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4-Chloro-*N*-(2,3-dichlorophenyl)-2-methylbenzenesulfonamide

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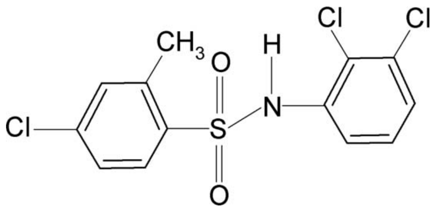
Key indicators: single-crystal X-ray study; *T* = 293 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.035; *wR* factor = 0.097; data-to-parameter ratio = 16.1.

The torsion angle of the C—SO$_2$—NH—C moiety in the title compound, $C_{13}H_{10}Cl_3NO_2S$, is 50.4 (2)°. The sulfonyl and aniline benzene rings are tilted relative to each other by 69.6 (1)°. The crystal structure is stabilized by N—H···O hydrogen bonds, linking the molecules into zigzag chains parallel to the *b* axis.

Related literature

For the preparation of the title compound, see: Savitha & Gowda (2006). For hydrogen-bonding preferences of sulfonamides, see: Adsmond & Grant (2001). For studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Gowda *et al.* (2000). For *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007). For *N*-(aryl)-arylsulfonamides, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006); Rodrigues *et al.* (2011); Shetty & Gowda (2005). For *N*-(chloro)-arylsulfonamides, see: Gowda & Kumar (2003).



Experimental

Crystal data

$C_{13}H_{10}Cl_3NO_2S$
M_r = 350.63
Monoclinic, $P2_1/c$
a = 9.2007 (9) Å
b = 9.8803 (9) Å
c = 16.163 (2) Å
β = 99.286 (9)°
V = 1450.1 (3) Å^3
Z = 4
Mo *K*α radiation
μ = 0.77 mm^{-1}
T = 293 K
0.48 × 0.44 × 0.36 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.708, T_{max} = 0.768
5387 measured reflections
2966 independent reflections
2381 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.097
S = 1.03
2966 reflections
184 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O2^i	0.85	2.64	3.446 (2)	161

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); 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, 2009); 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: BT5680).

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4-Chloro-*N*-(2,3-dichlorophenyl)-2-methylbenzenesulfonamide

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S1. Comment

The sulfonamide moiety is the constituent of many biologically significant compounds. The hydrogen bonding preferences of sulfonamides have been investigated (Adsmond & Grant, 2001). As part of our studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2000), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007), *N*-(aryl)-arylsulfonamides (Rodrigues *et al.*, 2011; Shetty & Gowda, 2005) and *N*-(chloro)-arylsulfonamides (Gowda & Kumar, 2003), in the present work, the crystal structure of 4-Chloro-2-methyl-*N*-(2,3-dichlorophenyl)benzenesulfonamide (I) has been determined (Fig. 1).

In (I), the conformation of the N—C bond in the C—SO_2—NH—C segment has *trans* and *gauche* torsion angles with the S═O bonds. Further, the the N—H bond is *syn* to the *ortho*-methyl group in the sulfonyl benzene ring as well as to the *ortho*-Cl and *meta*-Cl atoms in the anilino ring of (I). The molecule is bent at the S atom with the C—SO_2—NH—C torsion angle of 50.35 (20)°, compared to the value of -52.0 (2)° in 2,4-Dichloro-*N*-(2,3-dichlorophenyl)benzenesulfonamide (II) (Rodrigues *et al.*, 2011).

The sulfonyl and the aniline benzene rings are tilted relative to each other by 69.6 (1)°, compared to the value of 67.7 (1)° in (II).

The other bond parameters in (I) are similar to those observed in (II) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007).

The crystal structure is stabilized by N—H···O hydrogen bonds linking the molecules to zigzag chains parallel to the b-axis. Part of the crystal structure is shown in Fig. 2.

S2. Experimental

The solution of *m*-chlorotoluene (10 ml) in chloroform (40 ml) was treated dropwise with chlorosulfonic acid (25 ml) at 0 ° C. After the initial evolution of hydrogen chloride subsided, the reaction mixture was brought to room temperature and poured into crushed ice in a beaker. The chloroform layer was separated, washed with cold water and allowed to evaporate slowly. The residual 2-methyl-4-chlorobenzenesulfonylchloride was treated with 2,3-dichlorolaniline in the stoichiometric ratio and boiled for ten minutes. The reaction mixture was then cooled to room temperature and added to ice cold water (100 ml). The resultant solid 4-chloro-2-methyl-*N*- (2,3-dichlorophenyl)-benzenesulfonamide was filtered under suction and washed thoroughly with cold water. It was then recrystallized to constant melting point from dilute ethanol. The purity of the compound was checked and characterized by recording its infrared and NMR spectra (Savitha & Gowda, 2006).

