# organic compounds

# N-(3,5-Dichlorophenyl)methane-sulfonamide

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The structure of the title compound, $C_7H_7Cl_2NO_2S$, resembles those of other methanesulfonanilides, with similar geometric parameters. The amide H atom is roughly in the plane of the dichlorophenyl group with a deviation of −0.021 (7) Å, while the S atom is slightly above the plane with a deviation of 0.285 (9) Å. The dihedral angle between the benzene ring and the C—N—S plane is 14.4 (5)°. The molecules are packed into chains through N—H···O hydrogen bonds and weak (methyl)C—H···O interactions.

## Related literature

For related literature, see: Gowda *et al.* (2007*a*, 2007*b*); Jayalakshmi & Gowda (2004); Klug (1968).
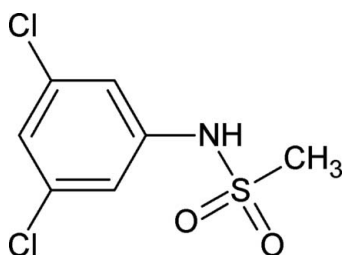


## Experimental

### Crystal data

| | |
|---|---|
| $C_7H_7Cl_2NO_2S$ | $V$ = 980.3 (5) Å$^3$ |
| $M_r$ = 240.10 | $Z$ = 4 |
| Monoclinic, $P2_1/c$ | Cu $K\alpha$ radiation |
| $a$ = 16.076 (3) Å | $\mu$ = 7.70 mm$^{-1}$ |
| $b$ = 5.053 (2) Å | $T$ = 299 (2) K |
| $c$ = 12.148 (3) Å | $0.75 \times 0.30 \times 0.03$ mm |
| $\beta$ = 96.60 (2)° | |

### Data collection

| | |
|---|---|
| Enraf–Nonius CAD4 diffractometer | 1731 independent reflections |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | 1424 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.043, $T_{max}$ = 0.597 (expected range = 0.057–0.794) | $R_{int}$ = 0.059 |
| 1828 measured reflections | 3 standard reflections frequency: 120 min intensity decay: 2.5% |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.092 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.255 | |
| $S$ = 1.09 | |
| 1731 reflections | $\Delta\rho_{max}$ = 1.17 e Å$^{-3}$ |
| 123 parameters | $\Delta\rho_{min}$ = −0.70 e Å$^{-3}$ |
| 1 restraint | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N5—H5···O3$^i$ | 0.85 (3) | 2.11 (4) | 2.949 (5) | 170 (4) |
| C1—H1$B$···O4$^{ii}$ | 0.96 | 2.45 | 3.233 (6) | 138 |

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

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC Software*; 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: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2186).

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**supplementary materials**

### *N*-(3,5-Dichlorophenyl)methanesulfonamide

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

#### Comment

The biological activity of sulfonanilides is thought to be due to the amide hydrogen portion of the molecules as it can align itself in relation to a receptor site. Thus the structural studies of sulfonanilides are of interest. In the present work, the structure of *N*-(3,5-dichlorophenyl)-methanesulfonamde (35DCPMSA) has been determined as part of our study of the substituent effects on the solid state structures of methanesulfonanilides (Gowda *et al.*, 2007*a*, *b*). The structure of 35DCPMSA (Fig. 1) is similar to those of other methanesulfonanilides (Gowda *et al.*, 2007*a*, *b*). 35DCPMSA crystallizes in monoclinic *P*2$_1$/*c* space group in contrast to the monoclinc *P*2$_1$/*c*, monoclinic *P*2$_1$/*n*, triclinic P-1 and triclinic P-1 space groups observed for *N*-(2,3-dichlorophenyl)-methanesulfonamide (23DCPMSA), *N*-(2,4-dichlorophenyl)-methanesulfonamide (24DCPMSA), *N*-(2,5-dichlorophenyl)-methanesulfonamide (25DCPMSA), *N*-(3,4-dichlorophenyl)-methanesulfonamide (34DCPMSA)(Gowda *et al.*, 2007*b*), respectively. The substitution of a Cl atom at the *meta* position of *N*-(phenyl)-methanesulfonamde (PMSA) (Klug, 1968) to produce *N*-(3-chlorophenyl)-methanesulfonamide (3CPMSA) changes its space group from monoclinic *P*2$_1$/*c* to C 2/c (Gowda *et al.*, 2007*a*). The substitution of an additional chloro group either at *ortho*, *para* or *meta* position of 3CPMSA to produce 23DCPMSA, 34DCPMSA or 35DCPMSA, respectively, changes the space group from monoclinc C 2/c to monoclinic *P*2$_1$/*c* with 23DCPMSA, triclinic P-1 with 34DCPMSA and monoclinic *P*2$_1$/*c* with 35DCPMSA. The geometric parameters in 35DCPMSA are similar to those in other methanesulfonanilides except for some difference in the bond and torsional angles. The N—H is roughly in the plane of the dichlorophenyl group with a deviation of −0.021 (7)Å from the plane, whereas the S atom is slightly above with a deviation of 0.285 (9) Å. The dihedral angle between the benzene ring and the C6—N5—S2 plane is 14.4 (5)°. The N—H⋯O hydrogen bonds (Table 1) build up chains which are further connected through weak C—H (methyl)⋯O interactions (Fig. 2).

