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N-(2-Chloro-2-nitro-1-phenylpropyl)-4-methylbenzenesulfonamide

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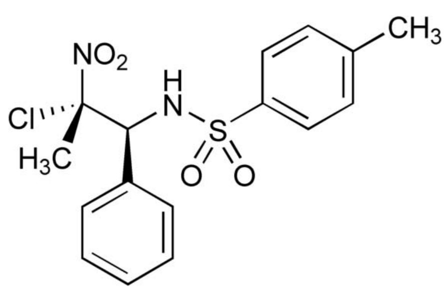
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.005 Å; R factor = 0.056; wR factor = 0.125; data-to-parameter ratio = 15.3.

In the title compound, $C_{16}H_{17}ClN_2O_4S$, the dihedral angle between the phenyl and benzene rings is 19.4 (2)°. The crystal packing is stabilized by intermolecular N—H···O hydrogen bonds, as well as by intra- and intermolecular C—H···O hydrogen bonds.

Related literature

For general background, see Kemp (1991); Qui & Silverman (2000); Orlek & Stemp (1991), Han *et al.* (2007); Li *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{17}ClN_2O_4S$
M_r = 368.83
Orthorhombic, *Pbca*
a = 7.8254 (8) Å
b = 19.610 (2) Å
c = 22.533 (3) Å
V = 3457.8 (7) $Å^3$
Z = 8
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 291 (2) K
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.901, T_{max} = 0.921
17527 measured reflections
3396 independent reflections
2467 reflections with $I > 2\sigma(I)$
R_{int} = 0.070

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.125
S = 1.01
3396 reflections
222 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.26 e $Å^{-3}$

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1	0.98	2.46	2.933 (4)	109
C3—H3···O3	0.98	2.42	2.779 (4)	101
C1—H1A···O2^{i}	0.96	2.52	3.369 (4)	147
C1—H1B···O1^{ii}	0.96	2.58	3.310 (4)	133
N2—H2A···O2^{i}	0.89 (2)	2.32 (3)	3.141 (3)	153

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2518).

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supplementary materials

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N-(2-Chloro-2-nitro-1-phenylpropyl)-4-methylbenzenesulfonamide

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Comment

Since the vicinal haloamines are important building blocks in organic and medicinal chemistry, many attentions are attracted to the aminohalogenation reactions of functionalized alkenes (Kemp, 1991; Qui & Silverman, 2000). In recent years, many new aminohalogenation processes of several kinds of functionalized alkenes have been developed (Han *et al.*, 2007), with the different nitrogen/halogen sources in the presence of metallic catalysts (Li *et al.*, 2007). However, the aminohalogenation reactions of 2-nitro-propenyl benzene was not been well documented. Recently, we synthesized the title compound (I) from 2-nitro-propenyl benzene. As part of this study, we have undertaken the X-ray crystallographic analysis of (I) in order to elucidate the conformation and configuration of this product.

The bond lengths and angles in (I) are in good agree with expected values (Allen *et al.*, 1987). The dihedral angle between the phenyl and benzene rings is 19.4 (2)°. The packing is stabilized by intermolecular N—H···O as well as intra and intermolecular C—H···O interactions in the crystal structure (Table 1).

Experimental

N-cChlorosuccinimide (400 mg, 3.0 mmol) was added into a solution of $MnSO_4$ (30.2 mg, 0.20 mmol), 1-benzyl-2-nitro-propene (163 mg, 1 mmol), tolunesulfonamide (513 mg, 3 mmol) and 4 Å molecular sieves (500 mg) in CH_2Cl_2 (5.0 ml) with nitrogen atmosphere. The resulting mixture was stirred at room temperature for 48 h. Reaction was quenched with saturated aqueous $Na_2S_2O_3$ solution. The solid precipitates were filtered off and washed with ethyl acetate (3 × 10 ml). The organic solution was concentrated and then purified *via* flash chromatography (ethyl acetate/ hexane, 1:4, *v*/*v*) provide the title compound (I) as white solid (276 mg) in yield of 75%. A colourless crystal of (I) for X-ray analysis was obtained by slow evaporation from ethyl acetate solution system.

Refinement

The H atom bonded to N was located in a difference map and refined with restraint of N—H = 0.89 (3) Å, and with U_{iso}(H) = 1.2U_{eq}(N). Other H atoms were geometrically placed and were treated as riding, with C—H distances of 0.93, 0.96 and 0.98 Å for aromatic, methyl and methine H atoms, respectively, and with U_{iso}(H) = 1.2U_{eq}(aromatic and methyne C) or 1.5U_{eq}(methyl C).