Prism like colourless single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and its coordinates were refined with the N—H distance restrained to 0.86 (2) %A. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93Å and methyl C—H = 0.96 Å. All H atoms were refined with isotropic displacement parameters. The U_{iso}(H) values were set at 1.2U_{eq}(C-aromatic, N) and 1.5U_{eq}(C-methyl).

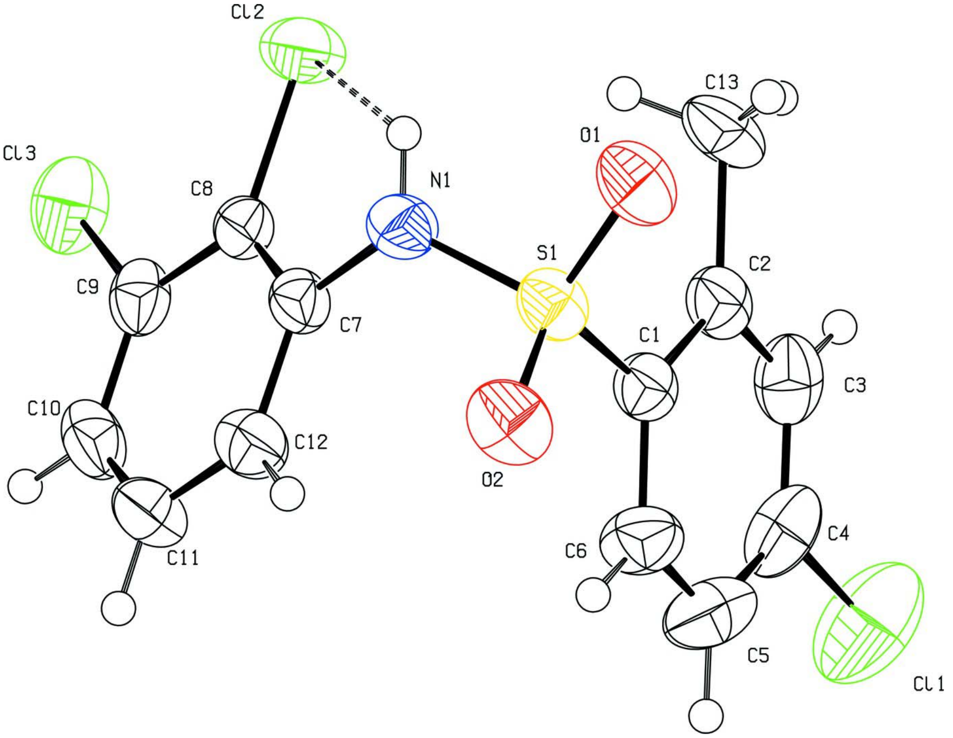

Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level.

