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

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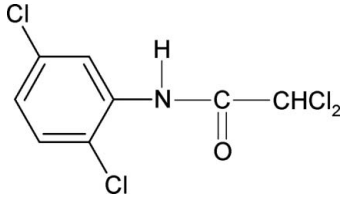
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.005 Å; R factor = 0.073; wR factor = 0.217; data-to-parameter ratio = 14.6.

The conformation of the N—H bond in the title compound, $C_8H_5Cl_4NO$, is *syn* to the 2-chloro substituent and *anti* to the 5-chloro substituent in the aromatic ring. The bond parameters are similar to those in 2,2-dichloro-*N*-phenylacetamide and other acetanilides. In the crystal structure, the molecules are linked into chains through N—H···O hydrogen bonding.

Related literature

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



Experimental

Crystal data

$C_8H_5Cl_4NO$
M_r = 272.93
Monoclinic, $P2_1/c$
a = 4.6977 (4) Å
b = 11.509 (2) Å
c = 19.888 (3) Å
β = 95.23 (1)°
V = 1070.8 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 9.77 mm^{-1}
T = 299 (2) K
0.60 × 0.08 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.367, T_{max} = 0.591
4168 measured reflections
1917 independent reflections
1420 reflections with $I > 2\sigma(I)$
R_{int} = 0.116
3 standard reflections
frequency: 120 min
intensity decay: 2.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.217
S = 1.03
1917 reflections
131 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.98 (6)	1.90 (6)	2.851 (4)	162 (4)

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

Data collection: *CAD-4-PC* Version (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC* Version; 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: BT2637).

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

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S1. Comment

In the present work, the structure of N-(2,5-dichlorophenyl)-2,2- dichloroacetamide (25DCPDCA) has been determined to study the effect of substituents on the structures of N-aromatic amides (Gowda et al., 2001, 2006; 2007a, b</i, c</i). The conformation of the N—H bond in 25DCPDCA is syn to the 2-chloro substituent and anti to the 5-chloro substituent in the aromatic ring (Fig. 1), in contrast to syn conformation observed with respect to both the 2- and 3-chloro substituents in N-(2,3-dichlorophenyl)-2,2-dichloroacetamide (23DCPDCA) (Gowda et al., 2007c), 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., 2007b),and 2- and 3-chloro substituents in N-(2,3-dichlorophenyl)-acetamide (23DCPA)(Gowda et al., 2007a), and 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 25DCPDCA are similar to those in N-(phenyl)-2,2-dichloroacetamide, 2CPDCA, 3CPDCA, 23DCPDCA, 34DCPDCA, 23DCPA and other acetanilides (Gowda et al., 2001, 2006; 2007a, b</i, b</i). The molecules in 25DCPDCA are linked into chains through N—H···O hydrogen bonding (Table 1 and Fig.2).

S2. 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.

S3. Refinement

The H atoms bonded to C were positioned with idealized geometry using a riding model with C—H = 0.93–0.98 Å. The NH atom was located in difference map and its coordinates were refined. U_{iso}(H) values were set equal to 1.2 U_{eq} of the parent atom.

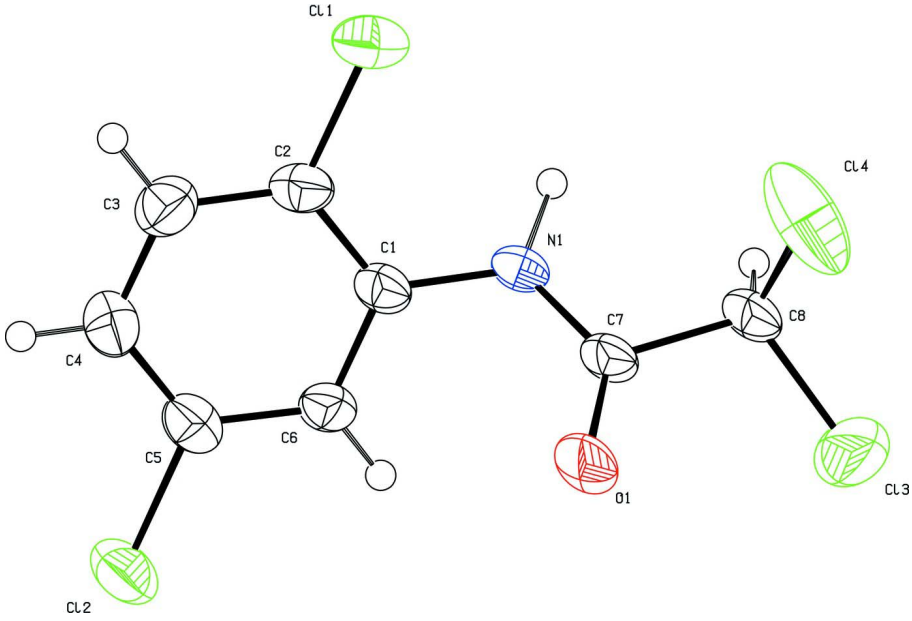

Figure 1

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.

