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N-(2,4-Dichlorophenyl)methanesulfonamide

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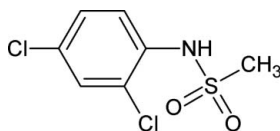
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.095; wR factor = 0.277; data-to-parameter ratio = 14.3.

The conformation of the N—H bond in the structure of the title compound, $\text{C}_7\text{H}_7\text{Cl}_2\text{NO}_2\text{S}$, is nearly *syn* to the *ortho*-chloro substituent, similar to the *syn* conformation observed for the *N*-(2,4-dimethylphenyl)methanesulfonamide. The geometric parameters are similar to those in other methanesulfonanilides, except for some differences in the bond and torsion angles. The amide H atom is readily available to a receptor molecule during its biological activity, as it lies on one side of the plane of the benzene ring, similar to those in other *N*-arylmethanesulfonamides. The molecules are packed into chains through both N—H...O and N—H...Cl hydrogen bonds.

Related literature

For related literature, see: Gowda *et al.* (2007*a,b,c,d,e,f,g,h,i,j,k,l*); Jayalakshmi & Gowda (2004); Klug (1968).



Experimental

Crystal data

$\text{C}_7\text{H}_7\text{Cl}_2\text{NO}_2\text{S}$
 $M_r = 240.10$
 Triclinic, $P\bar{1}$
 $a = 7.580$ (1) Å
 $b = 8.269$ (1) Å
 $c = 8.285$ (1) Å
 $\alpha = 83.70$ (1)°
 $\beta = 87.95$ (1)°

$\gamma = 68.57$ (1)°
 $V = 480.47$ (10) Å³
 $Z = 2$
 Cu $K\alpha$ radiation
 $\mu = 7.85$ mm⁻¹
 $T = 299$ (2) K
 $0.52 \times 0.32 \times 0.15$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.042$, $T_{\max} = 0.307$
 1831 measured reflections

1701 independent reflections
 1601 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.048$
 3 standard reflections
 frequency: 120 min
 intensity decay: 5.0%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.095$
 $wR(F^2) = 0.277$
 $S = 1.37$
 1701 reflections

119 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.84$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.65$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^{\text{i}}$	0.86	2.40	2.979 (5)	125
$\text{N1}-\text{H1N}\cdots\text{Cl2}^{\text{ii}}$	0.86	2.80	3.494 (3)	138

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

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: LW2023).

References

- Enraf–Nonius (1996). *CAD-4-PC* Software. Version 2.0. Enraf–Nonius, Delft, The Netherlands.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*a*). *Acta Cryst.* **E63**, o2337.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*b*). *Acta Cryst.* **E63**, o2338.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*c*). *Acta Cryst.* **E63**, o2339.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*d*). *Acta Cryst.* **E63**, o2340.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*e*). *Acta Cryst.* **E63**, o2569.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*f*). *Acta Cryst.* **E63**, o2570.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*g*). *Acta Cryst.* **E63**, o2597.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*h*). *Acta Cryst.* **E63**, o3014.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*i*). *Acta Cryst.* **E63**, o3015.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*j*). *Acta Cryst.* **E63**, o3085.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*k*). *Acta Cryst.* **E63**, o3086.
- Gowda, B. T., Foro, S. & Fuess, H. (2007*l*). Unpublished results.
- Jayalakshmi, K. L. & Gowda, B. T. (2004). *Z. Naturforsch. Teil A*, **59**, 491–500.
- Klug, H. P. (1968). *Acta Cryst.* **B24**, 792–802.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Stoe & Cie (1987). *REDU4*. Stoe & Cie GmbH, Darmstadt, Germany.

supplementary materials

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***N*-(2,4-Dichlorophenyl)methanesulfonamide**