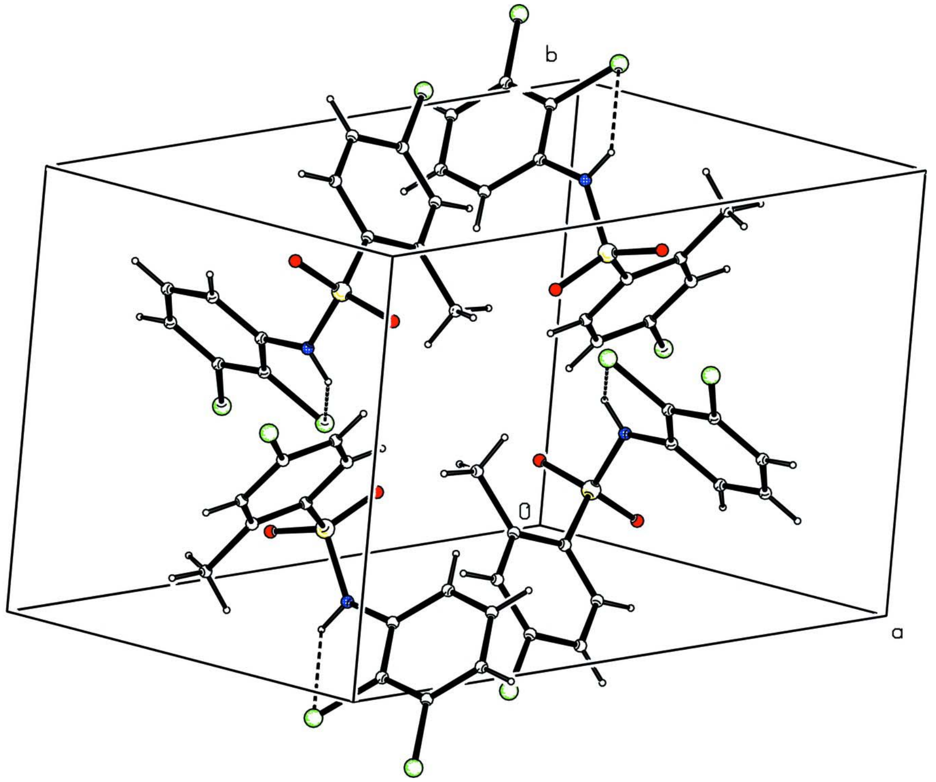

Figure 2

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

4-Chloro-*N*-(2,3-dichlorophenyl)-2-methylbenzenesulfonamide

Crystal data

$C_{13}H_{10}Cl_3NO_2S$
$M_r = 350.63$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.2007$ (9) Å
$b = 9.8803$ (9) Å
$c = 16.163$ (2) Å
$\beta = 99.286$ (9)°
$V = 1450.1$ (3) Å^3
$Z = 4$
$F(000) = 712$
$D_x = 1.606$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2185 reflections
$\theta = 2.6$–27.8°
$\mu = 0.77$ mm^{-1}
$T = 293$ K
Prism, colourless
0.48 × 0.44 × 0.36 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
Rotation method data acquisition using ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
$T_{min} = 0.708$, $T_{max} = 0.768$
5387 measured reflections
2966 independent reflections
2381 reflections with $I > 2\sigma(I)$
$R_{int} = 0.011$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.6°$
$h = -11 \rightarrow 7$
$k = -12 \rightarrow 5$
$l = -14 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.097$
$S = 1.03$
2966 reflections
184 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.5987P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	0.6255 (2)	0.1548 (2)	0.37135 (13)	0.0382 (4)
C2	0.5972 (2)	0.1912 (2)	0.45097 (13)	0.0420 (5)
C3	0.6623 (3)	0.1129 (2)	0.51881 (15)	0.0515 (6)
H3	0.6447	0.1335	0.5724	0.062*
C4	0.7520 (3)	0.0060 (3)	0.50742 (16)	0.0575 (6)
C5	0.7808 (3)	−0.0285 (3)	0.42980 (18)	0.0643 (7)
H5	0.8429	−0.1006	0.4233	0.077*
C6	0.7158 (3)	0.0459 (2)	0.36098 (16)	0.0519 (6)
H6	0.7329	0.0226	0.3076	0.062*
C7	0.7939 (2)	0.4027 (2)	0.29919 (12)	0.0357 (4)
C8	0.8590 (2)	0.5020 (2)	0.35427 (12)	0.0362 (4)
C9	1.0112 (2)	0.5177 (2)	0.36816 (13)	0.0405 (5)
C10	1.0985 (2)	0.4344 (3)	0.32855 (14)	0.0483 (6)
H10	1.2003	0.4445	0.3384	0.058*
C11	1.0338 (2)	0.3359 (3)	0.27425 (15)	0.0517 (6)
H11	1.0928	0.2796	0.2476	0.062*
C12	0.8825 (2)	0.3198 (2)	0.25888 (14)	0.0466 (5)
H12	0.8402	0.2537	0.2217	0.056*
C13	0.5027 (3)	0.3141 (3)	0.46785 (15)	0.0528 (6)
H13A	0.4052	0.3043	0.4367	0.063*
H13B	0.5465	0.3955	0.4508	0.063*
H13C	0.4975	0.3189	0.5266	0.063*
N1	0.63792 (19)	0.39045 (18)	0.28387 (12)	0.0434 (4)
H1N	0.593 (2)	0.459 (2)	0.2983 (15)	0.052*