#### 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 compound were obtained from a slow evaporation of its ethanolic solution and used for X-ray diffraction studies at room temperature.

#### Refinement

H atoms attached to C atoms were fixed geometrically and treated as riding, with C—H = 0.93 Å (aromatic) or 0.96 Å (CH$_3$) and with $U_{iso}$(H) = 1.2$U_{eq}$(aromatic) or $U_{iso}$(H) = 1.5$U_{eq}$(CH$_3$). H atom attched to N was refined using a N—H restraint of 0.85 (1) Å.
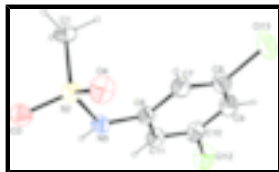
# supplementary materials

**Figures**



Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.
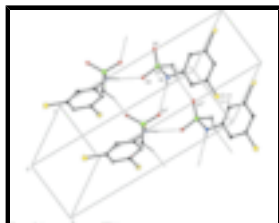


Fig. 2. Partial packing view showing the formation of the hydrogen bond network through N—H⋯O and C—H⋯O hydrogen bonding interactions. H bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code: (i) $-x + 1$, $1/2 + y$, $3/2 - z$; (ii) $x$, $1 + y$, $z$].

## *N*-(3,5-dichlorophenyl)methanesulfonamide

*Crystal data*

| | |
|---|---|
| $C_7H_7Cl_2NO_2S$ | $F_{000} = 488$ |
| $M_r = 240.10$ | $D_x = 1.627$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Cu $K\alpha$ radiation $\lambda = 1.54180$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 25 reflections |
| $a = 16.076\ (3)$ Å | $\theta = 2.8–19.9º$ |
| $b = 5.053\ (2)$ Å | $\mu = 7.70$ mm$^{-1}$ |
| $c = 12.148\ (3)$ Å | $T = 299\ (2)$ K |
| $\beta = 96.60\ (2)º$ | Long laminar, colourless |
| $V = 980.3\ (5)$ Å$^3$ | $0.75 \times 0.30 \times 0.03$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD4 diffractometer | $R_{int} = 0.059$ |
| Radiation source: fine-focus sealed tube | $\theta_{max} = 67.1º$ |
| Monochromator: graphite | $\theta_{min} = 2.8º$ |
| $T = 299(2)$ K | $h = -19 \rightarrow 19$ |
| $\omega$ scans | $k = 0 \rightarrow 6$ |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | $l = -14 \rightarrow 1$ |
| $T_{min} = 0.043$, $T_{max} = 0.597$ | 3 standard reflections |
| 1828 measured reflections | every 120 min |
| 1731 independent reflections | intensity decay: 2.5% |
| 1424 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.092$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.255$ | $w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.09$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 1731 reflections | $\Delta\rho_{max} = 1.17$ e Å$^{-3}$ |
| 123 parameters | $\Delta\rho_{min} = -0.69$ e Å$^{-3}$ |
| 1 restraint | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.6079 (3) | 0.3484 (10) | 1.0081 (4) | 0.0512 (12) |
| H1A | 0.5810 | 0.2657 | 1.0658 | 0.077* |
| H1B | 0.5894 | 0.5286 | 0.9994 | 0.077* |
| H1C | 0.6675 | 0.3447 | 1.0275 | 0.077* |
| C6 | 0.7071 (3) | 0.3919 (9) | 0.7811 (3) | 0.0415 (10) |
| C7 | 0.7711 (3) | 0.2524 (11) | 0.8417 (4) | 0.0525 (12) |
| H7 | 0.7596 | 0.1182 | 0.8901 | 0.063* |
| C8 | 0.8527 (4) | 0.3193 (11) | 0.8280 (5) | 0.0587 (13) |
| C9 | 0.8727 (3) | 0.5086 (12) | 0.7540 (5) | 0.0611 (13) |
| H9 | 0.9281 | 0.5488 | 0.7456 | 0.073* |
| C10 | 0.8076 (4) | 0.6353 (11) | 0.6932 (5) | 0.0562 (12) |
| C11 | 0.7251 (3) | 0.5846 (9) | 0.7054 (4) | 0.0482 (11) |
| H11 | 0.6822 | 0.6770 | 0.6641 | 0.058* |
| N5 | 0.6212 (2) | 0.3432 (8) | 0.7865 (3) | 0.0437 (9) |
| O3 | 0.4928 (2) | 0.1929 (7) | 0.8545 (3) | 0.0532 (9) |
| O4 | 0.6204 (2) | −0.0779 (7) | 0.8935 (3) | 0.0618 (10) |

| | | | |
|---|---|---|---|
| S2 | 0.58202 (7) | 0.1770 (2) | 0.88336 (8) | 0.0415 (5) |
| Cl12 | 0.83015 (11) | 0.8707 (3) | 0.59639 (16) | 0.0837 (6) |
| Cl13 | 0.93272 (10) | 0.1532 (5) | 0.90719 (17) | 0.0962 (8) |
| H5 | 0.586 (2) | 0.451 (7) | 0.754 (3) | 0.033 (11)* |