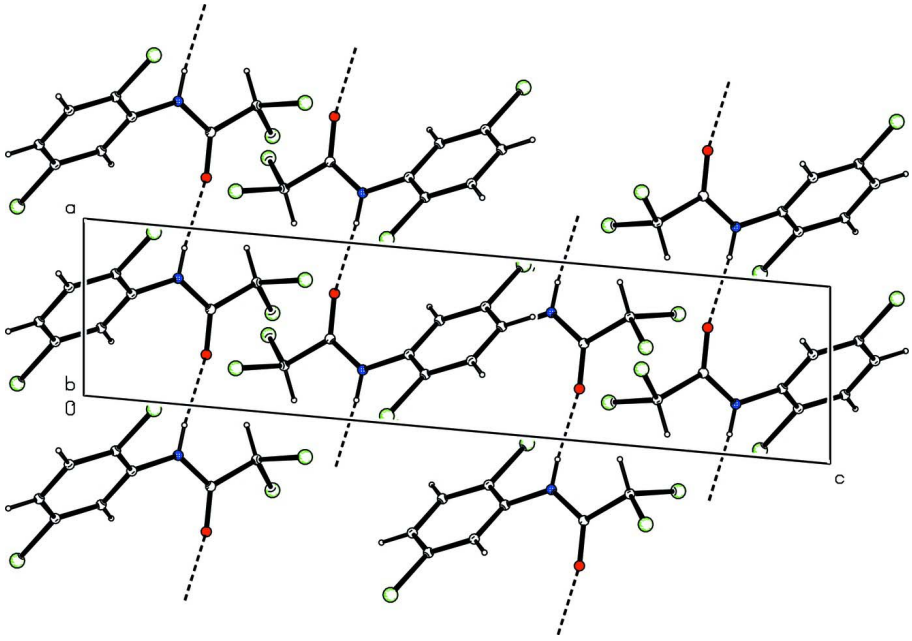

Figure 2

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

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

Crystal data

$C_8H_5Cl_4NO$
$M_r = 272.93$

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

a = 4.6977 (4) Å
b = 11.509 (2) Å
c = 19.888 (3) Å
β = 95.23 (1)°
V = 1070.8 (3) Å^3
Z = 4
F(000) = 544
D_x = 1.693 Mg m^{-3}

Cu $K\alpha$ radiation, λ = 1.54180 Å
Cell parameters from 25 reflections
θ = 7.7–20.2°
μ = 9.77 mm^{-1}
T = 299 K
Needle, colourless
0.60 × 0.08 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968
T_{min} = 0.367, T_{max} = 0.591
4168 measured reflections

1917 independent reflections
1420 reflections with $I > 2\sigma(I)$
R_{int} = 0.116
θ_{max} = 67.0°, θ_{min} = 4.4°
h = 0→5
k = −13→12
l = −23→23
3 standard reflections every 120 min
intensity decay: 2.0%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.217
S = 1.03
1917 reflections
131 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.144P)^2 + 0.518P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.033
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0038 (10)

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}
C1	0.3994 (8)	0.7062 (3)	0.43590 (17)	0.0324 (8)
C2	0.2953 (8)	0.8112 (3)	0.45736 (19)	0.0367 (8)
C3	0.3981 (10)	0.8573 (3)	0.5194 (2)	0.0439 (10)
H3	0.3256	0.9273	0.5337	0.053*
C4	0.6063 (10)	0.8001 (4)	0.55998 (19)	0.0440 (9)
H4	0.6764	0.8313	0.6014	0.053*

