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2,2-Dichloro-*N*-(4-chlorophenylsulfonyl)-acetamide

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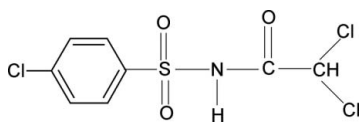
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 Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.027; wR factor = 0.073; data-to-parameter ratio = 15.9.

In the crystal structure of the title compound (N4CPSDCAA), $\text{C}_8\text{H}_6\text{Cl}_3\text{NO}_3\text{S}$, the conformations of the $\text{N}-\text{H}$ and $\text{C}=\text{O}$ bonds in the $\text{SO}_2-\text{NH}-\text{CO}-\text{C}$ group are *trans* to each other, similar to those observed in 2,2-dichloro-*N*-(phenylsulfonyl)-acetamide (NPSDCAA), 2,2-dichloro-*N*-(4-methylphenylsulfonyl)acetamide (N4MPSDCAA) and *N*-(4-chlorophenylsulfonyl)-2,2,2-trimethylacetamide (N4CPSTMAA), with similar bond parameters. The $-\text{SNHCOC}-$ unit in N4CPSDCAA is essentially planar and makes a dihedral angle of $79.67(5)^\circ$ with the benzene ring, comparable to $79.75(8)^\circ$ in NPSDCAA, $81.02(5)^\circ$ in N4MPSDCAA and $82.2(1)^\circ$ in N4CPSTMAA. The molecules in N4CPSDCAA are linked into layers parallel to the (001) plane by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For related literature, see: Gowda *et al.* (2003, 2006); Gowda, Foro, Nirmala *et al.* (2008); Gowda, Foro, Sowmya *et al.* (2008).



Experimental

Crystal data

$\text{C}_8\text{H}_6\text{Cl}_3\text{NO}_3\text{S}$
 $M_r = 302.55$
 Orthorhombic, *Pbca*
 $a = 9.5909(5)$ Å
 $b = 10.1750(5)$ Å
 $c = 23.256(1)$ Å
 $V = 2269.49(19)$ Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.98$ mm⁻¹
 $T = 299(2)$ K
 $0.32 \times 0.28 \times 0.08$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
 $T_{\min} = 0.745$, $T_{\max} = 0.926$
 10378 measured reflections
 2309 independent reflections
 1642 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.073$
 $S = 1.08$
 2309 reflections
 145 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.34$ e Å⁻³
 $\Delta\rho_{\min} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O3}^i$	0.86	1.97	2.814 (2)	169

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; 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: CI2630).

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supporting information

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2,2-Dichloro-*N*-(4-chlorophenylsulfonyl)acetamide

B. Thimme Gowda, Sabine Foro, P. G. Nirmala, B. P. Sowmya and Hartmut Fuess

S1. Comment

As part of a study of the substituent effects on the crystal structures of *N*-(aryl)-sulfonamides and substituted amides, in the present work, the structure of *N*-(4-chlorophenylsulfonyl)-2,2-dichloroacetamide (N4CPSDCAA) has been determined (Gowda *et al.*, 2003, 2006; Gowda, Foro, Nirmala *et al.*, 2008; Gowda, Foro, Sowmya *et al.*, 2008). The conformations of the N—H and C=O bonds of the SO₂—NH—CO—C group in N4CPSDCAA are *trans* to each other (Fig. 1), similar to those observed in *N*-(phenylsulfonyl)-2,2-dichloroacetamide (NPSDCAA), *N*-(4-methylphenylsulfonyl)-2,2-dichloroacetamide (N4MPSDCAA) (Gowda, Foro, Nirmala *et al.*, 2008) and (4-chlorophenylsulfonyl)-2,2,2-trimethylacetamide (N4CPSTMAA) (Gowda, Foro, Sowmya *et al.*, 2008). The bond parameters in N4CPSDCAA are similar to those in NPSDCAA, N4MPSDCAA, N4CPSTMAA (Gowda, Foro, Nirmala *et al.*, 2008; Gowda, Foro, Sowmya *et al.*, 2008), *N*-(aryl)-2,2-dichloroacetamides (Gowda *et al.*, 2006) and 4-chlorobenzene-sulfonamide (Gowda *et al.*, 2003).

The packing diagram of N4CPSDCAA showing the N—H···O hydrogen bonds (Table 1) involved in the formation of layers parallel to the (0 0 1) plane is shown in Fig. 2.

