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N-(2-Chlorobenzoyl)-2-nitrobenzene-sulfonamide

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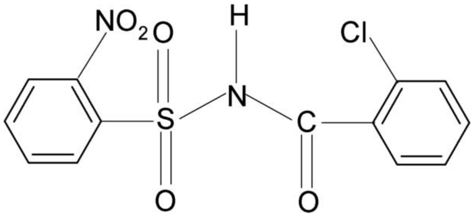
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.096; data-to-parameter ratio = 14.6.

In the title compound, $C_{13}H_9ClN_2O_5S$, the dihedral angle between the two rings is 71.2 (1)°. The crystal structure features inversion dimers linked by pairs of N—H⋯O(S) hydrogen bonds.

Related literature

For studies, including our studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Bowes *et al.* (2003); Gowda *et al.* (2006), on *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007), on *N*-(aryl)-arylsulfonamides, see: Shetty & Gowda (2005), on *N*-(substitutedbenzoyl)-arylsulfonamides, see: Suchetan *et al.* (2012) and on *N*-chloroarylamides, see: Gowda & Mahadevappa (1983).



Experimental

Crystal data

$C_{13}H_9ClN_2O_5S$
M_r = 340.73
Orthorhombic, *Pbca*
a = 12.266 (1) Å
b = 12.643 (1) Å
c = 18.738 (2) Å
V = 2905.9 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.43 mm^{-1}
T = 293 K
0.48 × 0.40 × 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} = 0.820, T_{max} = 0.919
7231 measured reflections
2958 independent reflections
2105 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.096
S = 1.04
2958 reflections
202 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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}	0.82 (1)	2.13 (1)	2.9465 (19)	172 (2)

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

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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5759).

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supporting information

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N-(2-Chlorobenzoyl)-2-nitrobenzenesulfonamide

P. A. Suchetan, Sabine Foro and B. Thimme Gowda

S1. Comment

Diaryl acylsulfonamides are known as potent antitumor agents. As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Bowes *et al.*, 2003; Gowda *et al.*, 2006), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007), *N*-(aryl)-arylsulfonamides (Shetty & Gowda, 2005); *N*-(substitutedbenzoyl)-arylsulfonamides (Suchetan *et al.*, 2012) and *N*-chloro-arylsulfonamides (Gowda & Mahadevappa, 1983), in the present work, the crystal structure of *N*-(2-chlorobenzoyl)-2-nitrobenzenesulfonamide (I) has been determined (Fig.1).

The conformation between the N—H and C=O bonds in the C—SO$_2$—NH—C(O) segment is *anti* and the N—C bond in the segment has *gauche* torsion with respect to the S═O bonds (Fig.1), similar to that observed in *N*-(3-chlorobenzoyl)-3-nitrobenzenesulfonamide (II)(Suchetan *et al.*, 2012). In (I), the conformation between the N—H bond and the *ortho*-nitro group in the sulfonyl benzene ring is *syn*, similar to that observed between the N—H bond and the *meta*-nitro group in (II). Further, the conformation of the C═O is *anti* to the *ortho*-Cl atom in the benzoyl ring, similar to that observed between the C═O and the *meta*-Cl atom in (II).

The molecule is twisted at the S—N bond with the torsional angle of -59.68 (17)°, compared to the value of -60.40 (29)° in (II).

The dihedral angle between the sulfonyl benzene ring and the —SO$_2$—NH—C—O segment is 77.5 (1)°, compared to the value of 77.0 (1)° in (II). Furthermore, the dihedral angle between the sulfonyl and the benzoyl benzene rings is 71.2 (1)°, compared to the value of 83.5 (1)° in (II).

In the crystal, the intermolecular N–H···O (S) hydrogen bonds (Table 1) link the molecules into centrosymmetric dimers. Part of the crystal structure is shown in Fig. 2.

S2. Experimental

The title compound was prepared by refluxing a mixture of 2-chlorobenzoic acid (0.02 mole), 2-nitrobenzenesulfonamide (0.02 mole) and excess phosphorous oxychloride for 3 h on a water bath. The resultant mixture was cooled and poured into crushed ice. The solid, *N*-(2-chlorobenzoyl)-2-nitrobenzenesulfonamide, obtained was filtered, washed thoroughly with water and then dissolved in a sodium bicarbonate solution. The compound was later reprecipitated by acidifying the filtered solution with dilute HCl. It was filtered, dried and recrystallized.

