organic compounds

N-(2-Chlorophenyl)-4-nitrobenzene-sulfonamide

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In the title compound, $C_{12}H_9ClN_2O_4S$, the dihedral angle between the benzene rings is 70.60 (11)°. An intramolecular N—H⋯Cl contact occurs. In the crystal, molecules form inversion dimers via pairs of N—H⋯O hydrogen bonds.

Related literature

For studies on the effects of substituents on the structures and other aspects of N-arylsulfonamides, see: Chaithanya et al. (2012); Gowda et al. (2005) and of N-chloroarylamides, see: Gowda & Shetty (2004); Gowda & Weiss (1994); Shetty & Gowda (2004). For hydrogen-bonding patterns and motifs, see: Adsmond et al. (2001); Allen et al. (1998); Bernstein et al. (1995); Etter (1990).
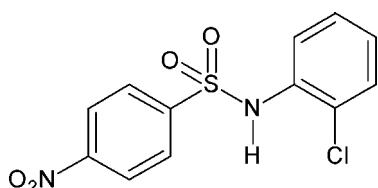
Experimental

Crystal data

$C_{12}H_9ClN_2O_4S$	V = 1345.9 (2) Å^3
M_r = 312.72	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 9.2762 (9) Å	μ = 0.45 mm^{-1}
b = 12.981 (1) Å	T = 293 K
c = 11.970 (1) Å	0.42 × 0.20 × 0.10 mm
β = 110.97 (1)°	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	Diffraction, 2009)
	T_{min} = 0.833, T_{max} = 0.956
Absorption correction: multi-scan (*CrysAlis RED*; Oxford	5256 measured reflections
	2748 independent reflections
	2019 reflections with $I > 2\sigma(I)$
	R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.110	
S = 1.16	
2748 reflections	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
185 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
1 restraint	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N⋯O1^i	0.84 (2)	2.20 (2)	3.003 (3)	159 (3)
N1—H1N⋯Cl1	0.84 (2)	2.56 (3)	2.984 (3)	112 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); 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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supporting information

N-(2-Chlorophenyl)-4-nitrobenzenesulfonamide

U. Chaithanya, Sabine Foro and B. Thimme Gowda

S1. Comment

As a part of our studies on the substituent effects on the structures and other aspects of *N*-arylsulfoamides (Chaithanya *et al.*, 2012; Gowda *et al.*, 2005) and *N*-chloroarylamides (Gowda & Shetty, 2004; Gowda & Weiss, 1994; Shetty & Gowda, 2004), in the present work, the crystal structure of *N*-(2-chlorophenyl)-4-nitrobenzenesulfonamide has been determined (Fig. 1).

The conformation of the N—C bond in the —SO$_2$—NH—C segment has *gauche* torsion with respect to the S═O bonds (Fig.1). Further, the conformation of the N—H bond in the —SO$_2$—NH— segment is *syn* to the *ortho*-Cl atom in the anilino ring, compared to *anti* conformation observed between the N—H bond and *ortho*- methyl group in *N*-(2-methyl-phenyl)-4-nitrobenzenesulfonamide (I) (Chaithanya *et al.*, 2012). The molecule is twisted at the S—N bond with the torsional angle of -59.12 (29)°, compared to the value of -58.97 (21)° in (II).

The dihedral angle between the sulfonyl and the anilino rings is 70.60 (11)°, compared to the value of 51.11 (10)° in (II).

The amide H-atom showed bifurcated intramolecular H-bonding with the *ortho*-Cl atom in the anilino benzene ring and the intermolecular H-bonding with the sulfonyl oxygen atom of the other molecule, generating C(6) and $R^2_2(8)$ motifs (Adsmond *et al.*, 2001; Allen *et al.*, 1998; Bernstein *et al.*, 1995; Etter, 1990).

In the crystal structure, the molecules form centrosymmetric dimers vi N—H⋯O hydrogen bonds. Part of the crystal structure is shown in Fig. 2.

S2. Experimental

The title compound was prepared by treating 4-nitrobenzenesulfonylchloride with 2-chloroaniline in the stoichiometric ratio and boiling the reaction mixture for 15 minutes. The reaction mixture was then cooled to room temperature and added to ice cold water (100 ml). The resultant solid *N*-(2-chlorophenyl)-4-nitrobenzenesulfonamide was filtered under suction and washed thoroughly with cold water and dilute HCl to remove the excess sulfonylchloride and aniline, respectively. It was then recrystallized to constant melting point from dilute ethanol. The purity of the compound was checked and characterized by its infrared spectra.

