

2-Chloro-*N*-(2-chlorophenyl)acetamideB. Thimme Gowda,^{a*} Sabine Foro^b and Hartmut Fuess^b

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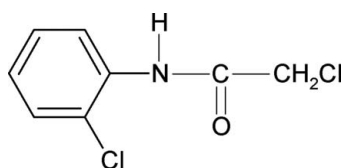
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.011$ Å; R factor = 0.091; wR factor = 0.247; data-to-parameter ratio = 15.8.

The conformation of the N—H bond in the structure of the title compound, $\text{C}_8\text{H}_7\text{Cl}_2\text{NO}$, is *syn* to the *ortho*-Cl substituent of the aromatic ring, similar to that observed in 2-chloro-*N*-(2-nitrophenyl)acetamide, *N*-(2-chlorophenyl)acetamide and *N*-(2-chlorophenyl)-2,2,2-trimethylacetamide. The bond parameters in these compounds have similar values. The molecules are packed into columns and are connected through N—H \cdots O hydrogen bonds.

Related literature

For related literature, see: Gowda *et al.* (2003, 2007, 2007*a,b*).



Experimental

Crystal data

$\text{C}_8\text{H}_7\text{Cl}_2\text{NO}$
 $M_r = 204.05$
Monoclinic, $P2_1/n$
 $a = 4.7699$ (7) Å
 $b = 10.859$ (2) Å
 $c = 16.599$ (3) Å
 $\beta = 91.48$ (2)°

$V = 859.5$ (3) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.70$ mm⁻¹
 $T = 100$ (2) K
 $0.50 \times 0.18 \times 0.12$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford

Diffraction, 2007)
 $T_{\min} = 0.733$, $T_{\max} = 0.881$
4931 measured reflections
1742 independent reflections
1588 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.091$
 $wR(F^2) = 0.247$
 $S = 1.30$
1742 reflections

110 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.86$ e Å⁻³
 $\Delta\rho_{\min} = -0.66$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.88	2.00	2.856 (8)	165

Symmetry code: (i) $x + 1, y, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); 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: BT2553).

References

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supplementary materials

Acta Cryst. (2007). E63, o4611 [doi:10.1107/S1600536807055201]

2-Chloro-*N*-(2-chlorophenyl)acetamide

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Comment

In the present work, as part of a study of the substituent effects on the structures of *N*-aromatic amides, the structure of *N*-(2-chlorophenyl)-2-chloroacetamide has been determined (Gowda *et al.*, 2007; Gowda *et al.*, 2007*a, b*). The conformation of the N—H bond in the structure is *syn* to the *ortho*-Cl substituent in the aniline ring (Fig. 1), similar to that observed in *N*-(2-nitrophenyl)-2-chloroacetamide (Gowda *et al.*, 2007), *N*-(2-chlorophenyl)-acetamide (Gowda *et al.*, 2007*b*) and *N*-(2-chlorophenyl)-2,2,2-trimethylacetamide (Gowda *et al.*, 2007*a*). The packing diagram molecules shows the hydrogen bonds N1—H1N \cdots O1 (Table 1) linking the molecules into chains (Fig. 2).

Experimental

The title compound was prepared according to the literature method (Gowda *et al.*, 2003). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Gowda *et al.*, 2003). Single crystals of the title compound were obtained from an ethanolic solution and used for X-ray diffraction studies at room temperature.

Refinement

The crystal was a non-merohedral twin (twin law: $-1\ 0\ 0/0\ -1\ 0/0.18\ 0\ 1$) and the contribution of the major domain refined to 0.201 (9). All H atoms were positioned geometrically and treated as riding atoms (C—H = 0.95–0.99 Å, N—H = 0.88 Å), with the $U_{\text{iso}}(\text{H})$ values set at 1.2 U_{eq} of the parent atom.

Figures

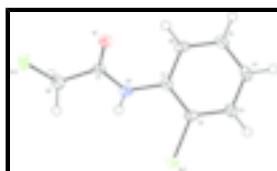


Fig. 1. Molecular structure of the title compound, showing the atom labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radius.

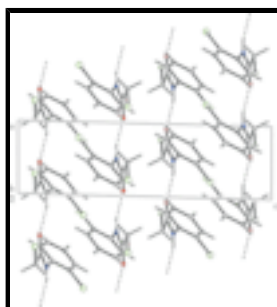


Fig. 2. Molecular packing and hydrogen bonding in the title compound. Hydrogen bonds are shown as dashed lines.