S2. Experimental

The title compound was prepared by refluxing 4-chlorobenzenesulfonamide (0.10 mole) with excess dichloroacetyl chloride (0.20 mole) for about an hour on a water bath. The reaction mixture was cooled and poured into ice cold water. The resulting solid was separated, washed thoroughly with water and dissolved in warm dilute sodium hydrogen carbonate solution. The title compound was precipitated by acidifying the filtered solution with glacial acetic acid. It was filtered, dried and recrystallized from ethanol. The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra. Single crystals of the title compound used for X-ray diffraction studies were obtained by slow evaporation of an ethanolic solution.

S3. Refinement

H atoms were positioned with idealized geometry (C—H = 0.93 or 0.98 Å, N—H = 0.86 Å) and were refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$. To improve R1, wR2 and S values (1 0 2) reflection was omitted during the refinement

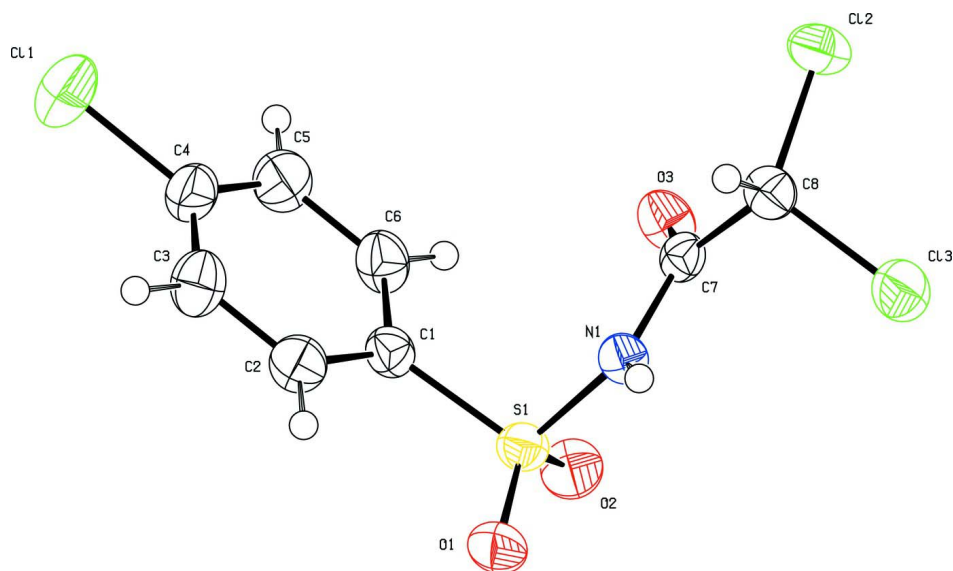


Figure 1

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 radii.

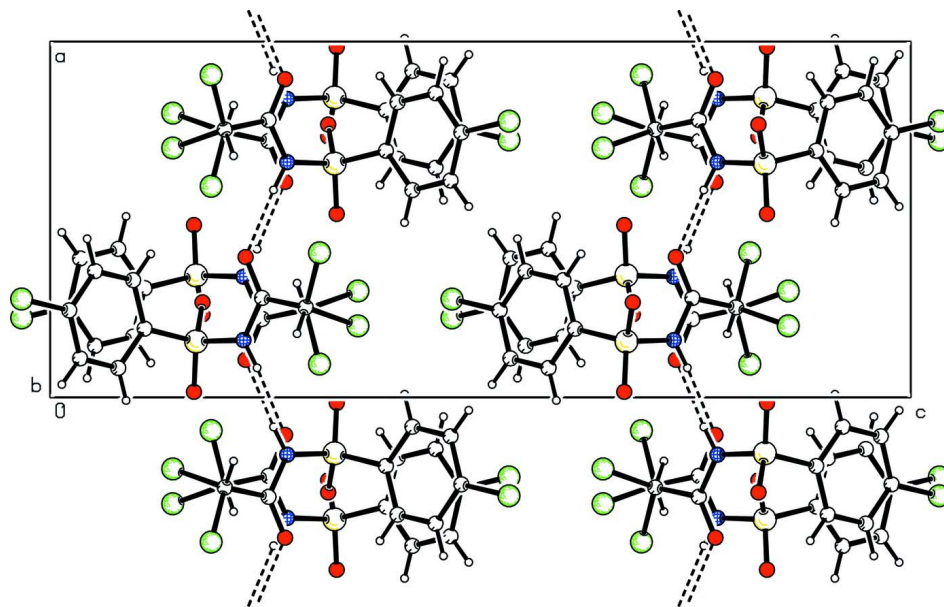


Figure 2

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

2,2-Dichloro-*N*-(4-chlorophenylsulfonyl)acetamide

Crystal data

$C_8H_6Cl_3NO_3S$

$M_r = 302.55$

Orthorhombic, *Pbca*

Hall symbol: $-P\ 2ac\ 2ab$

$a = 9.5909\ (5)\ \text{\AA}$

$b = 10.1750\ (5)\ \text{\AA}$

$c = 23.256\ (1)\ \text{\AA}$

$V = 2269.49\ (19)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1216$

$D_x = 1.771 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 5145 reflections
 $\theta = 2.2\text{--}28.0^\circ$

