

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

 B. Thimme Gowda,^{a*} Sabine Foro^b and Hartmut Fuess^b

^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany

Correspondence e-mail: gowdabt@yahoo.com

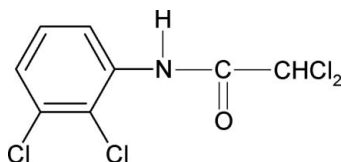
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.046; wR factor = 0.129; data-to-parameter ratio = 15.4.

The conformation of the N–H bond in the title compound, $\text{C}_8\text{H}_5\text{Cl}_4\text{NO}$, is *syn* to the 2-chloro substituent in the aromatic ring, similar to that of the 2-chloro substituent in 2,2-dichloro-*N*-(2-chlorophenyl)acetamide, the 3-chloro substituent in 2,2-dichloro-*N*-(3,4-dichlorophenyl)acetamide and the 2- and 3-chloro substituents in *N*-(2,3-dichlorophenyl)acetamide, but in contrast to the *anti* conformation observed with respect to the 3-chloro substituent in 2,2-dichloro-*N*-(3-chlorophenyl)acetamide. The bond parameters are similar to those in 2,2-dichloro-*N*-phenylacetamide and other acetanilides. The molecules are linked into chains through N–H \cdots O hydrogen bonding.

Related literature

For related literature, see: Gowda *et al.* (2001, 2006, 2007*a,b*); Shilpa & Gowda (2007).



Experimental

Crystal data

 $\text{C}_8\text{H}_5\text{Cl}_4\text{NO}$
 $M_r = 272.93$

 Monoclinic, $P2_1/n$
 $a = 4.674$ (1) Å

 $b = 11.804$ (2) Å

 $c = 19.833$ (3) Å

 $\beta = 95.05$ (1) $^\circ$
 $V = 1090.0$ (3) Å³
 $Z = 4$

 Cu $K\alpha$ radiation

 $\mu = 9.60$ mm⁻¹
 $T = 299$ (2) K

 $0.60 \times 0.08 \times 0.03$ mm

Data collection

Enraf–Nonius CAD-4

diffractometer

 Absorption correction: ψ scan

 (North *et al.*, 1968)

 $T_{\min} = 0.322$, $T_{\max} = 0.750$

2435 measured reflections

1950 independent reflections

 1421 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

3 standard reflections

frequency: 120 min

intensity decay: 1.0%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.129$
 $S = 1.05$

1950 reflections

127 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.44$ e Å⁻³
Table 1

 Hydrogen-bond geometry (Å, $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.86	1.95	2.790 (3)	167

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

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC Software*; data reduction: *REDU4* (Stoe & Cie, 1987); 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: BT2597).

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supplementary materials

Acta Cryst. (2007). E63, o4708 [doi:10.1107/S1600536807057054]

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

B. T. Gowda, S. Foro and H. Fues

Comment

In the present work, the structure of *N*-(2,3-dichlorophenyl)-2,2-dichloroacetamide (23DCPDCA) has been determined to study the substituent effects on the structures of *N*-aromatic amides (Gowda *et al.*, 2001, 2006; 2007*a, b*). The conformation of the N—H bond in 23DCPDCA is *syn* to both the 2-chloro and 3-chloro substituent (Fig. 1), similar to that of 2-chloro substituent in *N*-(2-chlorophenyl)-2,2-dichloroacetamide (2CPDCA)(Gowda *et al.*, 2001), 3-chloro substituent in *N*-(3,4-dichlorophenyl)-2,2-dichloroacetamide (34DCPDCA) (Gowda *et al.*, 2007*b*), and 2- and 3-chloro substituents in *N*-(2,3-dichlorophenyl)-acetamide (23DCPA)(Gowda *et al.*, 2007*a*), but in contrast to the *anti* conformation observed with respect to the 3-chloro substituent in the *N*-(3-chlorophenyl)-2,2-dichloroacetamide (3CPDCA)(Gowda *et al.*, 2006). The bond parameters in 23DCPDCA are similar to those in *N*-(phenyl)-2,2-dichloroacetamide, 2CPDCA, 3CPDCA, 34DCPDCA, 23DCPA and other acetanilides (Gowda *et al.*, 2001, 2006; 2007*a, b*). The molecules in 23DCPDCA are linked into chains through N—H···O hydrogen bonding (Table 1 and Fig.2).

Experimental

The title compound was prepared according to the literature method (Shilpa and 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 and 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 H atoms were positioned with idealized geometry using a riding model with N—H = 0.86 Å and C—H = 0.93–0.98 Å. $U_{\text{iso}}(\text{H})$ values were set equal to 1.2 U_{eq} of the parent atom.

