

N-(2,3-Dichlorophenyl)methanesulfonamide

 B. Thimme Gowda,^{a*} Sabine Foro^b and Hartmut Fuess^b

^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany

Correspondence e-mail: gowdabt@yahoo.com

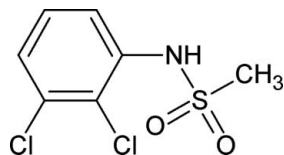
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Key indicators: single-crystal X-ray study; $T = 303$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.054; wR factor = 0.152; data-to-parameter ratio = 15.9.

In the structure of the title compound, $\text{C}_7\text{H}_7\text{Cl}_2\text{NO}_2\text{S}$, the conformation of the N—H bond is *syn* to both *ortho*- and *meta*-chloro substituents, in contrast to it lying between *syn* and *anti* to the methyl substituents at the *ortho*- and *meta*-positions in *N*-(2,3-dimethylphenyl)methanesulfonamide and the chloro substituents in *N*-(2-chlorophenyl)methanesulfonamide and *N*-(3-chlorophenyl)methanesulfonamide. The bond parameters are similar to those of other methylsulfonanilides, except for some differences in the bond and torsion angles. The amide H atom is available to a receptor molecule during its biological activity, as it lies on one side of the plane of the benzene ring, while the methanesulfonyl group is on the opposite side of the plane, similar to what is observed in other methylsulfonanilides. The molecules in the title compound are packed into chains through N—H...O hydrogen bonding.

Related literature

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



Experimental

Crystal data

$\text{C}_7\text{H}_7\text{Cl}_2\text{NO}_2\text{S}$
 $M_r = 240.10$
 Monoclinic, $P2_1/c$
 $a = 11.1299$ (9) Å
 $b = 5.1365$ (6) Å
 $c = 16.908$ (1) Å
 $\beta = 90.038$ (6)°

$V = 966.61$ (15) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.85$ mm⁻¹
 $T = 303$ (2) K
 $0.50 \times 0.15 \times 0.15$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector
 Absorption correction: none

5750 measured reflections
 1939 independent reflections
 1694 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.152$
 $S = 0.87$
 1939 reflections
 122 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.57$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.71$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N5}-\text{H5N}\cdots\text{O3}^i$	0.86 (4)	2.35 (4)	3.101 (3)	147 (3)
$\text{N5}-\text{H5N}\cdots\text{Cl12}$	0.86 (4)	2.43 (3)	2.937 (2)	118 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2003); 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: LW2022).

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supplementary materials

Acta Cryst. (2007). E63, o3086 [doi:10.1107/S160053680702555X]

***N*-(2,3-Dichlorophenyl)methanesulfonamide**

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

Comment

The structural studies of alkyl sulphonanilides are of interest as their biological activity is thought to be due to the hydrogen of the phenyl N—H portion of the sulphonanilide molecules as it can align itself, in relation to a receptor site. In the present work, the structure of *N*-(2,3-dichlorophenyl)-methanesulfonamide (23DCPMSA) has been determined to explore the substituent effects on the solid state structures of sulfonanilides (Gowda *et al.*, 2007*a-k*). The structure of 23DCPMSA (Fig. 1) resembles those of *N*-(phenyl)-methanesulfonamide (PMSA) (Klug, 1968) and other methylsulfonanilides (Gowda *et al.*, 2007*a-k*). The conformation of the N—H bond in 23DCPMSA is *syn* to both *ortho* and *meta* chloro substituents, in contrast to it lying between *syn* and *anti* conformations to the methyl substituents at *ortho* and *meta* positions, in *N*-(2,3-dimethylphenyl)-methanesulfonamide (23DMPMSA)(Gowda *et al.*, 2007*h*) and chloro substituents in *N*-(2-chlorophenyl)-methanesulfonamide (2CPMSA)(Gowda *et al.*, 2007*k*) and *N*-(3-chlorophenyl)-methanesulfonamide (3CPMSA)(Gowda *et al.*, 2007*e*). Chloro substitutions at both *ortho* and *meta* positions in PMSA do not change its space group, in contrast to change over from monoclinic $P2_1/c$ to orthorhombic $P2_12_12_1$ space group on methyl substitutions at both *ortho* and *meta* positions in PMSA to form 23DMPMSA (Gowda *et al.*, 2007*h*). The bond parameters in 23DCPMSA are similar to those in PMSA, 23DMPMSA and other methylsulfonanilides, except for some difference in the bond and torsional angles. The amide hydrogen is available to a receptor molecule during its biological activity as it sits alone on one side of the plane of the phenyl group, while the whole methanesulfonyl group is on the opposite side of the plane, similar to that in other methylsulfonanilides. The molecules in 23DCPMSA are packed into chains in the direction of *b* axis (Fig. 2) through N—H···O hydrogen bonds (Fig. 3 and 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

