Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2,5-Dichloro-*N*-cyclohexylbenzenesulfonamide

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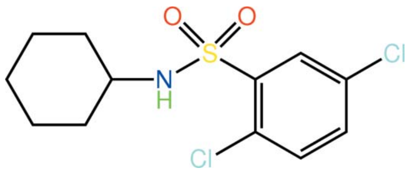
Received 21 September 2010; accepted 22 September 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.092; data-to-parameter ratio = 18.0.

The structure of the title sulfonamide, $C_{12}H_{15}Cl_2NO_2S$, features a distorted tetrahedral geometry for the S atom [maximum deviation: O—S—O = 120.23 (14)°]. One of the sulfonamide O atoms is coplanar with the benzene ring [C—C—S—O torsion angle = −174.5 (2)°], whereas the other lies well above the plane [C—C—S—O = 57.0 (3)°]. A chair conformation is found for the cyclohexyl ring. In the crystal, supramolecular chains aligned along the c axis are formed *via* N—H⋯O hydrogen bonds; these are consolidated in the three-dimensional packing by C—H⋯O contacts involving the second sulfonamide O atom.

Related literature

For background to the pharmacological uses of sulfonamides, see: Korolkovas (1988); Mandell & Sande (1992). For related structures, see: Khan *et al.* (2010); Sharif *et al.* (2010).



Experimental

Crystal data

$C_{12}H_{15}Cl_2NO_2S$
M_r = 308.21
Monoclinic, Cc
a = 17.4471 (12) Å
b = 10.7574 (8) Å
c = 8.2845 (6) Å
β = 111.956 (4)°
V = 1442.11 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.59 mm^{-1}
T = 293 K
0.28 × 0.14 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.692, $T_{\max}$ = 0.895
6491 measured reflections
2983 independent reflections
2492 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.092
S = 1.01
2983 reflections
166 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.24 e Å^{-3}
$\Delta\rho_{\min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 1327 Friedel pairs
Flack parameter: 0.06 (7)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1n⋯O2$^{\rm i}$	0.88 (2)	2.08 (2)	2.914 (3)	157 (2)
C4—H4⋯O1$^{\rm ii}$	0.93	2.60	3.246 (4)	127

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors are grateful to the Higher Education Commission of Pakistan for financial support to purchase the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2717).

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supporting information

Acta Cryst. (2010). E**66**, o2641   [doi:10.1107/S160053681003789X]

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S1. Comment

Sulfonamide drugs are widely used for the treatment of certain infections caused by Gram-positive and Gram-negative microorganisms, some fungi, and certain protozoa (Korolkovas, 1988; Mandell & Sande, 1992). In continuation of on-going structural studies of sulfonamide derivatives (Khan *et al.*, 2010, Sharif *et al.*, 2010), the crystal structure of title sulfonamide, (I), is described herein.

In (I), the S atom is tetrahedrally coordinated within a CNO_2 donor set with the greatest deviation manifested in the O1—S1—O2 angle of 120.23 (14) °. Whereas the sulfonamide-O1 atom is co-planar with the benzene ring [the O1—S1—C1—C2 torsion angle = -174.5 (2) °], the O2 atom lies well above the plane [O2—S1—C1—C2 = 57.0 (3) °]. The amide-H lies to the same side of the molecule as does the *ortho*-substituted Cl atom and approaches this atom at 2.85 (3) Å. The cyclohexyl ring adopts a chair conformation.

The presence of N1—H···O2 hydrogen bonding, Table 1, leads to the formation of supramolecular chains along the *c* axis, Fig. 2. Chains are consolidated in the 3-D packing by C4—H···O1 interactions, Fig. 3 and Table 1.

S2. Experimental

To 2,5-dichlorobenzenesulfonyl chloride (491 mg, 2 mmol) in 10 ml distilled water, was added cyclohexylamine (229 μl, 2 mmol) with stirring at room temperature while maintaining the pH of reaction mixture at 8 by using 3% sodium carbonate solution. The progress of reaction was monitored by TLC. After consumption of reactants, the precipitates were filtered, dried and crystallized from methanol

S3. Refinement

The C-bound H atoms were geometrically placed (C–H = 0.93–0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C). The N-bound H atom was refined with the distance restraint N–H = 0.88±0.01 Å, and with U_{iso}(H) = 1.2U_{eq}(N). In the final refinement two low angle reflections evidently effected by the beam stop were omitted, *i.e.* (110) and ($\bar{1}$10).