Prism like colourless single crystals of the title compound used in X-ray diffraction studies were obtained by slow evaporation of its toluene solution at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and its coordinates were refined with a distance restraint of 0.86 (2) %A. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93 Å. All H atoms were refined with isotropic displacement parameters set to 1.2 times of the U_{eq} of the parent atom.

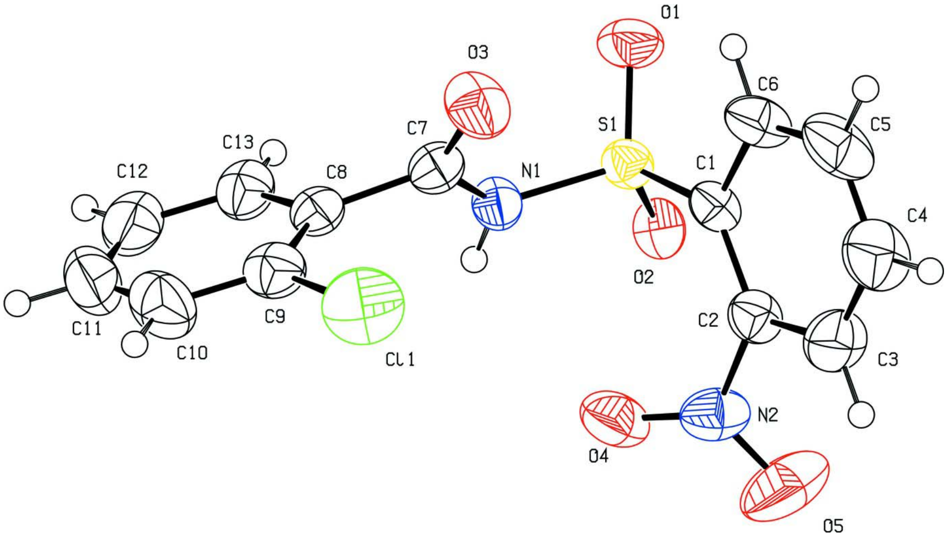

Figure 1

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

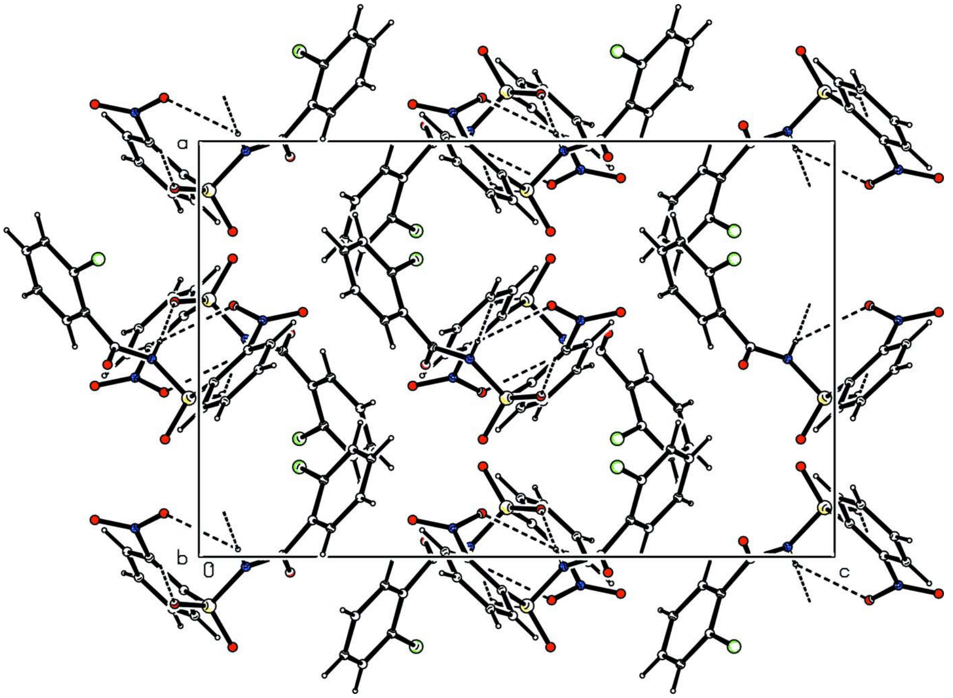

Figure 2

Molecular packing in the title compound. Hydrogen bonds are shown as dashed lines.