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

Comment

The structural studies of sulfonanilides are of interest due to their biological activity. In the present work, the structure of *N*-(2,4-dichlorophenyl)-methanesulfonamide (24DCPMSA) has been determined to explore the substituent effects on the solid state structures of sulfonanilides (Gowda *et al.*, 2007*a-l*). The structure of 24DCPMSA (Fig. 1) resembles those of *N*-(phenyl)-methanesulfonamide (PMSA) (Klug, 1968), *N*-(2-chlorophenyl)-methanesulfonamide (2CPMSA) (Gowda *et al.*, 2007*l*), *N*-(2,3-dichlorophenyl)-methanesulfonamide (23DCPMSA)(Gowda *et al.*, 2007*k*), *N*-(2,4-dimethylphenyl)-methanesulfonamide (24DMPMSA)(Gowda *et al.*, 2007*i*) and other methylsulfonanilides (Gowda *et al.*, 2007*a-h*). The conformation of the N—H bond in 24DCPMSA is nearly *syn* to the *ortho*-chloro substituent, similar to the *syn* conformation observed for 24DMPMSA (Gowda *et al.*, 2007*i*). The *ortho* substitution of either a chloro or methyl group in PMSA changes its space group from monoclinic $P2_1/c$ to triclinic P-1. But the substitution of an additional chloro group in the *para* position of 2CPMSA to produce 24DCPMSA does not further alter the space group, in contrast to the change over from triclinic P-1 to monoclinic $P2_1/n$ on substitution of an additional methyl group at the *para* position in *N*-(2-methylphenyl)-methanesulfonamide (2MPMSA) (Gowda *et al.*, 2007*d*) to form 24DMPMSA and monoclinic $P2_1/c$ space group observed for 23DCPMSA (Gowda *et al.*, 2007*k*). The geometric parameters in 24DCPMSA are similar to those in PMSA, 2CPMSA, 23DCPMSA, 24DMPMSA and other methanesulfonanilides except for some difference in the bond and torsional angles. The amide hydrogen is readily available to a receptor molecule during its biological activity as it sits alone on one side of the plane of the phenyl group similar to those in other *N*-(aryl)-methanesulfonamides. The molecules in 24DCPMSA are packed into chains in the direction of *b* axis (Fig. 2) through both N—H \cdots O and N—H \cdots Cl hydrogen bonds (Table 1).

Experimental

The title compound was prepared according to the literature method (Jayalakshmi & Gowda, 2004). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Jayalakshmi & Gowda, 2004). Single crystals of the title compound were obtained from a slow evaporation of its ethanolic solution and used for X-ray diffraction studied at room temperature.

Refinement

The H atom of the NH group was located in a difference map and its position refined. The carbon-bound H atoms were positioned with idealized geometry and refined using a riding model with C—H = 0.93 Å (CH aromatic) or 0.96 Å (CH₃). Isotropic displacement parameters for all H atoms were set equal to 1.2 U_{eq} (parent atom).

Figures

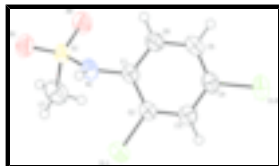


Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

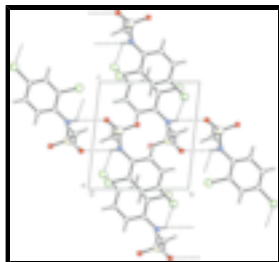


Fig. 2. The crystal packing of the title compound, viewed down the *b* axis.

N-(2,4-Dichlorophenyl)methanesulfonamide

Crystal data

$C_7H_7Cl_2NO_2S$

$M_r = 240.10$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.580$ (1) Å

$b = 8.269$ (1) Å

$c = 8.285$ (1) Å

$\alpha = 83.70$ (1)°

$\beta = 87.95$ (1)°

$\gamma = 68.57$ (1)°

$V = 480.47$ (10) Å³

$Z = 2$

$F_{000} = 244$

$D_x = 1.660$ Mg m⁻³

Cu $K\alpha$ radiation

$\lambda = 1.54180$ Å

Cell parameters from 25 reflections

$\theta = 6.8$ – 25.8 °

$\mu = 7.85$ mm⁻¹

$T = 299$ (2) K

Prism, colourless

$0.52 \times 0.32 \times 0.15$ 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)

$T_{\min} = 0.042$, $T_{\max} = 0.307$

1831 measured reflections

1701 independent reflections

1601 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\text{max}} = 66.9$ °

$\theta_{\text{min}} = 5.4$ °

$h = -9 \rightarrow 9$

$k = -9 \rightarrow 9$

$l = -9 \rightarrow 0$

3 standard reflections

every 120 min

intensity decay: 5.0%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.095$	$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$
$wR(F^2) = 0.277$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.37$	$(\Delta/\sigma)_{\max} < 0.001$
1701 reflections	$\Delta\rho_{\max} = 0.84 \text{ e } \text{\AA}^{-3}$
119 parameters	$\Delta\rho_{\min} = -1.65 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 1997), $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.130 (17)