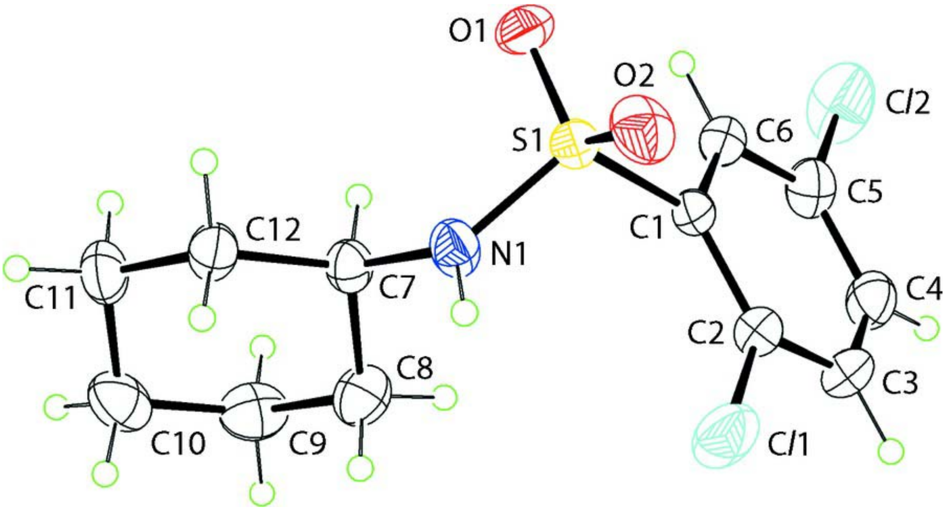

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

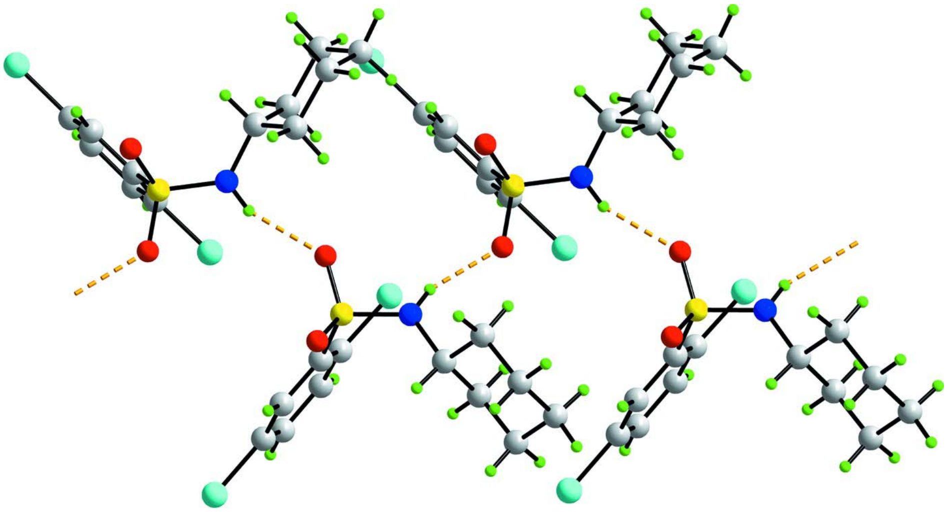

Figure 2

Supramolecular chain formation along *c* in (I) mediated by N—H···O hydrogen bonding (orange dashed lines).

