Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Bromomethyl-2-chloromethyl-1-phenylsulfonyl-1*H*-indole

G. Chakkaravarthi,[a]* V. Dhayalan,[b] A. K. Mohanakrishnan[b] and V. Manivannan[c]

[a]Department of Physics, CPCL Polytechnic College, Chennai 600 068, India, [b]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India, and [c]Department of Physics, Presidency College, Chennai 600 005, India
Correspondence e-mail: chakkaravarthi_2005@yahoo.com

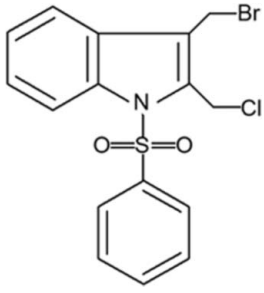
Received 19 March 2008; accepted 20 March 2008

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.010 Å; disorder in main residue; R factor = 0.066; wR factor = 0.232; data-to-parameter ratio = 13.3.

In the title compound, $C_{16}H_{13}BrClNO_2S$, the indole mean plane forms a dihedral angle of 73.59 (19)° with the phenyl ring. The molecular structure is stabilized by weak intramolecular C—H···O interactions. The Br atom is disordered over two positions with site occupancy factors of 0.7 and 0.3.

Related literature

For related crystal structures, see: Chakkaravarthi *et al.* (2007, 2008). For the biological activities of indole derivatives, see: Chai *et al.* (2006); Nieto *et al.* (2005); Olgen & Coban (2003).



Experimental

Crystal data

$C_{16}H_{13}BrClNO_2S$
M_r = 398.69
Monoclinic, $P2_1/c$
a = 11.8501 (9) Å
b = 16.3525 (13) Å
c = 8.5793 (6) Å
β = 108.766 (3)°
V = 1574.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.92 mm^{-1}
T = 295 (2) K
0.16 × 0.14 × 0.14 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.559, T_{max} = 0.665
14367 measured reflections
2770 independent reflections
1822 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.231
S = 1.06
2770 reflections
209 parameters
12 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.92 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O2	0.93	2.51	2.877 (9)	104
C13—H13···O1	0.93	2.31	2.873 (10)	118
C15—H15*B*···O2	0.97	2.17	2.939 (10)	136

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge the Sophisticated Analytical Instrument Facility, Indian Institute of Technology, Madras, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2689).

References

Bruker (2004). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chai, H., Zhao, C. & Gong, P. (2006). *Bioorg. Med. Chem.* **14**, 911–917.
Chakkaravarthi, G., Dhayalan, V., Mohanakrishnan, A. K. & Manivannan, V. (2008). *Acta Cryst.* E**64**, o542.
Chakkaravarthi, G., Ramesh, N., Mohanakrishnan, A. K. & Manivannan, V. (2007). *Acta Cryst.* E**63**, o3564.
Nieto, M. J., Alovero, F. L., Manzo, R. H. & Mazzieri, M. R. (2005). *Eur. J. Med. Chem.* **40**, 361–369.
Olgen, S. & Coban, T. (2003). *Biol. Pharm. Bull.* **26**, 736–738.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supplementary materials

Acta Cryst. (2008). E**64**, o749 [**doi:10.1107/S1600536808007678**]

3-Bromomethyl-2-chloromethyl-1-phenylsulfonyl-1*H*-indole

G. Chakkaravarthi, V. Dhayalan, A. K. Mohanakrishnan and V. Manivannan

Comment

In continuation of our studies of indole derivatives, which are known to exhibit anti-oxidant activity (Olgen & Coban, 2003), antihepatitis B virus activities (Chai *et al.*, 2006) and antibacterial (Nieto *et al.*, 2005) activities, we report the crystal structure of the title compound (I).

The geometric parameters of the molecule of (I) (Fig. 1) agree well with the reported structures (Chakkaravarthi *et al.*, 2007, 2008). The indole mean plane forms a dihedral angle of 73.59 (19)° with the phenyl ring. The N1—S1—C1 plane is almost orthogonal to indole ring (dihedral angle 82.30 (22)°) and makes 76.93 (22)° with the phenyl ring. The indole mean plane and C8—C16—BR1 plane are nearly orthogonal to each other forming a dihedral angle of 82.23 (0.29)°.