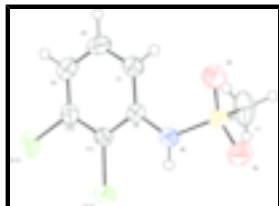


Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

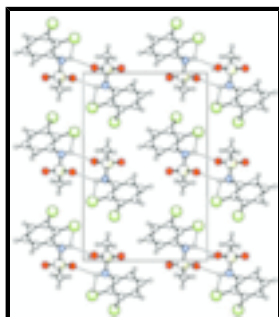


Fig. 2. The crystal packing of the title compound, viewed down the *b* axis.

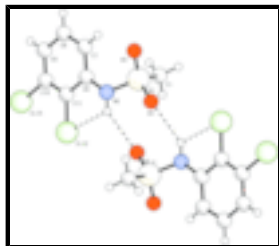


Fig. 3. Hydrogen bonding in the title compound. Hydrogen bonds are shown as dashed lines.

N-(2,3-dichlorophenyl)methanesulfonamide

Crystal data

$C_7H_7Cl_2NO_2S$

$M_r = 240.10$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 11.1299\ (9)\ \text{\AA}$

$b = 5.1365\ (6)\ \text{\AA}$

$c = 16.908\ (1)\ \text{\AA}$

$\beta = 90.038\ (6)^\circ$

$V = 966.61\ (15)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 488$

$D_x = 1.650\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2484 reflections

$\theta = 2.8\text{--}26.5^\circ$

$\mu = 0.85\ \text{mm}^{-1}$

$T = 303\ (2)\ \text{K}$

Long prism, colourless

$0.50 \times 0.15 \times 0.15\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur
diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

Monochromator: graphite

1694 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.058$

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

$T = 303(2)$ K $\theta_{\min} = 4.2^\circ$
 Rotation method data acquisition using ω scans $h = -13 \rightarrow 13$
 Absorption correction: none $k = -3 \rightarrow 6$
 5750 measured reflections $l = -21 \rightarrow 21$
 1939 independent reflections

Refinement

Refinement on F^2 Hydrogen site location: inferred from neighbouring sites
 Least-squares matrix: full H atoms treated by a mixture of independent and constrained refinement
 $R[F^2 > 2\sigma(F^2)] = 0.054$ $w = 1/[\sigma^2(F_o^2) + (0.1248P)^2 + 0.7632P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.152$ $(\Delta/\sigma)_{\max} = 0.011$
 $S = 0.87$ $\Delta\rho_{\max} = 0.57 \text{ e } \text{\AA}^{-3}$
 1939 reflections $\Delta\rho_{\min} = -0.71 \text{ e } \text{\AA}^{-3}$
 122 parameters Extinction correction: SHELXL97 (Sheldrick, 1997),
 $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.016 (4)
 Secondary atom site location: difference Fourier map

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	0.1880 (4)	0.2088 (7)	-0.08851 (19)	0.0657 (10)
H1A	0.1995	0.1405	-0.1408	0.099*
H1B	0.2505	0.3317	-0.0768	0.099*
H1C	0.1114	0.2940	-0.0855	0.099*
C6	0.2632 (2)	0.2694 (5)	0.10012 (14)	0.0341 (5)
C7	0.3847 (3)	0.2686 (6)	0.08165 (17)	0.0439 (6)
H7	0.4140	0.1514	0.0444	0.053*
C8	0.4626 (3)	0.4409 (6)	0.1182 (2)	0.0520 (8)
H8	0.5438	0.4368	0.1054	0.062*
C9	0.4221 (3)	0.6189 (6)	0.17337 (18)	0.0485 (7)

