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2,4-Dichloro-*N*-(3-methylphenyl)-benzenesulfonamide

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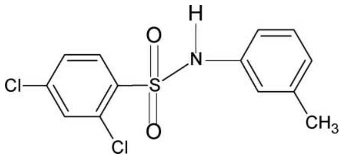
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.097; data-to-parameter ratio = 16.6.

In the title compound, $C_{13}H_{11}Cl_2NO_2S$, the conformations of the N—C bonds in the C—SO_2—NH—C segments have *gauche* torsions with respect to the S=O bonds. The dihedral angle between the two benzene rings is 68.6 (1)°. The crystal structure features inversion dimers linked by pairs of N—H···O hydrogen bonds.

Related literature

For the preparation of the title compound, see: Savitha & Gowda (2006). For our studies of the effect of substituents on the structures of *N*-(aryl)arylsulfonamides, see: Gowda *et al.* (2010*a*,*b*,*c*). For related structures, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006).



Experimental

Crystal data

$C_{13}H_{11}Cl_2NO_2S$
M_r = 316.19
Monoclinic, $P2_1/c$
a = 7.9031 (7) Å
b = 14.507 (1) Å
c = 12.715 (1) Å
β = 99.895 (8)°
V = 1436.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.59 mm^{-1}
T = 299 K
0.36 × 0.24 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
$T_{\min}$ = 0.815, $T_{\max}$ = 0.891
5737 measured reflections
2915 independent reflections
2389 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.012

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.03
2915 reflections
176 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.41 e Å^{-3}
$\Delta\rho_{\min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1$^{\rm i}$	0.85 (1)	2.17 (1)	2.940 (2)	151 (2)

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2216).

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supporting information

Acta Cryst. (2010). E**66**, o1520 [doi:10.1107/S1600536810020106]

2,4-Dichloro-*N*-(3-methylphenyl)benzenesulfonamide

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S1. Comment

As part of a study of the substituent effects on the structures of *N*-(aryl)arylsulfonamides (Gowda *et al.* , 2010*a,b,c*), the structure of 2,4-dichloro-*N*-(3-methylphenyl)-benzenesulfonamide (I) has been determined. The conformations of the N—C bonds in the C—SO_2—NH—C segment have *gauche* torsions with respect to the S═O bonds (Fig. 1).

The molecule is twisted at the S atom with the C1—SO_2—NH—C7 torsion angle of -60.2 (2)°, compared to the values of 60.6 (4)°, -59.7 (3)°, 63.9 (4)° and 53.0 (4)°, in the four molecules of 2,4-dichloro-*N*-(4-methylphenyl)benzenesulfonamide (II) (Gowda *et al.*, 2010*b*), 55.1 (3)° (molecule 1) and -48.3 (3)° (molecule 2) in 2,4-dichloro-*N*-(phenyl)-benzenesulfonamide (III) (Gowda *et al.*, 2010*c*) and 55.8 (2)° and -58.4 (3)°, in the 2 molecules of *N*-(3-methylphenyl)-benzenesulfonamide (IV) (Gowda *et al.*, 2010*a*).

The sulfonyl benzene and the aniline benzene rings in (I) are tilted relative to each other by 68.6 (1)°, compared to the values of 85.2 (1)° (molecule 1), 80.5 (2)° (molecule 2 A), 80.1 (2)° (molecule 2B), 87.5 (7) (molecule 3 A), 87.0 (6)° (molecule 3B) and 72.4 (1)° (molecule 4) in (II), 80.5 (2)° in the molecule 1 and 64.9 (1)° in molecule 2 of (III), and 67.9 (1)° in molecule 1 and 68.6 (1)° in molecule 2 of (IV).

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

In the crystal structure, the pairs of intermolecular N–H···O hydrogen bonds (Table 1) link the molecules through inversion-related dimers into infinite zigzag sequences running parallel to the *c*-axis. Part of the crystal structure is shown in Fig. 2.