The sum of bond angles around N1 (359.99°) shows that N1 is sp^2-hybridized. The torsion angles O1—S1—N1—C14 and O2—S1—N1—C7 [17.8 (6)° and -33.4 (6)°, respectively] indicate the *syn* conformation of the sulfonyl moiety. The molecular structure is stabilized by weak intramolecular C—H···O interactions.

Experimental

1-(Phenylsulfonyl)-3-(bromomethyl)-2-methylindole (0.5 g, 1.37 mmol) was dissolved in dry ccl_4(10 ml) and then powdered *N*-chloro succinimide was added. To this, azobisisobutyronitrle (50 mg) was also added and then refluxed for 2 h on a waterbath. After the reaction was completed, succinimide was floated on the surface of the reaction mixture. It was then filtered off and washed with CCl_4 (3 ml). The solvent was removed carefully under vacuo. The crude product was recrystallized from CCl_4. Yield:76 percentage.

Refinement

H atoms were positioned geometrically and refined using riding model with C—H = 0.93 Å and U_{iso}(H) = 1.2Ueq(C) for aromatic C—H and C—H = 0.97 Å and U_{iso}(H) = 1.2Ueq(C) for CH_2. The Br atom is disordered over two positions with the occupancies of 0.709 (16) and 0.291 (16), respectively. The distances C1—C2, C2—C3, C3—C4, C4—C5, C10—C11 and C11—C12 were restrained to 1.395 (1) Å, the distances C16—BR1 and C16—Br1A were restrained to 1.91 (10) Å and the distance CL1—C15 was restrained to 1.76 (5) Å. The anisotropic thermal parameters of C15, C16, BR1, BR1A, CL1 atoms were restrained with DELU in the final cycles of the refinement (Sheldrick, 2008).

Figures

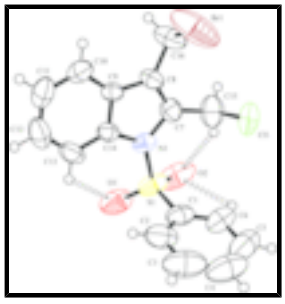


Fig. 1. The molecular structure of (I) showing the atomic labels and 50% probability displacement ellipsoids for non-H atoms. Only major parts of the disordered atoms are drawn. Intramolecular H-bonds are shown as dashed lines.

3-Bromomethyl-2-chloromethyl-1-phenylsulfonyl-1*H*-indole

Crystal data

$C_{16}H_{13}BrClNO_2S$	F_{000} = 800
M_r = 398.69	D_x = 1.682 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4037 reflections
a = 11.8501 (9) Å	θ = 2.5–25.0º
b = 16.3525 (13) Å	μ = 2.92 mm^{-1}
c = 8.5793 (6) Å	T = 295 (2) K
β = 108.766 (3)º	Block, colourless
V = 1574.1 (2) Å^3	0.16 × 0.14 × 0.14 mm
Z = 4	

Data collection

Bruker Kappa APEXII diffractometer	2770 independent reflections
Radiation source: fine-focus sealed tube	1822 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.045
T = 295(2) K	θ_{max} = 25.0º
ω and φ scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −14→13
T_{min} = 0.559, T_{max} = 0.665	k = −19→19
14367 measured reflections	l = −10→10