Figures

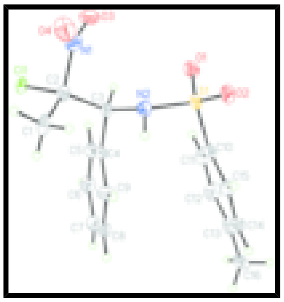


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

N-(2-Chloro-2-nitro-1-phenylpropyl)-4-methylbenzenesulfonamide

Crystal data

$C_{16}H_{17}ClN_2O_4S$	D_x = 1.417 Mg m^{-3}
M_r = 368.83	Melting point: 423.2 K
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 7468 reflections
a = 7.8254 (8) Å	θ = 2.3–27.9º
b = 19.610 (2) Å	μ = 0.36 mm^{-1}
c = 22.533 (3) Å	*T* = 291 (2) K
V = 3457.8 (7) Å^3	Block, colourless
Z = 8	0.30 × 0.26 × 0.24 mm
F_{000} = 1536	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3396 independent reflections
Radiation source: sealed tube	2467 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.070
T = 291(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	*h* = −9→8
T_{min} = 0.901, T_{max} = 0.921	*k* = −24→24
17527 measured reflections	*l* = −16→27

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.125$	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.58P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3396 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
222 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 > 2sigma(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}
C1	0.7916 (4)	0.10320 (17)	0.61740 (13)	0.0374 (7)
H1A	0.7709	0.1030	0.6594	0.056*
H1B	0.8606	0.1420	0.6072	0.056*
H1C	0.8503	0.0621	0.6064	0.056*
C2	0.6185 (4)	0.10723 (16)	0.58376 (13)	0.0333 (7)
C3	0.5059 (4)	0.16923 (14)	0.59888 (13)	0.0292 (6)
H3	0.3995	0.1645	0.5763	0.035*
C4	0.5853 (3)	0.23727 (15)	0.58138 (13)	0.0293 (6)
C5	0.5405 (4)	0.26704 (17)	0.52790 (13)	0.0362 (7)
H5	0.4651	0.2445	0.5028	0.043*
C6	0.6055 (4)	0.32954 (17)	0.51103 (14)	0.0415 (8)
H6	0.5765	0.3484	0.4745	0.050*
C7	0.7156 (4)	0.36407 (18)	0.54968 (15)	0.0437 (8)
H7	0.7570	0.4070	0.5396	0.052*
C8	0.7623 (4)	0.33466 (18)	0.60226 (15)	0.0444 (8)
H8	0.8381	0.3572	0.6273	0.053*
C9	0.6972 (4)	0.27113 (17)	0.61859 (14)	0.0414 (8)
H9	0.7290	0.2516	0.6545	0.050*
C10	0.3185 (3)	0.28495 (15)	0.69070 (12)	0.0314 (6)
C11	0.2653 (4)	0.32776 (18)	0.64579 (14)	0.0402 (7)
H11	0.2085	0.3099	0.6131	0.048*
C12	0.2959 (5)	0.39679 (18)	0.64904 (15)	0.0465 (8)
H12	0.2601	0.4252	0.6184	0.056*
C13	0.3804 (4)	0.42454 (18)	0.69822 (14)	0.0419 (8)
C14	0.4350 (4)	0.38054 (17)	0.74268 (15)	0.0423 (8)