Prism like light pink single crystals of the title compound used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation of the solvent at room temperature.

S3. Refinement

H atoms bonded to C were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å. The amino H atom was freely refined with the N—H distance restrained to 0.86 (2) Å. All H atoms were refined with isotropic displacement parameters set at 1.2 U_{eq} of the parent atom.
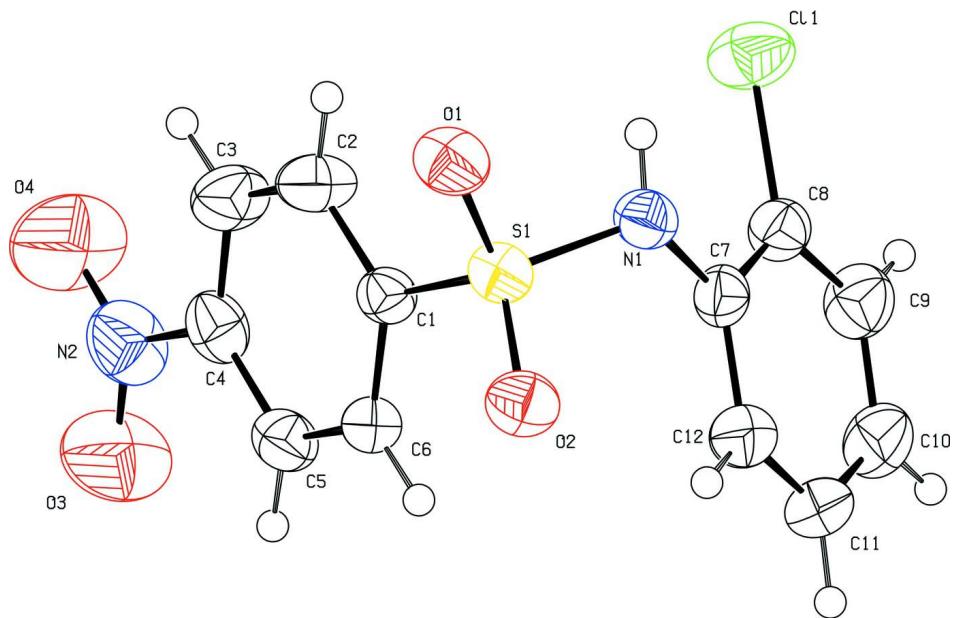
Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level.
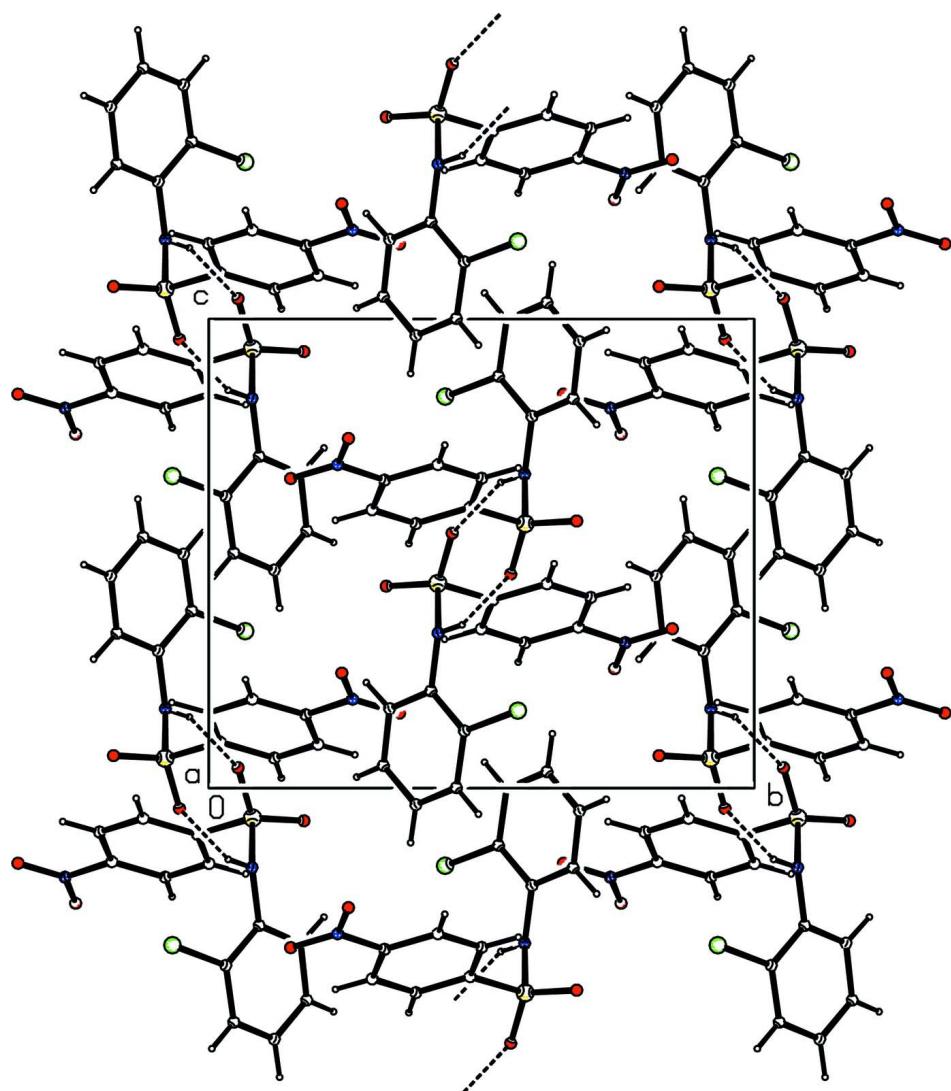
Figure 2

