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2-Chloro-*N*-(2,5-dichlorophenyl)-acetamide

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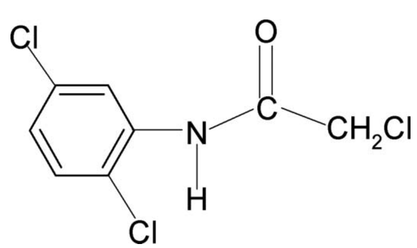
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.098; data-to-parameter ratio = 15.7.

The conformation of the N—H bond in the structure of the title compound, $C_8H_6Cl_3NO$, is *anti* to the C=O bond. The N—H H atom shows close intramolecular N—H···Cl hydrogen bonds with both the ring Cl atom in the *ortho* position and the side-chain Cl atom. The molecules crystallize in planes parallel to (221).

Related literature

For the preparation, see: Shilpa & Gowda (2007); Pies *et al.* (1971). For our work on the effect of ring and side-chain substitutions on the solid-state geometries of aromatic amides, see: Gowda Foro & Fuess (2008); Gowda, Kožíšek *et al.* (2008); Gowda *et al.* (2009).



Experimental

Crystal data

$C_8H_6Cl_3NO$
M_r = 238.49
Triclinic, $P\bar{1}$
a = 7.492 (2) Å
b = 8.496 (2) Å
c = 8.988 (2) Å
α = 69.68 (2)°
β = 67.54 (2)°
γ = 66.67 (2)°
V = 472.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.92 mm^{-1}
T = 299 K
0.38 × 0.28 × 0.22 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.720, T_{max} = 0.823
2735 measured reflections
1914 independent reflections
1359 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.098
S = 1.02
1914 reflections
122 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···Cl3	0.82 (3)	2.43 (3)	2.922 (2)	120 (2)
N1—H1*N*···Cl1	0.82 (3)	2.45 (3)	2.933 (2)	119 (2)

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2962).

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supplementary materials

Acta Cryst. (2009). E**65**, o1445 [**doi:10.1107/S1600536809019898**]

2-Chloro-*N*-(2,5-dichlorophenyl)acetamide

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Comment

As part of a study of the effect of ring and side chain substitutions on the solid state geometries of aromatic amides (Gowda Foro & Fuess, 2008; Gowda, Kožíšek *et al.*, 2008; Gowda *et al.*, 2009), in the present work, the structure of 2-chloro-*N*-(2,5-dichlorophenyl)acetamide (25DCPCA)(I) has been determined. The conformation of the N—H bond in the structure (Fig. 1) is *syn* to the *ortho*-chloro and *anti* to the *meta*-chloro substituents in the aromatic ring, in contrast to the *syn* conformation observed with respect to both the 2-chloro and 3-chloro groups in 2-chloro-*N*-(2,3-dichlorophenyl)acetamide (Gowda, Kožíšek *et al.*, 2008). Furthermore, the conformation of the C=O bond is *anti* to both the N—H bond and side chain Cl atom, compared to the *anti* conformation of the C=O bond with respect to the N–H bond and *syn* with respect to the side chain Cl atom, observed in 2-chloro-*N*-(2,3-dichlorophenyl)-acetamide (Gowda Foro & Fuess, 2008). But the conformations of the N–H bond and the side chain C–H bonds are anti to each other, while those of the ring C–Cl and the side chain C–Cl bonds are *syn* to each other. Further, the N—H H-atom shows simultaneous intramolecular hydrogen bonding with both the ring and side chain Cl atoms. The crystal packing is shown in Fig.2 (Table 1).

Experimental

The title compound was prepared according to the literature method (Shilpa & Gowda, 2007). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared, NMR and NQR spectra (Shilpa & Gowda, 2007; Pies *et al.*, 1971). Single crystals of the title compound used for X-ray diffraction studies were grown by a slow evaporation of its ethanolic solution at room temperature.