2-Chloro-*N*-(2-chlorophenyl)acetamide

Crystal data

C₈H₇Cl₂NO

$M_r = 204.05$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 4.7699$ (7) Å

$b = 10.859$ (2) Å

$c = 16.599$ (3) Å

$\beta = 91.48$ (2)°

$V = 859.5$ (3) Å³

$Z = 4$

$F_{000} = 416$

$D_x = 1.577$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1759 reflections

$\theta = 2.3$ – 27.3 °

$\mu = 0.70$ mm⁻¹

$T = 100$ (2) K

Needle, colourless

$0.50 \times 0.18 \times 0.12$ mm

Data collection

Oxford Diffraction Xcalibur
diffractometer with Sapphire CCD detector

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100$ (2) K

Rotation method data acquisition using ω and phi
scans.

Absorption correction: multi-scan
(CrysAlis RED; Oxford Diffraction, 2007)

$T_{\min} = 0.733$, $T_{\max} = 0.881$

4931 measured reflections

1742 independent reflections

1588 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\text{max}} = 26.4$ °

$\theta_{\text{min}} = 2.2$ °

$h = -5 \rightarrow 5$

$k = -13 \rightarrow 13$

$l = 0 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.091$

$wR(F^2) = 0.247$

$S = 1.30$

1742 reflections

110 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 13.468P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.86$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.66$ e Å⁻³

Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4183 (16)	-0.0268 (7)	0.3652 (4)	0.0156 (15)
C2	0.5352 (15)	-0.0816 (8)	0.2978 (4)	0.0167 (16)
C3	0.4611 (17)	-0.2001 (8)	0.2746 (5)	0.0203 (17)
H3	0.5443	-0.2368	0.2291	0.024*
C4	0.2656 (17)	-0.2647 (7)	0.3180 (5)	0.0195 (16)
H4	0.2095	-0.3450	0.3015	0.023*
C5	0.1513 (18)	-0.2108 (7)	0.3863 (5)	0.0206 (17)
H5	0.0210	-0.2560	0.4170	0.025*
C6	0.2254 (17)	-0.0925 (7)	0.4099 (4)	0.0188 (16)
H6	0.1454	-0.0566	0.4562	0.023*
C7	0.3208 (15)	0.1831 (7)	0.4107 (4)	0.0137 (15)
C8	0.4655 (17)	0.3052 (7)	0.4315 (5)	0.0210 (17)
H8A	0.6088	0.3230	0.3910	0.025*
H8B	0.5619	0.2981	0.4848	0.025*
N1	0.4995 (13)	0.0955 (6)	0.3881 (4)	0.0185 (14)
H1N	0.6789	0.1142	0.3873	0.022*
O1	0.0686 (11)	0.1699 (5)	0.4149 (3)	0.0190 (12)
Cl1	0.7771 (4)	-0.00167 (17)	0.24183 (10)	0.0177 (5)
Cl2	0.2236 (4)	0.42742 (19)	0.43354 (14)	0.0277 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.013 (4)	0.014 (4)	0.020 (4)	0.001 (3)	-0.003 (3)	0.004 (3)
C2	0.008 (3)	0.024 (4)	0.018 (4)	0.002 (3)	0.002 (3)	0.001 (3)
C3	0.016 (4)	0.018 (4)	0.027 (4)	-0.002 (3)	0.000 (3)	-0.004 (3)
C4	0.021 (4)	0.012 (3)	0.025 (4)	0.000 (3)	-0.005 (3)	0.005 (3)
C5	0.018 (4)	0.019 (4)	0.025 (4)	-0.004 (3)	0.002 (3)	0.003 (3)
C6	0.019 (4)	0.026 (4)	0.011 (3)	0.003 (3)	0.002 (3)	0.004 (3)
C7	0.011 (4)	0.022 (4)	0.007 (3)	-0.003 (3)	0.000 (3)	-0.003 (3)
C8	0.014 (4)	0.017 (4)	0.032 (4)	0.001 (3)	0.000 (3)	0.002 (3)
N1	0.005 (3)	0.025 (4)	0.026 (3)	0.001 (3)	0.001 (2)	-0.002 (3)

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O1	0.008 (3)	0.023 (3)	0.026 (3)	0.001 (2)	0.000 (2)	0.000 (2)
C11	0.0175 (9)	0.0189 (9)	0.0168 (8)	-0.0018 (8)	0.0048 (6)	0.0009 (7)
C12	0.0190 (10)	0.0190 (10)	0.0450 (13)	0.0038 (8)	-0.0022 (9)	-0.0097 (9)

Geometric parameters (\AA , $^\circ$)

