

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

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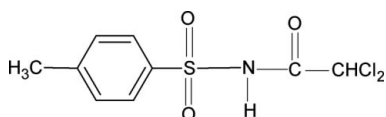
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.034; wR factor = 0.100; data-to-parameter ratio = 15.4.

The N—H and C=O bonds in the title compound, $\text{C}_9\text{H}_9\text{Cl}_2\text{NO}_3\text{S}$, are *trans* to each other, similar to what is observed in 2,2,2-trimethyl-*N*-(phenylsulfonyl)acetamide and 2,2,2-trimethyl-*N*-(4-methylphenylsulfonyl)acetamide. The bond parameters in the title compound are also similar to those in the aforementioned two structures. N—H...O hydrogen bonds connect the molecules into chains running along the *a* axis.

Related literature

For related literature, see: Gowda *et al.* (2006, 2007, 2008*a,b*).



Experimental

Crystal data

$\text{C}_9\text{H}_9\text{Cl}_2\text{NO}_3\text{S}$
 $M_r = 282.13$
Orthorhombic, *Pbca*
 $a = 9.6580$ (8) Å
 $b = 10.3177$ (8) Å
 $c = 23.067$ (2) Å

$V = 2298.6$ (3) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.74$ mm⁻¹
 $T = 299$ (2) K
 $0.48 \times 0.46 \times 0.32$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2007)
 $T_{\min} = 0.719$, $T_{\max} = 0.799$
8079 measured reflections
2313 independent reflections
1886 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.099$
 $S = 1.14$
2313 reflections
150 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O3}^i$	0.82 (3)	2.03 (3)	2.833 (2)	166 (2)

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: BT2744).

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supplementary materials

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

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Comment

As part of a study of the substituent effects on the solid state geometries of *N*-(aryl)-sulfonamides and substituted amides, the structure of *N*-(4-methylphenylsulfonyl)-2,2-dichloroacetamide (N4MPSDCAA) has been determined (Gowda *et al.*, 2006, 2007, 2008*a*, 2008*b*). The conformation of the N—H and C=O bonds in N4MPSDCAA are anti to each other (Fig. 1), similar to that observed in *N*-(phenylsulfonyl)-2,2,2-trimethylacetamide (NPSTMAA) (Gowda *et al.*, 2008*b*), *N*-(4-chlorophenylsulfonyl)-2,2,2-trimethylacetamide (N4CPSTMAA) and (4-methylphenylsulfonyl)-2,2,2-trimethylacetamide (N4MPSTMAA) (Gowda *et al.*, 2008*a*, *b*). The bond parameters in N4MPSDCAA are similar to those in NPSTMAA, N4MPSTMAA, N4CPSTMAA (Gowda *et al.*, 2008*a*, *b*), *N*-(aryl)-2,2-dichloroacetamides (Gowda *et al.*, 2006) and 4-methylbenzenesulfonamide and other arylsulfonamides (Gowda *et al.*, 2007). The N—H···O hydrogen bonds (Table 1) connect the molecules to chains running along the *a* axis (Fig. 2).

Experimental

The title compound was prepared by refluxing 4-methylbenzenesulfonamide (0.10 mole) with an 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 from a slow evaporation of an ethanolic solution.

Refinement

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

The five reflections most deviating reflections (1 0 2, 1 1 2, 0 2 0, 1 1 1, 0 0 4) were omitted from the refinement.

Figures

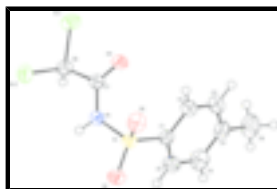


Fig. 1. Molecular structure of (I), showing the atom labeling scheme. The displacement ellipsoids are drawn at the 50% probability level. The H atoms are represented as small spheres of arbitrary radii.