O1	0.40072 (16)	0.28154 (19)	0.28184 (11)	0.0558 (4)
O2	0.58495 (18)	0.16949 (19)	0.21024 (10)	0.0552 (4)
Cl1	0.83346 (12)	−0.08759 (9)	0.59340 (6)	0.1014 (3)
Cl2	0.75060 (6)	0.60530 (6)	0.40496 (4)	0.05444 (18)
Cl3	1.09147 (7)	0.64184 (7)	0.43607 (4)	0.05939 (19)
S1	0.55050 (5)	0.24510 (6)	0.27992 (3)	0.04145 (16)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0350 (10)	0.0377 (11)	0.0424 (11)	−0.0063 (8)	0.0075 (8)	−0.0061 (9)
C2	0.0361 (11)	0.0435 (12)	0.0471 (12)	−0.0097 (9)	0.0092 (9)	−0.0092 (10)
C3	0.0536 (14)	0.0553 (14)	0.0454 (12)	−0.0198 (11)	0.0072 (10)	−0.0058 (11)
C4	0.0669 (16)	0.0425 (13)	0.0581 (15)	−0.0162 (12)	−0.0052 (12)	0.0066 (12)
C5	0.0714 (18)	0.0377 (13)	0.0811 (19)	0.0072 (12)	0.0043 (14)	−0.0030 (13)
C6	0.0614 (15)	0.0413 (12)	0.0536 (14)	0.0035 (11)	0.0114 (11)	−0.0101 (11)
C7	0.0299 (9)	0.0394 (11)	0.0382 (10)	0.0023 (8)	0.0064 (8)	0.0067 (9)
C8	0.0366 (10)	0.0351 (10)	0.0385 (10)	0.0038 (8)	0.0102 (8)	0.0084 (9)
C9	0.0359 (10)	0.0446 (12)	0.0400 (11)	−0.0040 (9)	0.0027 (8)	0.0117 (9)
C10	0.0295 (10)	0.0634 (15)	0.0532 (13)	0.0026 (10)	0.0108 (9)	0.0152 (12)
C11	0.0422 (12)	0.0577 (15)	0.0594 (14)	0.0086 (11)	0.0204 (10)	0.0010 (12)
C12	0.0451 (12)	0.0482 (13)	0.0486 (12)	0.0022 (10)	0.0142 (10)	−0.0042 (10)
C13	0.0462 (12)	0.0673 (16)	0.0492 (13)	0.0031 (11)	0.0207 (10)	−0.0198 (12)
N1	0.0309 (9)	0.0420 (10)	0.0571 (11)	0.0032 (8)	0.0064 (8)	0.0000 (9)
O1	0.0284 (7)	0.0726 (12)	0.0650 (10)	−0.0027 (7)	0.0030 (7)	−0.0022 (9)
O2	0.0537 (9)	0.0688 (11)	0.0425 (9)	−0.0053 (8)	0.0058 (7)	−0.0155 (8)
Cl1	0.1241 (8)	0.0777 (6)	0.0892 (6)	−0.0093 (5)	−0.0225 (5)	0.0294 (5)
Cl2	0.0470 (3)	0.0491 (3)	0.0684 (4)	0.0072 (2)	0.0129 (3)	−0.0117 (3)
Cl3	0.0540 (3)	0.0640 (4)	0.0561 (4)	−0.0173 (3)	−0.0032 (3)	0.0018 (3)
S1	0.0315 (3)	0.0493 (3)	0.0426 (3)	−0.0035 (2)	0.0030 (2)	−0.0064 (2)

Geometric parameters (Å, º)

C1—C6	1.386 (3)	C8—Cl2	1.724 (2)
C1—C2	1.401 (3)	C9—C10	1.377 (3)
C1—S1	1.768 (2)	C9—Cl3	1.730 (2)
C2—C3	1.396 (3)	C10—C11	1.379 (3)
C2—C13	1.543 (3)	C10—H10	0.9300
C3—C4	1.371 (4)	C11—C12	1.383 (3)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.366 (4)	C12—H12	0.9300
C4—Cl1	1.735 (3)	C13—H13A	0.9600
C5—C6	1.386 (4)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600
C6—H6	0.9300	N1—S1	1.6422 (19)
C7—C12	1.389 (3)	N1—H1N	0.847 (16)
C7—C8	1.393 (3)	O1—S1	1.4295 (16)
C7—N1	1.422 (2)	O2—S1	1.4291 (17)

