

N-(2,5-Dichlorophenyl)methanesulfonamide

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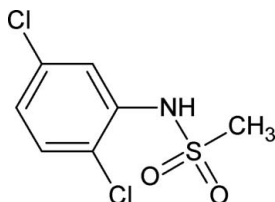
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.048; wR factor = 0.125; data-to-parameter ratio = 12.1.

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 group and *anti* to the *meta*-chloro group, similar to that in *N*-(2,5-dimethylphenyl)methanesulfonamide determined under identical conditions. This is in contrast to the *syn* conformations observed with respect to both *ortho*- and *meta*-chloro substituents in *N*-(2,3-dichlorophenyl)methanesulfonamide and the conformation lying between *syn* and *anti* to the chloro substituents at *ortho* or *meta* positions in *N*-(2-chlorophenyl)methanesulfonamide and *N*-(3-chlorophenyl)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 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 that in other methanesulfonanilides. The molecules in the title compound are packed into chains through N–H...O and N–H...Cl hydrogen bonding.

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, $P\bar{1}$
 $a = 5.8889$ (5) Å
 $b = 8.4810$ (6) Å
 $c = 9.937$ (1) Å
 $\alpha = 95.45$ (1)°
 $\beta = 103.30$ (1)°
 $\gamma = 90.16$ (1)°
 $V = 480.64$ (7) Å³
 $Z = 2$
 Cu $K\alpha$ radiation
 $\mu = 7.85$ mm⁻¹
 $T = 299$ (2) K
 $0.10 \times 0.10 \times 0.08$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.411$, $T_{\max} = 0.534$
 1804 measured reflections
 1683 independent reflections
 1271 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.051$
 3 standard reflections
 frequency: 120 min
 intensity decay: 1.2%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.125$
 $S = 1.03$
 1683 reflections
 139 parameters
 1 restraint
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.28$ e Å⁻³
 $\Delta\rho_{\min} = -0.47$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
N5—H5N...O3 ⁱ	0.857 (10)	2.13 (2)	2.923 (4)	153 (4)
N5—H5N...Cl12	0.857 (10)	2.63 (4)	3.012 (4)	108 (3)

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

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: LW2024).

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supplementary materials

Acta Cryst. (2007). E63, o3088-o3089 [doi:10.1107/S160053680702569X]

***N*-(2,5-Dichlorophenyl)methanesulfonamide**

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

Comment

The structural studies of sulfonanilides are of interest as their biological activity is thought to be due to the amide hydrogen portion of the molecules as it can align itself in relation to a receptor site. In the present work, the structure of *N*-(2,5-dichlorophenyl)-methanesulfonamide (25DCPMSA) has been determined to explore the substituent effects on the solid state structures of sulfonanilides (Gowda *et al.*, 2007*a-m*). The structure of 25DCPMSA (Fig. 1) is similar to those of *N*-(phenyl)-methanesulfonamide (PMSA) (Klug, 1968), *N*-(2-chlorophenyl)-methanesulfonamide (2CPMSA) (Gowda *et al.*, 2007*m*), *N*-(2,3-dichlorophenyl)-methanesulfonamide (23DCPMSA) (Gowda *et al.*, 2007*k*), *N*-(2,5-dimethylphenyl)-methanesulfonamide (25DMPMSA) (Gowda *et al.*, 2007*m*) and other alkyl sulfonanilides (Gowda *et al.*, 2007*a-i*). The conformation of the N—H bond in 25DCPMSA is nearly *syn* to the *ortho* chloro group and *anti* to the *meta* chloro group, similar to that in 25DMPMSA determined under identical conditions (Gowda *et al.*, 2007*m*). This is in contrast to the *syn* conformations observed with respect to both *ortho* and *meta* chloro substituents in 23DCPMSA (Gowda *et al.*, 2007*k*) and the conformation lying between *syn* and *anti* conformations to the chloro substituents at *ortho* or *meta* positions in 2CPMSA (Gowda *et al.*, 2007*m*) and *N*-(3-chlorophenyl)-methanesulfonamide (3CPMSA) (Gowda *et al.*, 2007*e*). 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 (Gowda *et al.*, 2007*d,m,k*). But the Substitution of an additional chloro group in the second *meta* position of 2CPMSA to produce 25DCPMSA does not further alter the space group, in contrast to the change over from triclinic P-1 to monoclinic $P2_1/c$ on Substitution of an additional methyl group at the second *meta* position in *N*-(2-methylphenyl)-methanesulfonamide (2MPMSA) to form 25DMPMSA (Gowda *et al.*, 2007*m*). Further, monoclinic $P2_1/c$ space group is observed with 23DCPMSA (Gowda *et al.*, 2007*k*). The geometric parameters in 25DCPMSA are similar to those in PMSA, 2CPMSA, 23DCPMSA, 25DMPMSA and other methanesulfonanilides except for some difference in the bond and torsional angles. The amide hydrogen 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 alkyl sulfonanilides. The amide hydrogen is thus available to a receptor molecule during its biological activity. The molecules in 25DCPMSA are packed into chains in the direction of *b* axis (Fig. 2) through N—H \cdots O and N—H \cdots Cl 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 atoms were located in difference map and their positions refined, with $U_{\text{iso}} = 1.2 U_{\text{eq}}$ of the parent atom.