Bifurcated hydrogen bonding in the title compound.

N-(2-Chlorophenyl)-4-nitrobenzenesulfonamide

Crystal data

$C_{12}H_9ClN_2O_4S$	$F(000) = 640$
$M_r = 312.72$	$D_x = 1.543$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1413 reflections
$a = 9.2762$ (9) Å	$\theta = 2.9–27.9°$
$b = 12.981$ (1) Å	$\mu = 0.45$ mm^{-1}
$c = 11.970$ (1) Å	$T = 293$ K
$\beta = 110.97$ (1)°	Prism, light pink
$V = 1345.9$ (2) Å^3	$0.42 \times 0.20 \times 0.10$ mm
$Z = 4$	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	5256 measured reflections
Radiation source: fine-focus sealed tube	2748 independent reflections
Graphite monochromator	2019 reflections with $I > 2\sigma(I)$
Rotation method data acquisition using ω scans	$R_{int} = 0.025$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	$\theta_{max} = 26.4°$, $\theta_{min} = 2.9°$
$T_{min} = 0.833$, $T_{max} = 0.956$	$h = -11\rightarrow10$
	$k = -11\rightarrow16$
	$l = -11\rightarrow14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.053$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.110$	
$S = 1.16$	$w = 1/[\sigma^2(F_o^2) + (0.0185P)^2 + 1.7246P]$ where $P = (F_o^2 + 2F_c^2)/3$
2748 reflections	
185 parameters	$(\Delta/\sigma)_{max} = 0.001$
1 restraint	$\Delta\rho_{max} = 0.29$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.30$ e Å^{-3}
Secondary atom site location: difference Fourier map	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
	Extinction coefficient: 0.0098 (7)

Special details

Experimental. Absorption correction: 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	0.1263 (3)	0.5209 (2)	0.4016 (3)	0.0343 (7)
C2	0.1763 (4)	0.6204 (3)	0.4357 (3)	0.0507 (9)
H2	0.2802	0.6331	0.4784	0.061*
C3	0.0722 (4)	0.7003 (3)	0.4066 (3)	0.0539 (9)
H3	0.1040	0.7676	0.4289	0.065*
C4	−0.0808 (4)	0.6778 (3)	0.3434 (3)	0.0444 (8)
C5	−0.1329 (4)	0.5798 (3)	0.3102 (3)	0.0430 (8)
H5	−0.2372	0.5673	0.2688	0.052*
C6	−0.0280 (3)	0.4996 (3)	0.3394 (3)	0.0375 (7)
H6	−0.0606	0.4324	0.3175	0.045*
C7	0.2457 (3)	0.4074 (2)	0.2074 (3)	0.0366 (7)
C8	0.2726 (3)	0.4687 (2)	0.1220 (3)	0.0401 (7)
C9	0.1904 (4)	0.4540 (3)	0.0011 (3)	0.0520 (9)

