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2-Chloro-*N*-(3-methoxybenzoyl)benzenesulfonamide

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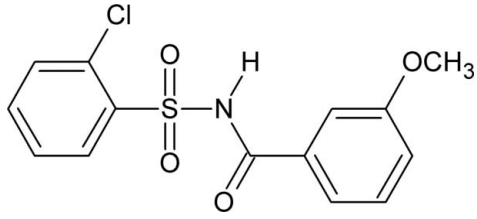
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.101; data-to-parameter ratio = 13.3.

In the title compound, $C_{14}H_{12}ClNO_4S$, the dihedral angle between the chloro- and methoxy-substituted benzene rings is 87.40 (1)°. In the crystal, adjacent molecules form inversion-related dimers through strong N—H···O hydrogen bonds, generating $R_2^2(8)$ loops. The dimers are further connected through two C—H···O interactions that form $C(11)$ chains and $R_2^2(14)$ loops. Aromatic π–π stacking interactions [centroid–centroid separation = 3.8574 (1) Å] are also observed.

Related literature

For a similar structure, see: Gowda *et al.* (2010)



Experimental

Crystal data

$C_{14}H_{12}ClNO_4S$ M_r = 325.76
Triclinic, $P\overline{1}$
a = 7.5731 (5) Å
b = 10.1861 (5) Å
c = 10.3636 (6) Å
α = 94.945 (4)°
β = 96.581 (5)°
γ = 110.974 (5)°
V = 734.56 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.42 mm^{-1}
T = 293 K
0.35 × 0.28 × 0.22 mm

Data collection

Bruker APEXII diffractometer
11351 measured reflections
2584 independent reflections
2133 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
2 standard reflections every 1 reflections
intensity decay: 10%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.101
S = 1.05
2584 reflections
195 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H*N*1···O1^{i}	0.78 (3)	2.14 (3)	2.926 (3)	170 (3)
C5—H5···O3^{ii}	0.93	2.53	3.417 (3)	160
C3—H3···O3^{iii}	0.93	2.60	3.338 (3)	137

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x, -y+1, -z+1$; (iii) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); 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: HG5328).

References

Bruker (2009). *APEX2*, *SAINT-Plus* and *XPREP*. Bruker AXS Inc., Madison, Wisconsin, USA.
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Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2013). E**69**, o1215 [doi:10.1107/S1600536813018291]

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S1. Comment

As a part of the efforts to study the crystal structures of *N*-(aroyl)-arylsulfonamides (Gowda *et al.*, 2010), the crystal structure of the title compound (I) was determined.

In the molecule, the conformation between the N—H bond and the *ortho*-chloro group in the sulfonyl bound benzene ring is *syn*. This is similar to that observed in *N*-(benzoyl)-2-chlorobenzenesulfonamide (II, Gowda *et al.* 2010).

In the structure, the adjacent molecules form inversion related dimers through strong N—H···O hydrogen bonds, generating $R_2^2(8)$ loops. The dimers are further connected through intermolecular C3—H3···O3 and C5—H5···O3 interactions that form C(11) chains and $R_2^2(14)$ loops. Aromatic π-π stacking interactions (centroid-centroid separation = 3.8574 (1) Å) are also observed in the structure.

S2. Experimental

The title compound was prepared by refluxing a mixture of 3-methoxybenzoic acid, 2-chlorobenzenesulfonamide and phosphorous oxychloride ($POCl_3$) for 2 h on a water bath. The resultant mixture was cooled and poured into ice cold water. The solid obtained was filtered and washed thoroughly with water and then dissolved in sodium bicarbonate solution. The compound was later reprecipitated by acidifying the filtered solution with dilute HCl. The filtered and dried solid was recrystallized to the constant melting point (443 K).

Colorless prisms of (I) were obtained from a slow evaporation of its ethanolic solution at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and later refined freely. 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 or 1.5 times of the U_{eq} of the parent atom).