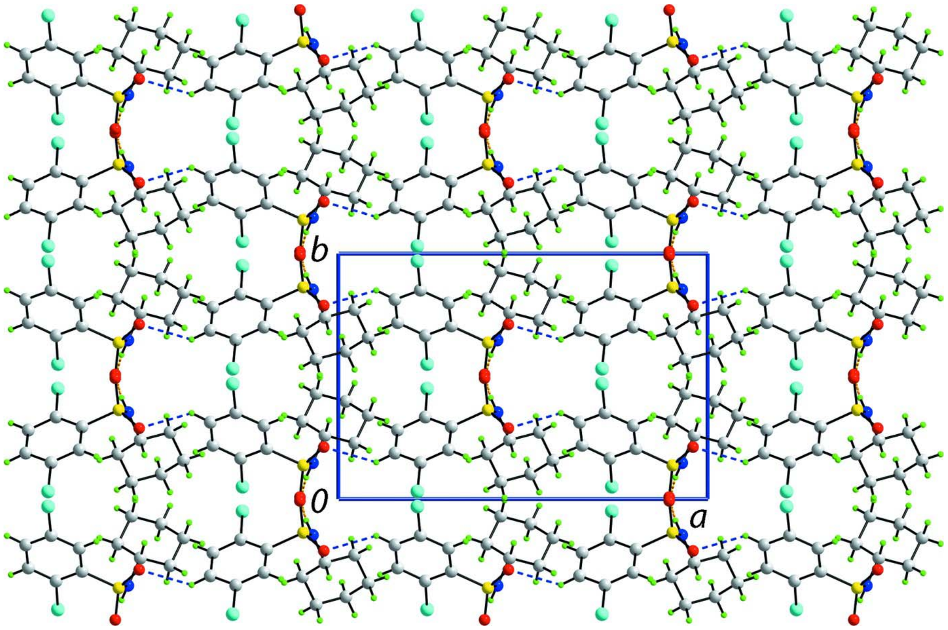

Figure 3

Unit-cell contents shown in projection down the *c* axis in (I). N—H···O hydrogen bonds (orange dashed lines) down the *c* axis are largely obscured. The C–H···O contacts are shown as blue dashed lines.

2,5-Dichloro-*N*-cyclohexylbenzenesulfonamide

Crystal data

$C_{12}H_{15}Cl_2NO_2S$
$M_r = 308.21$
Monoclinic, *Cc*
Hall symbol: C -2yc
$a = 17.4471$ (12) Å
$b = 10.7574$ (8) Å
$c = 8.2845$ (6) Å
$\beta = 111.956$ (4)°
$V = 1442.11$ (18) Å^3
$Z = 4$

$F(000) = 640$
$D_x = 1.420$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2154 reflections
$\theta = 2.3–25.8°$
$\mu = 0.59$ mm^{-1}
$T = 293$ K
Block, colourless
0.28 × 0.14 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.692$, $T_{\max} = 0.895$

6491 measured reflections
2983 independent reflections
2492 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.029$
$\theta_{\max} = 27.5°$, $\theta_{\min} = 3.8°$
$h = -22\rightarrow22$
$k = -13\rightarrow10$
$l = -10\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.092$

$S = 1.01$
2983 reflections
166 parameters
3 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

$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.24$ e Å^{-3}

$\Delta\rho_{min} = -0.19$ e Å^{-3}

Absolute structure: Flack (1983), 1327 Friedel pairs

Absolute structure parameter: 0.06 (7)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.74062 (5)	0.95016 (10)	0.40157 (10)	0.0731 (3)
Cl2	0.71411 (6)	0.53058 (11)	0.89049 (15)	0.0905 (4)
S1	0.90528 (4)	0.86294 (6)	0.72894 (8)	0.04118 (17)
O1	0.96062 (11)	0.7885 (2)	0.8655 (3)	0.0571 (6)
O2	0.89603 (14)	0.99251 (19)	0.7564 (3)	0.0608 (6)
N1	0.93057 (15)	0.8530 (2)	0.5645 (3)	0.0457 (6)
H1n	0.9127 (18)	0.912 (2)	0.486 (3)	0.055*
C1	0.80608 (14)	0.7947 (2)	0.6796 (3)	0.0373 (6)
C2	0.73570 (17)	0.8342 (3)	0.5422 (4)	0.0448 (7)
C3	0.66045 (17)	0.7810 (3)	0.5144 (4)	0.0540 (8)
H3	0.6137	0.8089	0.4232	0.065*
C4	0.65355 (18)	0.6871 (3)	0.6199 (4)	0.0533 (7)
H4	0.6027	0.6499	0.5995	0.064*
C5	0.72223 (19)	0.6489 (3)	0.7549 (4)	0.0510 (7)
C6	0.79885 (17)	0.7007 (3)	0.7871 (3)	0.0436 (6)
H6	0.8450	0.6729	0.8798	0.052*
C7	0.96379 (16)	0.7403 (2)	0.5145 (4)	0.0392 (6)
H7	0.9751	0.6793	0.6085	0.047*
C8	0.90286 (18)	0.6836 (3)	0.3500 (4)	0.0550 (8)
H8A	0.8874	0.7450	0.2575	0.066*
H8B	0.8533	0.6593	0.3689	0.066*
C9	0.9399 (2)	0.5697 (3)	0.2951 (5)	0.0608 (9)
H9A	0.9499	0.5050	0.3821	0.073*
H9B	0.9008	0.5379	0.1858	0.073*
C10	1.0206 (2)	0.6020 (3)	0.2741 (4)	0.0624 (9)
H10A	1.0442	0.5274	0.2457	0.075*
H10B	1.0099	0.6605	0.1791	0.075*
C11	1.0810 (2)	0.6580 (3)	0.4394 (4)	0.0564 (8)

