4-(5-Chloropentanamido)benzene-sulfonamide

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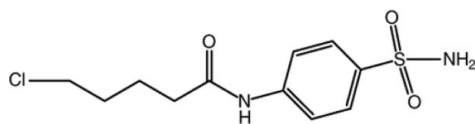
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The molecular conformation of the title compound, $C_{11}H_{15}ClN_2O_3S$, is stabilized by a C—H$\cdots$O hydrogen bond, forming an $S(6)$ ring motif. In the crystal, molecules are linked by two pairs of inversion-related N—H$\cdots$O hydrogen bonds, generating $R_2^2(8)$ and $R_2^2(20)$ ring motifs, resulting in chains running along $[0\bar{1}1]$. These chains are connected by N—H$\cdots$O hydrogen bonds along [100], forming layers parallel to (011). There are also C—H$\cdots\pi$ interactions between the layers, which consolidate the three-dimensional structure.

Related literature

Sulfonamides represent an important class of biologically active compounds. For their action as inhibitors of carbonic anhydrase enzyme, their antibacterial properties in chemotherapy, as antithyroid drugs, and for their antimicrobial properties, see: Maren (1987); Supuran (2008); Turkmen *et al.* (2005, 2011); Rami *et al.* (2011). For their antiviral properties, such as HIV protease inhibitors, see: De Clercq (2001) and as inhibitors of cysteine protease enzyme, see: Danial & Korsmeyer (2004). For related structures, see: Yalçın *et al.* (2012); Akkurt *et al.* (2010*a,b*). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).

Experimental

Crystal data

$C_{11}H_{15}ClN_2O_3S$	$a = 8.4872$ (1) Å
$M_r = 290.77$	$b = 8.7730$ (2) Å
Triclinic, $P\bar{1}$	$c = 10.4572$ (3) Å
$\alpha = 73.711$ (4)°	Mo $K\alpha$ radiation
$\beta = 85.281$ (4)°	$\mu = 0.44$ mm^{-1}
$\gamma = 63.393$ (3)°	$T = 294$ K
$V = 667.37$ (3) Å^3	$0.24 \times 0.15 \times 0.12$ mm
$Z = 2$	

Data collection

Rigaku R-AXIS RAPID-S diffractometer	20164 measured reflections
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)	4036 independent reflections
$T_{min} = 0.901$, $T_{max} = 0.949$	2815 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.068$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.169$	
$S = 1.05$	
4036 reflections	$\Delta\rho_{max} = 0.42$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
3 restraints	

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

$Cg1$ is the centroid of the C1–C6 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C5—H5$\cdots$O3	0.93	2.25	2.809 (4)	118
N1—H1$NA\cdots$O3^i	0.87 (2)	1.99 (3)	2.865 (3)	176 (3)
N1—H1$NB\cdots$O1^{ii}	0.87 (3)	2.11 (2)	2.963 (3)	166 (4)
N2—H2$N\cdots$O2^{iii}	0.88 (3)	2.17 (3)	3.021 (4)	166 (3)
C10—H10$A\cdots Cg1^{iv}$	0.97	2.96	3.771 (3)	142

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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supporting information

4-(5-Chloropentanamido)benzenesulfonamide

Hasan Türkmen, Şerife Pınar Yalçın, Mehmet Akkurt and Mustafa Durgun

S1. Comment

Sulfonamides represent an important class of biologically active compounds, with inhibitors of carbonic anhydrase enzyme, antibacterial properties in chemotherapy, antithyroid drugs, antimicrobial properties, (Maren, 1987; Rami *et al.*, 2011; Supuran, 2008; Turkmen *et al.*, 2005, 2011). Sulfonamides are also antiviral agents, such as as HIV protease inhibitors (De Clercq, 2001), and inhibitors of cysteine protease enzyme (Danial & Korsmeyer, 2004). The design and development of new sulfanilamide derivatives can help determine any structural requirements for improved biological activity. In this study, we have prepared and determined the crystal structure of the title compound.