Refinement

The N-bound H atom was located in difference map and its positional parameters were refined freely. The other H atoms were positioned with idealized geometry using a riding model [C—H = 0.93–0.97 Å]. All H atoms were refined with isotropic displacement parameters set to 1.2 times of the U_{eq} of the parent atom.

Figures

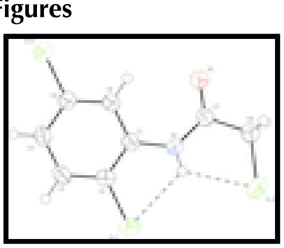

Fig. 1. Molecular structure of the title compound, showing the atom labelling scheme. The displacement ellipsoids are drawn at the 50% probability level.

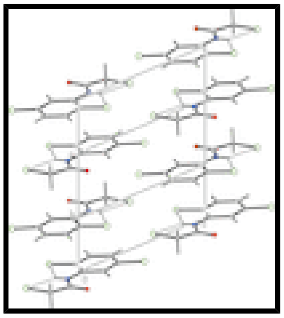

Fig. 2. Crystal packing of the title compound with hydrogen bonding shown as dashed lines.

2-Chloro-*N*-(2,5-dichlorophenyl)acetamide

Crystal data

$C_8H_6Cl_3NO$	$Z = 2$
$M_r = 238.49$	$F_{000} = 240$
Triclinic, $P\bar{1}$	$D_x = 1.677$ Mg m^{-3}
Hall symbol: -P 1	Mo *K*α radiation λ = 0.71073 Å
a = 7.492 (2) Å	Cell parameters from 698 reflections
b = 8.496 (2) Å	θ = 3.2–27.9º
c = 8.988 (2) Å	μ = 0.92 mm^{-1}
α = 69.68 (2)º	*T* = 299 K
β = 67.54 (2)º	Prism, colourless
γ = 66.67 (2)º	0.38 × 0.28 × 0.22 mm
V = 472.4 (2) Å^3	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	1914 independent reflections
Radiation source: fine-focus sealed tube	1359 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.018$
T = 299 K	$\theta_{max} = 26.4$º
Rotation method data acquisition using ω and φ scans	$\theta_{min} = 3.2$º
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	$h = -8 \rightarrow 9$
$T_{min} = 0.720$, $T_{max} = 0.823$	$k = -10 \rightarrow 9$
2735 measured reflections	$l = -10 \rightarrow 11$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.1195P]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.098$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.02$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
1914 reflections	$\Delta\rho_{min} = -0.31$ e Å^{-3}
122 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.043 (4)
Secondary atom site location: difference Fourier map	

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	1.03470 (12)	0.29507 (9)	−0.22746 (8)	0.0527 (2)
Cl2	0.72031 (12)	0.28513 (10)	0.52153 (8)	0.0594 (3)
Cl3	0.68240 (12)	0.81217 (11)	−0.40685 (9)	0.0608 (3)
O1	0.4705 (3)	0.7757 (2)	0.0703 (2)	0.0531 (6)
N1	0.7131 (3)	0.5825 (3)	−0.0831 (3)	0.0375 (5)
H1N	0.772 (4)	0.572 (3)	−0.178 (3)	0.045*
C1	0.7880 (4)	0.4371 (3)	0.0367 (3)	0.0322 (6)
C2	0.9420 (4)	0.2924 (3)	−0.0178 (3)	0.0335 (6)
C3	1.0238 (4)	0.1464 (3)	0.0928 (3)	0.0389 (6)
H3	1.1261	0.0506	0.0548	0.047*
C4	0.9541 (4)	0.1425 (3)	0.2590 (3)	0.0395 (6)
H4	1.0076	0.0443	0.3343	0.047*
C5	0.8036 (4)	0.2866 (3)	0.3124 (3)	0.0376 (6)
C6	0.7181 (4)	0.4339 (3)	0.2045 (3)	0.0370 (6)
H6	0.6158	0.5290	0.2436	0.044*
C7	0.5703 (4)	0.7374 (3)	−0.0617 (3)	0.0343 (6)
C8	0.5319 (4)	0.8743 (3)	−0.2174 (3)	0.0436 (7)
H8A	0.5540	0.9800	−0.2190	0.052*
H8B	0.3905	0.9042	−0.2109	0.052*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0643 (5)	0.0476 (4)	0.0356 (4)	−0.0068 (4)	−0.0059 (3)	−0.0184 (3)