H11A	1.1308	0.6823	0.4216	0.068*
H11B	1.0961	0.5961	0.5313	0.068*
C12	1.04439 (17)	0.7717 (3)	0.4958 (4)	0.0471 (7)
H12A	1.0834	0.8022	0.6061	0.056*
H12B	1.0352	0.8372	0.4101	0.056*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0545 (5)	0.0915 (7)	0.0708 (6)	0.0203 (4)	0.0206 (4)	0.0393 (5)
Cl2	0.0742 (6)	0.0882 (7)	0.1198 (9)	0.0010 (5)	0.0486 (6)	0.0425 (6)
S1	0.0346 (3)	0.0513 (4)	0.0368 (3)	−0.0019 (3)	0.0124 (2)	−0.0063 (3)
O1	0.0349 (10)	0.0900 (16)	0.0400 (11)	−0.0033 (10)	0.0066 (9)	0.0081 (10)
O2	0.0674 (14)	0.0549 (12)	0.0639 (14)	−0.0091 (11)	0.0290 (11)	−0.0221 (11)
N1	0.0516 (14)	0.0457 (14)	0.0473 (14)	0.0123 (10)	0.0273 (12)	0.0108 (10)
C1	0.0318 (12)	0.0465 (15)	0.0337 (13)	0.0026 (11)	0.0122 (10)	−0.0074 (11)
C2	0.0385 (14)	0.0551 (17)	0.0391 (15)	0.0093 (12)	0.0127 (12)	0.0014 (13)
C3	0.0327 (13)	0.076 (2)	0.0446 (18)	0.0084 (15)	0.0045 (13)	−0.0017 (15)
C4	0.0370 (15)	0.0645 (19)	0.0588 (18)	−0.0071 (14)	0.0183 (14)	−0.0115 (16)
C5	0.0482 (17)	0.0492 (18)	0.0612 (18)	0.0026 (13)	0.0267 (15)	0.0045 (15)
C6	0.0366 (14)	0.0536 (17)	0.0415 (15)	0.0052 (12)	0.0155 (12)	0.0037 (13)
C7	0.0409 (13)	0.0369 (14)	0.0438 (15)	0.0050 (11)	0.0204 (12)	0.0045 (11)
C8	0.0445 (16)	0.0543 (18)	0.0625 (19)	−0.0063 (13)	0.0159 (14)	−0.0042 (15)
C9	0.070 (2)	0.0468 (18)	0.0565 (19)	−0.0033 (15)	0.0132 (17)	−0.0089 (14)
C10	0.084 (2)	0.0563 (18)	0.0522 (19)	0.0169 (17)	0.0323 (18)	−0.0001 (15)
C11	0.0528 (17)	0.066 (2)	0.061 (2)	0.0148 (15)	0.0330 (16)	0.0026 (16)
C12	0.0381 (14)	0.0541 (17)	0.0512 (16)	0.0005 (12)	0.0192 (13)	−0.0029 (13)

Geometric parameters (Å, º)

Cl1—C2	1.731 (3)	C7—C8	1.508 (4)
Cl2—C5	1.738 (3)	C7—C12	1.510 (4)
S1—O1	1.427 (2)	C7—H7	0.9800
S1—O2	1.431 (2)	C8—C9	1.532 (5)
S1—N1	1.585 (2)	C8—H8A	0.9700
S1—C1	1.781 (3)	C8—H8B	0.9700
N1—C7	1.469 (3)	C9—C10	1.521 (5)
N1—H1n	0.88 (2)	C9—H9A	0.9700
C1—C6	1.384 (4)	C9—H9B	0.9700
C1—C2	1.392 (3)	C10—C11	1.507 (5)
C2—C3	1.370 (4)	C10—H10A	0.9700
C3—C4	1.371 (5)	C10—H10B	0.9700
C3—H3	0.9300	C11—C12	1.531 (4)
C4—C5	1.362 (4)	C11—H11A	0.9700
C4—H4	0.9300	C11—H11B	0.9700
C5—C6	1.379 (4)	C12—H12A	0.9700
C6—H6	0.9300	C12—H12B	0.9700