supplementary materials

H9	0.4750	0.7356	0.1970	0.058*
C10	0.3017 (3)	0.6200 (5)	0.19266 (15)	0.0382 (6)
C11	0.2221 (2)	0.4472 (5)	0.15705 (14)	0.0324 (5)
Cl12	0.07252 (6)	0.44392 (16)	0.18443 (4)	0.0490 (3)
Cl13	0.24995 (7)	0.84109 (15)	0.26233 (4)	0.0517 (3)
N5	0.1792 (2)	0.0930 (5)	0.06667 (14)	0.0425 (6)
H5N	0.107 (3)	0.137 (6)	0.077 (2)	0.051*
O3	0.0884 (2)	-0.2047 (4)	-0.02830 (14)	0.0581 (6)
O4	0.3061 (2)	-0.1694 (4)	-0.02436 (14)	0.0543 (6)
S2	0.19291 (6)	-0.04550 (12)	-0.02004 (4)	0.0371 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.106 (3)	0.0489 (17)	0.0418 (16)	0.0056 (18)	-0.0132 (18)	0.0018 (14)
C6	0.0320 (12)	0.0386 (12)	0.0318 (11)	0.0000 (10)	-0.0014 (9)	-0.0023 (10)
C7	0.0360 (14)	0.0521 (16)	0.0437 (14)	0.0054 (11)	0.0032 (11)	-0.0111 (12)
C8	0.0297 (14)	0.067 (2)	0.0591 (19)	0.0001 (12)	0.0020 (13)	-0.0091 (15)
C9	0.0394 (15)	0.0577 (17)	0.0485 (17)	-0.0056 (13)	-0.0093 (12)	-0.0050 (13)
C10	0.0453 (14)	0.0405 (13)	0.0288 (12)	0.0036 (11)	-0.0034 (10)	-0.0032 (10)
C11	0.0302 (12)	0.0393 (13)	0.0276 (11)	0.0039 (9)	-0.0001 (9)	0.0024 (9)
Cl12	0.0334 (4)	0.0687 (5)	0.0449 (5)	0.0027 (3)	0.0076 (3)	-0.0140 (3)
Cl13	0.0633 (5)	0.0521 (5)	0.0395 (4)	0.0029 (3)	-0.0015 (3)	-0.0138 (3)
N5	0.0334 (12)	0.0530 (13)	0.0412 (12)	-0.0036 (10)	0.0063 (10)	-0.0162 (10)
O3	0.0481 (12)	0.0551 (12)	0.0711 (15)	-0.0108 (10)	0.0010 (10)	-0.0263 (11)
O4	0.0479 (12)	0.0562 (12)	0.0588 (13)	0.0178 (10)	-0.0010 (10)	-0.0168 (10)
S2	0.0386 (4)	0.0353 (4)	0.0373 (4)	0.0039 (2)	-0.0014 (3)	-0.0079 (2)

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

C1—S2	1.746 (3)	C8—H8	0.9300
C1—H1A	0.9600	C9—C10	1.380 (4)
C1—H1B	0.9600	C9—H9	0.9300
C1—H1C	0.9600	C10—C11	1.390 (4)
C6—C7	1.388 (4)	C10—Cl13	1.735 (3)
C6—C11	1.404 (3)	C11—Cl12	1.729 (3)
C6—N5	1.420 (3)	N5—S2	1.637 (2)
C7—C8	1.384 (4)	N5—H5N	0.86 (4)
C7—H7	0.9300	O3—S2	1.428 (2)
C8—C9	1.382 (4)	O4—S2	1.413 (2)
S2—C1—H1A	109.5	C10—C9—H9	120.7
S2—C1—H1B	109.5	C11—C10—C9	120.9 (2)
H1A—C1—H1B	109.5	C11—C10—Cl13	120.0 (2)
S2—C1—H1C	109.5	C9—C10—Cl13	119.1 (2)
H1A—C1—H1C	109.5	C10—C11—C6	120.3 (2)
H1B—C1—H1C	109.5	C10—C11—Cl12	120.22 (19)
C7—C6—C11	118.3 (2)	C6—C11—Cl12	119.45 (19)
C7—C6—N5	123.4 (2)	C6—N5—S2	124.90 (19)

