organic compounds

N-(3-Chlorophenyl)-2,4-dimethyl-benzenesulfonamide

K. Shakuntala,[a] Sabine Foro[b] and B. Thimme Gowda[a]*

[a]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, Mangalore, India, and [b]Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

In the molecule of the title compound, $C_{14}H_{14}ClNO_2S$, the N—H bond is in a *syn* position with respect to the *meta*-Cl atom in the aniline ring. The molecule is twisted about the N—S bond with a C—SO_2—NH—C torsion angle of 44.55 (17)°. The two aromatic rings are inclined relative to each other by 66.2 (1)°. In the crystal, N—H···O hydrogen bonds link the molecules into infinite chains parallel to the *b* axis.

Related literature

For hydrogen bonding modes of sulfonamides, see: Adsmond & Grant (2001). For studies on the effects of substituents on the structures and other aspects of N-(aryl)-amides, see: Arjunan *et al.* (2004); Gowda *et al.* (1999), on N-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007) and on N-(aryl)arylsulfonamides, see: Gelbrich *et al.* (2007); Gowda *et al.* (2010); Perlovich *et al.* (2006). For the preparation of the title compound, See: Savitha & Gowda (2006)
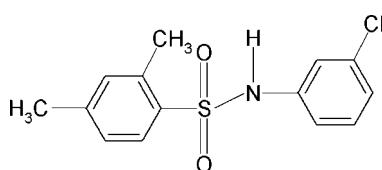
Experimental

Crystal data

$C_{14}H_{14}ClNO_2S$	$a = 9.0385$ (7) Å
$M_r = 295.77$	$b = 9.7830$ (7) Å
Monoclinic, $P2_1/c$	$c = 15.897$ (1) Å

$\beta = 97.284$ (7)°	$\mu = 0.42$ mm^{-1}
$V = 1394.33$ (17) Å^3	$T = 293$ K
$Z = 4$	$0.44 \times 0.40 \times 0.32$ mm
Mo $K\alpha$ radiation	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	Diffraction, 2009)
	$T_{min} = 0.837$, $T_{max} = 0.877$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford	5245 measured reflections
	2841 independent reflections
	2438 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.009$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.103$	
$S = 1.06$	
2841 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
177 parameters	$\Delta\rho_{min} = -0.36$ 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)	2.32 (2)	3.117 (2)	158 (2)

Symmetry code: (i) $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$.

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*.

References

Adsmond, D. A. & Grant, D. J. W. (2001). *J. Pharm. Sci.* **90**, 2058–2077.
Arjunan, V., Mohan, S., Subramanian, S. & Gowda, B. T. (2004). *Spectrochim. Acta Part A*, **60**, 1141–1159.
Gelbrich, T., Hursthouse, M. B. & Threlfall, T. L. (2007). *Acta Cryst.* B**63**, 621–632.
Gowda, B. T., Bhat, D. K., Fuess, H. & Weiss, A. (1999). *Z. Naturforsch. Teil A*, **54**, 261–267.
Gowda, B. T., Foro, S. & Fuess, H. (2007). *Acta Cryst.* E**63**, o2337.
Gowda, B. T., Foro, S., Nirmala, P. G. & Fuess, H. (2010). *Acta Cryst.* E**66**, o1282.
Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.
Perlovich, G. L., Tkachev, V. V., Schaper, K.-J. & Raevsky, O. A. (2006). *Acta Cryst.* E**62**, o780–o782.
Savitha, M. B. & Gowda, B. T. (2006). *Z. Naturforsch. Teil A*, **60**, 600–606.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