$\mu = 0.98 \text{ mm}^{-1}$
 $T = 299 \text{ K}$
 Plate, colourless
 $0.32 \times 0.28 \times 0.08 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω and ϕ scans
 Absorption correction: multi-scan
 (CrysAlis RED; Oxford Diffraction, 2007)
 $T_{\min} = 0.745$, $T_{\max} = 0.926$

10378 measured reflections
 2309 independent reflections
 1642 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 3.1^\circ$
 $h = -11 \rightarrow 11$
 $k = -10 \rightarrow 12$
 $l = -28 \rightarrow 29$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.073$
 $S = 1.09$
 2309 reflections
 145 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-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0279P)^2 + 1.2816P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.34 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.27 \text{ e \AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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	0.1931 (2)	0.2061 (2)	0.10986 (9)	0.0312 (5)
C2	0.0826 (2)	0.2583 (2)	0.07911 (10)	0.0397 (6)
H2	-0.0087	0.2353	0.0882	0.048*
C3	0.1095 (3)	0.3449 (3)	0.03482 (11)	0.0464 (6)
H3	0.0364	0.3802	0.0136	0.056*
C4	0.2445 (3)	0.3787 (2)	0.02219 (9)	0.0431 (6)
C5	0.3549 (3)	0.3264 (3)	0.05226 (10)	0.0471 (6)
H5	0.4458	0.3499	0.0430	0.056*
C6	0.3295 (2)	0.2384 (2)	0.09643 (10)	0.0405 (6)
H6	0.4031	0.2015	0.1168	0.049*
C7	0.2795 (2)	0.2659 (2)	0.24629 (9)	0.0275 (5)
C8	0.2532 (2)	0.3590 (2)	0.29700 (9)	0.0332 (5)

H8	0.1789	0.4210	0.2869	0.040*
N1	0.16103 (17)	0.21068 (16)	0.22462 (7)	0.0283 (4)
H1N	0.0830	0.2314	0.2404	0.034*
O1	0.01558 (16)	0.06430 (16)	0.16706 (7)	0.0436 (4)
O2	0.26697 (17)	0.01306 (15)	0.17698 (7)	0.0424 (4)
O3	0.39439 (14)	0.24435 (16)	0.22705 (6)	0.0379 (4)
Cl1	0.27646 (9)	0.48942 (8)	-0.03310 (3)	0.0686 (2)
Cl2	0.40650 (7)	0.44634 (6)	0.31294 (3)	0.04543 (17)
Cl3	0.20101 (7)	0.26485 (7)	0.35760 (2)	0.04963 (19)
S1	0.15710 (6)	0.10591 (5)	0.16960 (2)	0.03174 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0315 (12)	0.0341 (12)	0.0280 (11)	-0.0011 (9)	0.0005 (9)	-0.0033 (9)
C2	0.0314 (12)	0.0452 (14)	0.0425 (13)	-0.0007 (11)	-0.0017 (10)	0.0020 (11)
C3	0.0477 (15)	0.0485 (15)	0.0431 (14)	0.0070 (12)	-0.0074 (12)	0.0085 (12)
C4	0.0581 (16)	0.0403 (14)	0.0310 (12)	-0.0046 (12)	0.0046 (12)	0.0020 (11)
C5	0.0397 (14)	0.0629 (17)	0.0386 (13)	-0.0102 (13)	0.0071 (11)	-0.0001 (12)
C6	0.0312 (13)	0.0557 (15)	0.0346 (12)	-0.0004 (11)	-0.0008 (10)	0.0001 (11)
C7	0.0255 (12)	0.0289 (11)	0.0280 (10)	0.0023 (9)	-0.0023 (9)	0.0051 (9)
C8	0.0322 (12)	0.0322 (12)	0.0352 (11)	0.0030 (9)	-0.0028 (10)	-0.0014 (10)
N1	0.0200 (9)	0.0353 (10)	0.0296 (9)	0.0004 (7)	0.0020 (7)	-0.0014 (8)
O1	0.0366 (9)	0.0498 (10)	0.0443 (9)	-0.0153 (8)	-0.0038 (7)	0.0004 (8)
O2	0.0464 (10)	0.0329 (9)	0.0481 (10)	0.0080 (8)	-0.0005 (8)	-0.0007 (7)
O3	0.0220 (8)	0.0523 (10)	0.0394 (9)	0.0023 (7)	-0.0003 (7)	-0.0074 (8)
Cl1	0.0915 (6)	0.0667 (5)	0.0475 (4)	-0.0120 (4)	0.0065 (4)	0.0189 (4)
Cl2	0.0487 (4)	0.0371 (3)	0.0505 (4)	-0.0099 (3)	-0.0066 (3)	-0.0048 (3)
Cl3	0.0497 (4)	0.0641 (4)	0.0350 (3)	-0.0146 (3)	0.0070 (3)	-0.0037 (3)
S1	0.0303 (3)	0.0313 (3)	0.0337 (3)	-0.0030 (2)	-0.0011 (2)	-0.0007 (2)

