 (4)	156
C6—H6···O1^{ii}	0.95	2.55	3.415 (4)	151
C11—H11···Cl2^{iii}	0.95	2.83	3.680 (3)	150
C14—H14*C*···*Cg*2^{i}	0.98	2.87	3.528 (4)	125

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