N-(3-Chlorophenyl)-2,4-dimethylbenzenesulfonamide

K. Shakuntala, Sabine Foro and B. Thimme Gowda

S1. Comment

The amide and sulfonamide moieties are the constituents of many biologically important compounds. The hydrogen bonding preferences of sulfonamides have been investigated (Adsmond & Grant, 2001). As part of our work on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Arjunan *et al.*, 2004; Gowda *et al.*, 1999), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007) and *N*-(aryl)-arylsulfonamides (Gowda *et al.*, 2010), in the present work, the crystal structure of *N*-(3-chlorophenyl)-2,4-dimethylbenzene- sulfonamide (I) has been determined (Fig. 1). The conformation of the N—C bond in the C—SO$_2$—NH—C segment has *gauche* torsions with respect to the S═O bonds. The molecule is bent at the S atom with the C1—SO$_2$—NH—C7 torsion angle of 44.6 (2)°, compared to the values of -54.9 (2)° in *N*-(2-chlorophenyl)-2,4-dimethylbenzenesulfonamide (II) (Gowda *et al.*, 2010). Further, the conformation of the N—H bond in (I) is *syn* to the *meta*-chloro group in the anilino ring.

The two benzene rings in (I) are tilted relative to each other by 75.7 (1)°, compared to the value of 66.2 (1)° (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 packing of molecules in (I) *via* N—H···O(S) hydrogen bonds (Table 1) is shown in Fig.2.

S2. Experimental

The solution of 1,3-xylene (1,3-dimethylbenzene) (10 ml) in chloroform (40 ml) was treated dropwise with chloro-sulfonic 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 2,4-dimethylbenzenesulfonylchloride was treated with 3-chloro-aniline 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 *N*-(3-chlorophenyl)-2,4-dimethylbenzenesulfonamide 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).

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

S3. Refinement

The H atom of the NH group was located in a difference map and later restrained to the distance N—H = 0.86 (2) Å. 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 set to 1.2 times of the U_{eq} of the parent atom.
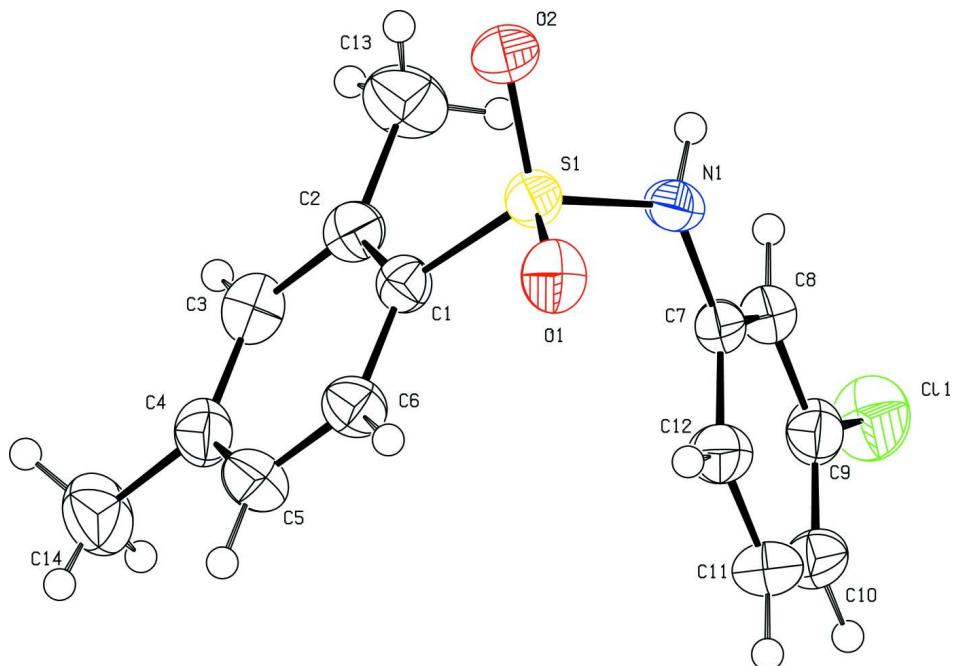
Figure 1