S2. Experimental

The solution of 1,3-dichlorobenzene (10 cc) in chloroform (40 cc) was treated dropwise with chlorosulfonic acid (25 cc) 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 2,4-dichlorobenzenesulfonylchloride was treated with *m*-toluidine 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 cc). The resultant solid 2,4-dichloro-*N*-(3-methylphenyl)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 colorless 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 later restrained to N—H = 0.86 (1)Å. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.96Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

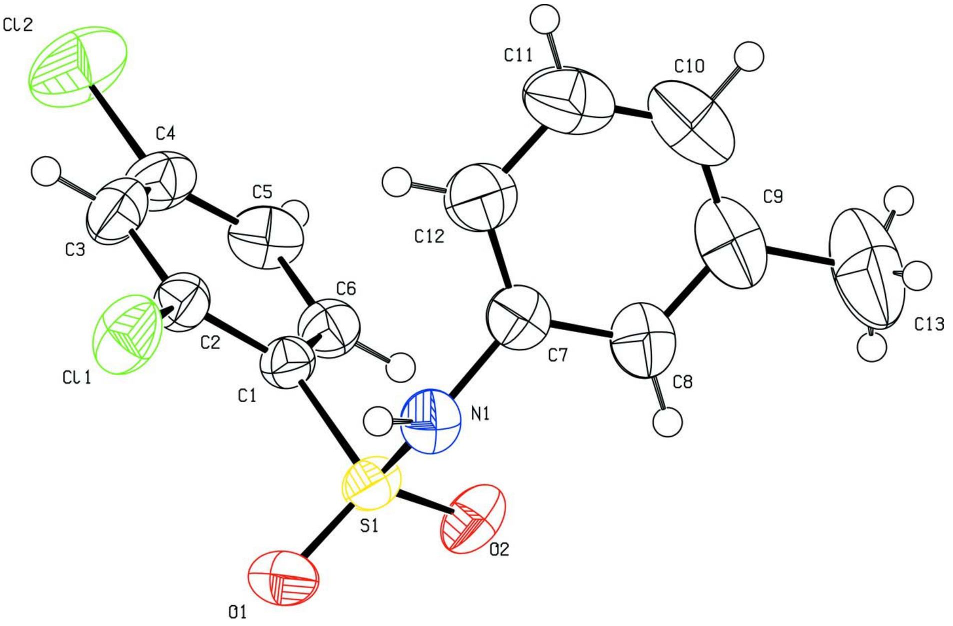

Figure 1

Molecular structure of (I), showing the atom labeling scheme and displacement ellipsoids are drawn at the 50% probability level.