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.066	H-atom parameters constrained
$wR(F^2)$ = 0.231	$w = 1/[\sigma^2(F_o^2) + (0.127P)^2 + 1.9456P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2770 reflections	$\Delta\rho_{max} = 0.42$ e Å^{-3}
209 parameters	$\Delta\rho_{min} = -0.91$ e Å^{-3}
12 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.90925 (16)	−0.10531 (12)	0.5085 (3)	0.0872 (6)	
Br1	0.8135 (6)	0.1043 (5)	0.3205 (5)	0.1572 (16)	0.709 (16)
Br1A	0.7901 (4)	0.1251 (3)	0.3139 (9)	0.093 (2)	0.291 (16)
S1	0.70049 (14)	−0.13649 (11)	0.8192 (2)	0.0743 (6)	
O1	0.6340 (5)	−0.1191 (4)	0.9258 (8)	0.114 (2)	
O2	0.6743 (5)	−0.2077 (3)	0.7212 (8)	0.109 (2)	
N1	0.6795 (4)	−0.0573 (3)	0.6922 (5)	0.0539 (11)	
C1	0.8512 (6)	−0.1369 (4)	0.9320 (7)	0.0655 (17)	
C2	0.8952 (7)	−0.0826 (5)	1.0604 (8)	0.097 (2)	
H2	0.8449	−0.0449	1.0854	0.117*	
C3	1.0161 (8)	−0.0849 (8)	1.1523 (11)	0.127 (4)	
H3	1.0474	−0.0485	1.2387	0.153*	
C4	1.0885 (9)	−0.1418 (7)	1.1133 (13)	0.122 (4)	
H4	1.1688	−0.1441	1.1753	0.147*	
C5	1.0442 (8)	−0.1951 (6)	0.9845 (13)	0.113 (3)	
H5	1.0947	−0.2325	0.9590	0.135*	
C6	0.9256 (7)	−0.1932 (5)	0.8934 (10)	0.086 (2)	
H6	0.8953	−0.2295	0.8065	0.103*	
C7	0.7000 (5)	−0.0556 (3)	0.5384 (7)	0.0574 (14)	
C8	0.6681 (5)	0.0197 (3)	0.4715 (6)	0.0535 (14)	
C9	0.6288 (5)	0.0680 (3)	0.5809 (5)	0.0463 (12)	
C10	0.5887 (6)	0.1477 (4)	0.5707 (7)	0.0622 (15)	
H10	0.5837	0.1796	0.4790	0.075*	
C11	0.5563 (6)	0.1790 (4)	0.6984 (8)	0.078 (2)	
H11	0.5295	0.2327	0.6939	0.094*	
C12	0.5633 (7)	0.1313 (5)	0.8332 (9)	0.083 (2)	
H12	0.5406	0.1537	0.9182	0.099*	
C13	0.6026 (6)	0.0519 (5)	0.8471 (6)	0.0687 (18)	
H13	0.6064	0.0203	0.9388	0.082*	
C14	0.6365 (4)	0.0206 (3)	0.7184 (6)	0.0468 (12)	
C15	0.7557 (4)	−0.1232 (4)	0.4743 (11)	0.086 (2)	
H15A	0.7151	−0.1294	0.3572	0.103*	
H15B	0.7464	−0.1739	0.5278	0.103*	
C16	0.6722 (5)	0.0443 (4)	0.3064 (7)	0.084 (2)	
H16A	0.6030	0.0777	0.2520	0.101*	0.709 (16)
H16B	0.6686	−0.0042	0.2400	0.101*	0.709 (16)
H16C	0.6052	0.0802	0.2560	0.101*	0.291 (16)