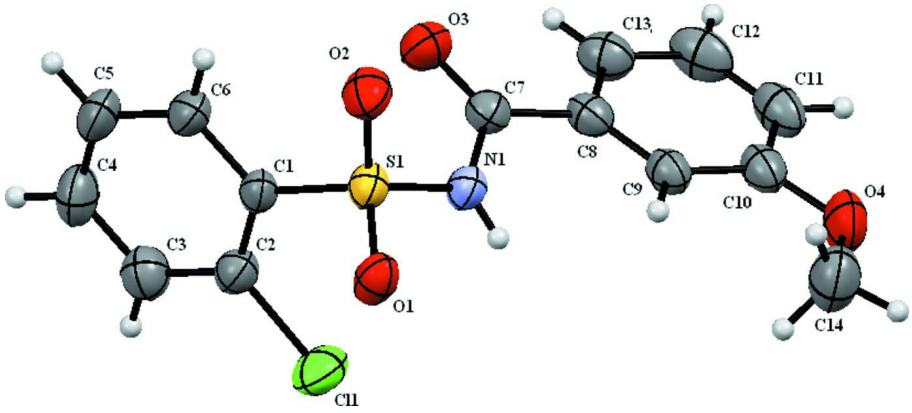

Figure 1

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

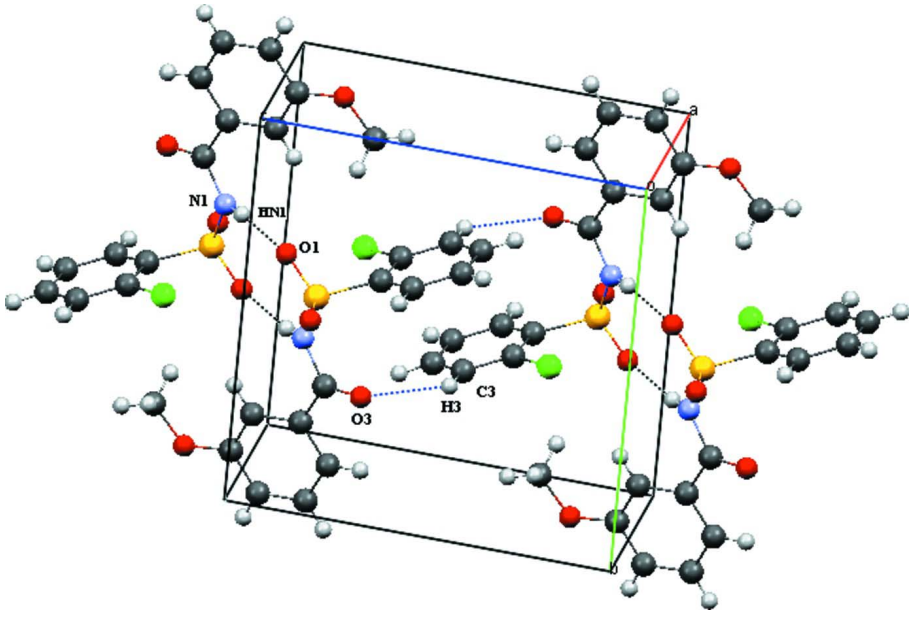

Figure 2

Molecular packing of (I) forming $R_2^2(8)$ loops and C(11) chains.

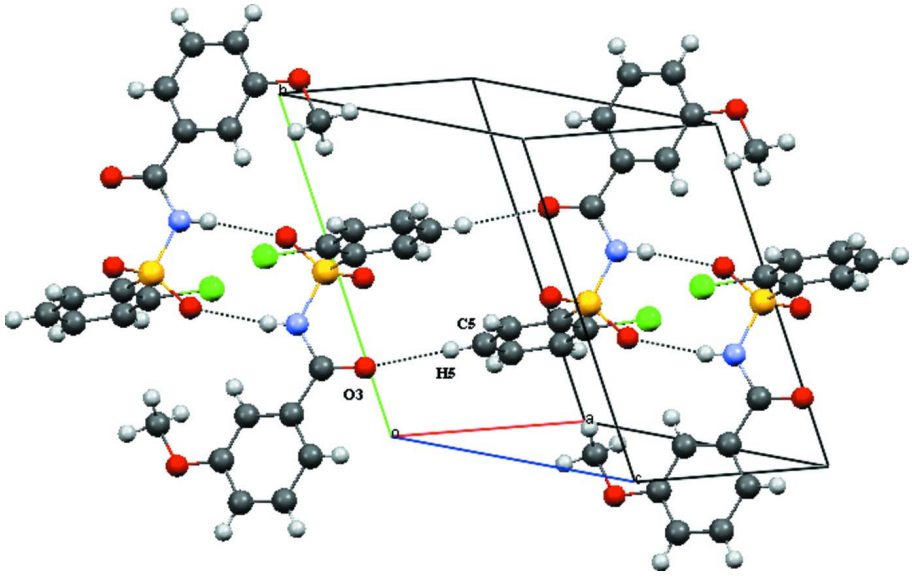

Figure 3

Molecular packing of (I) forming $R_2^2(14)$ loops.

