Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-*N*′-(5-Bromo-2-hydroxybenzylidene)-*p*-toluenesulfonohydrazide

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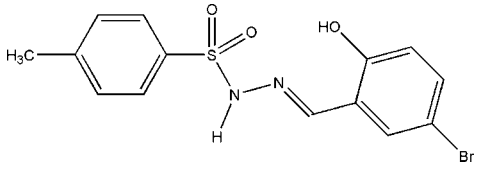
Received 3 November 2008; accepted 10 November 2008

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.079; data-to-parameter ratio = 35.5.

The title compound, $C_{14}H_{13}BrN_2O_3S$, features an intramolecular O—H···N hydrogen bond which generates an S(6) ring motif. The dihedral angle between the two benzene rings is 86.47 (6)°. Intermolecular N—H···O and C—H···O interactions link neighbouring molecules *via* $R_2^2(13)$ ring motifs, forming one-dimensional extended chains along the c axis.

Related literature

For background to sulfonamides, see: Kayser *et al.* (2004). For details of hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures and applications see: Tabatabaee *et al.* (2007); Mehrabi *et al.* (2008); Ali *et al.* (2007); Tierney *et al.* (2006); Krygowski *et al.* (1998).



Experimental

Crystal data

$C_{14}H_{13}BrN_2O_3S$
M_r = 369.23
Monoclinic, $P2_1/c$
a = 15.8890 (3) Å
b = 9.8502 (2) Å
c = 9.8702 (2) Å
β = 105.475 (1)°
V = 1488.78 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.91 mm^{-1}
T = 100.0 (1) K
0.45 × 0.34 × 0.31 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.308, T_{max} = 0.407
41486 measured reflections
7057 independent reflections
5961 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.079
S = 1.06
7057 reflections
199 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1*N2*···O2^{i}	0.851 (19)	2.002 (19)	2.8476 (13)	172.4 (19)
O1—H1*O1*···N1	0.84 (3)	1.94 (3)	2.6740 (14)	146 (3)
C7—H7*A*···O1^{i}	0.93	2.60	3.3793 (15)	142
C10—H10*A*···Br1^{ii}	0.93	2.91	3.8082 (12)	164
C12—H12*A*···O3^{iii}	0.93	2.49	3.3691 (15)	158

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

HKF and RK thank the Malaysian Government and Universiti Sains Malaysia for Science Fund grant No. 305/PFIZIK/613312. RK thanks Universiti Sains Malaysia for a post-doctoral research fellowship. HK thanks PNU for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2325).

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supporting information

Acta Cryst. (2008). E**64**, o2341 [doi:10.1107/S1600536808037069]

(*E*)-*N*′-(5-Bromo-2-hydroxybenzylidene)-*p*-toluenesulfonohydrazide

Reza Kia, Hoong-Kun Fun and Hadi Kargar

S1. Comment

Sulfonamides were the first class of antimicrobial agents to be discovered. They inhibit dihydropteroate synthetase in the bacterial folic acid pathway. Although their clinical role has diminished, they are still useful in certain situations, because of their efficacy and low cost (Krygowski *et al.*, 1998). Sulfonamides (e.g. sulfanilamide, sulfamethoxazole, sulfafurazole) are structural analogues of *p*-aminobenzoic acid (PABA) and compete with PABA to block its conversion to dihydrofolic acid. These agents are generally used in combination with other drugs to prevent or treat a number of bacterial and parasitic infections (Tierney *et al.*, 2006). With regard to all of the above, we report herein the crystal structure of the title compound, (I).

Bond lengths and angles in (I), Fig. 1, are comparable with those in related structures (Mehrabi *et al.*, 2008; Ali *et al.* 2007). An intramolecular O—H···N hydrogen bond is noted which generates a *S*(6) ring motif. The two benzene rings make the dihedral angle of 86.47 (6)°. Intermolecular N—H···O and C—H···O interactions link neighbouring molecules via $R_2^2(13)$ ring motifs to form 1-D extended chains along the *c*-axis, Fig. 2 and Table 1. The crystal structure is further stabilized by intermolecular N—H···O, C—H···Br, C—H···O and $\pi\cdots\pi$ [*Cg1*···*Cg1* = 3.9548 (8) Å; symmetry code: 2 - *x*, 1 - *y*, 2 - *z*] interactions.

S2. Experimental

p-Tosylhydrazine (2 mmol) was added to a refluxing ethanolic solution (50 ml) of 5-bromosalicylaldehyde (2 mmol). The mixture was stirred for 2 h. After cooling, the colorless crystalline solid was isolated by filtration, washed with cold ethanol, and recrystallized from an ethanol solution of (I).