H16D	0.6645	−0.0033	0.2363	0.101*	0.291 (16)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0663 (11)	0.0872 (13)	0.1176 (15)	0.0181 (9)	0.0429 (10)	−0.0109 (10)
Br1	0.235 (3)	0.159 (3)	0.106 (2)	−0.112 (2)	0.095 (2)	−0.0245 (15)
Br1A	0.063 (4)	0.105 (3)	0.098 (3)	−0.014 (2)	0.008 (2)	0.041 (3)
S1	0.0540 (9)	0.0688 (11)	0.0926 (12)	−0.0071 (8)	0.0129 (8)	0.0402 (9)
O1	0.082 (3)	0.142 (5)	0.130 (5)	0.015 (3)	0.053 (3)	0.087 (4)
O2	0.087 (3)	0.059 (3)	0.144 (5)	−0.029 (3)	−0.016 (3)	0.035 (3)
N1	0.057 (3)	0.049 (3)	0.052 (2)	0.000 (2)	0.012 (2)	0.013 (2)
C1	0.061 (4)	0.067 (4)	0.065 (4)	−0.010 (3)	0.015 (3)	0.028 (3)
C2	0.100 (6)	0.123 (7)	0.058 (4)	−0.002 (5)	0.010 (4)	0.011 (4)
C3	0.123 (8)	0.145 (10)	0.079 (6)	−0.039 (8)	−0.016 (6)	0.010 (6)
C4	0.077 (6)	0.138 (9)	0.121 (8)	−0.020 (6)	−0.010 (6)	0.051 (7)
C5	0.076 (6)	0.116 (7)	0.139 (8)	0.024 (5)	0.025 (6)	0.041 (7)
C6	0.072 (5)	0.077 (5)	0.096 (5)	0.011 (4)	0.009 (4)	0.019 (4)
C7	0.064 (4)	0.051 (3)	0.059 (3)	−0.005 (3)	0.023 (3)	−0.008 (3)
C8	0.070 (4)	0.053 (3)	0.039 (3)	−0.007 (3)	0.020 (2)	−0.002 (2)
C9	0.047 (3)	0.052 (3)	0.034 (2)	−0.002 (2)	0.005 (2)	−0.001 (2)
C10	0.066 (4)	0.050 (3)	0.061 (3)	0.006 (3)	0.007 (3)	0.005 (3)
C11	0.066 (4)	0.066 (4)	0.095 (5)	0.014 (3)	0.014 (4)	−0.021 (4)
C12	0.071 (4)	0.106 (6)	0.073 (4)	0.003 (4)	0.028 (4)	−0.036 (4)
C13	0.062 (4)	0.104 (5)	0.037 (3)	−0.001 (4)	0.013 (3)	−0.002 (3)
C14	0.043 (3)	0.057 (3)	0.038 (2)	−0.003 (2)	0.009 (2)	0.004 (2)
C15	0.079 (3)	0.074 (5)	0.110 (6)	−0.007 (4)	0.039 (4)	−0.026 (4)
C16	0.134 (5)	0.073 (4)	0.051 (3)	−0.026 (4)	0.038 (4)	0.001 (3)

Geometric parameters (Å, °)

Cl1—C15	1.772 (4)	C7—C8	1.359 (8)
Br1—C16	1.9114 (11)	C7—C15	1.481 (9)
Br1A—C16	1.9084 (11)	C8—C9	1.415 (8)
S1—O2	1.411 (6)	C8—C16	1.489 (7)
S1—O1	1.415 (6)	C9—C10	1.381 (8)
S1—N1	1.659 (4)	C9—C14	1.390 (7)
S1—C1	1.737 (6)	C10—C11	1.371 (7)
N1—C14	1.417 (7)	C10—H10	0.9300
N1—C7	1.417 (7)	C11—C12	1.375 (8)
C1—C2	1.380 (8)	C11—H11	0.9300
C1—C6	1.386 (10)	C12—C13	1.372 (10)
C2—C3	1.396 (8)	C12—H12	0.9300
C2—H2	0.9300	C13—C14	1.387 (8)
C3—C4	1.379 (9)	C13—H13	0.9300
C3—H3	0.9300	C15—H15A	0.9700
C4—C5	1.371 (9)	C15—H15B	0.9700
C4—H4	0.9300	C16—H16A	0.9700
C5—C6	1.371 (12)	C16—H16B	0.9700