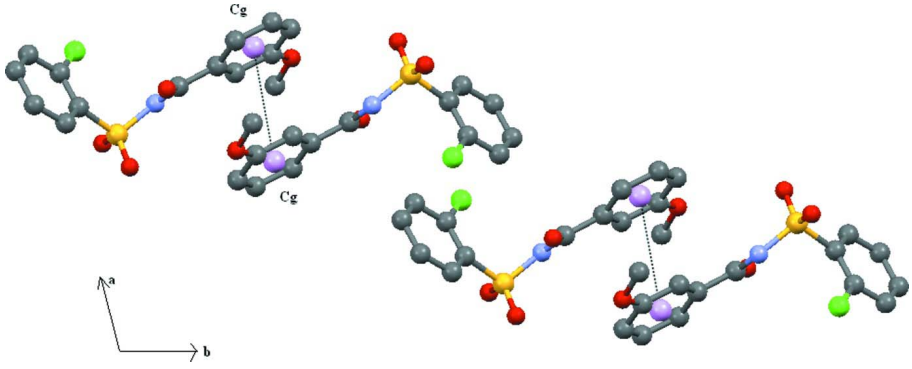

Figure 4

Stacking of molecules through *Cg*···*Cg* interactions. *Cg* is the centroid of the methoxy substituted benzene ring.

2-Chloro-*N*-(3-methoxybenzoyl)benzenesulfonamide

Crystal data

$C_{14}H_{12}ClNO_4S$
$M_r = 325.76$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.5731\ (5)$ Å
$b = 10.1861\ (5)$ Å
$c = 10.3636\ (6)$ Å
$\alpha = 94.945\ (4)°$
$\beta = 96.581\ (5)°$
$\gamma = 110.974\ (5)°$
$V = 734.56\ (7)$ Å^3
$Z = 2$
$F(000) = 336$
Prism
$D_x = 1.473$ Mg m^{-3}
Melting point: 443 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1029 reflections
$\theta = 2.7–25.0°$
$\mu = 0.42$ mm^{-1}
$T = 293$ K
Prism, colourless
0.35 × 0.28 × 0.22 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
11351 measured reflections
2584 independent reflections
2133 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.7°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 12$
2 standard reflections every 1 reflections
intensity decay: 10%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.101$
$S = 1.05$
2584 reflections
195 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2 + 0.1773P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
HN1	0.513 (4)	0.639 (3)	0.931 (3)	0.057 (8)*
C1	0.2590 (3)	0.4163 (2)	0.67778 (18)	0.0355 (5)
C2	0.4118 (3)	0.3814 (2)	0.6470 (2)	0.0404 (5)
C3	0.4049 (4)	0.3179 (3)	0.5217 (2)	0.0514 (6)
H3	0.5072	0.2947	0.5005	0.062*
C4	0.2458 (4)	0.2894 (3)	0.4285 (2)	0.0566 (7)
H4	0.2411	0.2460	0.3448	0.068*
C5	0.0939 (4)	0.3242 (3)	0.4578 (2)	0.0547 (6)
H5	−0.0122	0.3051	0.3940	0.066*
C6	0.0999 (3)	0.3876 (2)	0.5822 (2)	0.0450 (5)
H6	−0.0025	0.4111	0.6024	0.054*
C7	0.4531 (3)	0.7563 (2)	0.8139 (2)	0.0385 (5)
C8	0.6044 (3)	0.8929 (2)	0.8800 (2)	0.0390 (5)
C9	0.6794 (3)	0.9116 (2)	1.0120 (2)	0.0402 (5)
H9	0.6398	0.8371	1.0612	0.048*