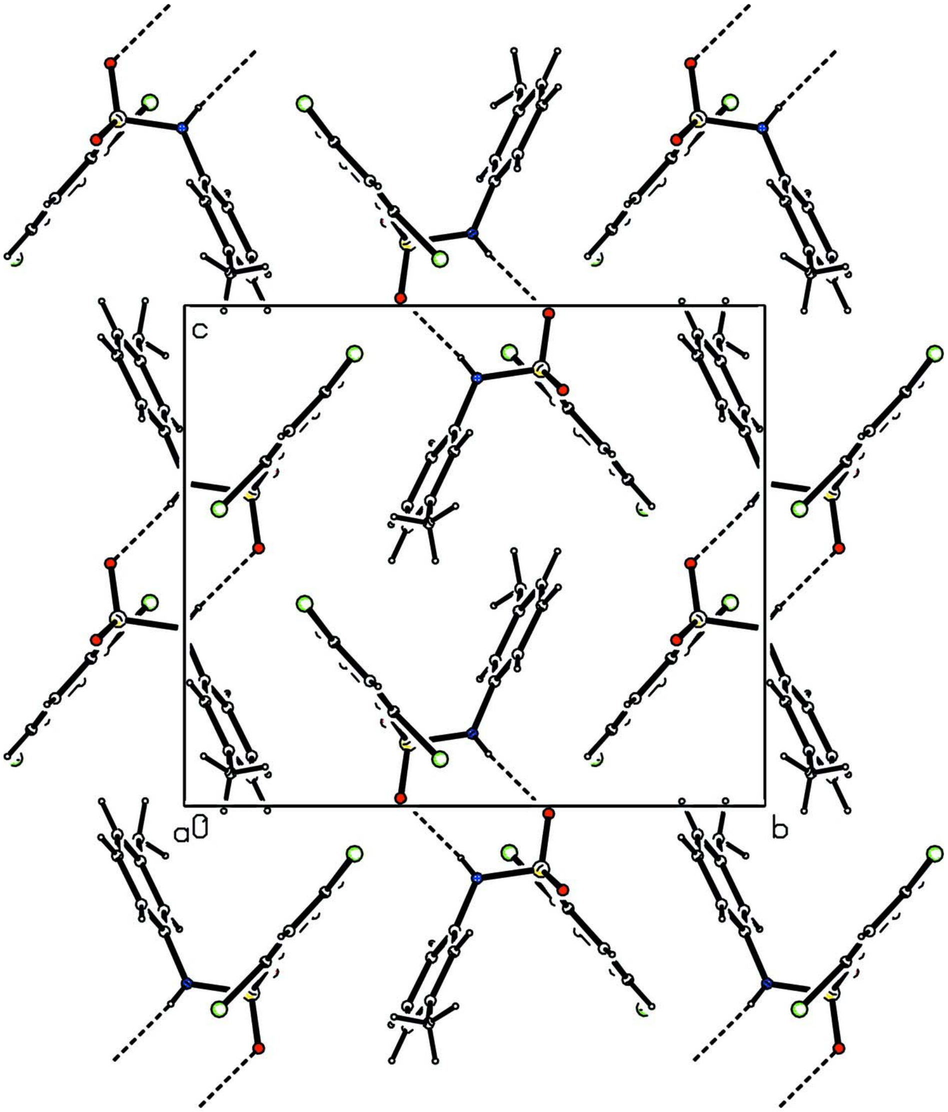

Figure 2

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

2,4-Dichloro-*N*-(3-methylphenyl)benzenesulfonamide

Crystal data

$C_{13}H_{11}Cl_2NO_2S$	$F(000) = 648$
$M_r = 316.19$	$D_x = 1.462$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3053 reflections
$a = 7.9031$ (7) Å	$\theta = 2.6$–27.8°
$b = 14.507$ (1) Å	$\mu = 0.59$ mm^{-1}
$c = 12.715$ (1) Å	$T = 299$ K
$\beta = 99.895$ (8)°	Prism, colorless
$V = 1436.1$ (2) Å^3	0.36 × 0.24 × 0.20 mm
$Z = 4$	

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 and phi scans.
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
$T_{\min} = 0.815$, $T_{\max} = 0.891$
5737 measured reflections
2915 independent reflections
2389 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.012$
$\theta_{\max} = 26.4°$, $\theta_{\min} = 2.6°$
$h = -9 \rightarrow 7$
$k = -18 \rightarrow 15$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.097$
$S = 1.03$
2915 reflections
176 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
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 0.717P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.41$ e Å^{-3}
$\Delta\rho_{\min} = -0.49$ e Å^{-3}

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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	0.6418 (2)	0.33869 (13)	0.20486 (15)	0.0350 (4)
C2	0.8068 (2)	0.36037 (14)	0.18846 (16)	0.0402 (4)
C3	0.9482 (3)	0.31950 (15)	0.24999 (18)	0.0509 (5)
H3	1.0586	0.3336	0.2389	0.061*
C4	0.9232 (3)	0.25777 (15)	0.32759 (18)	0.0520 (5)
C5	0.7617 (3)	0.23604 (15)	0.34720 (17)	0.0502 (5)
H5	0.7475	0.1948	0.4010	0.060*
C6	0.6213 (3)	0.27706 (14)	0.28493 (16)	0.0420 (4)
H6	0.5114	0.2631	0.2970	0.050*
C7	0.4604 (3)	0.53438 (14)	0.24891 (17)	0.0442 (5)
C8	0.3109 (3)	0.53560 (16)	0.2914 (2)	0.0549 (6)
H8	0.2117	0.5089	0.2538	0.066*
C9	0.3078 (4)	0.57624 (18)	0.3896 (2)	0.0714 (8)