Figures

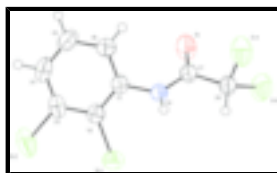


Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

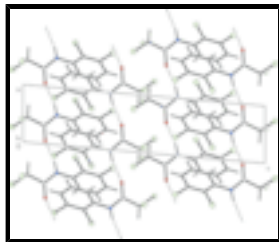


Fig. 2. Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

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

Crystal data

$C_8H_5Cl_4NO$

$M_r = 272.93$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2yn$

$a = 4.674\ (1)\ \text{\AA}$

$b = 11.804\ (2)\ \text{\AA}$

$c = 19.833\ (3)\ \text{\AA}$

$\beta = 95.05\ (1)^\circ$

$V = 1090.0\ (3)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 544$

$D_x = 1.663\ \text{Mg m}^{-3}$

Cu $K\alpha$ radiation

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

Cell parameters from 25 reflections

$\theta = 5.8\text{--}20.3^\circ$

$\mu = 9.60\ \text{mm}^{-1}$

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

Needle, colourless

$0.60 \times 0.08 \times 0.03\ \text{mm}$

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)

$T_{\min} = 0.322$, $T_{\max} = 0.750$

2435 measured reflections

1950 independent reflections

1421 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

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

$\theta_{\min} = 4.4^\circ$

$h = 0 \rightarrow 5$

$k = -14 \rightarrow 2$

$l = -23 \rightarrow 23$

3 standard reflections

every 120 min

intensity decay: 1.0%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.129$

$S = 1.05$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 0.5313P]$$

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

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

1950 reflections $\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$
 127 parameters $\Delta\rho_{\min} = -0.44 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods 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 $F^2 > 2\text{sigma}(F^2)$ 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.8086 (2)	-0.02347 (8)	0.21645 (5)	0.0564 (3)
Cl2	0.6830 (3)	-0.03704 (9)	0.36898 (5)	0.0763 (4)
Cl3	0.4053 (3)	0.33369 (10)	-0.00199 (6)	0.0782 (4)
Cl4	0.2454 (3)	0.10696 (11)	-0.04307 (5)	0.0761 (4)
O1	0.0589 (5)	0.1713 (3)	0.09146 (13)	0.0714 (9)
N1	0.5159 (5)	0.1646 (2)	0.13964 (13)	0.0395 (6)
H1N	0.6933	0.1633	0.1312	0.047*
C1	0.4524 (6)	0.1571 (3)	0.20853 (15)	0.0365 (7)
C2	0.5814 (7)	0.0733 (3)	0.24928 (15)	0.0378 (7)
C3	0.5257 (8)	0.0668 (3)	0.31709 (17)	0.0474 (8)
C4	0.3431 (9)	0.1429 (4)	0.34292 (17)	0.0572 (10)
H4	0.3052	0.1382	0.3881	0.069*
C5	0.2162 (9)	0.2260 (4)	0.30237 (19)	0.0587 (10)
H5	0.0922	0.2773	0.3203	0.070*
C6	0.2705 (8)	0.2346 (3)	0.23480 (18)	0.0478 (8)
H6	0.1856	0.2918	0.2076	0.057*
C7	0.3169 (7)	0.1735 (3)	0.08749 (16)	0.0414 (7)
C8	0.4369 (7)	0.1891 (3)	0.01915 (16)	0.0454 (8)
H8	0.6398	0.1671	0.0227	0.054*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0591 (6)	0.0535 (5)	0.0558 (5)	0.0144 (4)	-0.0003 (4)	-0.0054 (4)
Cl2	0.1161 (10)	0.0611 (6)	0.0478 (5)	-0.0047 (6)	-0.0142 (6)	0.0150 (5)
Cl3	0.1027 (9)	0.0732 (7)	0.0587 (6)	-0.0169 (6)	0.0076 (6)	0.0143 (5)
Cl4	0.0814 (8)	0.0907 (8)	0.0537 (5)	-0.0051 (6)	-0.0080 (5)	-0.0211 (5)
O1	0.0236 (12)	0.143 (3)	0.0475 (14)	-0.0033 (14)	0.0037 (10)	0.0094 (17)

supplementary materials

N1	0.0253 (12)	0.0599 (17)	0.0337 (13)	0.0029 (12)	0.0044 (10)	0.0049 (12)
C1	0.0301 (15)	0.0456 (17)	0.0337 (15)	-0.0059 (13)	0.0026 (12)	0.0014 (13)
C2	0.0359 (16)	0.0404 (16)	0.0366 (16)	-0.0065 (13)	-0.0003 (13)	-0.0046 (13)
C3	0.057 (2)	0.0499 (19)	0.0335 (15)	-0.0115 (17)	-0.0036 (15)	0.0044 (15)
C4	0.066 (2)	0.074 (3)	0.0332 (17)	-0.008 (2)	0.0136 (17)	-0.0058 (17)
C5	0.056 (2)	0.073 (3)	0.049 (2)	0.007 (2)	0.0141 (17)	-0.014 (2)
C6	0.0415 (18)	0.055 (2)	0.0468 (18)	0.0075 (16)	0.0043 (15)	0.0019 (16)
C7	0.0292 (15)	0.059 (2)	0.0363 (16)	0.0010 (14)	0.0048 (12)	0.0036 (15)
C8	0.0332 (16)	0.069 (2)	0.0340 (16)	0.0006 (15)	0.0031 (13)	0.0016 (16)