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 $F^2 > 2\sigma(F^2)$ 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	0.3441 (5)	0.4168 (5)	0.2568 (4)	0.0353 (9)
C2	0.2920 (5)	0.3970 (5)	0.1016 (4)	0.0366 (9)
C3	0.2380 (5)	0.5343 (5)	-0.0203 (5)	0.0402 (10)
H3	0.2052	0.5185	-0.1229	0.048*
C4	0.2337 (5)	0.6943 (5)	0.0135 (5)	0.0402 (10)
C5	0.2829 (6)	0.7203 (5)	0.1657 (5)	0.0435 (10)
H5	0.2785	0.8298	0.1870	0.052*
C6	0.3382 (6)	0.5817 (5)	0.2844 (5)	0.0423 (10)
H6	0.3727	0.5986	0.3860	0.051*
C7	0.1136 (6)	0.1897 (7)	0.4902 (7)	0.0582 (12)
H7A	0.1653	0.0805	0.4440	0.070*
H7B	0.0322	0.2772	0.4122	0.070*
H7C	0.0421	0.1765	0.5853	0.070*
N1	0.4140 (4)	0.2726 (4)	0.3754 (4)	0.0402 (9)
H1N	0.5211	0.1917	0.3592	0.048*
O1	0.4295 (5)	0.1117 (4)	0.6438 (4)	0.0533 (9)

supplementary materials

O2	0.2194 (5)	0.4216 (4)	0.6006 (4)	0.0528 (9)
S1	0.29870 (12)	0.25352 (12)	0.54332 (10)	0.0385 (6)
Cl1	0.29909 (18)	0.19478 (13)	0.05738 (13)	0.0544 (6)
Cl2	0.17636 (15)	0.86631 (14)	-0.14033 (13)	0.0542 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0286 (16)	0.0312 (19)	0.0447 (18)	-0.0097 (14)	0.0020 (13)	-0.0028 (15)
C2	0.0343 (17)	0.032 (2)	0.0433 (18)	-0.0125 (15)	0.0027 (14)	-0.0051 (16)
C3	0.0344 (18)	0.038 (2)	0.0462 (19)	-0.0111 (16)	0.0006 (15)	-0.0033 (17)
C4	0.0333 (18)	0.034 (2)	0.052 (2)	-0.0125 (16)	0.0045 (15)	0.0008 (17)
C5	0.046 (2)	0.0310 (19)	0.055 (2)	-0.0157 (16)	0.0037 (17)	-0.0058 (17)
C6	0.0416 (19)	0.038 (2)	0.0489 (19)	-0.0161 (16)	-0.0034 (16)	-0.0044 (17)
C7	0.044 (2)	0.055 (3)	0.080 (3)	-0.023 (2)	0.005 (2)	-0.008 (2)
N1	0.0335 (15)	0.0297 (16)	0.0499 (18)	-0.0041 (12)	0.0024 (13)	-0.0001 (14)
O1	0.062 (2)	0.0393 (17)	0.0464 (15)	-0.0057 (14)	-0.0051 (13)	0.0036 (13)
O2	0.0627 (19)	0.0360 (17)	0.0491 (16)	-0.0054 (14)	0.0043 (14)	-0.0063 (14)
S1	0.0398 (8)	0.0281 (8)	0.0423 (8)	-0.0068 (5)	0.0007 (5)	-0.0011 (5)
Cl1	0.0720 (9)	0.0341 (9)	0.0594 (9)	-0.0207 (6)	-0.0036 (6)	-0.0081 (5)
Cl2	0.0552 (9)	0.0405 (9)	0.0583 (9)	-0.0121 (6)	0.0016 (6)	0.0120 (5)

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

C1—C6	1.392 (5)	C5—H5	0.9300
C1—C2	1.402 (5)	C6—H6	0.9300
C1—N1	1.409 (5)	C7—S1	1.754 (4)
C2—C3	1.384 (6)	C7—H7A	0.9600
C2—Cl1	1.732 (4)	C7—H7B	0.9600
C3—C4	1.371 (6)	C7—H7C	0.9600
C3—H3	0.9300	N1—S1	1.640 (3)
C4—C5	1.388 (6)	N1—H1N	0.8600
C4—Cl2	1.739 (4)	O1—S1	1.431 (3)
C5—C6	1.374 (6)	O2—S1	1.426 (3)
C6—C1—C2	117.2 (4)	C1—C6—H6	119.1
C6—C1—N1	121.4 (3)	S1—C7—H7A	109.5
C2—C1—N1	121.2 (3)	S1—C7—H7B	109.5
C3—C2—C1	121.9 (4)	H7A—C7—H7B	109.5
C3—C2—Cl1	118.3 (3)	S1—C7—H7C	109.5
C1—C2—Cl1	119.8 (3)	H7A—C7—H7C	109.5
C4—C3—C2	118.6 (4)	H7B—C7—H7C	109.5
C4—C3—H3	120.7	C1—N1—S1	122.4 (2)
C2—C3—H3	120.7	C1—N1—H1N	118.8
C3—C4—C5	121.5 (4)	S1—N1—H1N	118.8
C3—C4—Cl2	119.3 (3)	O2—S1—O1	119.49 (19)
C5—C4—Cl2	119.1 (3)	O2—S1—N1	107.84 (18)
C6—C5—C4	119.0 (3)	O1—S1—N1	105.42 (18)
C6—C5—H5	120.5	O2—S1—C7	108.8 (2)