H14	0.4939	0.3980	0.7751	0.051*
C15	0.4037 (4)	0.31171 (16)	0.73966 (13)	0.0349 (7)
H15	0.4392	0.2831	0.7702	0.042*
C16	0.4064 (4)	0.49975 (18)	0.70326 (17)	0.0491 (9)
H16A	0.3241	0.5184	0.7304	0.074*
H16B	0.3922	0.5204	0.6650	0.074*
H16C	0.5196	0.5087	0.7177	0.074*
Cl1	0.65350 (9)	0.10276 (4)	0.50665 (3)	0.03580 (19)
N1	0.5083 (4)	0.04372 (13)	0.59707 (12)	0.0407 (6)
N2	0.4620 (3)	0.16217 (13)	0.66251 (11)	0.0334 (6)
H2A	0.547 (4)	0.1794 (17)	0.6841 (15)	0.040*
O1	0.1557 (3)	0.18367 (12)	0.64347 (10)	0.0439 (6)
O2	0.2635 (3)	0.17162 (12)	0.74582 (9)	0.0432 (6)
O3	0.3667 (3)	0.04155 (12)	0.57591 (12)	0.0547 (7)
O4	0.5674 (3)	−0.00057 (13)	0.62846 (12)	0.0595 (7)
S1	0.28433 (9)	0.19677 (4)	0.68662 (3)	0.03081 (19)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0406 (16)	0.0483 (18)	0.0233 (14)	0.0236 (14)	−0.0020 (12)	−0.0033 (14)
C2	0.0365 (16)	0.0373 (16)	0.0261 (14)	0.0020 (13)	0.0056 (12)	−0.0015 (13)
C3	0.0296 (15)	0.0309 (14)	0.0270 (14)	−0.0002 (12)	0.0055 (11)	−0.0002 (12)
C4	0.0267 (14)	0.0322 (15)	0.0290 (15)	−0.0027 (11)	0.0033 (12)	−0.0024 (12)
C5	0.0344 (15)	0.0448 (17)	0.0295 (15)	−0.0005 (14)	−0.0042 (12)	0.0001 (13)
C6	0.056 (2)	0.0446 (19)	0.0242 (16)	0.0022 (15)	0.0026 (14)	0.0134 (14)
C7	0.0534 (19)	0.0383 (18)	0.0393 (19)	−0.0083 (15)	0.0079 (15)	0.0100 (15)
C8	0.0513 (19)	0.0471 (19)	0.0349 (18)	−0.0214 (16)	0.0037 (15)	0.0031 (15)
C9	0.0496 (18)	0.0467 (19)	0.0279 (15)	−0.0154 (15)	−0.0060 (14)	0.0084 (14)
C10	0.0265 (14)	0.0402 (17)	0.0275 (15)	0.0008 (12)	0.0057 (11)	0.0011 (12)
C11	0.0452 (17)	0.0461 (19)	0.0293 (16)	0.0124 (14)	−0.0042 (14)	0.0062 (14)
C12	0.059 (2)	0.0433 (19)	0.0368 (18)	0.0120 (16)	−0.0016 (16)	0.0137 (16)
C13	0.0474 (19)	0.0432 (18)	0.0350 (17)	−0.0008 (14)	0.0099 (14)	0.0068 (15)
C14	0.0435 (18)	0.0471 (19)	0.0363 (17)	−0.0077 (14)	−0.0042 (14)	−0.0025 (15)
C15	0.0373 (15)	0.0392 (16)	0.0281 (15)	−0.0009 (13)	−0.0026 (12)	0.0021 (13)
C16	0.0394 (17)	0.050 (2)	0.058 (2)	−0.0181 (15)	0.0056 (17)	0.0087 (18)
Cl1	0.0403 (4)	0.0396 (4)	0.0275 (4)	0.0017 (3)	0.0128 (3)	−0.0108 (3)
N1	0.0507 (17)	0.0366 (15)	0.0347 (15)	−0.0014 (13)	0.0048 (13)	−0.0007 (12)
N2	0.0326 (13)	0.0325 (14)	0.0351 (15)	0.0000 (10)	0.0028 (11)	−0.0005 (11)
O1	0.0323 (10)	0.0607 (15)	0.0387 (12)	−0.0070 (10)	−0.0006 (10)	−0.0059 (11)
O2	0.0565 (14)	0.0451 (13)	0.0281 (11)	−0.0041 (10)	0.0182 (11)	0.0042 (10)
O3	0.0564 (15)	0.0482 (14)	0.0597 (17)	−0.0259 (12)	0.0007 (13)	−0.0081 (12)
O4	0.0644 (16)	0.0474 (15)	0.0667 (18)	0.0107 (12)	0.0152 (14)	0.0303 (14)
S1	0.0299 (3)	0.0364 (4)	0.0261 (4)	−0.0028 (3)	0.0059 (3)	0.0004 (3)

Geometric parameters (Å, °)

C1—C2	1.555 (4)	C10—C11	1.379 (4)
C1—H1A	0.9600	C10—C15	1.392 (4)

C1—H1B	0.9600	C10—S1	1.752 (3)
C1—H1C	0.9600	C11—C12	1.377 (5)
C2—C3	1.539 (4)	C11—H11	0.9300
C2—N1	1.544 (4)	C12—C13	1.400 (5)
C2—Cl1	1.761 (3)	C12—H12	0.9300
C3—N2	1.481 (4)	C13—C14	1.390 (5)
C3—C4	1.524 (4)	C13—C16	1.493 (5)
C3—H3	0.9800	C14—C15	1.373 (4)
C4—C9	1.382 (4)	C14—H14	0.9300
C4—C5	1.384 (4)	C15—H15	0.9300
C5—C6	1.380 (4)	C16—H16A	0.9600
C5—H5	0.9300	C16—H16B	0.9600
C6—C7	1.400 (5)	C16—H16C	0.9600
C6—H6	0.9300	N1—O3	1.207 (3)
C7—C8	1.367 (5)	N1—O4	1.212 (3)
C7—H7	0.9300	N2—S1	1.639 (3)
C8—C9	1.395 (4)	N2—H2A	0.89 (3)
C8—H8	0.9300	O1—S1	1.423 (2)
C9—H9	0.9300	O2—S1	1.432 (2)