Molecular structure of (I), showing the atom labelling scheme and displacement ellipsoids are drawn at the 50% probability level.
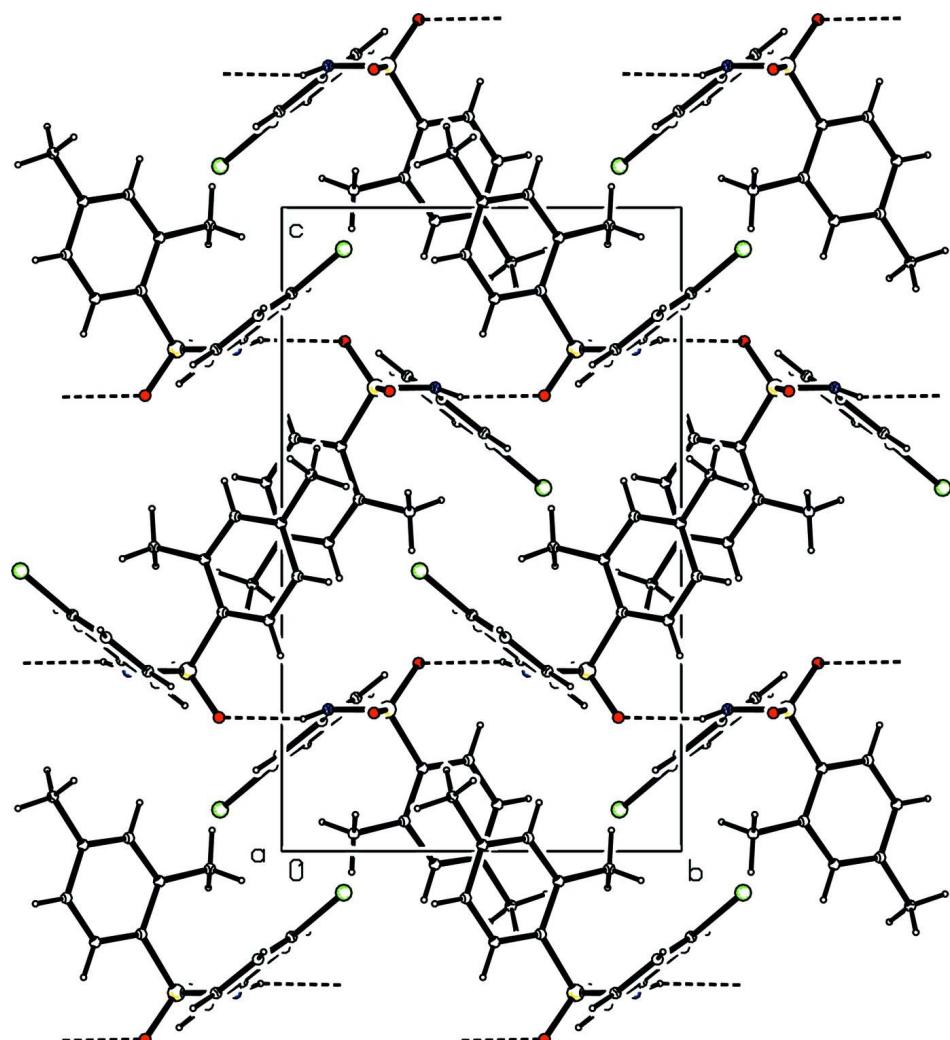
Figure 2
Molecular packing of (I) with hydrogen bonding shown as dashed lines.