Figures

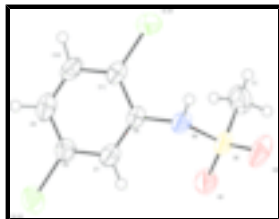


Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

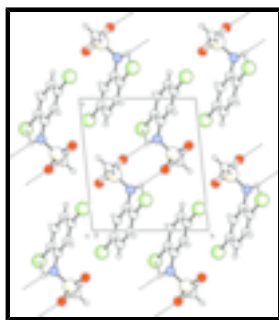


Fig. 2. The crystal packing of the title compound, viewed down the *b* axis.

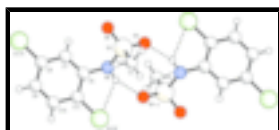


Fig. 3. Hydrogen bonding in the title compound. Hydrogen bonds are shown as dashed lines.

N-(2,5-dichlorophenyl)methanesulfonamide

Crystal data

$C_7H_7Cl_2NO_2S$

$M_r = 240.10$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 5.8889$ (5) Å

$b = 8.4810$ (6) Å

$c = 9.9370$ (10) Å

$\alpha = 95.450$ (10)°

$\beta = 103.300$ (10)°

$\gamma = 90.160$ (10)°

$V = 480.64$ (7) Å³

$Z = 2$

$F_{000} = 244$

$D_x = 1.659$ Mg m⁻³

Cu $K\alpha$ radiation

$\lambda = 1.54180$ Å

Cell parameters from 25 reflections

$\theta = 6.6$ – 23.8 °

$\mu = 7.85$ mm⁻¹

$T = 299$ (2) K

Block, colourless

$0.10 \times 0.10 \times 0.08$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 299$ (2) K

$\omega/2\theta$ scans

$R_{int} = 0.051$

$\theta_{max} = 66.9$ °

$\theta_{min} = 4.6$ °

$h = -6 \rightarrow 7$

$k = -10 \rightarrow 10$

Absorption correction: Psi-scan
(North *et al.*, 1968) $l = -11 \rightarrow 1$
 $T_{\min} = 0.411$, $T_{\max} = 0.534$ 3 standard reflections
 1804 measured reflections every 120 min
 1683 independent reflections intensity decay: 1.2%
 1271 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
 $R[F^2 > 2\sigma(F^2)] = 0.048$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.125$ $w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.4897P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.03$ $(\Delta/\sigma)_{\max} = 0.018$
 1683 reflections $\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$
 139 parameters $\Delta\rho_{\min} = -0.47 \text{ e } \text{\AA}^{-3}$
 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2881 (10)	0.1936 (7)	0.4766 (6)	0.0609 (14)
H1A	0.312 (10)	0.114 (6)	0.549 (6)	0.073*
H1B	0.370 (10)	0.286 (7)	0.510 (6)	0.073*
H1C	0.323 (10)	0.138 (6)	0.395 (6)	0.073*
C6	-0.0009 (7)	0.3162 (4)	0.1698 (4)	0.0323 (8)
C7	-0.1735 (8)	0.2222 (5)	0.0805 (4)	0.0381 (9)
H7	-0.305 (8)	0.192 (5)	0.106 (5)	0.046*
C8	-0.1542 (8)	0.1785 (5)	-0.0540 (4)	0.0396 (9)
C9	0.0319 (9)	0.2282 (5)	-0.1024 (4)	0.0487 (11)
H9	0.028 (9)	0.204 (5)	-0.196 (5)	0.058*