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

Cl1—C2	1.726 (3)	C2—C3	1.394 (4)
Cl2—C3	1.723 (4)	C3—C4	1.369 (5)
Cl3—C8	1.761 (4)	C4—C5	1.370 (6)
Cl4—C8	1.752 (4)	C4—H4	0.9300
O1—C7	1.216 (4)	C5—C6	1.389 (5)
N1—C7	1.333 (4)	C5—H5	0.9300
N1—C1	1.426 (4)	C6—H6	0.9300
N1—H1N	0.8600	C7—C8	1.523 (4)
C1—C2	1.382 (4)	C8—H8	0.9800
C1—C6	1.382 (5)		
C7—N1—C1	123.9 (3)	C4—C5—C6	120.8 (4)
C7—N1—H1N	118.0	C4—C5—H5	119.6
C1—N1—H1N	118.0	C6—C5—H5	119.6
C2—C1—C6	120.3 (3)	C1—C6—C5	119.1 (3)
C2—C1—N1	119.2 (3)	C1—C6—H6	120.5
C6—C1—N1	120.5 (3)	C5—C6—H6	120.5
C1—C2—C3	119.6 (3)	O1—C7—N1	125.2 (3)
C1—C2—Cl1	120.3 (2)	O1—C7—C8	120.4 (3)
C3—C2—Cl1	120.1 (3)	N1—C7—C8	114.4 (3)
C4—C3—C2	120.0 (3)	C7—C8—Cl4	110.8 (2)
C4—C3—Cl2	119.4 (3)	C7—C8—Cl3	107.4 (2)
C2—C3—Cl2	120.6 (3)	Cl4—C8—Cl3	109.98 (19)
C3—C4—C5	120.1 (3)	C7—C8—H8	109.5
C3—C4—H4	119.9	Cl4—C8—H8	109.5
C5—C4—H4	119.9	Cl3—C8—H8	109.5
C7—N1—C1—C2	131.1 (3)	Cl2—C3—C4—C5	180.0 (3)
C7—N1—C1—C6	-50.7 (5)	C3—C4—C5—C6	-0.1 (6)
C6—C1—C2—C3	0.4 (5)	C2—C1—C6—C5	-0.9 (5)
N1—C1—C2—C3	178.6 (3)	N1—C1—C6—C5	-179.1 (3)
C6—C1—C2—Cl1	-180.0 (3)	C4—C5—C6—C1	0.8 (6)
N1—C1—C2—Cl1	-1.8 (4)	C1—N1—C7—O1	-3.4 (6)
C1—C2—C3—C4	0.3 (5)	C1—N1—C7—C8	175.7 (3)
Cl1—C2—C3—C4	-179.3 (3)	O1—C7—C8—Cl4	-42.7 (4)
C1—C2—C3—Cl2	179.9 (2)	N1—C7—C8—Cl4	138.2 (3)
Cl1—C2—C3—Cl2	0.3 (4)	O1—C7—C8—Cl3	77.5 (4)
C2—C3—C4—C5	-0.4 (6)	N1—C7—C8—Cl3	-101.6 (3)

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O1^i$	0.86	1.95	2.790 (3)	167

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

Fig. 1

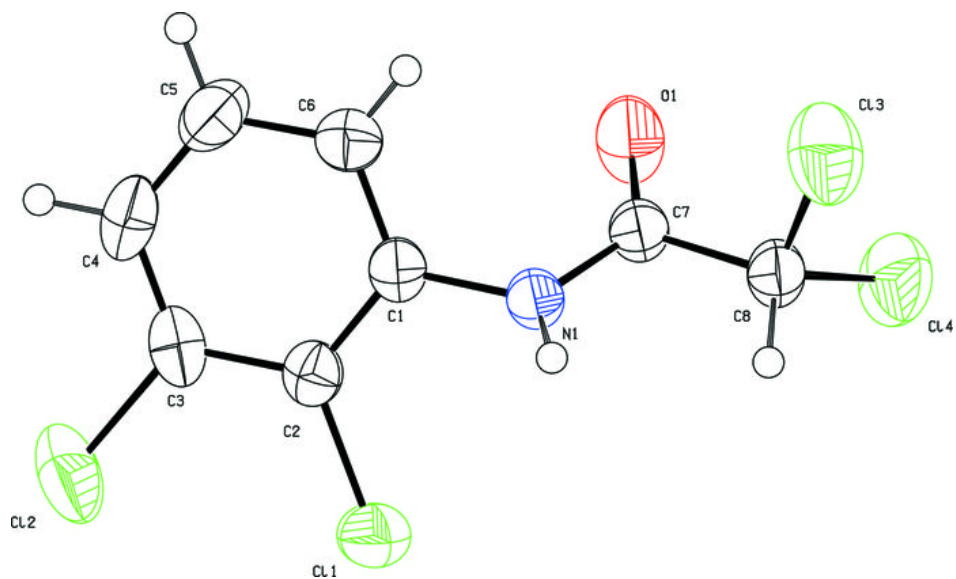


Fig. 2