C8—C9	1.391 (3)		

C6—C1—C2	121.0 (2)	C8—C9—Cl3	119.82 (17)
C6—C1—S1	116.83 (17)	C9—C10—C11	119.6 (2)
C2—C1—S1	122.18 (17)	C9—C10—H10	120.2
C3—C2—C1	117.3 (2)	C11—C10—H10	120.2
C3—C2—C13	118.7 (2)	C10—C11—C12	120.9 (2)
C1—C2—C13	124.0 (2)	C10—C11—H11	119.6
C4—C3—C2	120.9 (2)	C12—C11—H11	119.6
C4—C3—H3	119.6	C11—C12—C7	119.8 (2)
C2—C3—H3	119.6	C11—C12—H12	120.1
C5—C4—C3	121.7 (2)	C7—C12—H12	120.1
C5—C4—Cl1	118.7 (2)	C2—C13—H13A	109.5
C3—C4—Cl1	119.6 (2)	C2—C13—H13B	109.5
C4—C5—C6	118.9 (2)	H13A—C13—H13B	109.5
C4—C5—H5	120.6	C2—C13—H13C	109.5
C6—C5—H5	120.6	H13A—C13—H13C	109.5
C5—C6—C1	120.3 (2)	H13B—C13—H13C	109.5
C5—C6—H6	119.9	C7—N1—S1	123.75 (15)
C1—C6—H6	119.9	C7—N1—H1N	114.1 (17)
C12—C7—C8	119.38 (18)	S1—N1—H1N	116.9 (17)
C12—C7—N1	121.35 (19)	O2—S1—O1	119.07 (10)
C8—C7—N1	119.24 (18)	O2—S1—N1	108.56 (11)
C9—C8—C7	119.94 (19)	O1—S1—N1	104.29 (10)
C9—C8—Cl2	120.11 (17)	O2—S1—C1	106.77 (10)
C7—C8—Cl2	119.94 (15)	O1—S1—C1	110.90 (10)
C10—C9—C8	120.4 (2)	N1—S1—C1	106.62 (9)
C10—C9—Cl3	119.81 (17)		

C6—C1—C2—C3	−0.5 (3)	C7—C8—C9—Cl3	179.56 (15)
S1—C1—C2—C3	−179.90 (15)	Cl2—C8—C9—Cl3	−0.3 (2)
C6—C1—C2—C13	177.7 (2)	C8—C9—C10—C11	0.6 (3)
S1—C1—C2—C13	−1.7 (3)	Cl3—C9—C10—C11	−179.77 (17)
C1—C2—C3—C4	0.9 (3)	C9—C10—C11—C12	0.2 (4)
C13—C2—C3—C4	−177.4 (2)	C10—C11—C12—C7	−0.7 (4)
C2—C3—C4—C5	−0.2 (4)	C8—C7—C12—C11	0.5 (3)
C2—C3—C4—Cl1	179.02 (17)	N1—C7—C12—C11	179.0 (2)
C3—C4—C5—C6	−0.8 (4)	C12—C7—N1—S1	45.0 (3)
Cl1—C4—C5—C6	179.9 (2)	C8—C7—N1—S1	−136.50 (18)
C4—C5—C6—C1	1.2 (4)	C7—N1—S1—O2	−64.3 (2)
C2—C1—C6—C5	−0.5 (4)	C7—N1—S1—O1	167.74 (17)
S1—C1—C6—C5	178.9 (2)	C7—N1—S1—C1	50.3 (2)
C12—C7—C8—C9	0.3 (3)	C6—C1—S1—O2	8.5 (2)
N1—C7—C8—C9	−178.25 (18)	C2—C1—S1—O2	−172.06 (17)
C12—C7—C8—Cl2	−179.84 (16)	C6—C1—S1—O1	139.71 (18)
N1—C7—C8—Cl2	1.6 (3)	C2—C1—S1—O1	−40.9 (2)
C7—C8—C9—C10	−0.8 (3)	C6—C1—S1—N1	−107.36 (18)
Cl2—C8—C9—C10	179.27 (16)	C2—C1—S1—N1	72.04 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2^{i}	0.85	2.64	3.446 (2)	161

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.