C10	0.8142 (3)	1.0429 (2)	1.0698 (2)	0.0450 (5)
C11	0.8727 (3)	1.1534 (2)	0.9965 (3)	0.0533 (6)
H11	0.9626	1.2412	1.0355	0.064*
C12	0.7984 (4)	1.1338 (3)	0.8665 (3)	0.0585 (7)
H12	0.8384	1.2087	0.8177	0.070*
C13	0.6639 (3)	1.0035 (2)	0.8062 (2)	0.0506 (6)
H13	0.6146	0.9907	0.7176	0.061*
C14	0.8344 (4)	0.9647 (3)	1.2797 (3)	0.0710 (8)
H14A	0.6997	0.9395	1.2805	0.106*
H14B	0.9025	0.9995	1.3673	0.106*
H14C	0.8580	0.8825	1.2465	0.106*
O1	0.2842 (2)	0.39742 (15)	0.92753 (14)	0.0450 (4)
O2	0.0743 (2)	0.51231 (17)	0.83161 (15)	0.0508 (4)
O3	0.3527 (2)	0.74552 (17)	0.70992 (15)	0.0526 (4)
O4	0.8980 (3)	1.07160 (17)	1.19821 (17)	0.0628 (5)
S1	0.24743 (7)	0.48701 (5)	0.83723 (5)	0.03761 (17)
N1	0.4298 (3)	0.63820 (18)	0.87817 (18)	0.0398 (4)
Cl1	0.61560 (9)	0.41609 (7)	0.75991 (6)	0.0578 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0390 (12)	0.0294 (10)	0.0326 (10)	0.0097 (9)	−0.0032 (9)	0.0000 (8)
C2	0.0387 (13)	0.0383 (12)	0.0398 (11)	0.0116 (10)	−0.0013 (9)	0.0044 (9)
C3	0.0547 (15)	0.0508 (14)	0.0481 (13)	0.0199 (12)	0.0083 (11)	0.0019 (11)
C4	0.0656 (18)	0.0565 (15)	0.0368 (12)	0.0152 (13)	−0.0003 (11)	−0.0072 (11)
C5	0.0583 (16)	0.0535 (15)	0.0409 (13)	0.0166 (13)	−0.0149 (11)	−0.0053 (11)
C6	0.0412 (13)	0.0404 (12)	0.0469 (13)	0.0134 (10)	−0.0088 (10)	0.0000 (10)
C7	0.0385 (12)	0.0381 (12)	0.0400 (11)	0.0164 (10)	0.0054 (9)	0.0027 (9)
C8	0.0383 (12)	0.0318 (11)	0.0489 (12)	0.0145 (9)	0.0096 (10)	0.0056 (9)
C9	0.0384 (12)	0.0303 (11)	0.0499 (13)	0.0110 (9)	0.0064 (10)	0.0030 (9)
C10	0.0380 (13)	0.0344 (12)	0.0598 (14)	0.0123 (10)	0.0065 (10)	−0.0022 (10)
C11	0.0426 (14)	0.0327 (12)	0.0795 (18)	0.0084 (10)	0.0126 (12)	0.0023 (12)
C12	0.0550 (16)	0.0392 (13)	0.0849 (19)	0.0141 (12)	0.0252 (14)	0.0237 (13)
C13	0.0508 (15)	0.0462 (14)	0.0584 (14)	0.0196 (12)	0.0124 (11)	0.0142 (11)
C14	0.079 (2)	0.0590 (17)	0.0580 (16)	0.0125 (15)	−0.0047 (14)	−0.0001 (13)
O1	0.0472 (9)	0.0376 (8)	0.0398 (8)	0.0055 (7)	−0.0025 (7)	0.0085 (6)
O2	0.0379 (9)	0.0558 (10)	0.0545 (9)	0.0164 (8)	0.0025 (7)	−0.0040 (8)
O3	0.0556 (10)	0.0529 (10)	0.0475 (9)	0.0212 (8)	−0.0036 (8)	0.0079 (7)
O4	0.0652 (12)	0.0421 (10)	0.0614 (11)	0.0051 (9)	−0.0064 (9)	−0.0090 (8)
S1	0.0354 (3)	0.0351 (3)	0.0362 (3)	0.0091 (2)	−0.0017 (2)	0.0003 (2)
N1	0.0388 (11)	0.0319 (10)	0.0411 (10)	0.0091 (8)	−0.0079 (8)	0.0010 (8)
Cl1	0.0433 (4)	0.0770 (5)	0.0540 (4)	0.0283 (3)	−0.0046 (3)	0.0028 (3)

Geometric parameters (Å, º)

C1—C2	1.388 (3)	C9—C10	1.390 (3)
C1—C6	1.394 (3)	C9—H9	0.9300

C1—S1	1.769 (2)	C10—O4	1.365 (3)
C2—C3	1.387 (3)	C10—C11	1.382 (3)
C2—Cl1	1.732 (2)	C11—C12	1.368 (4)
C3—C4	1.379 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.390 (3)
C4—C5	1.376 (4)	C12—H12	0.9300
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.380 (3)	C14—O4	1.417 (3)
C5—H5	0.9300	C14—H14A	0.9600
C6—H6	0.9300	C14—H14B	0.9600
C7—O3	1.218 (2)	C14—H14C	0.9600
C7—N1	1.391 (3)	O1—S1	1.4337 (14)
C7—C8	1.494 (3)	O2—S1	1.4200 (16)
C8—C13	1.386 (3)	S1—N1	1.6402 (18)
C8—C9	1.389 (3)	N1—HN1	0.79 (3)