In the 5-chloropentanamide moiety of the title compound, Fig. 1, the N2—C7—C8—C9, Cl1—C11—C10—C9 and O3 —C7—C8—C9 torsion angles are 165.7 (2), 63.2 (3) and -16.5 (3) °, respectively. The bond lengths and bond angles are within the normal range and are comparable to those reported previously for the isomer 4-(3-chloro-2,2-dimethyl-propanoylamino)-benzenesulfonamide (Yalçın *et al.*, 2012) and other related compounds (Akkurt *et al.*, 2010*a*,*b*).

A C—H⋯O hydrogen bond stabilizes the molecular conformation of the title molecule, forming a S(6) ring motif (Bernstein *et al.*, 1995; Table 1). In the crystal, neighbouring molecules are linked by two pairs of intermolecular N— H⋯O hydrogen bonds (Table 1 & Fig. 2), forming inversion dimers with $R^2_2(8)$ and $R^2_2(20)$ ring motifs, into chains running along [0 -1 1]. These chains are connected by N—H⋯O hydrogen bonds along the [100] direction, forming layers parallel to the (011) plane. C—H⋯π interactions between these layers further help in stabilizing the supramolecular structure (Table 1).

S2. Experimental

The title compound was prepared by a nucleophilic acyl substitution reaction of sulfanilamide with 5-chloropentano-ylchloride. To a solution of 3.00 g (17.42 mmol) of sulfanilamide in 50.0 ml of THF, 4.41 ml (34.84 mmol) of NEM [N-ethylmaleimide] was added. A solution of 4.47 ml (34.84 mmol) of 5-chloropentanoylchloride in 20 ml of THF was added with stirring. A white precipitate of NEM.HCl salt was immediately observed. The reaction mixture was stirred at room temperature for 24 h, the progress of which was monitored by TLC (dichloromethane/methanol 6/1 *v*/*v*). The precipitate was filtered out and the filtrate collected was evaporated *in vacuo* to leave a residue. The residue was dissolved in ethyl acetate. The organic extract was washed with 3 *M* hydrochloric acid, then with saturated sodium bicarbonate solution and finally with brine. The extract was dried (MgSO₄) and concentrated by evaporation *in vacuo* to give a residue. Recrystallization (ethanol) afforded 3.80 g (75%) the title compound as a white solid [*M*.p. 458–461 K]. Crystals suitable for X-ray diffraction were grown by slow evaporation of a solution in ethanol/chloroform/dichloro-methane (4/3/3 *v*/*v*).

S3. Refinement

The H atoms on the NH and NH_2 groups were located from a difference Fourier map and refined with distance restraints of N—H = 0.88 (1) Å, with $U_{iso}(H) = 1.2U_{eq}(N)$. The C-bound H atoms were positioned geometrically, with C—H = 0.93 and 0.97 Å for CH and CH_2 H atoms, respectively, and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.
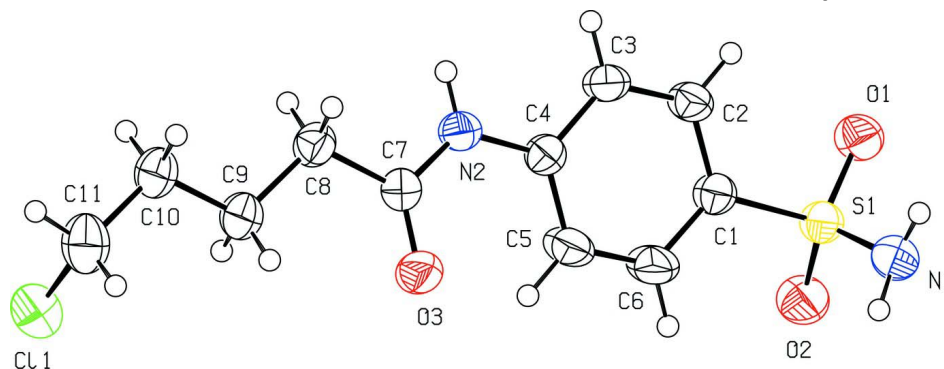
Figure 1