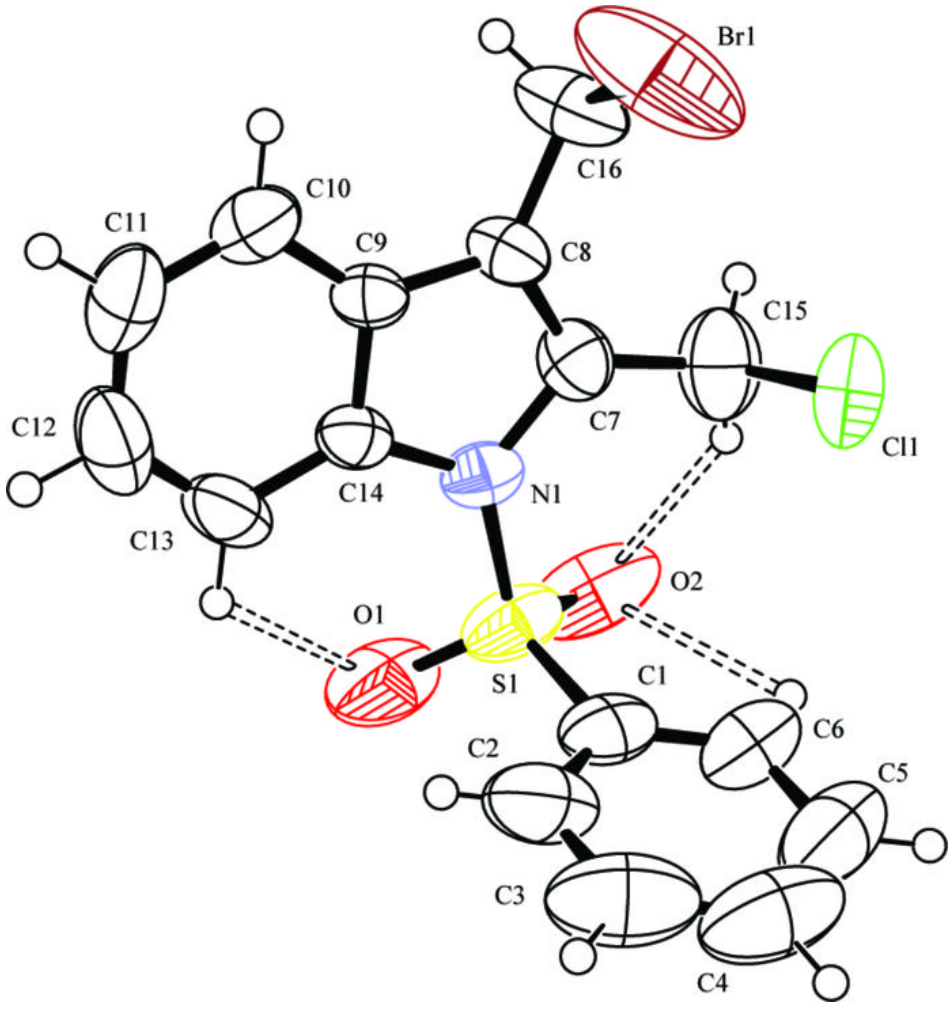
C5—H5	0.9300	C16—H16C	0.9700
C6—H6	0.9300	C16—H16D	0.9700
O2—S1—O1	119.3 (4)	C9—C10—H10	120.6
O2—S1—N1	107.2 (3)	C10—C11—C12	120.3 (6)
O1—S1—N1	105.7 (3)	C10—C11—H11	119.9
O2—S1—C1	108.5 (3)	C12—C11—H11	119.9
O1—S1—C1	109.1 (4)	C13—C12—C11	122.5 (6)
N1—S1—C1	106.2 (3)	C13—C12—H12	118.8
C14—N1—C7	108.0 (4)	C11—C12—H12	118.8
C14—N1—S1	125.7 (4)	C12—C13—C14	117.1 (6)
C7—N1—S1	126.3 (4)	C12—C13—H13	121.4
C2—C1—C6	120.8 (7)	C14—C13—H13	121.4
C2—C1—S1	119.9 (6)	C13—C14—C9	120.9 (5)
C6—C1—S1	119.3 (5)	C13—C14—N1	131.9 (5)
C1—C2—C3	119.3 (8)	C9—C14—N1	107.2 (4)
C1—C2—H2	120.4	C7—C15—Cl1	111.8 (4)
C3—C2—H2	120.4	C7—C15—H15A	109.3
C4—C3—C2	119.1 (9)	Cl1—C15—H15A	109.3
C4—C3—H3	120.4	C7—C15—H15B	109.3
C2—C3—H3	120.4	Cl1—C15—H15B	109.3
C5—C4—C3	121.2 (9)	H15A—C15—H15B	107.9
C5—C4—H4	119.4	C8—C16—Br1A	113.8 (4)
C3—C4—H4	119.4	C8—C16—Br1	112.0 (4)
C6—C5—C4	120.1 (9)	C8—C16—H16A	109.2
C6—C5—H5	119.9	Br1A—C16—H16A	97.1
C4—C5—H5	119.9	Br1—C16—H16A	109.2
C5—C6—C1	119.5 (8)	C8—C16—H16B	109.2
C5—C6—H6	120.3	Br1A—C16—H16B	118.6
C1—C6—H6	120.3	Br1—C16—H16B	109.2
C8—C7—N1	107.6 (5)	H16A—C16—H16B	107.9
C8—C7—C15	128.1 (6)	C8—C16—H16C	108.4
N1—C7—C15	124.2 (6)	Br1A—C16—H16C	94.7
C7—C8—C9	109.4 (5)	Br1—C16—H16C	107.0
C7—C8—C16	123.8 (6)	H16B—C16—H16C	111.0
C9—C8—C16	126.7 (5)	C8—C16—H16D	110.3
C10—C9—C14	120.5 (5)	Br1A—C16—H16D	119.9
C10—C9—C8	131.7 (5)	Br1—C16—H16D	110.9
C14—C9—C8	107.8 (5)	H16A—C16—H16D	104.9
C11—C10—C9	118.7 (5)	H16C—C16—H16D	108.0
C11—C10—H10	120.6		
O2—S1—N1—C14	146.0 (5)	C15—C7—C8—C16	7.1 (9)
O1—S1—N1—C14	17.8 (6)	C7—C8—C9—C10	179.3 (6)
C1—S1—N1—C14	−98.1 (5)	C16—C8—C9—C10	−2.1 (10)
O2—S1—N1—C7	−33.4 (6)	C7—C8—C9—C14	−0.3 (6)
O1—S1—N1—C7	−161.6 (5)	C16—C8—C9—C14	178.3 (5)
C1—S1—N1—C7	82.5 (5)	C14—C9—C10—C11	−0.2 (8)
O2—S1—C1—C2	−167.5 (6)	C8—C9—C10—C11	−179.8 (6)
O1—S1—C1—C2	−36.0 (6)	C9—C10—C11—C12	−0.4 (10)