N-(2-Chlorobenzoyl)-2-nitrobenzenesulfonamide

Crystal data

$C_{13}H_9ClN_2O_5S$	Hall symbol: -P 2ac 2ab
M_r = 340.73	*a* = 12.266 (1) Å
Orthorhombic, *Pbca*	*b* = 12.643 (1) Å

c = 18.738 (2) Å
V = 2905.9 (5) Å^3
Z = 8
F(000) = 1392
D_x = 1.558 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2278 reflections
θ = 2.5–27.7°
μ = 0.43 mm^{-1}
T = 293 K
Prism, colourless
0.48 × 0.40 × 0.20 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)
$T_{\min}$ = 0.820, $T_{\max}$ = 0.919
7231 measured reflections
2958 independent reflections
2105 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.015
$\theta_{\max}$ = 26.4°, $\theta_{\min}$ = 2.6°
$h = -13 \rightarrow 15$
$k = -12 \rightarrow 15$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.096
S = 1.04
2958 reflections
202 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
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.3259P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ < 0.001
$\Delta\rho_{\max}$ = 0.23 e Å^{-3}
$\Delta\rho_{\min}$ = −0.29 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	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.08013 (14)	0.23007 (14)	0.52361 (9)	0.0378 (4)
C2	0.00268 (15)	0.21928 (14)	0.57758 (10)	0.0414 (4)
C3	−0.01230 (19)	0.12473 (16)	0.61266 (12)	0.0579 (6)
H3	−0.0641	0.1194	0.6487	0.069*
C4	0.0493 (2)	0.03845 (17)	0.59424 (14)	0.0698 (7)
H4	0.0393	−0.0256	0.6178	0.084*
C5	0.12540 (19)	0.04664 (18)	0.54136 (14)	0.0679 (7)
H5	0.1667	−0.0122	0.5288	0.081*