*Atomic displacement parameters ($Å^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.063 (3) | 0.046 (3) | 0.043 (2) | −0.002 (2) | 0.003 (2) | −0.0037 (19) |
| C6 | 0.046 (2) | 0.035 (2) | 0.043 (2) | 0.0028 (18) | 0.0066 (17) | −0.0026 (17) |
| C7 | 0.056 (3) | 0.051 (3) | 0.051 (2) | 0.007 (2) | 0.008 (2) | 0.006 (2) |
| C8 | 0.053 (3) | 0.061 (3) | 0.061 (3) | 0.006 (2) | 0.003 (2) | 0.006 (2) |
| C9 | 0.051 (3) | 0.060 (3) | 0.074 (3) | −0.006 (2) | 0.017 (2) | 0.002 (3) |
| C10 | 0.063 (3) | 0.046 (3) | 0.063 (3) | −0.001 (2) | 0.022 (2) | 0.005 (2) |
| C11 | 0.056 (2) | 0.036 (2) | 0.053 (2) | 0.004 (2) | 0.012 (2) | 0.0040 (19) |
| N5 | 0.043 (2) | 0.045 (2) | 0.0418 (19) | 0.0047 (15) | 0.0016 (16) | 0.0088 (15) |
| O3 | 0.0509 (18) | 0.048 (2) | 0.0596 (19) | −0.0087 (14) | 0.0031 (15) | −0.0066 (14) |
| O4 | 0.076 (2) | 0.0288 (18) | 0.082 (2) | 0.0097 (17) | 0.0151 (19) | 0.0060 (16) |
| S2 | 0.0479 (7) | 0.0299 (7) | 0.0466 (7) | −0.0017 (4) | 0.0061 (5) | −0.0010 (4) |
| Cl12 | 0.0841 (11) | 0.0712 (11) | 0.1022 (12) | −0.0009 (8) | 0.0379 (9) | 0.0311 (9) |
| Cl13 | 0.0531 (9) | 0.1287 (19) | 0.1048 (13) | 0.0228 (8) | 0.0006 (8) | 0.0388 (11) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—S2 | 1.754 (5) | C8—Cl13 | 1.732 (6) |
| C1—H1A | 0.9600 | C9—C10 | 1.369 (8) |
| C1—H1B | 0.9600 | C9—H9 | 0.9300 |
| C1—H1C | 0.9600 | C10—C11 | 1.376 (7) |
| C6—C7 | 1.387 (7) | C10—Cl12 | 1.740 (5) |
| C6—C11 | 1.392 (7) | C11—H11 | 0.9300 |
| C6—N5 | 1.412 (6) | N5—S2 | 1.630 (4) |
| C7—C8 | 1.382 (8) | N5—H5 | 0.85 (3) |
| C7—H7 | 0.9300 | O3—S2 | 1.439 (4) |
| C8—C9 | 1.376 (8) | O4—S2 | 1.427 (4) |
| S2—C1—H1A | 109.5 | C8—C9—H9 | 121.4 |
| S2—C1—H1B | 109.5 | C9—C10—C11 | 122.8 (5) |
| H1A—C1—H1B | 109.5 | C9—C10—Cl12 | 118.6 (4) |
| S2—C1—H1C | 109.5 | C11—C10—Cl12 | 118.6 (4) |
| H1A—C1—H1C | 109.5 | C10—C11—C6 | 118.5 (5) |
| H1B—C1—H1C | 109.5 | C10—C11—H11 | 120.7 |
| C7—C6—C11 | 120.6 (4) | C6—C11—H11 | 120.7 |
| C7—C6—N5 | 123.8 (4) | C6—N5—S2 | 125.8 (3) |
| C11—C6—N5 | 115.5 (4) | C6—N5—H5 | 118 (3) |
| C8—C7—C6 | 117.9 (5) | S2—N5—H5 | 112 (3) |
| C8—C7—H7 | 121.1 | O4—S2—O3 | 118.7 (2) |
| C6—C7—H7 | 121.1 | O4—S2—N5 | 109.2 (2) |
| C9—C8—C7 | 123.0 (5) | O3—S2—N5 | 104.6 (2) |
| C9—C8—Cl13 | 119.0 (4) | O4—S2—C1 | 108.3 (2) |

| | | | |
|---|---|---|---|
| C7—C8—Cl13 | 117.9 (4) | O3—S2—C1 | 108.3 (2) |
| C10—C9—C8 | 117.1 (5) | N5—S2—C1 | 107.2 (2) |
| C10—C9—H9 | 121.4 | | |

*Hydrogen-bond geometry (Å, °)*

| D—H⋯A | D—H | H⋯A | D⋯A | D—H⋯A |
|---|---|---|---|---|
| N5—H5⋯O3[i] | 0.85 (3) | 2.11 (4) | 2.949 (5) | 170 (4) |
| C1—H1B⋯O4[ii] | 0.96 | 2.45 | 3.233 (6) | 138 |

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

**Fig. 1**

**Fig. 2**