C5	0.7093 (9)	0.6956 (4)	0.53810 (19)	0.0389 (9)
C6	0.6085 (8)	0.6475 (3)	0.47678 (18)	0.0345 (8)
H6	0.6797	0.5768	0.4630	0.041*
C7	0.4478 (8)	0.6066 (3)	0.33005 (18)	0.0344 (8)
C8	0.2734 (8)	0.5542 (4)	0.26817 (19)	0.0411 (9)
H8	0.0932	0.5238	0.2822	0.049*
N1	0.2888 (7)	0.6573 (3)	0.37354 (16)	0.0379 (7)
H1N	0.080 (12)	0.653 (4)	0.365 (2)	0.045*
O1	0.7058 (6)	0.5995 (4)	0.33605 (15)	0.0578 (10)
Cl1	0.0411 (3)	0.88522 (9)	0.40600 (6)	0.0526 (4)
Cl2	0.9670 (3)	0.62104 (10)	0.58918 (5)	0.0529 (4)
Cl3	0.4607 (4)	0.4414 (2)	0.23486 (12)	0.1290 (11)
Cl4	0.1977 (5)	0.66320 (18)	0.20858 (7)	0.0964 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0219 (16)	0.0374 (17)	0.0371 (17)	−0.0020 (15)	−0.0014 (13)	−0.0001 (14)
C2	0.0262 (18)	0.0368 (18)	0.047 (2)	0.0029 (15)	0.0014 (15)	0.0014 (15)
C3	0.043 (2)	0.0387 (19)	0.050 (2)	0.0025 (19)	0.0064 (19)	−0.0080 (17)
C4	0.044 (2)	0.049 (2)	0.0380 (19)	−0.002 (2)	−0.0037 (17)	−0.0059 (16)
C5	0.0312 (19)	0.046 (2)	0.0383 (18)	−0.0034 (17)	−0.0028 (15)	0.0030 (15)
C6	0.0234 (18)	0.0380 (17)	0.0415 (19)	0.0014 (16)	0.0002 (15)	−0.0038 (15)
C7	0.0174 (17)	0.0484 (19)	0.0366 (18)	−0.0005 (15)	−0.0015 (14)	−0.0015 (15)
C8	0.0202 (17)	0.059 (2)	0.0424 (19)	−0.0010 (18)	−0.0059 (15)	−0.0060 (18)
N1	0.0157 (14)	0.0525 (17)	0.0442 (17)	0.0034 (15)	−0.0044 (12)	−0.0095 (14)
O1	0.0155 (14)	0.104 (3)	0.0529 (18)	0.0009 (16)	−0.0025 (12)	−0.0211 (16)
Cl1	0.0420 (7)	0.0528 (6)	0.0619 (7)	0.0160 (5)	−0.0019 (5)	0.0066 (4)
Cl2	0.0484 (7)	0.0613 (7)	0.0456 (6)	0.0052 (5)	−0.0138 (5)	0.0056 (4)
Cl3	0.0671 (10)	0.1554 (19)	0.1553 (17)	0.0449 (12)	−0.0398 (11)	−0.1137 (16)
Cl4	0.1098 (15)	0.1129 (13)	0.0588 (9)	−0.0326 (12)	−0.0344 (9)	0.0310 (8)

Geometric parameters (Å, º)

C1—C2	1.386 (5)	C5—Cl2	1.735 (4)
C1—C6	1.392 (5)	C6—H6	0.9300
C1—N1	1.417 (5)	C7—O1	1.209 (5)
C2—C3	1.389 (6)	C7—N1	1.328 (5)
C2—Cl1	1.724 (4)	C7—C8	1.538 (5)
C3—C4	1.377 (6)	C8—Cl3	1.734 (4)
C3—H3	0.9300	C8—Cl4	1.740 (4)
C4—C5	1.381 (6)	C8—H8	0.9800
C4—H4	0.9300	N1—H1N	0.98 (6)
C5—C6	1.383 (5)		
C2—C1—C6	119.6 (3)	C5—C6—C1	119.2 (3)
C2—C1—N1	120.3 (3)	C5—C6—H6	120.4
C6—C1—N1	120.1 (3)	C1—C6—H6	120.4

C1—C2—C3	120.2 (4)	O1—C7—N1	125.7 (4)
C1—C2—Cl1	119.6 (3)	O1—C7—C8	120.5 (4)
C3—C2—Cl1	120.2 (3)	N1—C7—C8	113.8 (3)
C4—C3—C2	120.6 (4)	C7—C8—Cl3	110.3 (3)
C4—C3—H3	119.7	C7—C8—Cl4	108.9 (3)
C2—C3—H3	119.7	Cl3—C8—Cl4	111.0 (2)
C3—C4—C5	118.8 (4)	C7—C8—H8	108.9
C3—C4—H4	120.6	Cl3—C8—H8	108.9
C5—C4—H4	120.6	Cl4—C8—H8	108.9
C4—C5—C6	121.7 (4)	C7—N1—C1	124.1 (3)
C4—C5—Cl2	119.5 (3)	C7—N1—H1N	119 (3)
C6—C5—Cl2	118.9 (3)	C1—N1—H1N	117 (3)
C6—C1—C2—C3	−0.3 (6)	C2—C1—C6—C5	−0.2 (6)
N1—C1—C2—C3	178.1 (4)	N1—C1—C6—C5	−178.7 (3)
C6—C1—C2—Cl1	178.8 (3)	O1—C7—C8—Cl3	25.6 (5)
N1—C1—C2—Cl1	−2.8 (5)	N1—C7—C8—Cl3	−154.3 (3)
C1—C2—C3—C4	0.7 (6)	O1—C7—C8—Cl4	−96.5 (4)
Cl1—C2—C3—C4	−178.3 (3)	N1—C7—C8—Cl4	83.6 (4)
C2—C3—C4—C5	−0.6 (7)	O1—C7—N1—C1	−3.3 (7)
C3—C4—C5—C6	0.0 (7)	C8—C7—N1—C1	176.5 (3)
C3—C4—C5—Cl2	−178.8 (3)	C2—C1—N1—C7	138.6 (4)
C4—C5—C6—C1	0.4 (6)	C6—C1—N1—C7	−43.0 (6)
Cl2—C5—C6—C1	179.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.98 (6)	1.90 (6)	2.851 (4)	162 (4)

Symmetry code: (i) *x*−1, *y*, *z*.