The view of the molecular structure of the title molecule, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.
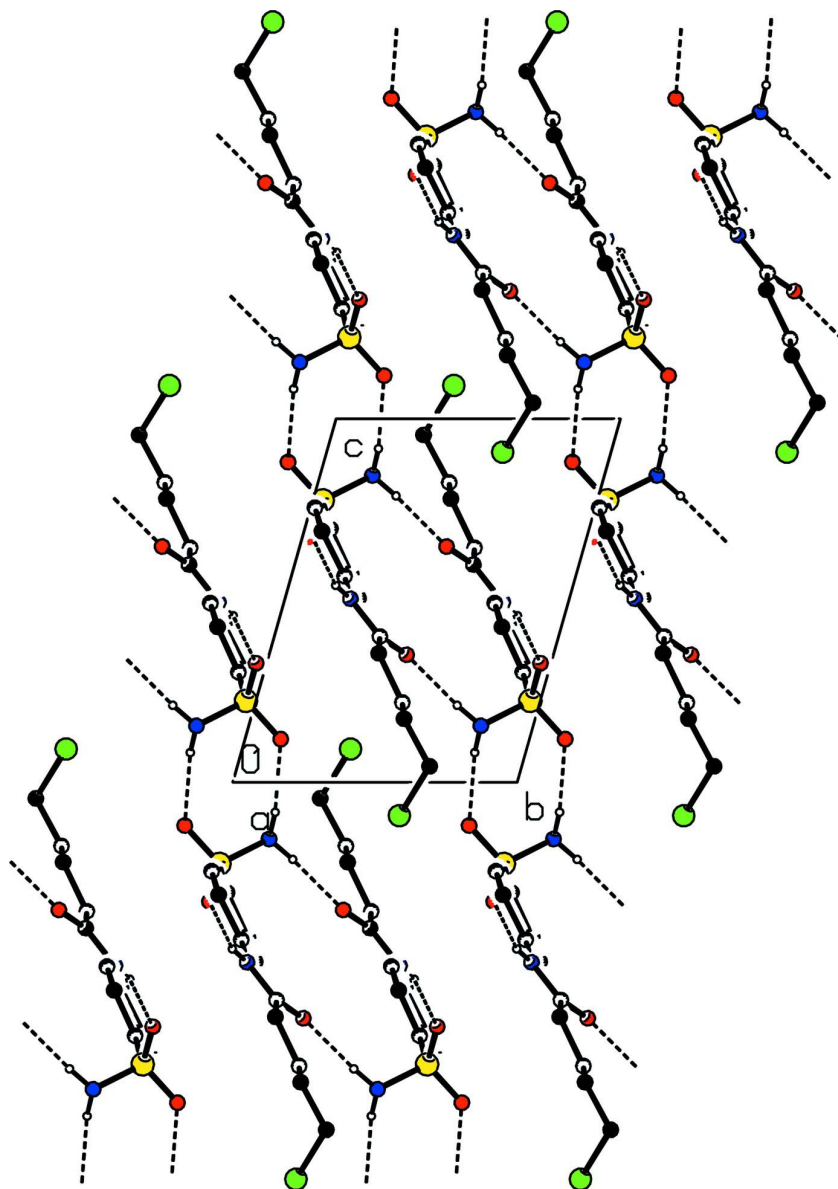
Figure 2

A partial view along the *a* axis of the crystal packing of the title compound, showing the inversion dimers formed by N—H···O hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted for clarity.