C6	0.14160 (17)	0.14199 (16)	0.50620 (12)	0.0523 (5)
H6	0.1942	0.1467	0.4706	0.063*
C7	−0.00470 (16)	0.32241 (14)	0.36776 (9)	0.0409 (4)
C8	−0.08836 (15)	0.37301 (15)	0.32102 (9)	0.0396 (4)
C9	−0.18466 (17)	0.32223 (16)	0.30359 (11)	0.0504 (5)
C10	−0.2584 (2)	0.3683 (2)	0.25752 (13)	0.0705 (7)
H10	−0.3228	0.3334	0.2459	0.085*
C11	−0.2364 (2)	0.4656 (2)	0.22895 (13)	0.0741 (7)
H11	−0.2859	0.4962	0.1976	0.089*
C12	−0.1425 (2)	0.51844 (19)	0.24594 (11)	0.0638 (7)
H12	−0.1283	0.5846	0.2263	0.077*
C13	−0.06890 (18)	0.47285 (16)	0.29256 (10)	0.0496 (5)
H13	−0.0058	0.5092	0.3050	0.060*
N1	0.02375 (13)	0.38393 (11)	0.42595 (8)	0.0391 (4)
H1N	−0.0179 (15)	0.4312 (12)	0.4385 (10)	0.047*
N2	−0.06860 (14)	0.30632 (13)	0.60028 (10)	0.0515 (4)
O1	0.21732 (10)	0.33502 (11)	0.44685 (8)	0.0546 (4)
O2	0.11350 (10)	0.43253 (10)	0.53775 (7)	0.0425 (3)
O3	0.03763 (13)	0.23796 (11)	0.35641 (8)	0.0616 (4)
O4	−0.10433 (11)	0.36567 (12)	0.55488 (9)	0.0617 (4)
O5	−0.08870 (17)	0.31359 (14)	0.66363 (9)	0.0915 (6)
Cl1	−0.21604 (5)	0.20111 (5)	0.34182 (4)	0.0802 (2)
S1	0.11830 (4)	0.35192 (4)	0.48410 (2)	0.03738 (15)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0299 (9)	0.0363 (9)	0.0473 (11)	0.0011 (8)	−0.0029 (8)	0.0011 (8)
C2	0.0403 (10)	0.0352 (9)	0.0487 (11)	0.0010 (8)	0.0007 (9)	0.0025 (8)
C3	0.0635 (14)	0.0468 (12)	0.0634 (13)	−0.0006 (11)	0.0106 (11)	0.0138 (10)
C4	0.0751 (17)	0.0445 (12)	0.0897 (19)	0.0078 (12)	0.0053 (15)	0.0198 (12)
C5	0.0591 (14)	0.0412 (12)	0.103 (2)	0.0199 (11)	0.0002 (14)	0.0047 (12)
C6	0.0400 (11)	0.0453 (11)	0.0716 (14)	0.0073 (9)	0.0082 (10)	−0.0014 (10)
C7	0.0458 (11)	0.0393 (10)	0.0377 (10)	−0.0012 (9)	0.0036 (9)	−0.0050 (8)
C8	0.0442 (10)	0.0427 (10)	0.0319 (9)	0.0040 (9)	0.0023 (8)	−0.0100 (8)
C9	0.0492 (12)	0.0554 (12)	0.0467 (11)	−0.0016 (10)	0.0015 (10)	−0.0117 (10)
C10	0.0534 (14)	0.0891 (18)	0.0689 (14)	0.0072 (14)	−0.0158 (12)	−0.0157 (15)
C11	0.0748 (18)	0.0890 (19)	0.0585 (14)	0.0365 (16)	−0.0122 (14)	−0.0051 (13)
C12	0.0891 (19)	0.0533 (13)	0.0490 (12)	0.0234 (13)	0.0107 (13)	0.0015 (11)
C13	0.0622 (13)	0.0445 (11)	0.0420 (10)	0.0046 (10)	0.0013 (10)	−0.0058 (9)
N1	0.0427 (9)	0.0377 (8)	0.0369 (8)	0.0057 (7)	−0.0033 (7)	−0.0040 (7)
N2	0.0510 (10)	0.0386 (9)	0.0648 (12)	−0.0051 (9)	0.0179 (9)	−0.0005 (8)
O1	0.0371 (8)	0.0643 (9)	0.0626 (9)	−0.0019 (7)	0.0130 (7)	0.0056 (7)
O2	0.0455 (7)	0.0375 (7)	0.0444 (7)	−0.0043 (6)	−0.0091 (6)	−0.0018 (5)
O3	0.0694 (10)	0.0515 (9)	0.0638 (10)	0.0196 (8)	−0.0084 (8)	−0.0189 (7)
O4	0.0478 (9)	0.0516 (9)	0.0857 (11)	0.0121 (7)	0.0126 (8)	0.0112 (9)
O5	0.1402 (18)	0.0662 (11)	0.0681 (12)	0.0066 (11)	0.0497 (12)	−0.0041 (9)
Cl1	0.0745 (5)	0.0688 (4)	0.0972 (5)	−0.0255 (3)	0.0024 (4)	−0.0002 (3)

S1	0.0315 (2)	0.0384 (3)	0.0423 (3)	−0.0029 (2)	0.0002 (2)	0.0017 (2)

Geometric parameters (Å, º)

C1—C6	1.384 (3)	C8—C13	1.391 (3)
C1—C2	1.394 (2)	C9—C10	1.379 (3)
C1—S1	1.7721 (18)	C9—Cl1	1.734 (2)
C2—C3	1.376 (3)	C10—C11	1.369 (3)
C2—N2	1.468 (2)	C10—H10	0.9300
C3—C4	1.371 (3)	C11—C12	1.369 (3)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.365 (3)	C12—C13	1.382 (3)
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.388 (3)	C13—H13	0.9300
C5—H5	0.9300	N1—S1	1.6420 (16)
C6—H6	0.9300	N1—H1N	0.820 (14)
C7—O3	1.206 (2)	N2—O5	1.216 (2)
C7—N1	1.384 (2)	N2—O4	1.216 (2)
C7—C8	1.493 (3)	O1—S1	1.4171 (13)
C8—C9	1.384 (3)	O2—S1	1.4327 (13)