C2—C1—C6	119.67 (19)	O4—C10—C9	123.9 (2)
C2—C1—S1	123.23 (15)	C11—C10—C9	120.2 (2)
C6—C1—S1	116.98 (17)	C12—C11—C10	120.0 (2)
C3—C2—C1	119.8 (2)	C12—C11—H11	120.0
C3—C2—Cl1	117.89 (18)	C10—C11—H11	120.0
C1—C2—Cl1	122.28 (16)	C11—C12—C13	120.9 (2)
C4—C3—C2	119.8 (2)	C11—C12—H12	119.5
C4—C3—H3	120.1	C13—C12—H12	119.5
C2—C3—H3	120.1	C8—C13—C12	119.1 (2)
C5—C4—C3	120.9 (2)	C8—C13—H13	120.5
C5—C4—H4	119.5	C12—C13—H13	120.5
C3—C4—H4	119.5	O4—C14—H14A	109.5
C4—C5—C6	119.7 (2)	O4—C14—H14B	109.5
C4—C5—H5	120.2	H14A—C14—H14B	109.5
C6—C5—H5	120.2	O4—C14—H14C	109.5
C5—C6—C1	120.2 (2)	H14A—C14—H14C	109.5
C5—C6—H6	119.9	H14B—C14—H14C	109.5
C1—C6—H6	119.9	C10—O4—C14	118.15 (18)
O3—C7—N1	120.3 (2)	O2—S1—O1	119.16 (10)
O3—C7—C8	123.37 (19)	O2—S1—N1	109.37 (10)
N1—C7—C8	116.29 (18)	O1—S1—N1	104.24 (9)
C13—C8—C9	120.4 (2)	O2—S1—C1	107.73 (10)
C13—C8—C7	117.8 (2)	O1—S1—C1	108.22 (9)
C9—C8—C7	121.78 (19)	N1—S1—C1	107.61 (10)
C8—C9—C10	119.4 (2)	C7—N1—S1	123.60 (15)
C8—C9—H9	120.3	C7—N1—HN1	119.7 (19)
C10—C9—H9	120.3	S1—N1—HN1	116.2 (19)
O4—C10—C11	115.9 (2)		

C6—C1—C2—C3	−0.1 (3)	O4—C10—C11—C12	179.1 (2)
S1—C1—C2—C3	175.85 (17)	C9—C10—C11—C12	−0.2 (4)
C6—C1—C2—Cl1	179.10 (16)	C10—C11—C12—C13	0.0 (4)

S1—C1—C2—Cl1	−4.9 (3)	C9—C8—C13—C12	−0.6 (3)
C1—C2—C3—C4	−0.3 (3)	C7—C8—C13—C12	177.2 (2)
Cl1—C2—C3—C4	−179.55 (18)	C11—C12—C13—C8	0.4 (4)
C2—C3—C4—C5	0.6 (4)	C11—C10—O4—C14	176.2 (2)
C3—C4—C5—C6	−0.5 (4)	C9—C10—O4—C14	−4.5 (3)
C4—C5—C6—C1	0.1 (4)	C2—C1—S1—O2	178.82 (17)
C2—C1—C6—C5	0.2 (3)	C6—C1—S1—O2	−5.11 (19)
S1—C1—C6—C5	−175.99 (18)	C2—C1—S1—O1	−51.1 (2)
O3—C7—C8—C13	−15.7 (3)	C6—C1—S1—O1	124.97 (16)
N1—C7—C8—C13	164.6 (2)	C2—C1—S1—N1	60.99 (19)
O3—C7—C8—C9	162.0 (2)	C6—C1—S1—N1	−122.94 (17)
N1—C7—C8—C9	−17.6 (3)	O3—C7—N1—S1	−11.6 (3)
C13—C8—C9—C10	0.4 (3)	C8—C7—N1—S1	167.99 (15)
C7—C8—C9—C10	−177.34 (19)	O2—S1—N1—C7	−52.2 (2)
C8—C9—C10—O4	−179.3 (2)	O1—S1—N1—C7	179.30 (18)
C8—C9—C10—C11	0.0 (3)	C1—S1—N1—C7	64.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H*N*1···O1^{i}	0.78 (3)	2.14 (3)	2.926 (3)	170 (3)
C5—H5···O3^{ii}	0.93	2.53	3.417 (3)	160
C3—H3···O3^{iii}	0.93	2.60	3.338 (3)	137

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) −*x*, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1.