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C10	0.2017 (9)	0.3234 (5)	-0.0152 (4)	0.0456 (11)
H10	0.344 (9)	0.354 (5)	-0.036 (5)	0.055*
C11	0.1899 (7)	0.3666 (4)	0.1211 (4)	0.0370 (9)
N5	-0.0228 (7)	0.3644 (4)	0.3072 (3)	0.0387 (8)
H5N	0.042 (7)	0.455 (3)	0.338 (4)	0.046*
O3	-0.0774 (7)	0.3234 (4)	0.5373 (3)	0.0672 (11)
O4	-0.1353 (6)	0.0990 (4)	0.3569 (3)	0.0585 (9)
S2	-0.0073 (2)	0.23883 (12)	0.42304 (10)	0.0421 (3)
Cl12	0.4089 (2)	0.48370 (14)	0.23125 (12)	0.0552 (3)
Cl13	-0.3795 (2)	0.06441 (14)	-0.16473 (11)	0.0585 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.066 (4)	0.069 (3)	0.044 (3)	0.000 (3)	0.000 (2)	0.014 (2)
C6	0.041 (2)	0.0312 (18)	0.0277 (18)	0.0049 (16)	0.0132 (16)	0.0047 (14)
C7	0.044 (3)	0.038 (2)	0.035 (2)	-0.0001 (18)	0.0144 (18)	0.0044 (17)
C8	0.049 (3)	0.037 (2)	0.033 (2)	0.0007 (18)	0.0107 (18)	0.0021 (16)
C9	0.070 (3)	0.048 (2)	0.033 (2)	0.006 (2)	0.022 (2)	0.0046 (18)
C10	0.045 (3)	0.056 (3)	0.043 (2)	0.000 (2)	0.024 (2)	0.008 (2)
C11	0.043 (3)	0.035 (2)	0.037 (2)	0.0021 (17)	0.0161 (18)	0.0083 (16)
N5	0.055 (2)	0.0314 (17)	0.0336 (17)	-0.0078 (15)	0.0196 (16)	-0.0023 (14)
O3	0.115 (3)	0.0538 (19)	0.0468 (19)	-0.007 (2)	0.051 (2)	-0.0047 (15)
O4	0.085 (3)	0.0476 (17)	0.0419 (17)	-0.0273 (17)	0.0133 (16)	0.0029 (14)
S2	0.0621 (7)	0.0392 (5)	0.0283 (5)	-0.0106 (5)	0.0193 (4)	-0.0022 (4)
Cl12	0.0469 (7)	0.0661 (7)	0.0535 (7)	-0.0147 (5)	0.0155 (5)	0.0020 (5)
Cl13	0.0740 (9)	0.0577 (7)	0.0393 (6)	-0.0099 (6)	0.0091 (5)	-0.0080 (5)

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

C1—S2	1.753 (6)	C8—Cl13	1.739 (4)
C1—H1A	1.02 (6)	C9—C10	1.367 (7)
C1—H1B	0.91 (6)	C9—H9	0.93 (5)
C1—H1C	0.96 (6)	C10—C11	1.386 (6)
C6—C7	1.380 (6)	C10—H10	0.95 (5)
C6—C11	1.402 (5)	C11—Cl12	1.728 (4)
C6—N5	1.422 (4)	N5—S2	1.629 (3)
C7—C8	1.384 (5)	N5—H5N	0.857 (10)
C7—H7	0.91 (5)	O3—S2	1.427 (3)
C8—C9	1.374 (6)	O4—S2	1.423 (3)
S2—C1—H1A	111 (3)	C8—C9—H9	118 (3)
S2—C1—H1B	108 (4)	C9—C10—C11	120.7 (4)
H1A—C1—H1B	112 (5)	C9—C10—H10	125 (3)
S2—C1—H1C	104 (3)	C11—C10—H10	114 (3)
H1A—C1—H1C	105 (4)	C10—C11—C6	120.1 (4)
H1B—C1—H1C	117 (5)	C10—C11—Cl12	120.2 (3)
C7—C6—C11	118.9 (3)	C6—C11—Cl12	119.7 (3)
C7—C6—N5	119.8 (3)	C6—N5—S2	121.9 (3)