4-(5-Chloropentanamido)benzenesulfonamide

Crystal data

$C_{11}H_{15}ClN_2O_3S$	$\gamma = 63.393\ (3)°$
$M_r = 290.77$	$V = 667.37\ (3)$ Å^3
Triclinic, $P\overline{1}$	$Z = 2$
Hall symbol: -P 1	$F(000) = 304$
$a = 8.4872\ (1)$ Å	$D_x = 1.447$ Mg m^{-3}
$b = 8.7730\ (2)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$c = 10.4572\ (3)$ Å	Cell parameters from 3036 reflections
$\alpha = 73.711\ (4)°$	$\theta = 2.7–30.5°$
$\beta = 85.281\ (4)°$	$\mu = 0.44$ mm^{-1}

T = 294 K

Needle, pale yellow

0.24 × 0.15 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer	20164 measured reflections
Radiation source: Sealed Tube	4036 independent reflections
Graphite Monochromator monochromator	2815 reflections with $I > 2\sigma(I)$
Detector resolution: 10.0000 pixels mm⁻¹	R_{int} = 0.068

Rigaku R-AXIS RAPID-S
  diffractometer

Radiation source: Sealed Tube

Graphite Monochromator monochromator

Detector resolution: 10.0000 pixels mm^{-1}

dtprofit.ref scans

Absorption correction: multi-scan
  (*SORTAV*; Blessing, 1995)

T_{min} = 0.901, T_{max} = 0.949

20164 measured reflections

4036 independent reflections

2815 reflections with $I > 2\sigma(I)$

R_{int} = 0.068

θ_{max} = 30.5°, θ_{min} = 2.7°

$h = -12 \rightarrow 10$

$k = -12 \rightarrow 12$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.065

$wR(F^2)$ = 0.169

S = 1.05

4036 reflections

172 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

$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 0.2759P]$
  where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 0.42 e Å⁻³

$\Delta\rho_{min}$ = −0.35 e Å⁻³

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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	0.72069 (11)	0.61911 (12)	−0.09175 (8)	0.0692 (3)
S1	−0.03920 (8)	0.03480 (8)	0.77585 (6)	0.0419 (2)
O1	0.0449 (2)	−0.1257 (2)	0.88113 (18)	0.0500 (6)
O2	−0.1417 (3)	0.0370 (3)	0.6723 (2)	0.0572 (7)
O3	0.3519 (2)	0.4839 (2)	0.35111 (19)	0.0552 (6)
N1	−0.1676 (3)	0.1842 (3)	0.8443 (2)	0.0535 (8)
N2	0.5163 (3)	0.2301 (3)	0.5050 (2)	0.0416 (6)
C1	0.1271 (3)	0.0902 (3)	0.6970 (2)	0.0398 (7)
C2	0.2959 (3)	0.0145 (3)	0.7555 (2)	0.0426 (7)
C3	0.4213 (3)	0.0629 (3)	0.6908 (2)	0.0434 (7)
C4	0.3823 (3)	0.1871 (3)	0.5670 (2)	0.0385 (7)
C5	0.2142 (4)	0.2581 (4)	0.5068 (3)	0.0586 (9)
C6	0.0901 (4)	0.2088 (4)	0.5723 (3)	0.0611 (10)