H9	0.2115	0.4943	−0.0554	0.062*
C10	0.0775 (4)	0.3798 (3)	−0.0351 (3)	0.0573 (10)
H10	0.0215	0.3701	−0.1161	0.069*
C11	0.0477 (4)	0.3202 (3)	0.0484 (3)	0.0550 (10)
H11	−0.0307	0.2713	0.0237	0.066*
C12	0.1327 (4)	0.3317 (3)	0.1695 (3)	0.0486 (9)
H12	0.1141	0.2889	0.2251	0.058*
N1	0.3366 (3)	0.4207 (2)	0.3307 (2)	0.0399 (6)
H1N	0.407 (3)	0.465 (2)	0.348 (3)	0.048*
N2	−0.1922 (4)	0.7636 (3)	0.3102 (3)	0.0653 (9)
O1	0.3880 (2)	0.44640 (19)	0.54360 (19)	0.0490 (6)
O2	0.1828 (2)	0.32555 (17)	0.4305 (2)	0.0460 (6)
O3	−0.3263 (3)	0.7440 (3)	0.2546 (3)	0.0967 (11)
O4	−0.1448 (4)	0.8497 (3)	0.3400 (4)	0.1119 (14)
Cl1	0.40864 (11)	0.56660 (7)	0.16532 (8)	0.0596 (3)
S1	0.26320 (9)	0.41991 (6)	0.43645 (7)	0.0372 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0355 (16)	0.0356 (17)	0.0334 (16)	−0.0036 (14)	0.0144 (13)	0.0015 (13)
C2	0.0380 (18)	0.045 (2)	0.063 (2)	−0.0078 (16)	0.0104 (17)	0.0001 (18)
C3	0.053 (2)	0.0350 (19)	0.072 (2)	−0.0039 (17)	0.0198 (19)	−0.0011 (18)
C4	0.0448 (18)	0.043 (2)	0.050 (2)	0.0092 (16)	0.0232 (16)	0.0072 (16)
C5	0.0344 (16)	0.051 (2)	0.0433 (18)	−0.0011 (16)	0.0141 (14)	0.0003 (17)
C6	0.0348 (16)	0.0393 (18)	0.0392 (17)	−0.0051 (14)	0.0144 (14)	−0.0047 (14)
C7	0.0358 (15)	0.0373 (17)	0.0371 (16)	0.0028 (14)	0.0136 (13)	−0.0044 (14)
C8	0.0391 (17)	0.0385 (18)	0.0419 (18)	0.0003 (15)	0.0135 (14)	−0.0026 (15)
C9	0.058 (2)	0.056 (2)	0.042 (2)	−0.0009 (19)	0.0178 (17)	−0.0003 (17)
C10	0.056 (2)	0.069 (3)	0.042 (2)	−0.004 (2)	0.0126 (18)	−0.0138 (19)
C11	0.047 (2)	0.058 (2)	0.060 (2)	−0.0154 (18)	0.0185 (18)	−0.023 (2)
C12	0.0500 (19)	0.048 (2)	0.051 (2)	−0.0104 (17)	0.0220 (17)	−0.0074 (17)
N1	0.0346 (14)	0.0450 (16)	0.0398 (14)	−0.0029 (13)	0.0131 (12)	−0.0010 (13)
N2	0.061 (2)	0.053 (2)	0.086 (3)	0.0140 (18)	0.033 (2)	0.0111 (19)
O1	0.0384 (12)	0.0669 (17)	0.0355 (12)	−0.0016 (11)	0.0056 (10)	0.0057 (11)
O2	0.0487 (13)	0.0389 (13)	0.0511 (14)	−0.0024 (11)	0.0188 (11)	0.0085 (11)
O3	0.0542 (18)	0.076 (2)	0.145 (3)	0.0226 (17)	0.018 (2)	0.014 (2)
O4	0.090 (2)	0.0459 (19)	0.187 (4)	0.0172 (18)	0.034 (3)	0.002 (2)
Cl1	0.0682 (6)	0.0530 (6)	0.0543 (6)	−0.0196 (5)	0.0179 (5)	0.0023 (4)
S1	0.0343 (4)	0.0411 (5)	0.0348 (4)	0.0007 (4)	0.0106 (3)	0.0052 (4)

Geometric parameters (Å, º)

C1—C2	1.383 (4)	C8—C9	1.386 (4)
C1—C6	1.385 (4)	C8—Cl1	1.735 (3)
C1—S1	1.768 (3)	C9—C10	1.374 (5)
C2—C3	1.375 (5)	C9—H9	0.9300
C2—H2	0.9300	C10—C11	1.367 (5)