N-(3-Chlorophenyl)-2,4-dimethylbenzenesulfonamide

Crystal data

$C_{14}H_{14}ClNO_2S$	$F(000) = 616$
$M_r = 295.77$	$D_x = 1.409$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2954 reflections
$a = 9.0385$ (7) Å	$\theta = 2.6$–27.8°
$b = 9.7830$ (7) Å	$\mu = 0.42$ mm^{-1}
$c = 15.897$ (1) Å	$T = 293$ K
$\beta = 97.284$ (7)°	Prism, colourless
$V = 1394.33$ (17) Å^3	$0.44 \times 0.40 \times 0.32$ mm
$Z = 4$	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	5245 measured reflections
	2841 independent reflections
Radiation source: fine-focus sealed tube	2438 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.009$
Rotation method data acquisition using ω and φ scans	$\theta_{max} = 26.4°$, $\theta_{min} = 2.6°$
	$h = -11 \rightarrow 5$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	$k = -12 \rightarrow 5$
	$l = -19 \rightarrow 19$
$T_{min} = 0.837$, $T_{max} = 0.877$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.036$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.103$	
$S = 1.06$	H atoms treated by a mixture of independent and constrained refinement
2841 reflections	
177 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.5258P]$ where $P = (F_o^2 + 2F_c^2)/3$
1 restraint	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 0.28$ e Å⁻³
	$\Delta\rho_{min} = -0.36$ 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 (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.89077 (18)	0.14785 (17)	0.62875 (11)	0.0313 (4)
C2	0.90726 (19)	0.19319 (18)	0.54672 (11)	0.0359 (4)
C3	0.8365 (2)	0.1167 (2)	0.47948 (12)	0.0407 (4)
H3	0.8471	0.1439	0.4245	0.049*
C4	0.7512 (2)	0.0022 (2)	0.49045 (12)	0.0413 (4)
C5	0.7383 (2)	−0.0396 (2)	0.57271 (13)	0.0453 (5)
H5	0.6825	−0.1170	0.5816	0.054*
C6	0.8074 (2)	0.03244 (19)	0.64125 (12)	0.0385 (4)
H6	0.7980	0.0035	0.6960	0.046*
C7	0.73064 (18)	0.40051 (18)	0.70252 (11)	0.0328 (4)
C8	0.67615 (19)	0.50629 (19)	0.64977 (12)	0.0371 (4)
H8	0.7410	0.5643	0.6259	0.045*
C9	0.5229 (2)	0.5243 (2)	0.63306 (12)	0.0402 (4)
C10	0.4248 (2)	0.4403 (2)	0.66791 (13)	0.0459 (5)
H10	0.3224	0.4542	0.6563	0.055*