C11—C6—N5	121.3 (4)	C6—N5—H5N	112 (3)
C6—C7—C8	119.6 (4)	S2—N5—H5N	115 (3)
C6—C7—H7	122 (3)	O4—S2—O3	118.8 (2)
C8—C7—H7	118 (3)	O4—S2—N5	107.90 (18)
C9—C8—C7	121.7 (4)	O3—S2—N5	105.64 (18)
C9—C8—C113	120.1 (3)	O4—S2—C1	108.9 (3)
C7—C8—C113	118.2 (3)	O3—S2—C1	108.4 (3)
C10—C9—C8	119.0 (4)	N5—S2—C1	106.5 (2)
C10—C9—H9	123 (3)		
C11—C6—C7—C8	0.5 (6)	C7—C6—C11—C10	0.8 (6)
N5—C6—C7—C8	178.6 (4)	N5—C6—C11—C10	-177.2 (4)
C6—C7—C8—C9	-0.9 (6)	C7—C6—C11—C112	-179.7 (3)
C6—C7—C8—C113	-178.2 (3)	N5—C6—C11—C112	2.3 (5)
C7—C8—C9—C10	-0.1 (7)	C7—C6—N5—S2	68.5 (5)
C113—C8—C9—C10	177.2 (3)	C11—C6—N5—S2	-113.6 (4)
C8—C9—C10—C11	1.4 (7)	C6—N5—S2—O4	-42.0 (4)
C9—C10—C11—C6	-1.8 (7)	C6—N5—S2—O3	-170.0 (3)
C9—C10—C11—C112	178.7 (4)	C6—N5—S2—C1	74.8 (4)

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	0.857 (10)	2.13 (2)	2.923 (4)	153 (4)
N5—H5N \cdots C112	0.857 (10)	2.63 (4)	3.012 (4)	108 (3)