Cl2	0.0708 (5)	0.0553 (5)	0.0319 (4)	0.0043 (4)	−0.0182 (3)	−0.0098 (3)
Cl3	0.0635 (5)	0.0648 (5)	0.0333 (4)	−0.0041 (4)	−0.0140 (3)	−0.0049 (3)
O1	0.0577 (13)	0.0443 (11)	0.0350 (10)	0.0045 (10)	−0.0100 (10)	−0.0104 (9)
N1	0.0410 (13)	0.0352 (12)	0.0268 (11)	−0.0026 (10)	−0.0082 (10)	−0.0081 (9)
C1	0.0309 (13)	0.0310 (13)	0.0336 (13)	−0.0067 (11)	−0.0102 (10)	−0.0079 (10)
C2	0.0337 (13)	0.0358 (14)	0.0321 (13)	−0.0114 (11)	−0.0053 (11)	−0.0126 (11)
C3	0.0341 (14)	0.0336 (14)	0.0468 (16)	−0.0013 (11)	−0.0138 (12)	−0.0146 (12)
C4	0.0402 (15)	0.0333 (14)	0.0431 (15)	−0.0042 (12)	−0.0196 (12)	−0.0060 (11)
C5	0.0394 (15)	0.0384 (14)	0.0327 (13)	−0.0063 (12)	−0.0129 (11)	−0.0090 (11)
C6	0.0356 (14)	0.0358 (14)	0.0338 (13)	−0.0018 (11)	−0.0105 (11)	−0.0111 (11)
C7	0.0334 (14)	0.0313 (13)	0.0352 (14)	−0.0073 (11)	−0.0095 (11)	−0.0077 (11)
C8	0.0405 (15)	0.0421 (15)	0.0369 (15)	−0.0039 (13)	−0.0109 (12)	−0.0062 (12)

Geometric parameters (Å, °)

Cl1—C2	1.737 (2)	C3—C4	1.374 (4)
Cl2—C5	1.737 (3)	C3—H3	0.9300
Cl3—C8	1.771 (3)	C4—C5	1.379 (4)
O1—C7	1.212 (3)	C4—H4	0.9300
N1—C7	1.348 (3)	C5—C6	1.383 (3)
N1—C1	1.410 (3)	C6—H6	0.9300
N1—H1N	0.82 (3)	C7—C8	1.518 (3)
C1—C6	1.389 (3)	C8—H8A	0.9700
C1—C2	1.395 (3)	C8—H8B	0.9700
C2—C3	1.382 (3)		
C7—N1—C1	128.8 (2)	C4—C5—C6	122.2 (2)
C7—N1—H1N	117.0 (19)	C4—C5—Cl2	119.14 (19)
C1—N1—H1N	114.0 (19)	C6—C5—Cl2	118.7 (2)
C6—C1—C2	119.1 (2)	C5—C6—C1	118.8 (2)
C6—C1—N1	122.9 (2)	C5—C6—H6	120.6
C2—C1—N1	117.9 (2)	C1—C6—H6	120.6
C3—C2—C1	120.9 (2)	O1—C7—N1	125.7 (2)
C3—C2—Cl1	119.2 (2)	O1—C7—C8	117.8 (2)
C1—C2—Cl1	119.91 (19)	N1—C7—C8	116.5 (2)
C4—C3—C2	120.1 (2)	C7—C8—Cl3	115.98 (18)
C4—C3—H3	120.0	C7—C8—H8A	108.3
C2—C3—H3	120.0	Cl3—C8—H8A	108.3
C3—C4—C5	119.0 (2)	C7—C8—H8B	108.3
C3—C4—H4	120.5	Cl3—C8—H8B	108.3
C5—C4—H4	120.5	H8A—C8—H8B	107.4
C7—N1—C1—C6	0.5 (4)	C3—C4—C5—Cl2	−178.1 (2)
C7—N1—C1—C2	−178.1 (3)	C4—C5—C6—C1	−0.7 (4)
C6—C1—C2—C3	0.6 (4)	Cl2—C5—C6—C1	178.5 (2)
N1—C1—C2—C3	179.3 (2)	C2—C1—C6—C5	−0.1 (4)
C6—C1—C2—Cl1	−178.90 (19)	N1—C1—C6—C5	−178.7 (2)
N1—C1—C2—Cl1	−0.2 (3)	C1—N1—C7—O1	−4.4 (5)
C1—C2—C3—C4	−0.2 (4)	C1—N1—C7—C8	175.9 (2)
Cl1—C2—C3—C4	179.3 (2)	O1—C7—C8—Cl3	179.7 (2)
C2—C3—C4—C5	−0.6 (4)	N1—C7—C8—Cl3	−0.5 (3)