C11	0.4810 (2)	0.3354 (2)	0.72028 (14)	0.0467 (5)
H11	0.4157	0.2778	0.7440	0.056*
C12	0.6335 (2)	0.3141 (2)	0.73829 (13)	0.0410 (4)
H12	0.6703	0.2430	0.7738	0.049*
C13	0.9916 (3)	0.3198 (2)	0.52740 (15)	0.0575 (6)
H13A	1.0910	0.3159	0.5569	0.069*
H13B	0.9415	0.3991	0.5455	0.069*
H13C	0.9960	0.3252	0.4675	0.069*
C14	0.6723 (3)	−0.0736 (3)	0.41520 (15)	0.0614 (6)
H14A	0.7398	−0.0875	0.3740	0.074*
H14B	0.5881	−0.0212	0.3904	0.074*
H14C	0.6387	−0.1606	0.4334	0.074*
N1	0.88789 (16)	0.38382 (16)	0.72030 (11)	0.0376 (4)
H1N	0.943 (2)	0.4465 (19)	0.7057 (13)	0.045*
O1	0.93966 (15)	0.15746 (14)	0.79211 (8)	0.0437 (3)
O2	1.12517 (14)	0.26929 (15)	0.71487 (9)	0.0469 (4)
Cl1	0.45335 (6)	0.65483 (6)	0.56450 (4)	0.06029 (19)
S1	0.97232 (4)	0.23553 (4)	0.72011 (3)	0.03353 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0310 (8)	0.0294 (8)	0.0333 (8)	0.0027 (6)	0.0027 (6)	0.0007 (7)
C2	0.0360 (9)	0.0325 (9)	0.0395 (9)	0.0025 (7)	0.0061 (7)	0.0050 (7)
C3	0.0440 (10)	0.0439 (10)	0.0339 (9)	0.0081 (8)	0.0037 (7)	0.0030 (8)
C4	0.0393 (9)	0.0408 (10)	0.0427 (10)	0.0060 (8)	0.0005 (8)	−0.0079 (8)
C5	0.0475 (11)	0.0349 (10)	0.0534 (12)	−0.0084 (8)	0.0064 (9)	−0.0023 (8)
C6	0.0422 (9)	0.0347 (9)	0.0386 (9)	−0.0038 (8)	0.0055 (7)	0.0041 (7)
C7	0.0273 (8)	0.0326 (8)	0.0384 (9)	−0.0005 (7)	0.0042 (7)	−0.0097 (7)
C8	0.0346 (9)	0.0342 (9)	0.0432 (10)	−0.0012 (7)	0.0074 (7)	−0.0047 (8)
C9	0.0373 (9)	0.0402 (10)	0.0419 (10)	0.0053 (8)	0.0006 (7)	−0.0080 (8)
C10	0.0284 (9)	0.0547 (12)	0.0540 (12)	0.0006 (8)	0.0032 (8)	−0.0118 (10)
C11	0.0338 (9)	0.0515 (12)	0.0563 (12)	−0.0088 (8)	0.0110 (9)	−0.0037 (10)
C12	0.0367 (9)	0.0386 (10)	0.0483 (11)	−0.0019 (8)	0.0077 (8)	0.0010 (8)
C13	0.0710 (14)	0.0507 (13)	0.0517 (13)	−0.0151 (11)	0.0110 (11)	0.0133 (10)
C14	0.0632 (14)	0.0641 (15)	0.0537 (13)	−0.0016 (12)	−0.0052 (11)	−0.0189 (12)
N1	0.0270 (7)	0.0298 (8)	0.0558 (10)	−0.0027 (6)	0.0037 (7)	−0.0041 (7)
O1	0.0500 (8)	0.0452 (8)	0.0348 (7)	0.0053 (6)	0.0014 (6)	0.0012 (6)
O2	0.0261 (6)	0.0531 (8)	0.0605 (9)	0.0008 (6)	0.0013 (6)	−0.0101 (7)
Cl1	0.0576 (3)	0.0566 (3)	0.0637 (4)	0.0163 (3)	−0.0036 (3)	0.0070 (3)
S1	0.0284 (2)	0.0337 (2)	0.0376 (2)	0.00265 (16)	0.00062 (16)	−0.00264 (17)

Geometric parameters (Å, º)

C1—C6	1.386 (2)	C9—C10	1.376 (3)
C1—C2	1.403 (2)	C9—Cl1	1.744 (2)
C1—S1	1.7655 (17)	C10—C11	1.378 (3)
C2—C3	1.392 (3)	C10—H10	0.9300