N1—S1—C1—C2	77.6 (6)	C10—C11—C12—C13	0.3 (11)
O2—S1—C1—C6	11.3 (6)	C11—C12—C13—C14	0.4 (10)
O1—S1—C1—C6	142.8 (5)	C12—C13—C14—C9	−0.9 (8)
N1—S1—C1—C6	−103.7 (5)	C12—C13—C14—N1	−179.4 (6)
C6—C1—C2—C3	−0.2 (11)	C10—C9—C14—C13	0.9 (8)
S1—C1—C2—C3	178.6 (7)	C8—C9—C14—C13	−179.4 (5)
C1—C2—C3—C4	−0.5 (15)	C10—C9—C14—N1	179.7 (5)
C2—C3—C4—C5	1.1 (17)	C8—C9—C14—N1	−0.6 (6)
C3—C4—C5—C6	−1.0 (16)	C7—N1—C14—C13	179.9 (6)
C4—C5—C6—C1	0.3 (13)	S1—N1—C14—C13	0.4 (8)
C2—C1—C6—C5	0.2 (11)	C7—N1—C14—C9	1.3 (6)
S1—C1—C6—C5	−178.5 (6)	S1—N1—C14—C9	−178.2 (4)
C14—N1—C7—C8	−1.5 (6)	C8—C7—C15—Cl1	76.0 (8)
S1—N1—C7—C8	178.0 (4)	N1—C7—C15—Cl1	−98.7 (7)
C14—N1—C7—C15	174.1 (5)	C7—C8—C16—Br1A	−112.1 (6)
S1—N1—C7—C15	−6.4 (8)	C9—C8—C16—Br1A	69.5 (8)
N1—C7—C8—C9	1.1 (6)	C7—C8—C16—Br1	−98.2 (7)
C15—C7—C8—C9	−174.3 (5)	C9—C8—C16—Br1	83.4 (8)
N1—C7—C8—C16	−177.5 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2	0.93	2.51	2.877 (9)	104
C13—H13···O1	0.93	2.31	2.873 (10)	118
C15—H15B···O2	0.97	2.17	2.939 (10)	136

Fig. 1


Br1
C16
C10
C11
C9
C8
C15
C12
C7
Cl1
C14
N1
C13
O2
O1
S1
C1
C6
C2
C5
C3
C4