O1—S1—O2	120.23 (14)	C12—C7—H7	108.2
O1—S1—N1	108.69 (13)	C7—C8—C9	111.0 (2)
O2—S1—N1	106.64 (13)	C7—C8—H8A	109.4
O1—S1—C1	105.20 (13)	C9—C8—H8A	109.4
O2—S1—C1	106.27 (13)	C7—C8—H8B	109.4
N1—S1—C1	109.51 (13)	C9—C8—H8B	109.4
C7—N1—S1	124.32 (19)	H8A—C8—H8B	108.0
C7—N1—H1N	117 (2)	C10—C9—C8	111.3 (3)
S1—N1—H1N	117 (2)	C10—C9—H9A	109.4
C6—C1—C2	119.0 (2)	C8—C9—H9A	109.4
C6—C1—S1	117.89 (19)	C10—C9—H9B	109.4
C2—C1—S1	123.1 (2)	C8—C9—H9B	109.4
C3—C2—C1	120.4 (3)	H9A—C9—H9B	108.0
C3—C2—Cl1	118.2 (2)	C11—C10—C9	110.5 (3)
C1—C2—Cl1	121.3 (2)	C11—C10—H10A	109.6
C2—C3—C4	120.5 (3)	C9—C10—H10A	109.6
C2—C3—H3	119.7	C11—C10—H10B	109.6
C4—C3—H3	119.7	C9—C10—H10B	109.6
C5—C4—C3	119.1 (3)	H10A—C10—H10B	108.1
C5—C4—H4	120.4	C10—C11—C12	111.5 (3)
C3—C4—H4	120.4	C10—C11—H11A	109.3
C4—C5—C6	121.9 (3)	C12—C11—H11A	109.3
C4—C5—Cl2	119.5 (2)	C10—C11—H11B	109.3
C6—C5—Cl2	118.6 (2)	C12—C11—H11B	109.3
C5—C6—C1	119.1 (3)	H11A—C11—H11B	108.0
C5—C6—H6	120.5	C7—C12—C11	111.3 (3)
C1—C6—H6	120.5	C7—C12—H12A	109.4
N1—C7—C8	111.7 (2)	C11—C12—H12A	109.4
N1—C7—C12	109.0 (2)	C7—C12—H12B	109.4
C8—C7—C12	111.5 (2)	C11—C12—H12B	109.4
N1—C7—H7	108.2	H12A—C12—H12B	108.0
C8—C7—H7	108.2		

O1—S1—N1—C7	34.9 (3)	C3—C4—C5—C6	−1.0 (5)
O2—S1—N1—C7	165.9 (2)	C3—C4—C5—Cl2	179.6 (2)
C1—S1—N1—C7	−79.5 (2)	C4—C5—C6—C1	0.4 (4)
O1—S1—C1—C6	7.6 (2)	Cl2—C5—C6—C1	179.8 (2)
O2—S1—C1—C6	−120.9 (2)	C2—C1—C6—C5	0.1 (4)
N1—S1—C1—C6	124.3 (2)	S1—C1—C6—C5	178.1 (2)
O1—S1—C1—C2	−174.5 (2)	S1—N1—C7—C8	110.4 (3)
O2—S1—C1—C2	57.0 (3)	S1—N1—C7—C12	−126.0 (2)
N1—S1—C1—C2	−57.8 (2)	N1—C7—C8—C9	177.4 (3)
C6—C1—C2—C3	0.2 (4)	C12—C7—C8—C9	55.2 (4)
S1—C1—C2—C3	−177.7 (2)	C7—C8—C9—C10	−55.8 (4)
C6—C1—C2—Cl1	−179.3 (2)	C8—C9—C10—C11	56.1 (4)
S1—C1—C2—Cl1	2.8 (3)	C9—C10—C11—C12	−55.8 (4)
C1—C2—C3—C4	−0.9 (4)	N1—C7—C12—C11	−178.7 (2)
Cl1—C2—C3—C4	178.6 (2)	C8—C7—C12—C11	−55.0 (3)

C2—C3—C4—C5	1.3 (5)	C10—C11—C12—C7	55.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*n*···O2^{i}	0.88 (2)	2.08 (2)	2.914 (3)	157 (2)
C4—H4···O1^{ii}	0.93	2.60	3.246 (4)	127

Symmetry codes: (i) $x, -y+2, z-1/2$; (ii) $x-1/2, -y+3/2, z-1/2$.