C2—C13	1.506 (3)	C11—C12	1.388 (3)
C3—C4	1.383 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.389 (3)	C13—H13A	0.9600
C4—C14	1.508 (3)	C13—H13B	0.9600
C5—C6	1.379 (3)	C13—H13C	0.9600
C5—H5	0.9300	C14—H14A	0.9600
C6—H6	0.9300	C14—H14B	0.9600
C7—C8	1.383 (3)	C14—H14C	0.9600
C7—C12	1.391 (3)	N1—S1	1.6394 (15)
C7—N1	1.423 (2)	N1—H1N	0.844 (15)
C8—C9	1.388 (2)	O1—S1	1.4375 (14)
C8—H8	0.9300	O2—S1	1.4331 (13)
C6—C1—C2	120.98 (16)	C11—C10—H10	120.6
C6—C1—S1	117.11 (13)	C10—C11—C12	121.16 (18)
C2—C1—S1	121.91 (13)	C10—C11—H11	119.4
C3—C2—C1	116.81 (16)	C12—C11—H11	119.4
C3—C2—C13	118.72 (17)	C11—C12—C7	119.09 (18)
C1—C2—C13	124.44 (18)	C11—C12—H12	120.5
C4—C3—C2	123.20 (17)	C7—C12—H12	120.5
C4—C3—H3	118.4	C2—C13—H13A	109.5
C2—C3—H3	118.4	C2—C13—H13B	109.5
C3—C4—C5	118.18 (17)	H13A—C13—H13B	109.5
C3—C4—C14	120.90 (19)	C2—C13—H13C	109.5
C5—C4—C14	120.92 (19)	H13A—C13—H13C	109.5
C6—C5—C4	120.60 (18)	H13B—C13—H13C	109.5
C6—C5—H5	119.7	C4—C14—H14A	109.5
C4—C5—H5	119.7	C4—C14—H14B	109.5
C5—C6—C1	120.23 (17)	H14A—C14—H14B	109.5
C5—C6—H6	119.9	C4—C14—H14C	109.5
C1—C6—H6	119.9	H14A—C14—H14C	109.5
C8—C7—C12	120.54 (16)	H14B—C14—H14C	109.5
C8—C7—N1	118.44 (16)	C7—N1—S1	123.69 (12)
C12—C7—N1	121.01 (17)	C7—N1—H1N	118.4 (15)
C7—C8—C9	118.76 (17)	S1—N1—H1N	110.5 (15)
C7—C8—H8	120.6	O2—S1—O1	117.75 (9)
C9—C8—H8	120.6	O2—S1—N1	104.39 (8)
C10—C9—C8	121.73 (18)	O1—S1—N1	109.03 (8)
C10—C9—Cl1	119.26 (15)	O2—S1—C1	111.66 (8)
C8—C9—Cl1	119.00 (15)	O1—S1—C1	106.89 (8)
C9—C10—C11	118.72 (17)	N1—S1—C1	106.59 (8)
C9—C10—H10	120.6		
C6—C1—C2—C3	−0.1 (3)	C8—C9—C10—C11	−0.4 (3)
S1—C1—C2—C3	−179.26 (13)	Cl1—C9—C10—C11	178.36 (15)
C6—C1—C2—C13	177.94 (19)	C9—C10—C11—C12	0.3 (3)
S1—C1—C2—C13	−1.2 (3)	C10—C11—C12—C7	−0.1 (3)

C1—C2—C3—C4	1.0 (3)	C8—C7—C12—C11	0.0 (3)
C13—C2—C3—C4	−177.20 (19)	N1—C7—C12—C11	179.00 (17)
C2—C3—C4—C5	−1.3 (3)	C8—C7—N1—S1	−136.09 (15)
C2—C3—C4—C14	177.68 (18)	C12—C7—N1—S1	44.9 (2)
C3—C4—C5—C6	0.8 (3)	C7—N1—S1—O2	162.85 (15)
C14—C4—C5—C6	−178.21 (19)	C7—N1—S1—O1	−70.50 (17)
C4—C5—C6—C1	0.0 (3)	C7—N1—S1—C1	44.55 (17)
C2—C1—C6—C5	−0.3 (3)	C6—C1—S1—O2	133.91 (14)
S1—C1—C6—C5	178.83 (15)	C2—C1—S1—O2	−46.93 (17)
C12—C7—C8—C9	−0.2 (3)	C6—C1—S1—O1	3.82 (16)
N1—C7—C8—C9	−179.16 (16)	C2—C1—S1—O1	−177.03 (14)
C7—C8—C9—C10	0.4 (3)	C6—C1—S1—N1	−112.67 (14)
C7—C8—C9—Cl1	−178.42 (13)	C2—C1—S1—N1	66.48 (16)

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)	2.32 (2)	3.117 (2)	158 (2)

Symmetry code: (i) −*x*+2, *y*+1/2, −*z*+3/2.