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2,2-Dichloro-*N*-(3,4-dichlorophenyl)acetamide

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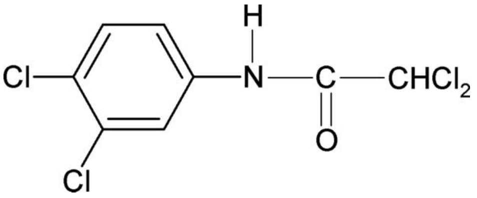
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.106; data-to-parameter ratio = 14.3.

The conformation of the N—H bond in the title compound, $C_8H_5Cl_4NO$, is *syn* to the 3-chloro substituent, in contrast to the *anti* conformation observed in *N*-(3,4-dichlorophenyl)acetamide, 2,2-dichloro-*N*-(3-chlorophenyl)acetamide and 2,2,2-trichloro-*N*-(3,4-dichlorophenyl)acetamide. The bond parameters in the title compound are similar to those in other acetanilides. The molecules are linked into zigzag chains running along the *c* axis through N—H···O hydrogen bonding.

Related literature

For related literature, see: Gowda *et al.* (2006); Gowda, Kozisek, Svoboda & Fuess (2007); Gowda, Kožíšek, Tokarčík & Fuess (2007); Jones *et al.* (1990); Shilpa & Gowda (2007).



Experimental

Crystal data

$C_8H_5Cl_4NO$
M_r = 272.93
Monoclinic, $P2_1/c$
a = 11.898 (5) Å
b = 10.310 (3) Å
c = 9.212 (2) Å
β = 111.23 (3)°
V = 1053.3 (6) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 9.93 mm^{-1}
T = 299 (2) K
0.25 × 0.23 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min}$ = 0.120, $T_{\max}$ = 0.138
2954 measured reflections
1877 independent reflections
1690 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.033
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.106
S = 1.07
1877 reflections
131 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.56 e Å^{-3}
$\Delta\rho_{\min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1$^{\rm i}$	0.78 (3)	2.08 (3)	2.852 (3)	171 (3)

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

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, 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: BT2485).

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supplementary materials

Acta Cryst. (2007). E**63**, o3875 [**doi:10.1107/S1600536807041025**]

2,2-Dichloro-*N*-(3,4-dichlorophenyl)acetamide

B. T. Gowda, **S. Foro** and **H. Fuess**

Comment

In the present work, the structure of *N*-(3,4-dichlorophenyl)-2,2- dichloroacetamide (34DCPDCA) has been determined to study the substituent effects on the structures of *N*-aromatic amides (Gowda *et al.*, 2006; Gowda, Kozisek, Svoboda & Fuess, 2007; Gowda, Kožíšek, Tokarčík & Fuess, 2007). The conformation of the N—H bond in 34DCPDCA is *syn* to the 3-chloro substituent (Fig. 1), compared to the *anti* conformation observed in the chain unsubstituted *N*-(3,4-dichlorophenyl)- acetamide (34DCPA)(Jones *et al.*, 1990), *N*-(3-chlorophenyl)- 2,2-dichloroacetamide (3CPDCA)(Gowda *et al.*, 2006) and *N*-(3,4-dichlorophenyl)-2,2,2-trichloroacetamide (34DCPTCA)(Gowda, Kožíšek, Tokarčík & Fuess, 2007). The bond parameters in 34DCPDCA are similar to those in 34DCPA, 3CPDCA, 34DCPTCA and other acetanilides (Gowda *et al.*, 2006; Gowda, Kozisek, Svoboda & Fuess, 2007; Gowda, Kožíšek, Tokarčík & Fuess, 2007). The molecules in 34DCPDCA are linked into chains *via* N—H···O hydrogen bonding (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 studied at room temperature.

Refinement

The amino H atom was located in difference map and was freely refined. The other H atoms were positioned with idealized geometry using a riding model (C—H = 0.93–0.98 Å). All H atoms bonded to C were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

Figures

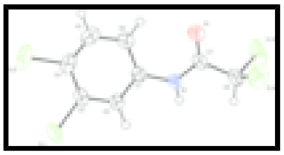

Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

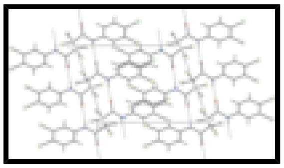

Fig. 2. Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

2,2-Dichloro-*N*-(3,4-dichlorophenyl)acetamide

Crystal data

$C_8H_5Cl_4NO$

$M_r = 272.93$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.898$ (5) Å

$b = 10.310$ (3) Å

$c = 9.212$ (2) Å

$\beta = 111.23$ (3)°

$V = 1053.3$ (6) Å^3

$Z = 4$

$F_{000} = 544$

$D_x = 1.721$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54180$ Å

Cell parameters from 25 reflections

$\theta = 5.7–23.9$°

$\mu = 9.93$ mm^{-1}

$T = 299$ (2) K

Prism, colourless

0.25 × 0.23 × 0.20 mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 299(2)$ K