C2—C1—H1A	109.5	C11—C10—C15	119.8 (3)
C2—C1—H1B	109.5	C11—C10—S1	121.1 (2)
H1A—C1—H1B	109.5	C15—C10—S1	119.1 (2)
C2—C1—H1C	109.5	C12—C11—C10	120.5 (3)
H1A—C1—H1C	109.5	C12—C11—H11	119.8
H1B—C1—H1C	109.5	C10—C11—H11	119.8
C3—C2—N1	105.9 (2)	C11—C12—C13	120.4 (3)
C3—C2—C1	115.5 (2)	C11—C12—H12	119.8
N1—C2—C1	110.5 (2)	C13—C12—H12	119.8
C3—C2—Cl1	110.3 (2)	C14—C13—C12	118.3 (3)
N1—C2—Cl1	103.78 (19)	C14—C13—C16	121.1 (3)
C1—C2—Cl1	110.1 (2)	C12—C13—C16	120.6 (3)
N2—C3—C4	115.3 (2)	C15—C14—C13	121.3 (3)
N2—C3—C2	105.9 (2)	C15—C14—H14	119.3
C4—C3—C2	113.6 (2)	C13—C14—H14	119.3
N2—C3—H3	107.2	C14—C15—C10	119.7 (3)
C4—C3—H3	107.2	C14—C15—H15	120.2
C2—C3—H3	107.2	C10—C15—H15	120.2
C9—C4—C5	119.1 (3)	C13—C16—H16A	109.5
C9—C4—C3	121.5 (3)	C13—C16—H16B	109.5
C5—C4—C3	119.4 (3)	H16A—C16—H16B	109.5
C6—C5—C4	121.4 (3)	C13—C16—H16C	109.5
C6—C5—H5	119.3	H16A—C16—H16C	109.5
C4—C5—H5	119.3	H16B—C16—H16C	109.5
C5—C6—C7	119.0 (3)	O3—N1—O4	123.8 (3)
C5—C6—H6	120.5	O3—N1—C2	117.7 (3)
C7—C6—H6	120.5	O4—N1—C2	118.6 (3)
C8—C7—C6	120.0 (3)	C3—N2—S1	118.6 (2)
C8—C7—H7	120.0	C3—N2—H2A	109 (2)
C6—C7—H7	120.0	S1—N2—H2A	107 (2)

C7—C8—C9	120.5 (3)	O1—S1—O2	119.59 (14)
C7—C8—H8	119.8	O1—S1—N2	107.37 (14)
C9—C8—H8	119.8	O2—S1—N2	105.25 (14)
C4—C9—C8	120.0 (3)	O1—S1—C10	108.79 (14)
C4—C9—H9	120.0	O2—S1—C10	107.97 (14)
C8—C9—H9	120.0	N2—S1—C10	107.24 (13)