Geometric parameters (Å, °)

C1—C6	1.385 (3)	C6—H6	0.93
C1—C2	1.385 (3)	C7—O3	1.209 (2)
C1—S1	1.757 (2)	C7—N1	1.364 (3)
C2—C3	1.380 (3)	C7—C8	1.534 (3)
C2—H2	0.93	C8—Cl2	1.758 (2)
C3—C4	1.371 (4)	C8—Cl3	1.776 (2)
C3—H3	0.93	C8—H8	0.98
C4—C5	1.376 (4)	N1—S1	1.6659 (17)
C4—Cl1	1.737 (2)	N1—H1N	0.86
C5—C6	1.384 (3)	O1—S1	1.4230 (16)
C5—H5	0.93	O2—S1	1.4257 (16)
C6—C1—C2	121.0 (2)	O3—C7—N1	123.20 (19)
C6—C1—S1	120.11 (17)	O3—C7—C8	123.12 (19)
C2—C1—S1	118.73 (17)	N1—C7—C8	113.68 (17)

C3—C2—C1	119.2 (2)	C7—C8—C12	109.69 (15)
C3—C2—H2	120.4	C7—C8—C13	108.86 (15)
C1—C2—H2	120.4	C12—C8—C13	109.94 (12)
C4—C3—C2	119.8 (2)	C7—C8—H8	109.4
C4—C3—H3	120.1	C12—C8—H8	109.4
C2—C3—H3	120.1	C13—C8—H8	109.4
C3—C4—C5	121.3 (2)	C7—N1—S1	124.49 (15)
C3—C4—C11	119.2 (2)	C7—N1—H1N	117.8
C5—C4—C11	119.4 (2)	S1—N1—H1N	117.8
C4—C5—C6	119.5 (2)	O1—S1—O2	120.86 (10)
C4—C5—H5	120.3	O1—S1—N1	104.11 (9)
C6—C5—H5	120.3	O2—S1—N1	108.35 (9)
C5—C6—C1	119.2 (2)	O1—S1—C1	109.09 (10)
C5—C6—H6	120.4	O2—S1—C1	109.54 (10)
C1—C6—H6	120.4	N1—S1—C1	103.40 (9)
C6—C1—C2—C3	-0.7 (3)	N1—C7—C8—C13	70.2 (2)
S1—C1—C2—C3	175.18 (18)	O3—C7—N1—S1	0.5 (3)
C1—C2—C3—C4	-0.5 (4)	C8—C7—N1—S1	179.96 (14)
C2—C3—C4—C5	1.1 (4)	C7—N1—S1—O1	173.55 (16)
C2—C3—C4—C11	-179.21 (19)	C7—N1—S1—O2	43.71 (19)
C3—C4—C5—C6	-0.4 (4)	C7—N1—S1—C1	-72.47 (18)
C11—C4—C5—C6	179.85 (19)	C6—C1—S1—O1	-167.61 (18)
C4—C5—C6—C1	-0.7 (4)	C2—C1—S1—O1	16.5 (2)
C2—C1—C6—C5	1.3 (3)	C6—C1—S1—O2	-33.3 (2)
S1—C1—C6—C5	-174.49 (18)	C2—C1—S1—O2	150.84 (17)
O3—C7—C8—C12	10.0 (3)	C6—C1—S1—N1	82.0 (2)
N1—C7—C8—C12	-169.47 (14)	C2—C1—S1—N1	-93.83 (19)
O3—C7—C8—C13	-110.4 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O3 ⁱ	0.86	1.97	2.814 (2)	169

Symmetry code: (i) $x-1/2, y, -z+1/2$.