$\omega/2\theta$ scans

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

$T_{min} = 0.120$, $T_{max} = 0.138$

2954 measured reflections

1877 independent reflections

1690 reflections with $I > 2\sigma(I)$

$R_{int} = 0.033$

$\theta_{max} = 67.0$°

$\theta_{min} = 4.0$°

$h = -14 \rightarrow 14$

$k = -12 \rightarrow 0$

$l = -10 \rightarrow 4$

3 standard reflections

every 120 min

intensity decay: 1.0%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.106$

$S = 1.07$

1877 reflections

131 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of
independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.967P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.56$ e Å^{-3}

$\Delta\rho_{min} = -0.37$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 1997),
$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0037 (5)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3114 (2)	0.6392 (2)	0.1238 (3)	0.0323 (5)
C2	0.3922 (2)	0.6851 (3)	0.0593 (3)	0.0357 (5)
H2	0.3670	0.7446	−0.0220	0.043*
C3	0.5100 (2)	0.6426 (3)	0.1154 (3)	0.0388 (6)
C4	0.5474 (2)	0.5531 (3)	0.2341 (3)	0.0441 (6)
C5	0.4653 (3)	0.5052 (3)	0.2945 (3)	0.0467 (7)
H5	0.4901	0.4433	0.3732	0.056*
C6	0.3476 (2)	0.5471 (3)	0.2409 (3)	0.0400 (6)
H6	0.2932	0.5140	0.2828	0.048*
C7	0.1198 (2)	0.7060 (2)	0.1425 (3)	0.0330 (5)
C8	0.0002 (2)	0.7693 (3)	0.0475 (3)	0.0403 (6)
H8	0.0030	0.8002	−0.0517	0.048*
N1	0.19265 (18)	0.6887 (2)	0.0630 (2)	0.0339 (5)
H1N	0.174 (3)	0.720 (3)	−0.020 (4)	0.041*
O1	0.14306 (18)	0.6745 (2)	0.2774 (2)	0.0470 (5)
Cl1	0.60911 (7)	0.70548 (8)	0.03597 (11)	0.0602 (3)
Cl2	0.69516 (7)	0.50195 (9)	0.30933 (12)	0.0738 (3)
Cl3	−0.11360 (7)	0.65230 (11)	0.01221 (13)	0.0785 (3)
Cl4	−0.02442 (9)	0.90102 (9)	0.15321 (10)	0.0679 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0321 (12)	0.0373 (13)	0.0287 (11)	−0.0023 (10)	0.0124 (9)	−0.0040 (9)
C2	0.0330 (13)	0.0393 (13)	0.0371 (12)	0.0025 (10)	0.0155 (10)	0.0017 (10)
C3	0.0327 (13)	0.0401 (14)	0.0477 (14)	−0.0009 (10)	0.0195 (11)	−0.0059 (11)
C4	0.0361 (13)	0.0401 (14)	0.0497 (15)	0.0074 (11)	0.0079 (11)	−0.0067 (12)
C5	0.0548 (17)	0.0397 (15)	0.0413 (14)	0.0073 (12)	0.0122 (12)	0.0047 (11)
C6	0.0460 (14)	0.0402 (14)	0.0370 (13)	−0.0021 (11)	0.0188 (11)	0.0008 (11)
C7	0.0309 (12)	0.0394 (13)	0.0315 (12)	−0.0074 (10)	0.0146 (9)	−0.0060 (10)
C8	0.0336 (13)	0.0530 (16)	0.0383 (13)	−0.0014 (11)	0.0177 (11)	−0.0040 (11)
N1	0.0290 (10)	0.0485 (13)	0.0258 (9)	−0.0016 (9)	0.0120 (8)	0.0026 (9)

O1	0.0481 (11)	0.0659 (13)	0.0317 (9)	0.0038 (9)	0.0201 (8)	0.0024 (8)
Cl1	0.0420 (4)	0.0638 (5)	0.0887 (6)	0.0026 (3)	0.0403 (4)	0.0032 (4)
Cl2	0.0412 (4)	0.0708 (6)	0.0955 (7)	0.0202 (4)	0.0081 (4)	0.0067 (5)
Cl3	0.0354 (4)	0.0872 (7)	0.1111 (8)	−0.0204 (4)	0.0241 (4)	−0.0139 (6)
Cl4	0.0855 (6)	0.0608 (5)	0.0616 (5)	0.0230 (4)	0.0318 (4)	−0.0043 (4)

Geometric parameters (Å, °)