C1—C6	1.394 (11)	C5—H5	0.9500
C1—C2	1.396 (11)	C6—H6	0.9500
C1—N1	1.433 (10)	C7—O1	1.216 (9)
C2—C3	1.386 (11)	C7—N1	1.337 (10)
C2—C11	1.732 (8)	C7—C8	1.530 (11)
C3—C4	1.384 (11)	C8—C12	1.759 (8)
C3—H3	0.9500	C8—H8A	0.9900
C4—C5	1.400 (11)	C8—H8B	0.9900
C4—H4	0.9500	N1—H1N	0.8800
C5—C6	1.386 (11)		
C6—C1—C2	119.5 (7)	C5—C6—C1	119.4 (7)
C6—C1—N1	120.7 (7)	C5—C6—H6	120.3
C2—C1—N1	119.8 (7)	C1—C6—H6	120.3
C3—C2—C1	120.9 (7)	O1—C7—N1	124.8 (7)
C3—C2—C11	119.0 (6)	O1—C7—C8	122.0 (7)
C1—C2—C11	120.1 (6)	N1—C7—C8	113.1 (6)
C4—C3—C2	119.8 (8)	C7—C8—C12	111.5 (5)
C4—C3—H3	120.1	C7—C8—H8A	109.3
C2—C3—H3	120.1	C12—C8—H8A	109.3
C3—C4—C5	119.4 (7)	C7—C8—H8B	109.3
C3—C4—H4	120.3	C12—C8—H8B	109.3
C5—C4—H4	120.3	H8A—C8—H8B	108.0
C6—C5—C4	121.0 (7)	C7—N1—C1	124.4 (6)
C6—C5—H5	119.5	C7—N1—H1N	117.8
C4—C5—H5	119.5	C1—N1—H1N	117.8
C6—C1—C2—C3	0.3 (11)	C2—C1—C6—C5	-0.5 (11)
N1—C1—C2—C3	179.7 (7)	N1—C1—C6—C5	-179.9 (7)
C6—C1—C2—C11	-179.6 (6)	O1—C7—C8—C12	-16.4 (9)
N1—C1—C2—C11	-0.2 (10)	N1—C7—C8—C12	163.7 (5)
C1—C2—C3—C4	1.0 (12)	O1—C7—N1—C1	0.4 (12)
C11—C2—C3—C4	-179.2 (6)	C8—C7—N1—C1	-179.7 (7)
C2—C3—C4—C5	-2.0 (12)	C6—C1—N1—C7	-44.3 (11)
C3—C4—C5—C6	1.7 (12)	C2—C1—N1—C7	136.3 (8)
C4—C5—C6—C1	-0.5 (12)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O1 ⁱ	0.88	2.00	2.856 (8)	165

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

Fig. 1

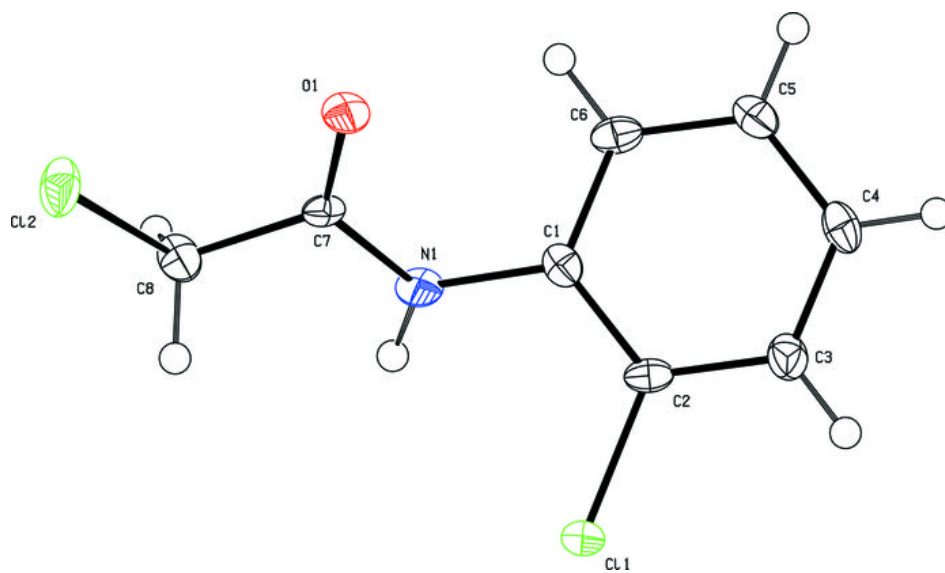


Fig. 2

