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2-Chloro-*N*-(2,3-dichlorophenyl)-acetamide

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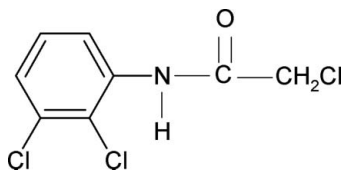
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.080; wR factor = 0.231; data-to-parameter ratio = 13.9.

The conformation of the N—H bond in the title compound (23DCPCA), $\text{C}_8\text{H}_6\text{Cl}_3\text{NO}$, is *syn* to both the 2- and 3-chloro substituents in the aromatic ring, similar to the 2-chloro substituent in 2-chloro-*N*-(2-chlorophenyl)acetamide (2CPCA), the 2- and 3-chloro substituents in *N*-(2,3-dichlorophenyl)acetamide (23DCPA) and in 2,2-dichloro-*N*-(2,3-dichlorophenyl)acetamide (23DCPDCA). The bond parameters in 23DCPCA are similar to those in 2-chloro-*N*-(phenyl)acetamide, 2CPCA, 23DCPA, 23DCPDCA and other acetanilides. The molecules in 23DCPCA are linked into chains through N—H...O hydrogen bonding.

Related literature

For related literature, see: Gowda *et al.* (2007*a,b,c*); Shilpa & Gowda (2007).



Experimental

Crystal data

 $\text{C}_8\text{H}_6\text{Cl}_3\text{NO}$
 $M_r = 238.49$

 Monoclinic, $P2_1/n$
 $a = 11.704$ (3) Å

 $b = 4.712$ (1) Å

 $c = 17.503$ (4) Å

 $\beta = 99.76$ (2)°

 $V = 951.3$ (4) Å³
 $Z = 4$

 Cu $K\alpha$ radiation

 $\mu = 8.38$ mm⁻¹
 $T = 299$ (2) K

 $0.50 \times 0.35 \times 0.28$ mm

Data collection

Enraf–Nonius CAD-4

diffractometer

 Absorption correction: ψ scan

 (North *et al.*, 1968)

 $T_{\min} = 0.014$, $T_{\max} = 0.096$

1832 measured reflections

1692 independent reflections

 1625 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

3 standard reflections

frequency: 120 min

intensity decay: 2.0%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.080$
 $wR(F^2) = 0.231$
 $S = 1.07$

1692 reflections

122 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.87$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.04$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.85 (5)	2.05 (5)	2.862 (4)	161 (4)

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

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); 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: OM2201).

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supplementary materials

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2-Chloro-*N*-(2,3-dichlorophenyl)acetamide

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Comment

In the present work, the structure of 2-chloro-*N*-(2,3-dichlorophenyl)- acetamide (23DCPCA) has been determined to study the effect of substituents on the structures of *N*-aromatic amides (Gowda *et al.*, 2007*a, b, c*). The conformation of the N—H bond in 23DCPCA is *syn* to both the 2-chloro and 3-chloro substituent (Fig. 1), similar to that of 2-chloro substituent in 2-chloro-*N*-(2-chlorophenyl)acetamide (2CPCA)(Gowda *et al.*, 2007*b*), 2- and 3-chloro substituents in *N*-(2,3-dichlorophenyl)-acetamide (23DCPA) (Gowda *et al.*, 2007*a*) and in 2,2-dichloro-*N*-(2,3-dichlorophenyl)acetamide (23DCPDCA)(Gowda *et al.*, 2007*c*). The bond parameters in 23DCPCA are similar to those in 2-chloro-*N*-(phenyl)acetamide, 2CPCA, 23DCPA, 23DCPDCA and other acetanilides (Gowda *et al.*, 2007*a, b, c*). The molecules in the structure of 23DCPCA are stabilized through N—H···O hydrogen bonding (Table 1 and Fig.2).

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 and NMR spectra (Shilpa & Gowda, 2007). Single crystals of the title compound were obtained from an ethanolic solution and used for X-ray diffraction studies at room temperature.

Refinement

The CH atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.97 Å. The NH atom was located in difference map with N—H = 0.85 (5) Å. $U_{\text{iso}}(\text{H})$ values were set equal to 1.2 U_{eq} of the parent atom.

The residual electron-density features are located in the region of C11. The highest peak and deepest hole are 1.04 and 0.80%Å from C11, respectively.

Figures

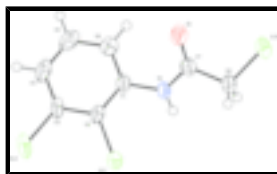


Fig. 1. Molecular structure of the title compound, showing the atom labeling scheme. The displacement ellipsoids are drawn at the 50% probability level.