C10	0.4569 (5)	0.6157 (2)	0.4429 (2)	0.0811 (9)
H10	0.4570	0.6428	0.5092	0.097*
C11	0.6042 (4)	0.6158 (2)	0.4005 (2)	0.0768 (8)
H11	0.7026	0.6439	0.4373	0.092*
C12	0.6080 (3)	0.57441 (17)	0.3031 (2)	0.0589 (6)
H12	0.7087	0.5736	0.2745	0.071*
C13	0.1436 (6)	0.5793 (3)	0.4345 (4)	0.1194 (15)
H13A	0.0657	0.5334	0.4000	0.143*
H13B	0.0922	0.6392	0.4223	0.143*
H13C	0.1682	0.5672	0.5098	0.143*
N1	0.4582 (2)	0.49567 (12)	0.14485 (14)	0.0433 (4)
H1N	0.516 (3)	0.5237 (15)	0.1041 (16)	0.052*
O1	0.46312 (19)	0.37240 (11)	0.01548 (11)	0.0509 (4)
O2	0.31399 (17)	0.34804 (11)	0.16821 (13)	0.0538 (4)
S1	0.45443 (6)	0.38552 (3)	0.12621 (4)	0.03929 (14)
Cl1	0.84366 (7)	0.44023 (5)	0.09354 (5)	0.06074 (19)
Cl2	1.10203 (10)	0.20696 (6)	0.40428 (7)	0.0889 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0317 (9)	0.0341 (9)	0.0383 (10)	−0.0004 (7)	0.0031 (7)	−0.0038 (8)
C2	0.0354 (9)	0.0417 (10)	0.0439 (11)	−0.0010 (8)	0.0076 (8)	−0.0012 (8)
C3	0.0337 (10)	0.0520 (12)	0.0659 (14)	0.0041 (9)	0.0056 (9)	−0.0013 (11)
C4	0.0461 (12)	0.0456 (12)	0.0579 (13)	0.0081 (9)	−0.0093 (10)	−0.0004 (10)
C5	0.0605 (13)	0.0409 (11)	0.0462 (12)	−0.0013 (10)	0.0006 (10)	0.0059 (9)
C6	0.0418 (10)	0.0390 (10)	0.0448 (11)	−0.0047 (8)	0.0068 (8)	−0.0016 (8)
C7	0.0510 (12)	0.0348 (10)	0.0459 (11)	0.0067 (9)	0.0059 (9)	0.0047 (9)
C8	0.0598 (14)	0.0464 (12)	0.0615 (14)	0.0002 (10)	0.0194 (11)	0.0008 (11)
C9	0.100 (2)	0.0550 (15)	0.0682 (17)	0.0041 (14)	0.0391 (16)	0.0008 (13)
C10	0.126 (3)	0.0642 (17)	0.0560 (16)	0.0059 (18)	0.0231 (17)	−0.0095 (14)
C11	0.093 (2)	0.0638 (17)	0.0661 (17)	−0.0007 (15)	−0.0074 (16)	−0.0131 (14)
C12	0.0566 (14)	0.0536 (13)	0.0639 (15)	0.0025 (11)	0.0033 (11)	−0.0041 (11)
C13	0.143 (4)	0.114 (3)	0.125 (3)	−0.007 (3)	0.089 (3)	−0.019 (2)
N1	0.0453 (9)	0.0410 (9)	0.0441 (10)	0.0038 (7)	0.0092 (7)	0.0050 (7)
O1	0.0512 (9)	0.0578 (9)	0.0401 (8)	−0.0029 (7)	−0.0021 (6)	−0.0060 (7)
O2	0.0320 (7)	0.0580 (9)	0.0711 (10)	−0.0050 (7)	0.0083 (7)	0.0028 (8)
S1	0.0313 (2)	0.0429 (3)	0.0420 (3)	−0.00170 (19)	0.00161 (18)	−0.0009 (2)
Cl1	0.0471 (3)	0.0713 (4)	0.0669 (4)	−0.0056 (3)	0.0187 (3)	0.0193 (3)
Cl2	0.0670 (4)	0.0819 (5)	0.1043 (6)	0.0210 (4)	−0.0233 (4)	0.0194 (4)

Geometric parameters (Å, º)

C1—C6	1.385 (3)	C8—H8	0.9300
C1—C2	1.392 (3)	C9—C10	1.379 (5)
C1—S1	1.7734 (18)	C9—C13	1.505 (4)
C2—C3	1.383 (3)	C10—C11	1.365 (5)
C2—Cl1	1.733 (2)	C10—H10	0.9300