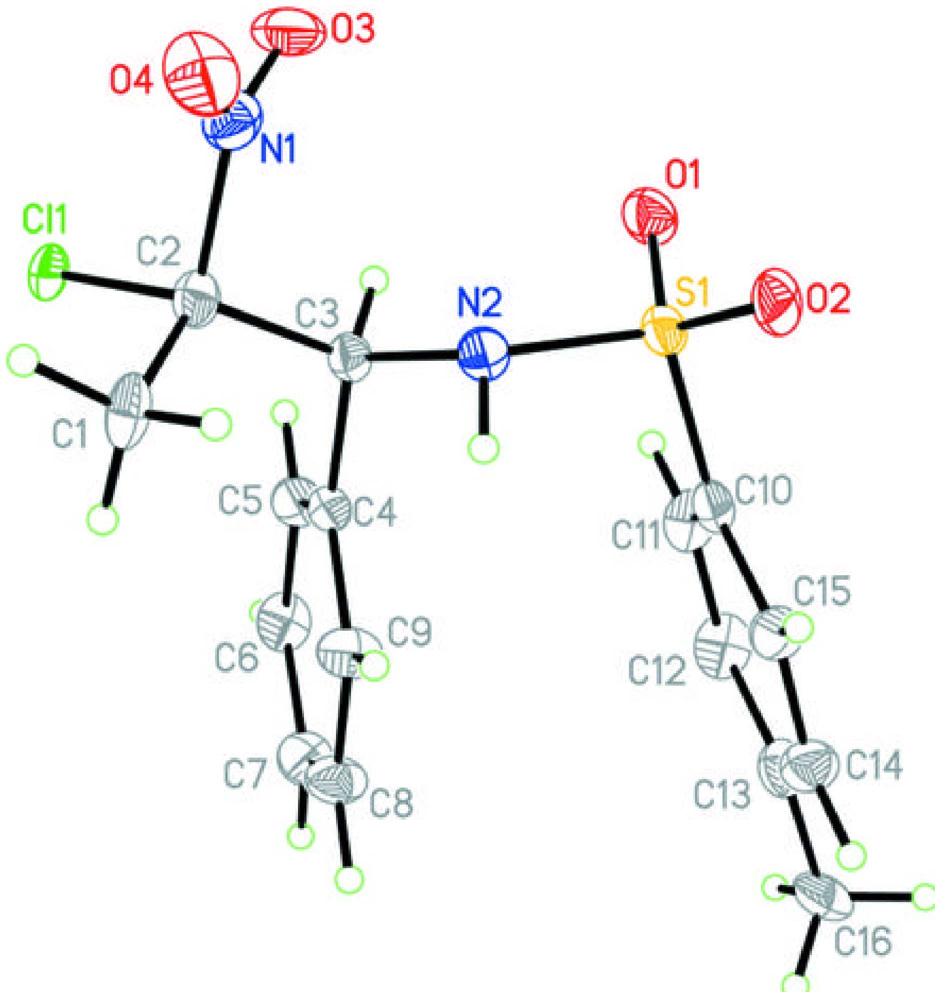
N1—C2—C3—N2	59.6 (3)	C12—C13—C14—C15	−1.7 (5)
C1—C2—C3—N2	−63.2 (3)	C16—C13—C14—C15	176.7 (3)
Cl1—C2—C3—N2	171.28 (19)	C13—C14—C15—C10	1.2 (5)
N1—C2—C3—C4	−172.9 (2)	C11—C10—C15—C14	−0.4 (4)
C1—C2—C3—C4	64.4 (3)	S1—C10—C15—C14	178.4 (2)
Cl1—C2—C3—C4	−61.2 (3)	C3—C2—N1—O3	50.9 (3)
N2—C3—C4—C9	36.7 (4)	C1—C2—N1—O3	176.7 (3)
C2—C3—C4—C9	−85.7 (3)	Cl1—C2—N1—O3	−65.3 (3)
N2—C3—C4—C5	−141.2 (3)	C3—C2—N1—O4	−128.4 (3)
C2—C3—C4—C5	96.3 (3)	C1—C2—N1—O4	−2.5 (4)
C9—C4—C5—C6	0.2 (5)	Cl1—C2—N1—O4	115.5 (3)
C3—C4—C5—C6	178.1 (3)	C4—C3—N2—S1	80.2 (3)
C4—C5—C6—C7	−1.6 (5)	C2—C3—N2—S1	−153.3 (2)
C5—C6—C7—C8	2.4 (5)	C3—N2—S1—O1	41.9 (3)
C6—C7—C8—C9	−1.8 (5)	C3—N2—S1—O2	170.3 (2)
C5—C4—C9—C8	0.5 (5)	C3—N2—S1—C10	−74.9 (2)
C3—C4—C9—C8	−177.4 (3)	C11—C10—S1—O1	−18.0 (3)
C7—C8—C9—C4	0.4 (5)	C15—C10—S1—O1	163.2 (2)
C15—C10—C11—C12	0.0 (5)	C11—C10—S1—O2	−149.2 (2)
S1—C10—C11—C12	−178.8 (3)	C15—C10—S1—O2	32.0 (3)
C10—C11—C12—C13	−0.4 (5)	C11—C10—S1—N2	97.8 (3)
C11—C12—C13—C14	1.2 (5)	C15—C10—S1—N2	−80.9 (2)
C11—C12—C13—C16	−177.1 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1	0.98	2.46	2.933 (4)	109
C3—H3···O3	0.98	2.42	2.779 (4)	101
C1—H1A···O2^{i}	0.96	2.52	3.369 (4)	147
C1—H1B···O1^{ii}	0.96	2.58	3.310 (4)	133
N2—H2A···O2^{i}	0.89 (2)	2.32 (3)	3.141 (3)	153

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

Fig. 1


O3
O4
N1
O1
Cl1
C2
S1
O2
C3
N2
C1
C5
C4
C10
C11
C15
C6
C9
C12
C7
C8
C13
C14
C16