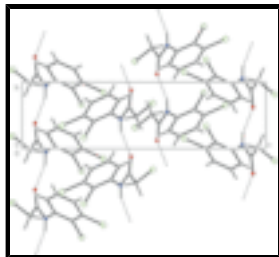


Fig. 2. Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

2-Chloro-N-(2,3-dichlorophenyl)acetamide

Crystal data

$C_8H_6Cl_3NO$

$M_r = 238.49$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 11.704 (3) \text{ \AA}$

$b = 4.712 (1) \text{ \AA}$

$c = 17.503 (4) \text{ \AA}$

$\beta = 99.76 (2)^\circ$

$V = 951.3 (4) \text{ \AA}^3$

$Z = 4$

$F_{000} = 480$

$D_x = 1.665 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation

$\lambda = 1.54180 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 4.3\text{--}23.9^\circ$

$\mu = 8.38 \text{ mm}^{-1}$

$T = 299 (2) \text{ K}$

Prism, colourless

$0.50 \times 0.35 \times 0.28 \text{ mm}$

Data collection

Enraf-Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 299(2) \text{ K}$

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.014$, $T_{\max} = 0.096$

1832 measured reflections

1692 independent reflections

1625 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 66.9^\circ$

$\theta_{\min} = 4.2^\circ$

$h = -13 \rightarrow 1$

$k = -5 \rightarrow 0$

$l = -20 \rightarrow 20$

3 standard reflections

every 120 min

intensity decay: 2.0%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.080$

$wR(F^2) = 0.231$

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.181P)^2 + 0.9328P]$$

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

$(\Delta/\sigma)_{\max} = 0.025$

$S = 1.07$
 1692 reflections
 122 parameters
 Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map

$\Delta\rho_{\max} = 0.87 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.04 \text{ e } \text{\AA}^{-3}$
 Extinction correction: SHELXL,
 $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.018 (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 $F^2 > 2\sigma(F^2)$ 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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4488 (3)	0.5307 (7)	0.36495 (19)	0.0315 (8)
C2	0.4556 (3)	0.4122 (7)	0.2935 (2)	0.0316 (8)
C3	0.5442 (3)	0.4955 (9)	0.2544 (2)	0.0395 (9)
C4	0.6240 (4)	0.6936 (10)	0.2854 (3)	0.0502 (11)
H4	0.6825	0.7486	0.2586	0.060*
C5	0.6172 (4)	0.8099 (9)	0.3558 (3)	0.0502 (11)
H5	0.6714	0.9453	0.3768	0.060*
C6	0.5313 (3)	0.7298 (9)	0.3964 (2)	0.0416 (9)
H6	0.5284	0.8089	0.4447	0.050*
C7	0.2947 (4)	0.6252 (8)	0.4390 (2)	0.0376 (9)
C8	0.1935 (4)	0.4823 (8)	0.4667 (2)	0.0430 (10)
H8A	0.2178	0.2983	0.4884	0.052*
H8B	0.1320	0.4519	0.4228	0.052*
N1	0.3582 (3)	0.4477 (7)	0.40382 (17)	0.0347 (8)
H1N	0.346 (4)	0.271 (11)	0.405 (3)	0.042*
O1	0.3132 (3)	0.8774 (6)	0.4466 (2)	0.0556 (9)
Cl1	0.35541 (7)	0.16288 (19)	0.25447 (5)	0.0389 (5)
Cl2	0.55325 (12)	0.3482 (3)	0.16544 (7)	0.0653 (6)
Cl3	0.14026 (11)	0.6855 (3)	0.53663 (7)	0.0602 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0320 (17)	0.0313 (17)	0.0342 (16)	-0.0005 (13)	0.0143 (13)	0.0009 (13)
C2	0.0245 (16)	0.0345 (17)	0.0383 (18)	-0.0019 (13)	0.0126 (13)	0.0041 (14)

supplementary materials

C3	0.0316 (17)	0.050 (2)	0.0428 (19)	0.0012 (16)	0.0234 (14)	0.0043 (16)
C4	0.034 (2)	0.056 (2)	0.067 (3)	-0.0074 (17)	0.0248 (19)	0.006 (2)
C5	0.033 (2)	0.049 (2)	0.070 (3)	-0.0125 (17)	0.0109 (19)	-0.0012 (19)
C6	0.040 (2)	0.045 (2)	0.042 (2)	-0.0058 (16)	0.0097 (16)	-0.0031 (16)
C7	0.050 (2)	0.0314 (18)	0.0354 (18)	-0.0010 (15)	0.0188 (16)	0.0010 (14)
C8	0.048 (2)	0.040 (2)	0.049 (2)	-0.0055 (16)	0.0304 (17)	-0.0054 (16)
N1	0.0431 (16)	0.0293 (15)	0.0376 (16)	-0.0022 (12)	0.0236 (13)	-0.0005 (12)
O1	0.074 (2)	0.0305 (15)	0.076 (2)	-0.0035 (13)	0.0499 (18)	-0.0054 (13)
Cl1	0.0360 (7)	0.0433 (7)	0.0411 (7)	-0.0057 (3)	0.0170 (4)	-0.0078 (3)
Cl2	0.0670 (9)	0.0878 (10)	0.0529 (8)	-0.0123 (6)	0.0438 (6)	-0.0074 (5)
Cl3	0.0646 (9)	0.0640 (9)	0.0639 (8)	-0.0105 (5)	0.0454 (6)	-0.0179 (5)