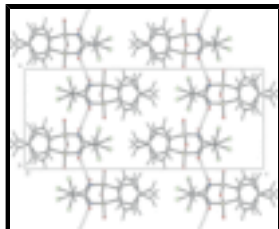


Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

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Crystal data

$C_9H_9Cl_2NO_3S$

$M_r = 282.13$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 9.6580$ (8) Å

$b = 10.3177$ (8) Å

$c = 23.067$ (2) Å

$V = 2298.6$ (3) Å³

$Z = 8$

$F_{000} = 1152$

$D_x = 1.631$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3825 reflections

$\theta = 2.3$ – 28.0°

$\mu = 0.74$ mm⁻¹

$T = 299$ (2) K

Prism, colourless

$0.48 \times 0.46 \times 0.32$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 299$ (2) K

Rotation method data acquisition using ω and φ scans $\theta_{\min} = 3.9^\circ$

Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)

$T_{\min} = 0.719$, $T_{\max} = 0.799$

8079 measured reflections

2313 independent reflections

1886 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\max} = 26.4^\circ$

$h = -12 \rightarrow 11$

$k = -12 \rightarrow 11$

$l = -28 \rightarrow 28$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.099$

$S = 1.14$

2313 reflections

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 1.5357P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.34 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\min} = -0.33 \text{ e \AA}^{-3}$$

150 parameters

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.0216 (12)

Secondary atom site location: difference Fourier map

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2007) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1940 (2)	0.2089 (2)	0.10903 (9)	0.0321 (5)
C2	0.3304 (3)	0.2325 (3)	0.09411 (11)	0.0419 (6)
H2	0.4022	0.1916	0.1138	0.050*
C3	0.3581 (3)	0.3181 (3)	0.04945 (11)	0.0487 (6)
H3	0.4497	0.3348	0.0395	0.058*
C4	0.2532 (3)	0.3796 (2)	0.01912 (10)	0.0440 (6)
C5	0.1181 (3)	0.3514 (3)	0.03428 (12)	0.0480 (6)
H5	0.0463	0.3897	0.0136	0.058*
C6	0.0866 (3)	0.2678 (2)	0.07928 (11)	0.0403 (6)
H6	-0.0050	0.2515	0.0893	0.048*
C7	0.2763 (2)	0.27000 (19)	0.24611 (9)	0.0279 (4)
C8	0.2502 (2)	0.3642 (2)	0.29677 (9)	0.0331 (5)
H8	0.1773	0.4255	0.2859	0.040*
C9	0.2837 (4)	0.4760 (3)	-0.02853 (13)	0.0626 (8)
H9A	0.3703	0.4545	-0.0466	0.075*
H9B	0.2892	0.5616	-0.0124	0.075*
H9C	0.2111	0.4731	-0.0569	0.075*
N1	0.15881 (18)	0.21543 (18)	0.22498 (8)	0.0292 (4)
H1N	0.084 (3)	0.238 (2)	0.2380 (11)	0.035*
O1	0.01360 (18)	0.07170 (18)	0.16642 (7)	0.0438 (4)
O2	0.26226 (19)	0.01713 (16)	0.17765 (7)	0.0433 (4)
O3	0.39079 (15)	0.24827 (17)	0.22726 (7)	0.0385 (4)
Cl1	0.40283 (7)	0.45024 (6)	0.31278 (3)	0.0454 (2)
Cl2	0.19595 (7)	0.27418 (7)	0.35847 (3)	0.0508 (2)
S1	0.15508 (6)	0.11055 (5)	0.16929 (2)	0.03130 (18)