S3. Refinement

H atoms bound to O1 and N2 were located from a difference Fourier map and refined freely. The remaining H atoms were positioned geometrically and refined as riding model with C—H = 0.93 - 0.96 Å, and with U_{iso}(H) = 1.2-1.5U_{eq}(C). A rotating group model was used for the methyl group. The highest residual electron density peak (1.28 eÅ^{-3}) was located 0.64 Å from Br1 and the deepest hole (-0.59 eÅ^{-3}) was located 0.59 Å from Br1.

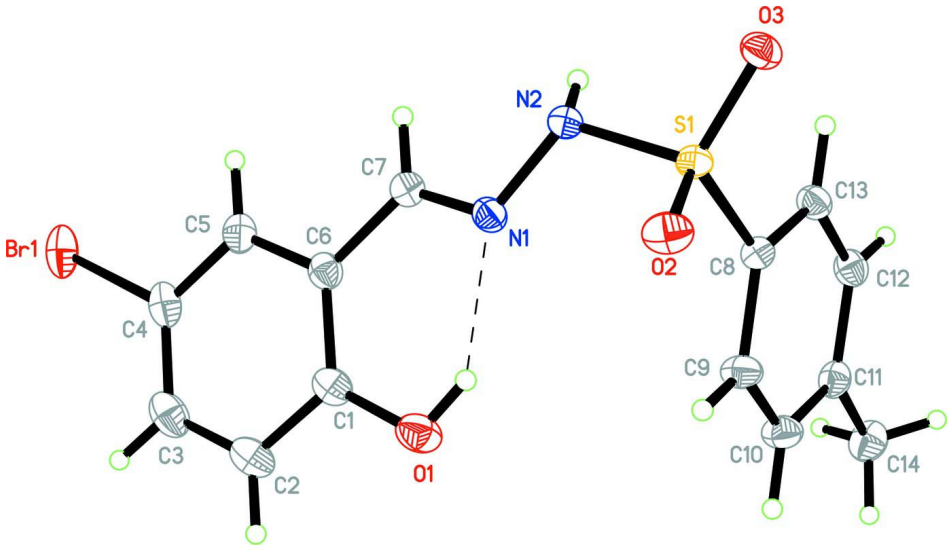

Figure 1

The molecular structure of (I), showing 50% displacement ellipsoids and the atomic numbering. The intramolecular hydrogen bond is shown as a dashed line.

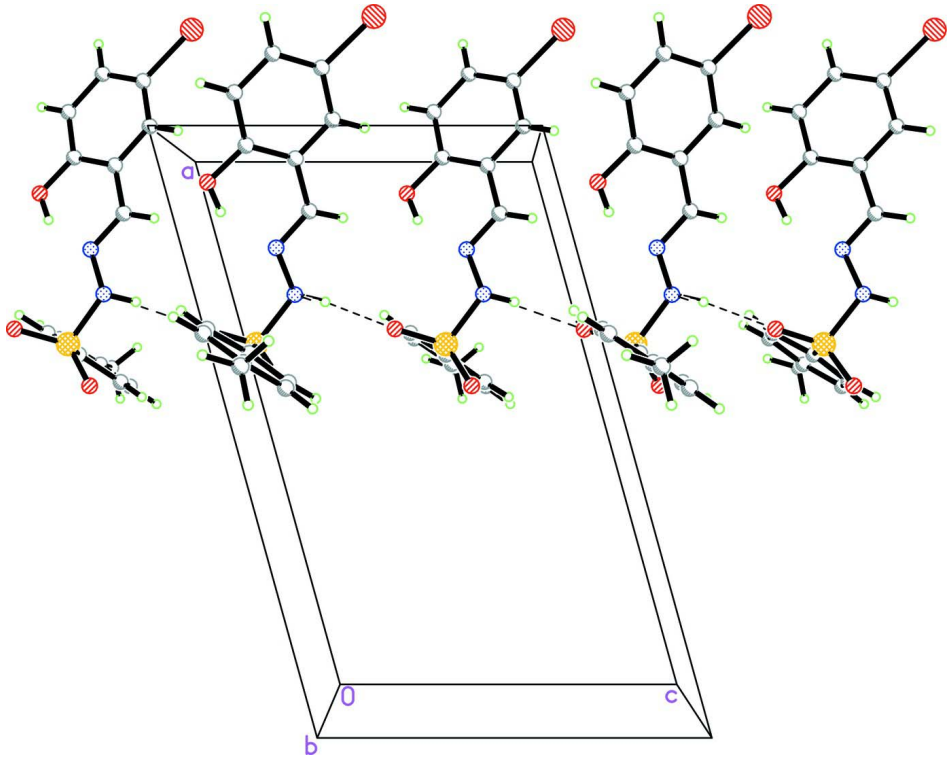

Figure 2

The crystal packing of (I) viewed down the *b*-axis, showing 1-D extended chains along the *c*-axis through N—H···O hydrogen bonding. Intermolecular hydrogen bonds are shown as dashed lines.