C1—C6	1.384 (4)	C5—H5	0.9300
C1—C2	1.384 (4)	C6—H6	0.9300
C1—N1	1.413 (3)	C7—O1	1.215 (3)
C2—C3	1.378 (4)	C7—N1	1.334 (3)
C2—H2	0.9300	C7—C8	1.521 (4)
C3—C4	1.376 (4)	C8—Cl3	1.754 (3)
C3—Cl1	1.723 (3)	C8—Cl4	1.756 (3)
C4—C5	1.378 (4)	C8—H8	0.9800
C4—Cl2	1.723 (3)	N1—H1N	0.78 (3)
C5—C6	1.375 (4)		

C6—C1—C2	120.0 (2)	C5—C6—C1	119.0 (2)
C6—C1—N1	122.8 (2)	C5—C6—H6	120.5
C2—C1—N1	117.2 (2)	C1—C6—H6	120.5
C3—C2—C1	120.1 (2)	O1—C7—N1	125.0 (2)
C3—C2—H2	120.0	O1—C7—C8	121.4 (2)
C1—C2—H2	120.0	N1—C7—C8	113.6 (2)
C4—C3—C2	120.2 (2)	C7—C8—Cl3	108.0 (2)
C4—C3—Cl1	121.1 (2)	C7—C8—Cl4	108.81 (17)
C2—C3—Cl1	118.7 (2)	Cl3—C8—Cl4	111.21 (14)
C3—C4—C5	119.3 (2)	C7—C8—H8	109.6
C3—C4—Cl2	120.9 (2)	Cl3—C8—H8	109.6
C5—C4—Cl2	119.8 (2)	Cl4—C8—H8	109.6
C6—C5—C4	121.4 (3)	C7—N1—C1	125.8 (2)
C6—C5—H5	119.3	C7—N1—H1N	118 (2)
C4—C5—H5	119.3	C1—N1—H1N	115 (2)

C6—C1—C2—C3	−2.3 (4)	C2—C1—C6—C5	1.8 (4)
N1—C1—C2—C3	178.7 (2)	N1—C1—C6—C5	−179.3 (2)
C1—C2—C3—C4	0.9 (4)	O1—C7—C8—Cl3	−69.7 (3)
C1—C2—C3—Cl1	−178.1 (2)	N1—C7—C8—Cl3	109.7 (2)
C2—C3—C4—C5	0.9 (4)	O1—C7—C8—Cl4	51.1 (3)
Cl1—C3—C4—C5	180.0 (2)	N1—C7—C8—Cl4	−129.4 (2)
C2—C3—C4—Cl2	−178.3 (2)	O1—C7—N1—C1	−4.4 (4)
Cl1—C3—C4—Cl2	0.7 (3)	C8—C7—N1—C1	176.2 (2)
C3—C4—C5—C6	−1.4 (4)	C6—C1—N1—C7	32.8 (4)
Cl2—C4—C5—C6	177.8 (2)	C2—C1—N1—C7	−148.3 (3)
C4—C5—C6—C1	0.0 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O1$^{\mathrm{i}}$	0.78 (3)	2.08 (3)	2.852 (3)	171 (3)

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

Fig. 1

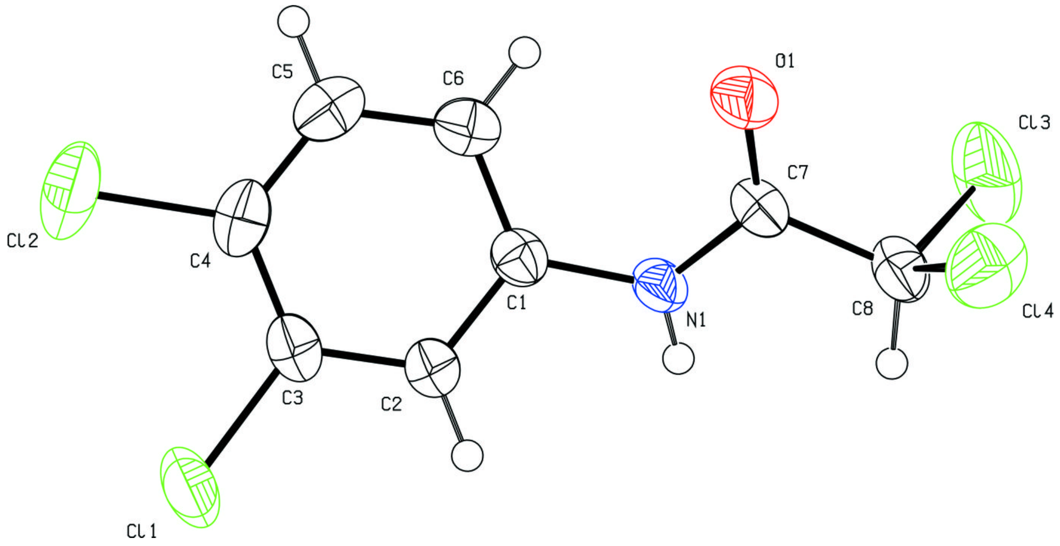

Fig. 2