C3—C4	1.372 (3)	C11—C12	1.381 (4)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.379 (3)	C12—H12	0.9300
C4—Cl2	1.737 (2)	C13—H13A	0.9600
C5—C6	1.382 (3)	C13—H13B	0.9600
C5—H5	0.9300	C13—H13C	0.9600
C6—H6	0.9300	N1—S1	1.6150 (18)
C7—C12	1.377 (3)	N1—H1N	0.852 (10)
C7—C8	1.381 (3)	O1—S1	1.4340 (15)
C7—N1	1.435 (3)	O2—S1	1.4201 (15)
C8—C9	1.385 (4)		

C6—C1—C2	119.14 (17)	C8—C9—C13	120.3 (3)
C6—C1—S1	118.04 (14)	C11—C10—C9	121.4 (3)
C2—C1—S1	122.82 (15)	C11—C10—H10	119.3
C3—C2—C1	120.37 (19)	C9—C10—H10	119.3
C3—C2—Cl1	117.66 (16)	C10—C11—C12	120.3 (3)
C1—C2—Cl1	121.96 (15)	C10—C11—H11	119.8
C4—C3—C2	118.96 (19)	C12—C11—H11	119.8
C4—C3—H3	120.5	C7—C12—C11	119.0 (3)
C2—C3—H3	120.5	C7—C12—H12	120.5
C3—C4—C5	122.14 (19)	C11—C12—H12	120.5
C3—C4—Cl2	118.47 (18)	C9—C13—H13A	109.5
C5—C4—Cl2	119.39 (18)	C9—C13—H13B	109.5
C4—C5—C6	118.4 (2)	H13A—C13—H13B	109.5
C4—C5—H5	120.8	C9—C13—H13C	109.5
C6—C5—H5	120.8	H13A—C13—H13C	109.5
C5—C6—C1	121.01 (19)	H13B—C13—H13C	109.5
C5—C6—H6	119.5	C7—N1—S1	121.30 (14)
C1—C6—H6	119.5	C7—N1—H1N	116.9 (16)
C12—C7—C8	120.5 (2)	S1—N1—H1N	112.4 (16)
C12—C7—N1	120.1 (2)	O2—S1—O1	119.39 (9)
C8—C7—N1	119.3 (2)	O2—S1—N1	108.64 (9)
C7—C8—C9	120.5 (3)	O1—S1—N1	105.82 (9)
C7—C8—H8	119.8	O2—S1—C1	105.78 (9)
C9—C8—H8	119.8	O1—S1—C1	109.07 (9)
C10—C9—C8	118.3 (3)	N1—S1—C1	107.68 (9)
C10—C9—C13	121.4 (3)		

C6—C1—C2—C3	1.2 (3)	C8—C9—C10—C11	0.6 (4)
S1—C1—C2—C3	−177.90 (16)	C13—C9—C10—C11	−177.7 (3)
C6—C1—C2—Cl1	−177.39 (15)	C9—C10—C11—C12	−1.3 (5)
S1—C1—C2—Cl1	3.5 (2)	C8—C7—C12—C11	0.0 (4)
C1—C2—C3—C4	−0.3 (3)	N1—C7—C12—C11	176.4 (2)
Cl1—C2—C3—C4	178.37 (17)	C10—C11—C12—C7	1.0 (4)
C2—C3—C4—C5	−0.9 (3)	C12—C7—N1—S1	107.6 (2)
C2—C3—C4—Cl2	180.00 (17)	C8—C7—N1—S1	−76.0 (2)
C3—C4—C5—C6	1.1 (3)	C7—N1—S1—O2	53.93 (18)

Cl2—C4—C5—C6	−179.77 (16)	C7—N1—S1—O1	−176.73 (15)
C4—C5—C6—C1	−0.1 (3)	C7—N1—S1—C1	−60.20 (18)
C2—C1—C6—C5	−1.0 (3)	C6—C1—S1—O2	1.58 (18)
S1—C1—C6—C5	178.18 (16)	C2—C1—S1—O2	−179.27 (16)
C12—C7—C8—C9	−0.7 (3)	C6—C1—S1—O1	−128.01 (16)
N1—C7—C8—C9	−177.1 (2)	C2—C1—S1—O1	51.14 (18)
C7—C8—C9—C10	0.4 (4)	C6—C1—S1—N1	117.60 (16)
C7—C8—C9—C13	178.6 (3)	C2—C1—S1—N1	−63.25 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.85 (1)	2.17 (1)	2.940 (2)	151 (2)

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