C11—C6—N5	118.3 (2)	C6—N5—H5N	112 (2)
C6—C7—C8	120.5 (3)	S2—N5—H5N	113 (2)
C6—C7—H7	119.8	O4—S2—O3	117.55 (14)
C8—C7—H7	119.8	O4—S2—N5	109.03 (13)
C9—C8—C7	121.3 (3)	O3—S2—N5	105.07 (12)
C9—C8—H8	119.3	O4—S2—C1	109.26 (18)
C7—C8—H8	119.3	O3—S2—C1	109.79 (18)
C8—C9—C10	118.6 (3)	N5—S2—C1	105.41 (15)
C8—C9—H9	120.7		
C11—C6—C7—C8	-0.6 (4)	C7—C6—C11—C10	1.0 (4)
N5—C6—C7—C8	-178.0 (3)	N5—C6—C11—C10	178.6 (2)
C6—C7—C8—C9	-0.4 (5)	C7—C6—C11—C112	-177.3 (2)
C7—C8—C9—C10	1.0 (5)	N5—C6—C11—C112	0.3 (3)
C8—C9—C10—C11	-0.5 (4)	C7—C6—N5—S2	-27.1 (4)
C8—C9—C10—C113	179.6 (2)	C11—C6—N5—S2	155.4 (2)
C9—C10—C11—C6	-0.5 (4)	C6—N5—S2—O4	53.1 (3)
C113—C10—C11—C6	179.42 (19)	C6—N5—S2—O3	179.9 (2)
C9—C10—C11—C112	177.8 (2)	C6—N5—S2—C1	-64.1 (3)
C113—C10—C11—C112	-2.3 (3)		

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

<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>
N5—H5N \cdots O3 ⁱ	0.86 (4)	2.35 (4)	3.101 (3)	147 (3)
N5—H5N \cdots C112	0.86 (4)	2.43 (3)	2.937 (2)	118 (3)