C7	0.4969 (3)	0.3716 (3)	0.4010 (2)	0.0389 (7)
C8	0.6648 (3)	0.3843 (3)	0.3549 (3)	0.0424 (7)
C9	0.6398 (3)	0.5164 (4)	0.2196 (3)	0.0479 (8)
C10	0.8041 (4)	0.5387 (4)	0.1751 (3)	0.0481 (8)
C11	0.7774 (5)	0.6762 (4)	0.0441 (3)	0.0614 (11)
H1NA	−0.226 (4)	0.288 (2)	0.788 (3)	0.0830*
H2	0.32410	−0.06880	0.83840	0.0510*
H1NB	−0.123 (4)	0.179 (5)	0.918 (2)	0.0830*
H2N	0.623 (2)	0.165 (4)	0.542 (3)	0.0830*
H3	0.53400	0.01160	0.73060	0.0520*
H5	0.18640	0.33860	0.42260	0.0700*
H6	−0.02150	0.25670	0.53150	0.0730*
H8A	0.75400	0.26840	0.35040	0.0510*
H8B	0.70720	0.41920	0.41990	0.0510*
H9A	0.60360	0.47760	0.15390	0.0570*
H9B	0.54550	0.63050	0.22290	0.0570*
H10A	0.84370	0.57130	0.24300	0.0580*
H10B	0.89650	0.42580	0.16730	0.0580*
H11A	0.88480	0.69000	0.02530	0.0740*
H11B	0.68420	0.78900	0.05110	0.0740*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0689 (5)	0.0792 (6)	0.0525 (4)	−0.0331 (4)	0.0030 (4)	−0.0072 (4)
S1	0.0379 (3)	0.0438 (3)	0.0432 (3)	−0.0228 (3)	−0.0011 (2)	−0.0018 (2)
O1	0.0490 (10)	0.0413 (9)	0.0534 (10)	−0.0225 (8)	−0.0011 (8)	0.0015 (8)
O2	0.0533 (11)	0.0741 (13)	0.0549 (11)	−0.0404 (10)	−0.0057 (9)	−0.0101 (10)
O3	0.0387 (10)	0.0531 (11)	0.0557 (11)	−0.0175 (9)	−0.0005 (8)	0.0077 (9)
N1	0.0487 (14)	0.0476 (13)	0.0476 (13)	−0.0144 (11)	0.0006 (11)	−0.0003 (10)
N2	0.0343 (10)	0.0437 (11)	0.0411 (11)	−0.0180 (9)	−0.0022 (8)	−0.0009 (9)
C1	0.0373 (12)	0.0411 (12)	0.0416 (12)	−0.0213 (10)	−0.0008 (10)	−0.0047 (10)
C2	0.0409 (13)	0.0424 (12)	0.0399 (12)	−0.0211 (11)	−0.0056 (10)	0.0023 (10)
C3	0.0342 (12)	0.0422 (12)	0.0455 (13)	−0.0153 (10)	−0.0078 (10)	0.0000 (10)
C4	0.0357 (12)	0.0432 (12)	0.0378 (11)	−0.0203 (10)	0.0003 (9)	−0.0076 (10)
C5	0.0488 (15)	0.0720 (18)	0.0456 (14)	−0.0362 (14)	−0.0149 (12)	0.0185 (13)
C6	0.0435 (15)	0.077 (2)	0.0528 (15)	−0.0348 (15)	−0.0160 (12)	0.0159 (14)
C7	0.0376 (12)	0.0420 (12)	0.0365 (11)	−0.0177 (10)	0.0035 (10)	−0.0101 (10)
C8	0.0365 (12)	0.0432 (12)	0.0464 (13)	−0.0186 (10)	0.0051 (10)	−0.0100 (10)
C9	0.0426 (14)	0.0565 (15)	0.0474 (14)	−0.0287 (12)	0.0048 (11)	−0.0076 (12)
C10	0.0458 (14)	0.0527 (15)	0.0517 (14)	−0.0292 (12)	0.0095 (12)	−0.0126 (12)
C11	0.0676 (19)	0.0628 (18)	0.0634 (18)	−0.0410 (16)	0.0158 (15)	−0.0143 (15)

Geometric parameters (Å, º)

Cl1—C11	1.797 (4)	C7—C8	1.508 (4)
S1—O1	1.4365 (18)	C8—C9	1.516 (4)
S1—O2	1.435 (2)	C9—C10	1.512 (5)