C6—C1—C2	117.61 (17)	C8—C9—Cl1	120.13 (16)
C6—C1—S1	117.21 (15)	C11—C10—C9	119.6 (2)
C2—C1—S1	124.62 (14)	C11—C10—H10	120.2
C3—C2—C1	121.49 (18)	C9—C10—H10	120.2
C3—C2—N2	115.66 (17)	C10—C11—C12	120.9 (2)
C1—C2—N2	122.85 (16)	C10—C11—H11	119.5
C4—C3—C2	119.8 (2)	C12—C11—H11	119.5
C4—C3—H3	120.1	C11—C12—C13	119.5 (2)
C2—C3—H3	120.1	C11—C12—H12	120.2
C5—C4—C3	120.0 (2)	C13—C12—H12	120.2
C5—C4—H4	120.0	C12—C13—C8	120.6 (2)
C3—C4—H4	120.0	C12—C13—H13	119.7
C4—C5—C6	120.5 (2)	C8—C13—H13	119.7
C4—C5—H5	119.7	C7—N1—S1	124.22 (13)
C6—C5—H5	119.7	C7—N1—H1N	118.5 (14)
C1—C6—C5	120.6 (2)	S1—N1—H1N	115.4 (14)
C1—C6—H6	119.7	O5—N2—O4	124.28 (19)
C5—C6—H6	119.7	O5—N2—C2	117.40 (18)
O3—C7—N1	121.86 (18)	O4—N2—C2	118.31 (17)
O3—C7—C8	124.88 (17)	O1—S1—O2	119.21 (8)
N1—C7—C8	113.24 (15)	O1—S1—N1	108.40 (8)
C9—C8—C13	118.50 (19)	O2—S1—N1	105.14 (7)
C9—C8—C7	121.76 (18)	O1—S1—C1	107.53 (8)
C13—C8—C7	119.73 (17)	O2—S1—C1	108.33 (8)
C10—C9—C8	120.8 (2)	N1—S1—C1	107.76 (8)
C10—C9—Cl1	119.05 (18)		

C6—C1—C2—C3	0.2 (3)	C9—C10—C11—C12	−0.6 (4)
S1—C1—C2—C3	−170.90 (16)	C10—C11—C12—C13	0.1 (3)
C6—C1—C2—N2	−179.14 (18)	C11—C12—C13—C8	1.4 (3)
S1—C1—C2—N2	9.8 (3)	C9—C8—C13—C12	−2.3 (3)
C1—C2—C3—C4	−0.5 (3)	C7—C8—C13—C12	176.28 (17)
N2—C2—C3—C4	178.9 (2)	O3—C7—N1—S1	1.6 (3)
C2—C3—C4—C5	0.1 (4)	C8—C7—N1—S1	−176.52 (12)
C3—C4—C5—C6	0.4 (4)	C3—C2—N2—O5	38.4 (3)
C2—C1—C6—C5	0.4 (3)	C1—C2—N2—O5	−142.2 (2)
S1—C1—C6—C5	172.14 (18)	C3—C2—N2—O4	−141.0 (2)
C4—C5—C6—C1	−0.7 (4)	C1—C2—N2—O4	38.4 (3)
O3—C7—C8—C9	56.4 (3)	C7—N1—S1—O1	56.41 (17)
N1—C7—C8—C9	−125.56 (19)	C7—N1—S1—O2	−175.07 (15)
O3—C7—C8—C13	−122.1 (2)	C7—N1—S1—C1	−59.68 (17)
N1—C7—C8—C13	56.0 (2)	C6—C1—S1—O1	−6.83 (18)
C13—C8—C9—C10	1.7 (3)	C2—C1—S1—O1	164.30 (16)
C7—C8—C9—C10	−176.79 (18)	C6—C1—S1—O2	−136.91 (15)
C13—C8—C9—Cl1	−176.28 (14)	C2—C1—S1—O2	34.23 (18)
C7—C8—C9—Cl1	5.2 (2)	C6—C1—S1—N1	109.83 (16)
C8—C9—C10—C11	−0.3 (3)	C2—C1—S1—N1	−79.04 (17)
Cl1—C9—C10—C11	177.70 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2^{i}	0.82 (1)	2.13 (1)	2.9465 (19)	172 (2)

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