C3—C4—C5—C6	1.1 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···Cl3	0.82 (3)	2.43 (3)	2.922 (2)	120 (2)
N1—H1N···Cl1	0.82 (3)	2.45 (3)	2.933 (2)	119 (2)

Fig. 1

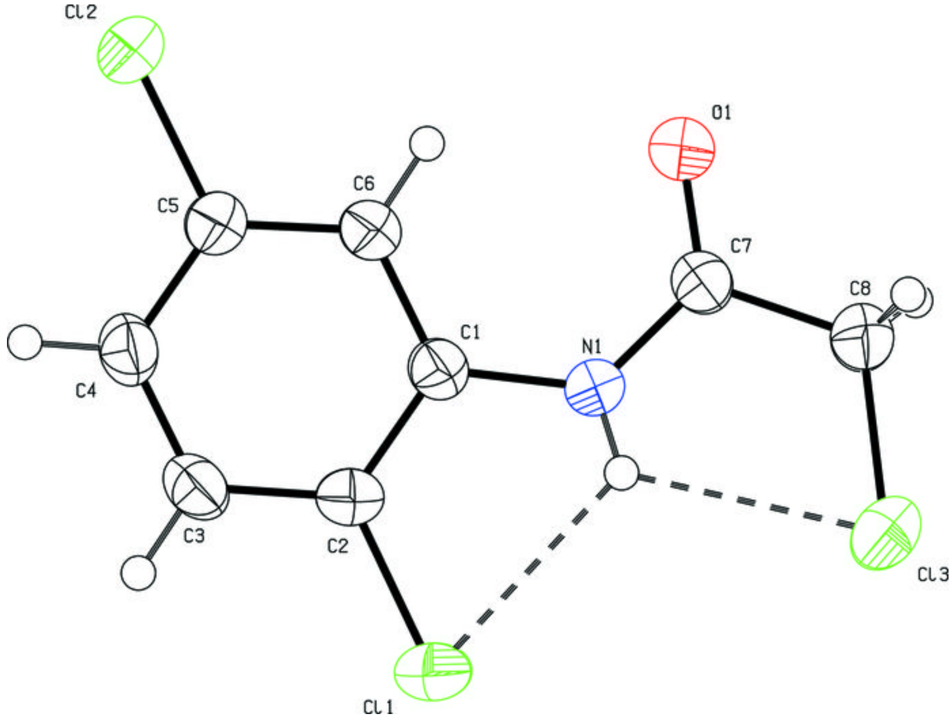

Fig. 2