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0352 (11)	0.0326 (11)	0.0285 (10)	-0.0022 (9)	-0.0002 (8)	-0.0039 (9)
C2	0.0335 (12)	0.0556 (16)	0.0366 (12)	-0.0025 (11)	0.0009 (10)	0.0018 (11)
C3	0.0431 (14)	0.0605 (17)	0.0424 (13)	-0.0122 (12)	0.0083 (11)	0.0013 (13)
C4	0.0633 (16)	0.0372 (13)	0.0315 (11)	-0.0068 (11)	0.0046 (11)	-0.0024 (10)
C5	0.0556 (16)	0.0421 (14)	0.0463 (14)	0.0060 (12)	-0.0039 (12)	0.0077 (12)
C6	0.0353 (12)	0.0431 (14)	0.0424 (13)	0.0005 (10)	-0.0013 (10)	0.0029 (11)
C7	0.0282 (11)	0.0269 (10)	0.0285 (10)	0.0026 (8)	-0.0035 (8)	0.0052 (8)
C8	0.0323 (11)	0.0314 (11)	0.0355 (11)	0.0042 (9)	-0.0043 (9)	-0.0018 (9)
C9	0.087 (2)	0.0531 (18)	0.0476 (15)	-0.0120 (16)	0.0097 (15)	0.0089 (13)
N1	0.0233 (9)	0.0331 (10)	0.0312 (9)	0.0009 (7)	0.0022 (7)	-0.0022 (8)
O1	0.0407 (10)	0.0470 (10)	0.0436 (9)	-0.0165 (8)	-0.0027 (7)	-0.0009 (8)
O2	0.0524 (10)	0.0314 (9)	0.0462 (9)	0.0083 (8)	0.0008 (8)	-0.0002 (7)
O3	0.0230 (8)	0.0514 (10)	0.0412 (9)	0.0030 (7)	-0.0015 (6)	-0.0083 (7)
Cl1	0.0513 (4)	0.0353 (3)	0.0496 (4)	-0.0104 (3)	-0.0075 (3)	-0.0046 (3)
Cl2	0.0564 (4)	0.0610 (4)	0.0348 (3)	-0.0153 (3)	0.0085 (3)	-0.0048 (3)
S1	0.0327 (3)	0.0290 (3)	0.0322 (3)	-0.0035 (2)	-0.0011 (2)	-0.0005 (2)

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

C1—C2	1.384 (3)	C7—N1	1.357 (3)
C1—C6	1.384 (3)	C7—C8	1.540 (3)
C1—S1	1.761 (2)	C8—C11	1.760 (2)
C2—C3	1.383 (4)	C8—Cl2	1.778 (2)
C2—H2	0.9300	C8—H8	0.9800
C3—C4	1.385 (4)	C9—H9A	0.9600
C3—H3	0.9300	C9—H9B	0.9600
C4—C5	1.381 (4)	C9—H9C	0.9600
C4—C9	1.512 (4)	N1—S1	1.6800 (19)
C5—C6	1.384 (4)	N1—H1N	0.82 (3)
C5—H5	0.9300	O1—S1	1.4256 (17)
C6—H6	0.9300	O2—S1	1.4276 (17)
C7—O3	1.210 (3)		
C2—C1—C6	120.8 (2)	C7—C8—C11	109.94 (15)
C2—C1—S1	120.07 (18)	C7—C8—Cl2	109.02 (14)
C6—C1—S1	118.98 (18)	C11—C8—Cl2	110.02 (12)
C3—C2—C1	118.8 (2)	C7—C8—H8	109.3
C3—C2—H2	120.6	C11—C8—H8	109.3
C1—C2—H2	120.6	Cl2—C8—H8	109.3
C2—C3—C4	121.8 (2)	C4—C9—H9A	109.5
C2—C3—H3	119.1	C4—C9—H9B	109.5
C4—C3—H3	119.1	H9A—C9—H9B	109.5
C5—C4—C3	117.8 (2)	C4—C9—H9C	109.5
C5—C4—C9	120.5 (3)	H9A—C9—H9C	109.5
C3—C4—C9	121.7 (3)	H9B—C9—H9C	109.5