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

C1—C2	1.384 (5)	C5—H5	0.9300
C1—C6	1.391 (5)	C6—H6	0.9300
C1—N1	1.409 (4)	C7—O1	1.211 (5)
C2—C3	1.392 (5)	C7—N1	1.337 (5)
C2—Cl1	1.718 (3)	C7—C8	1.512 (5)
C3—C4	1.366 (6)	C8—Cl3	1.750 (4)
C3—Cl2	1.725 (4)	C8—H8A	0.9700
C4—C5	1.363 (7)	C8—H8B	0.9700
C4—H4	0.9300	N1—H1N	0.85 (5)
C5—C6	1.379 (6)		
C2—C1—C6	119.2 (3)	C5—C6—C1	119.9 (4)
C2—C1—N1	119.2 (3)	C5—C6—H6	120.0
C6—C1—N1	121.7 (3)	C1—C6—H6	120.0
C1—C2—C3	119.5 (3)	O1—C7—N1	124.2 (4)
C1—C2—Cl1	119.7 (3)	O1—C7—C8	122.5 (4)
C3—C2—Cl1	120.8 (3)	N1—C7—C8	113.3 (3)
C4—C3—C2	120.9 (4)	C7—C8—Cl3	111.8 (3)
C4—C3—Cl2	119.4 (3)	C7—C8—H8A	109.3
C2—C3—Cl2	119.7 (3)	Cl3—C8—H8A	109.3
C5—C4—C3	119.5 (4)	C7—C8—H8B	109.3
C5—C4—H4	120.2	Cl3—C8—H8B	109.3
C3—C4—H4	120.2	H8A—C8—H8B	107.9
C4—C5—C6	121.0 (4)	C7—N1—C1	124.9 (3)
C4—C5—H5	119.5	C7—N1—H1N	119 (3)
C6—C5—H5	119.5	C1—N1—H1N	116 (3)
C6—C1—C2—C3	-0.3 (5)	C3—C4—C5—C6	0.2 (7)
N1—C1—C2—C3	179.3 (3)	C4—C5—C6—C1	-1.0 (7)
C6—C1—C2—Cl1	179.3 (3)	C2—C1—C6—C5	1.0 (6)
N1—C1—C2—Cl1	-1.1 (5)	N1—C1—C6—C5	-178.6 (4)
C1—C2—C3—C4	-0.4 (6)	O1—C7—C8—Cl3	-20.3 (5)
Cl1—C2—C3—C4	-180.0 (3)	N1—C7—C8—Cl3	161.7 (3)
C1—C2—C3—Cl2	179.9 (3)	O1—C7—N1—C1	-5.7 (6)
Cl1—C2—C3—Cl2	0.3 (5)	C8—C7—N1—C1	172.3 (3)
C2—C3—C4—C5	0.4 (6)	C2—C1—N1—C7	-135.6 (4)
Cl2—C3—C4—C5	-179.9 (3)	C6—C1—N1—C7	44.0 (5)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N···O1 ⁱ	0.85 (5)	2.05 (5)	2.862 (4)	161 (4)

Symmetry codes: (i) *x*, *y*-1, *z*.

Fig. 1

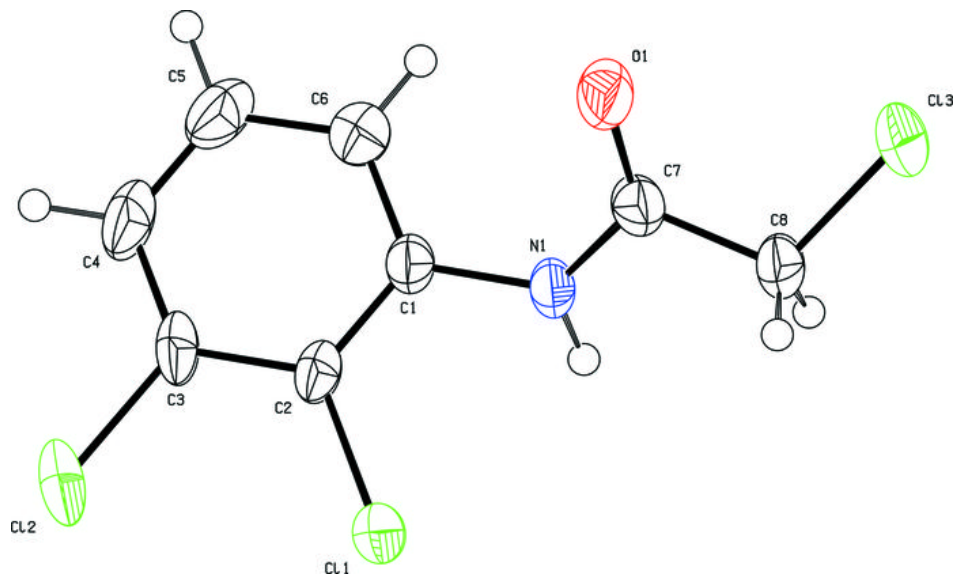


Fig. 2