C4—C5—H5	120.5	O1—S1—C7	108.1 (2)
C5—C6—C1	121.9 (3)	N1—S1—C7	106.5 (2)
C5—C6—H6	119.1		
C6—C1—C2—C3	0.4 (5)	Cl2—C4—C5—C6	-176.5 (3)
N1—C1—C2—C3	-174.6 (3)	C4—C5—C6—C1	-0.8 (6)
C6—C1—C2—Cl1	179.1 (3)	C2—C1—C6—C5	0.4 (6)
N1—C1—C2—Cl1	4.1 (5)	N1—C1—C6—C5	175.3 (3)
C1—C2—C3—C4	-0.7 (6)	C6—C1—N1—S1	70.8 (4)
Cl1—C2—C3—C4	-179.4 (3)	C2—C1—N1—S1	-114.4 (3)
C2—C3—C4—C5	0.3 (6)	C1—N1—S1—O2	-40.3 (3)
C2—C3—C4—Cl2	177.2 (3)	C1—N1—S1—O1	-169.0 (3)
C3—C4—C5—C6	0.4 (6)	C1—N1—S1—C7	76.4 (3)

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 O1 ⁱ	0.86	2.40	2.979 (5)	125
N1—H1N \cdots Cl2 ⁱⁱ	0.86	2.80	3.494 (3)	138

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

Fig. 1

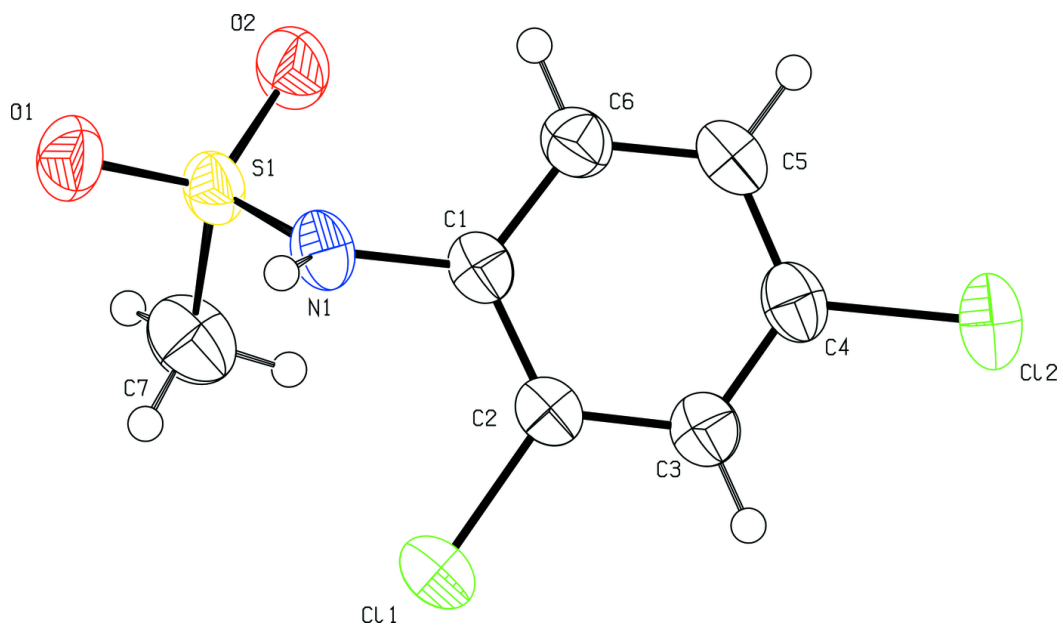


Fig. 2