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

Fig. 1

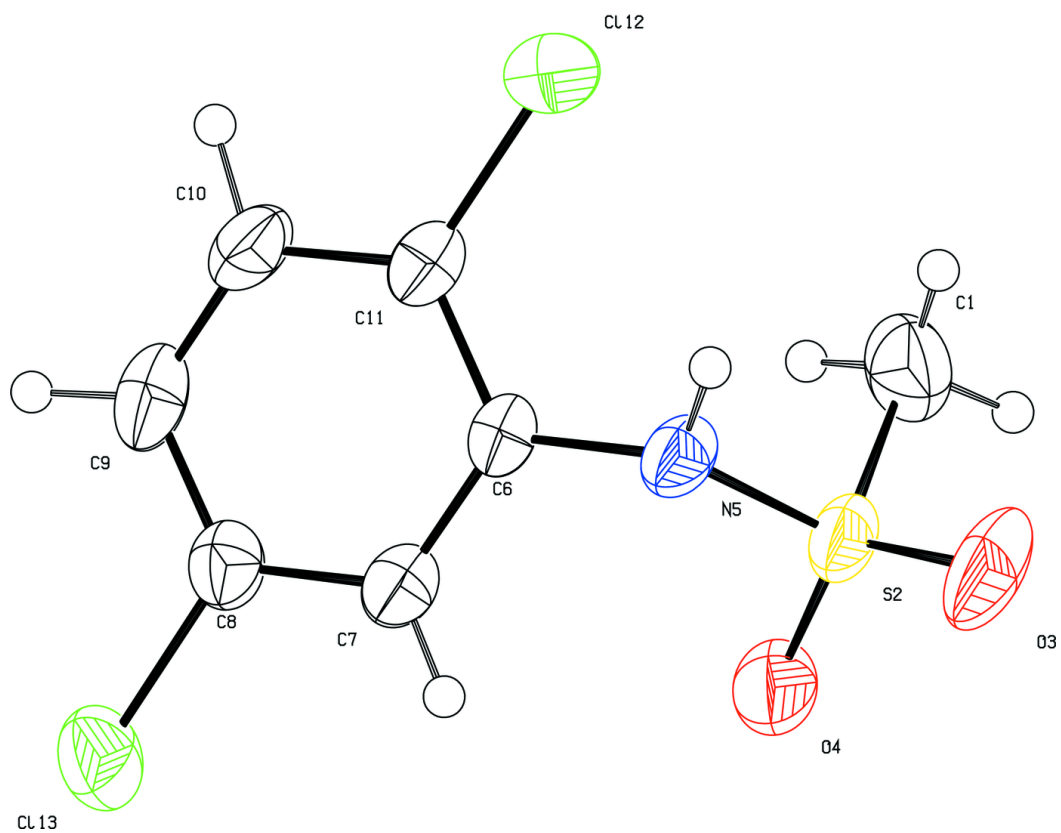


Fig. 2

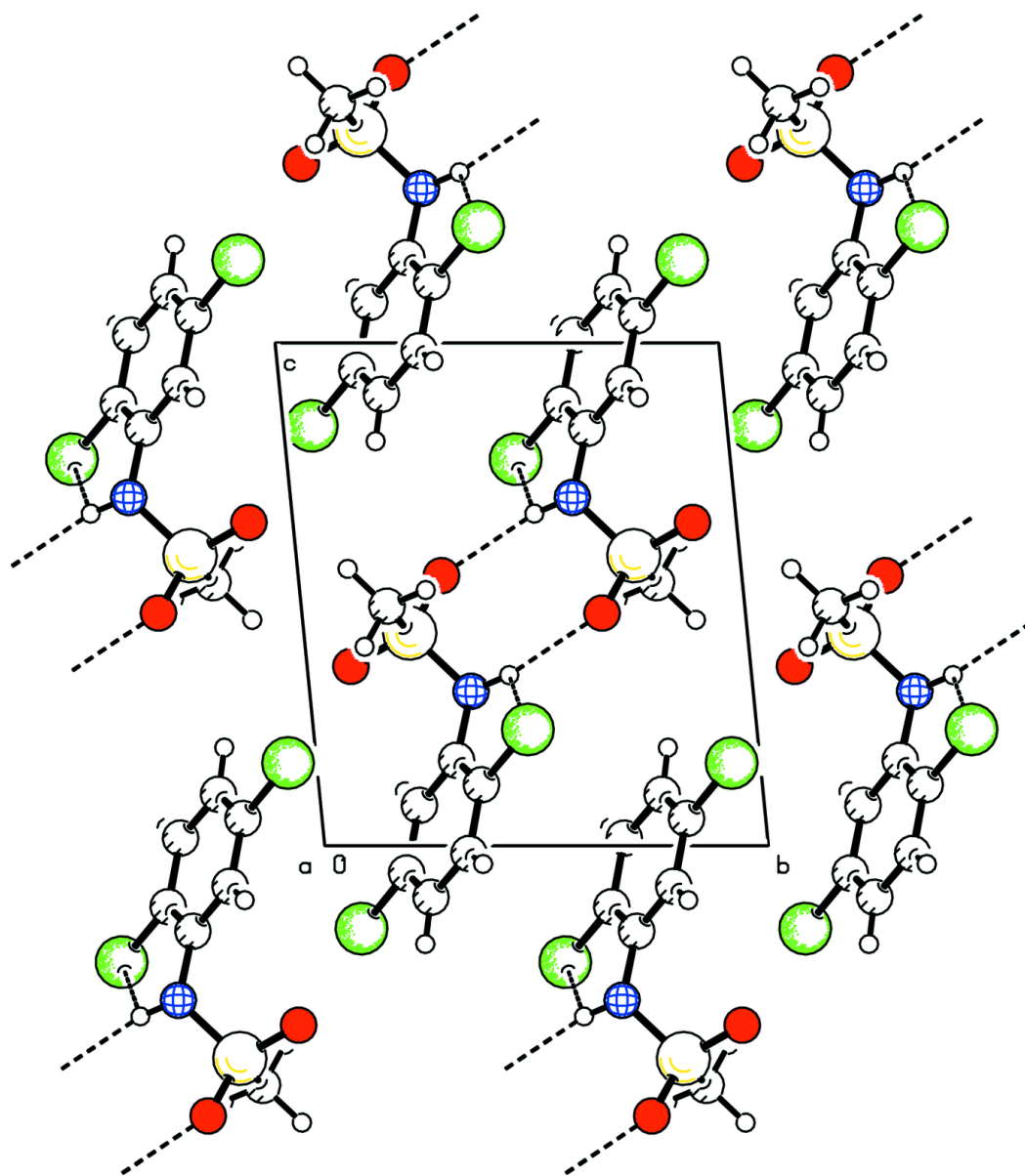


Fig. 3