C3—C4	1.380 (5)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.388 (5)
C4—C5	1.369 (5)	C11—H11	0.9300
C4—N2	1.474 (4)	C12—H12	0.9300
C5—C6	1.381 (4)	N1—S1	1.637 (3)
C5—H5	0.9300	N1—H1N	0.837 (18)
C6—H6	0.9300	N2—O4	1.207 (4)
C7—C8	1.386 (4)	N2—O3	1.210 (4)
C7—C12	1.390 (4)	O1—S1	1.429 (2)
C7—N1	1.425 (4)	O2—S1	1.423 (2)
C2—C1—C6	121.2 (3)	C10—C9—C8	119.9 (3)
C2—C1—S1	119.1 (2)	C10—C9—H9	120.1
C6—C1—S1	119.7 (2)	C8—C9—H9	120.1
C3—C2—C1	119.9 (3)	C11—C10—C9	119.8 (3)
C3—C2—H2	120.0	C11—C10—H10	120.1
C1—C2—H2	120.0	C9—C10—H10	120.1
C2—C3—C4	118.0 (3)	C10—C11—C12	120.9 (3)
C2—C3—H3	121.0	C10—C11—H11	119.5
C4—C3—H3	121.0	C12—C11—H11	119.5
C5—C4—C3	122.9 (3)	C11—C12—C7	119.8 (3)
C5—C4—N2	119.0 (3)	C11—C12—H12	120.1
C3—C4—N2	118.1 (3)	C7—C12—H12	120.1
C4—C5—C6	118.9 (3)	C7—N1—S1	123.0 (2)
C4—C5—H5	120.5	C7—N1—H1N	117 (2)
C6—C5—H5	120.5	S1—N1—H1N	108 (2)
C5—C6—C1	119.0 (3)	O4—N2—O3	123.4 (4)
C5—C6—H6	120.5	O4—N2—C4	118.4 (4)
C1—C6—H6	120.5	O3—N2—C4	118.2 (4)
C8—C7—C12	118.6 (3)	O2—S1—O1	119.60 (14)
C8—C7—N1	120.0 (3)	O2—S1—N1	108.81 (14)
C12—C7—N1	121.4 (3)	O1—S1—N1	105.37 (13)
C9—C8—C7	120.9 (3)	O2—S1—C1	107.88 (14)
C9—C8—Cl1	118.9 (3)	O1—S1—C1	108.43 (15)
C7—C8—Cl1	120.2 (2)	N1—S1—C1	105.99 (14)
C6—C1—C2—C3	−0.8 (5)	C10—C11—C12—C7	2.6 (5)
S1—C1—C2—C3	178.2 (3)	C8—C7—C12—C11	−1.2 (5)
C1—C2—C3—C4	0.0 (5)	N1—C7—C12—C11	−179.2 (3)
C2—C3—C4—C5	0.9 (5)	C8—C7—N1—S1	137.5 (3)
C2—C3—C4—N2	−179.0 (3)	C12—C7—N1—S1	−44.6 (4)
C3—C4—C5—C6	−1.1 (5)	C5—C4—N2—O4	180.0 (4)
N2—C4—C5—C6	178.8 (3)	C3—C4—N2—O4	−0.1 (6)
C4—C5—C6—C1	0.3 (5)	C5—C4—N2—O3	0.0 (5)
C2—C1—C6—C5	0.6 (5)	C3—C4—N2—O3	179.9 (4)
S1—C1—C6—C5	−178.4 (2)	C7—N1—S1—O2	56.7 (3)
C12—C7—C8—C9	−0.9 (5)	C7—N1—S1—O1	−173.9 (3)
N1—C7—C8—C9	177.1 (3)	C7—N1—S1—C1	−59.1 (3)

C12—C7—C8—Cl1	178.1 (2)	C2—C1—S1—O2	160.4 (3)
N1—C7—C8—Cl1	−3.9 (4)	C6—C1—S1—O2	−20.6 (3)
C7—C8—C9—C10	1.8 (5)	C2—C1—S1—O1	29.5 (3)
Cl1—C8—C9—C10	−177.3 (3)	C6—C1—S1—O1	−151.5 (2)
C8—C9—C10—C11	−0.5 (6)	C2—C1—S1—N1	−83.2 (3)
C9—C10—C11—C12	−1.7 (6)	C6—C1—S1—N1	95.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1[i]	0.84 (2)	2.20 (2)	3.003 (3)	159 (3)
N1—H1*N*···Cl1	0.84 (2)	2.56 (3)	2.984 (3)	112 (3)

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