(*E*)-N′-(5-Bromo-2-hydroxybenzylidene)-*p*-toluenesulfonohydrazide

Crystal data

$C_{14}H_{13}BrN_2O_3S$
$M_r = 369.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.8890\ (3)$ Å
$b = 9.8502\ (2)$ Å
$c = 9.8702\ (2)$ Å
$\beta = 105.475\ (1)°$
$V = 1488.78\ (5)$ Å^3
$Z = 4$
$F(000) = 744$
$D_x = 1.647$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9995 reflections
$\theta = 2.5–37.4°$
$\mu = 2.91$ mm^{-1}
$T = 100$ K
Block, colourless
0.45 × 0.34 × 0.31 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.308$, $T_{max} = 0.407$
41486 measured reflections
7057 independent reflections
5961 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 36.0°$, $\theta_{min} = 1.3°$
$h = -26\rightarrow25$
$k = -16\rightarrow16$
$l = -16\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.079$
$S = 1.06$
7057 reflections
199 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2 + 0.5647P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 1.28$ e Å^{-3}
$\Delta\rho_{min} = -0.59$ e Å^{-3}

Special details

Experimental. The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	1.191754 (8)	0.636650 (14)	1.149579 (15)	0.02941 (4)
S1	0.645085 (17)	0.74186 (3)	0.60027 (3)	0.01571 (5)

O1	0.91105 (6)	0.56509 (11)	0.60615 (10)	0.02610 (17)
O2	0.67053 (6)	0.78504 (9)	0.47792 (9)	0.02295 (16)
O3	0.57441 (5)	0.80755 (8)	0.63807 (9)	0.02088 (15)
N1	0.80775 (6)	0.71072 (9)	0.72484 (10)	0.01769 (15)
N2	0.73002 (6)	0.77064 (9)	0.73584 (10)	0.01728 (15)
C1	0.97310 (8)	0.58383 (12)	0.72937 (13)	0.02124 (19)
C2	1.05653 (9)	0.53277 (14)	0.74093 (15)	0.0272 (2)
H2A	1.0683	0.4878	0.6652	0.033*
C3	1.12225 (8)	0.54873 (14)	0.86523 (15)	0.0279 (2)
H3A	1.1778	0.5144	0.8729	0.033*
C4	1.10429 (8)	0.61625 (12)	0.97777 (14)	0.0237 (2)
C5	1.02200 (8)	0.66843 (12)	0.96797 (13)	0.02211 (19)
H5A	1.0110	0.7138	1.0441	0.027*
C6	0.95532 (7)	0.65296 (11)	0.84359 (12)	0.01914 (18)
C7	0.86985 (7)	0.70900 (11)	0.83885 (12)	0.01990 (18)
H7A	0.8600	0.7445	0.9206	0.024*
C8	0.62834 (7)	0.56588 (10)	0.59177 (11)	0.01615 (16)
C9	0.66712 (8)	0.48649 (11)	0.50865 (12)	0.02087 (19)
H9A	0.6983	0.5265	0.4518	0.025*
C10	0.65842 (8)	0.34621 (11)	0.51211 (13)	0.0223 (2)
H10A	0.6841	0.2924	0.4567	0.027*
C11	0.61190 (7)	0.28427 (11)	0.59702 (12)	0.01949 (18)
C12	0.57195 (8)	0.36678 (11)	0.67689 (13)	0.02032 (18)
H12A	0.5393	0.3271	0.7318	0.024*
C13	0.58016 (7)	0.50714 (11)	0.67562 (12)	0.01874 (17)
H13A	0.5539	0.5612	0.7300	0.022*
C14	0.60620 (9)	0.13240 (11)	0.60308 (15)	0.0257 (2)
H14A	0.6230	0.0935	0.5250	0.039*
H14B	0.5473	0.1063	0.5986	0.039*
H14C	0.6446	0.1003	0.6894	0.039*
H1N2	0.7171 (12)	0.7519 (19)	0.812 (2)	0.027 (4)*
H1O1	0.8634 (18)	0.596 (3)	0.615 (3)	0.057 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01916 (6)	0.02972 (7)	0.03454 (8)	−0.00490 (4)	−0.00119 (5)	0.00973 (5)
S1	0.01756 (10)	0.01561 (10)	0.01476 (10)	0.00098 (7)	0.00568 (8)	0.00033 (7)
O1	0.0248 (4)	0.0345 (5)	0.0198 (4)	0.0064 (3)	0.0074 (3)	0.0009 (3)
O2	0.0322 (4)	0.0216 (3)	0.0178 (4)	0.0002 (3)	0.0114 (3)	0.0031 (3)
O3	0.0191 (3)	0.0196 (3)	0.0246 (4)	0.0042 (3)	0.0069 (3)	−0.0005 (3)
N1	0.0164 (4)	0.0173 (3)	0.0206 (4)	0.0000 (3)	0.0071 (3)	0.0001 (3)
N2	0.0165 (4)	0.0191 (4)	0.0171 (4)	−0.0003 (3)	0.0059 (3)	−0.0025 (3)
C1	0.0217 (5)	0.0216 (4)	0.0217 (5)	0.0026 (4)	0.0081 (4)	0.0037 (4)
C2	0.0245 (5)	0.0295 (5)	0.0305 (6)	0.0072 (4)	0.0122 (5)	0.0035 (5)
C3	0.0208 (5)	0.0282 (5)	0.0359 (7)	0.0051 (4)	0.0095 (5)	0.0075 (5)
C4	0.0172 (4)	0.0233 (5)	0.0290 (6)	−0.0010 (4)	0.0035 (4)	0.0069 (4)
C5	0.0189 (5)	0.0213 (4)	0.0251 (5)	−0.0017 (4)	0.0042 (4)	0.0007 (4)