C4—C5—C6	121.9 (2)	C7—N1—S1	124.04 (15)
C4—C5—H5	119.0	C7—N1—H1N	119.4 (18)
C6—C5—H5	119.0	S1—N1—H1N	116.4 (18)
C5—C6—C1	118.7 (2)	O1—S1—O2	120.76 (11)
C5—C6—H6	120.6	O1—S1—N1	103.72 (10)
C1—C6—H6	120.6	O2—S1—N1	108.43 (10)
O3—C7—N1	124.0 (2)	O1—S1—C1	109.25 (11)
O3—C7—C8	122.60 (19)	O2—S1—C1	109.93 (11)
N1—C7—C8	113.45 (18)	N1—S1—C1	103.17 (10)
C6—C1—C2—C3	1.1 (4)	N1—C7—C8—C12	69.4 (2)
S1—C1—C2—C3	-175.0 (2)	O3—C7—N1—S1	-0.4 (3)
C1—C2—C3—C4	-0.5 (4)	C8—C7—N1—S1	179.19 (14)
C2—C3—C4—C5	-0.9 (4)	C7—N1—S1—O1	174.97 (17)
C2—C3—C4—C9	178.2 (3)	C7—N1—S1—O2	45.5 (2)
C3—C4—C5—C6	1.9 (4)	C7—N1—S1—C1	-71.09 (19)
C9—C4—C5—C6	-177.2 (3)	C2—C1—S1—O1	-164.08 (19)
C4—C5—C6—C1	-1.4 (4)	C6—C1—S1—O1	19.7 (2)
C2—C1—C6—C5	-0.2 (4)	C2—C1—S1—O2	-29.4 (2)
S1—C1—C6—C5	175.97 (19)	C6—C1—S1—O2	154.39 (18)
O3—C7—C8—C11	9.7 (3)	C2—C1—S1—N1	86.1 (2)
N1—C7—C8—C11	-169.93 (15)	C6—C1—S1—N1	-90.13 (19)
O3—C7—C8—C12	-111.0 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots O3 ⁱ	0.82 (3)	2.03 (3)	2.833 (2)	166 (2)

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

Fig. 1

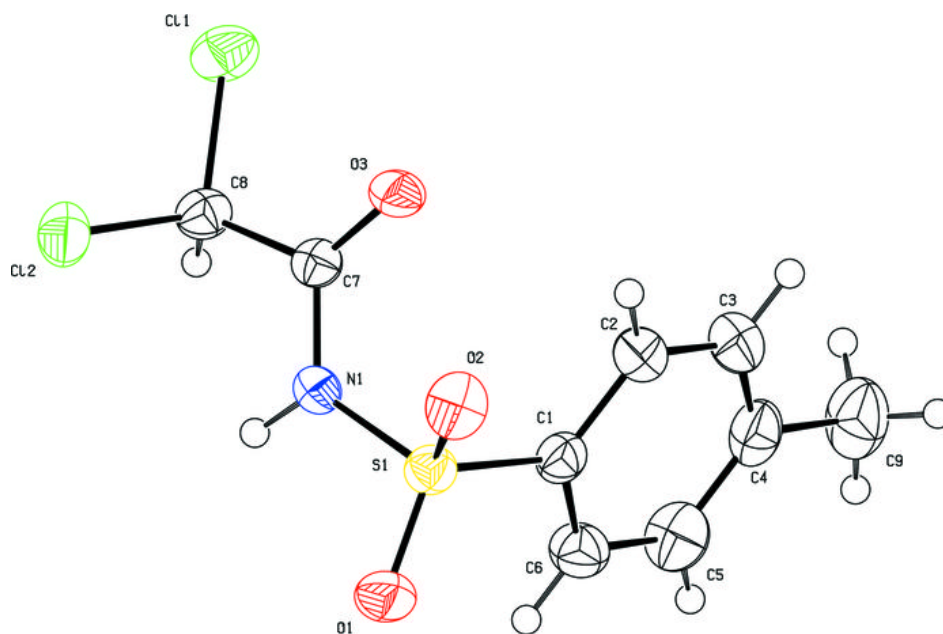


Fig. 2