S1—N1	1.593 (2)	C10—C11	1.504 (4)
S1—C1	1.763 (3)	C2—H2	0.9300
O3—C7	1.220 (3)	C3—H3	0.9300
N2—C4	1.408 (4)	C5—H5	0.9300
N2—C7	1.356 (3)	C6—H6	0.9300
N1—H1NA	0.87 (2)	C8—H8A	0.9700
N1—H1NB	0.87 (3)	C8—H8B	0.9700
N2—H2N	0.88 (3)	C9—H9A	0.9700
C1—C2	1.390 (4)	C9—H9B	0.9700
C1—C6	1.377 (4)	C10—H10A	0.9700
C2—C3	1.377 (4)	C10—H10B	0.9700
C3—C4	1.389 (3)	C11—H11A	0.9700
C4—C5	1.397 (4)	C11—H11B	0.9700
C5—C6	1.374 (5)		
O1—S1—O2	118.85 (13)	C1—C2—H2	120.00
O1—S1—N1	106.87 (11)	C3—C2—H2	120.00
O1—S1—C1	107.57 (11)	C2—C3—H3	120.00
O2—S1—N1	107.06 (14)	C4—C3—H3	119.00
O2—S1—C1	106.58 (13)	C4—C5—H5	120.00
N1—S1—C1	109.75 (13)	C6—C5—H5	120.00
C4—N2—C7	127.3 (2)	C1—C6—H6	119.00
S1—N1—H1NB	114 (3)	C5—C6—H6	119.00
H1NA—N1—H1NB	119 (3)	C7—C8—H8A	109.00
S1—N1—H1NA	114.0 (18)	C7—C8—H8B	109.00
C7—N2—H2N	115.5 (19)	C9—C8—H8A	109.00
C4—N2—H2N	116.9 (18)	C9—C8—H8B	109.00
S1—C1—C6	119.1 (2)	H8A—C8—H8B	108.00
S1—C1—C2	121.99 (17)	C8—C9—H9A	109.00
C2—C1—C6	118.9 (3)	C8—C9—H9B	109.00
C1—C2—C3	120.0 (2)	C10—C9—H9A	109.00
C2—C3—C4	121.1 (2)	C10—C9—H9B	109.00
C3—C4—C5	118.6 (3)	H9A—C9—H9B	108.00
N2—C4—C5	122.8 (2)	C9—C10—H10A	109.00
N2—C4—C3	118.6 (2)	C9—C10—H10B	109.00
C4—C5—C6	119.8 (3)	C11—C10—H10A	109.00
C1—C6—C5	121.5 (3)	C11—C10—H10B	109.00
O3—C7—C8	122.4 (2)	H10A—C10—H10B	108.00
N2—C7—C8	115.8 (2)	Cl1—C11—H11A	109.00
O3—C7—N2	121.8 (3)	Cl1—C11—H11B	109.00
C7—C8—C9	112.6 (2)	C10—C11—H11A	109.00
C8—C9—C10	113.4 (2)	C10—C11—H11B	109.00
C9—C10—C11	113.6 (3)	H11A—C11—H11B	108.00
Cl1—C11—C10	112.5 (3)		
O1—S1—C1—C2	−15.8 (2)	C6—C1—C2—C3	2.2 (4)
O2—S1—C1—C2	−144.3 (2)	C1—C2—C3—C4	0.0 (4)
N1—S1—C1—C2	100.2 (2)	C2—C3—C4—N2	−179.4 (2)

O1—S1—C1—C6	162.6 (2)	C2—C3—C4—C5	−2.2 (4)
O2—S1—C1—C6	34.2 (3)	N2—C4—C5—C6	179.2 (3)
N1—S1—C1—C6	−81.5 (2)	C3—C4—C5—C6	2.1 (4)
C7—N2—C4—C3	−164.6 (2)	C4—C5—C6—C1	0.1 (5)
C7—N2—C4—C5	18.3 (4)	O3—C7—C8—C9	−16.5 (3)
C4—N2—C7—O3	2.2 (4)	N2—C7—C8—C9	165.7 (2)
C4—N2—C7—C8	180.0 (2)	C7—C8—C9—C10	176.7 (2)
S1—C1—C2—C3	−179.42 (18)	C8—C9—C10—C11	−177.1 (3)
S1—C1—C6—C5	179.3 (2)	C9—C10—C11—Cl1	−63.2 (3)
C2—C1—C6—C5	−2.2 (4)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O3	0.93	2.25	2.809 (4)	118
N1—H1*NA*···O3[i]	0.87 (2)	1.99 (3)	2.865 (3)	176 (3)
N1—H1*NB*···O1[ii]	0.87 (3)	2.11 (2)	2.963 (3)	166 (4)
N2—H2*N*···O2[iii]	0.88 (3)	2.17 (3)	3.021 (4)	166 (3)
C10—H10*A*···Cg1[iv]	0.97	2.96	3.771 (3)	142

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