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

Fig. 1

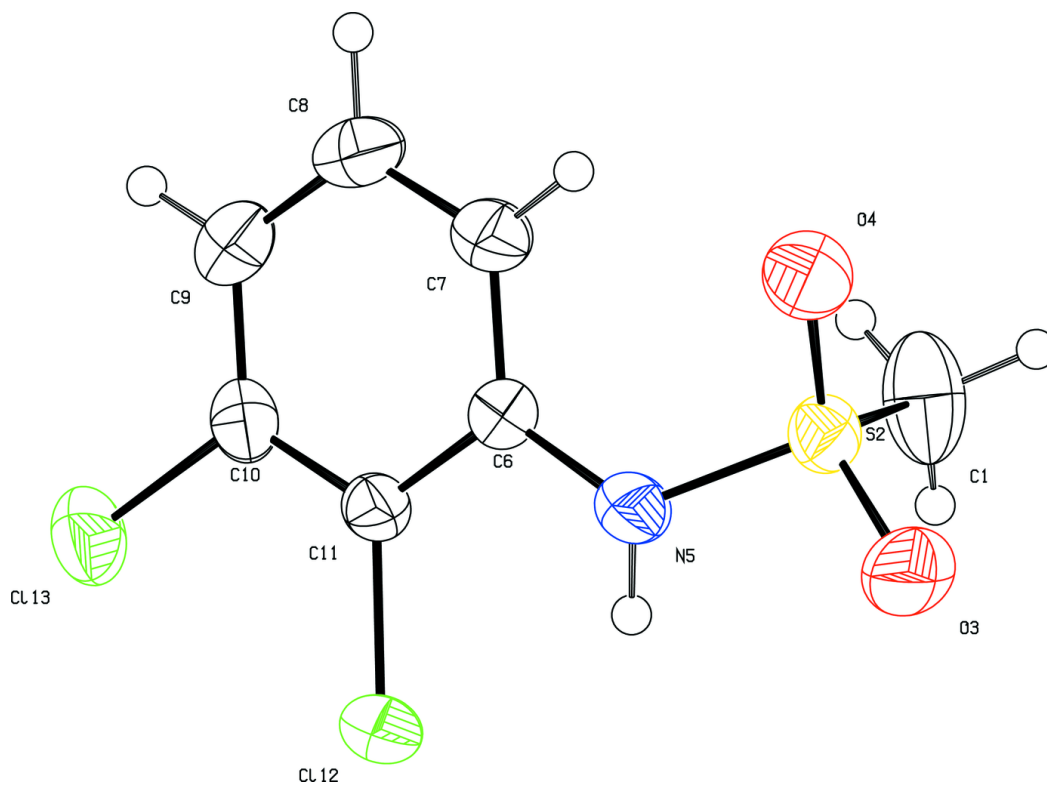


Fig. 2

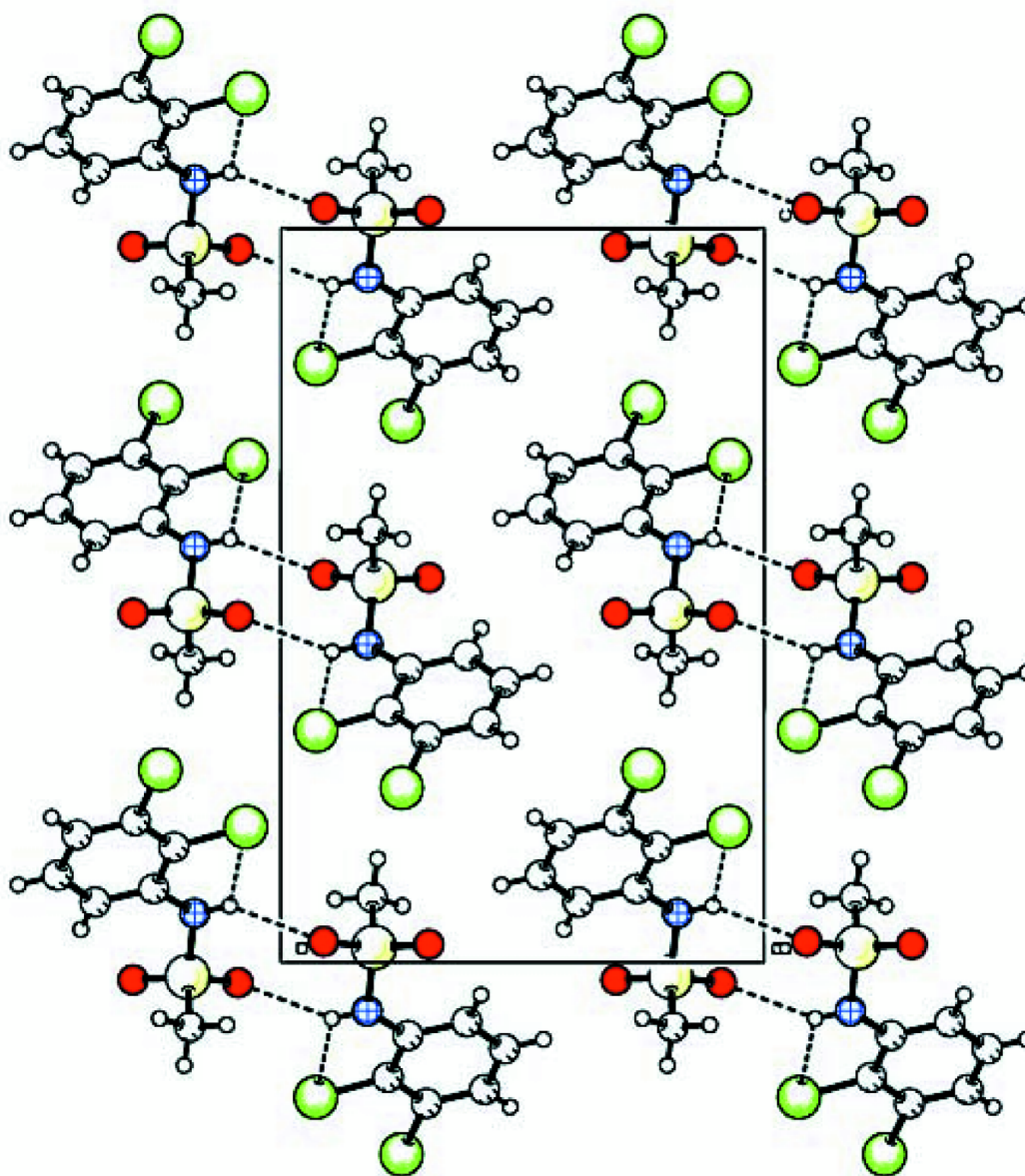


Fig. 3