C6	0.0178 (4)	0.0181 (4)	0.0218 (5)	0.0002 (3)	0.0057 (4)	0.0017 (3)
C7	0.0181 (4)	0.0199 (4)	0.0219 (5)	−0.0005 (3)	0.0057 (4)	−0.0023 (4)
C8	0.0168 (4)	0.0161 (4)	0.0154 (4)	−0.0003 (3)	0.0040 (3)	−0.0008 (3)
C9	0.0253 (5)	0.0193 (4)	0.0211 (5)	−0.0006 (4)	0.0114 (4)	−0.0023 (4)
C10	0.0263 (5)	0.0184 (4)	0.0238 (5)	0.0005 (4)	0.0094 (4)	−0.0037 (4)
C11	0.0181 (4)	0.0174 (4)	0.0209 (5)	−0.0002 (3)	0.0015 (4)	0.0004 (3)
C12	0.0187 (4)	0.0201 (4)	0.0228 (5)	−0.0018 (3)	0.0067 (4)	0.0016 (4)
C13	0.0175 (4)	0.0197 (4)	0.0205 (4)	−0.0001 (3)	0.0075 (3)	−0.0002 (3)
C14	0.0291 (6)	0.0171 (4)	0.0286 (6)	−0.0001 (4)	0.0035 (5)	0.0009 (4)

Geometric parameters (Å, º)

Br1—C4	1.8947 (13)	C5—H5A	0.9300
S1—O3	1.4292 (8)	C6—C7	1.4551 (16)
S1—O2	1.4363 (9)	C7—H7A	0.9300
S1—N2	1.6518 (10)	C8—C9	1.3907 (15)
S1—C8	1.7524 (10)	C8—C13	1.3943 (15)
O1—C1	1.3585 (16)	C9—C10	1.3900 (16)
O1—H1O1	0.84 (3)	C9—H9A	0.9300
N1—C7	1.2837 (15)	C10—C11	1.3971 (17)
N1—N2	1.3989 (13)	C10—H10A	0.9300
N2—H1N2	0.852 (19)	C11—C12	1.3972 (16)
C1—C2	1.3937 (17)	C11—C14	1.5008 (16)
C1—C6	1.4087 (16)	C12—C13	1.3891 (15)
C2—C3	1.392 (2)	C12—H12A	0.9300
C2—H2A	0.9300	C13—H13A	0.9300
C3—C4	1.388 (2)	C14—H14A	0.9600
C3—H3A	0.9300	C14—H14B	0.9600
C4—C5	1.3840 (17)	C14—H14C	0.9600
C5—C6	1.3995 (17)		
O3—S1—O2	120.16 (5)	C1—C6—C7	122.85 (11)
O3—S1—N2	103.88 (5)	N1—C7—C6	121.72 (10)
O2—S1—N2	106.13 (5)	N1—C7—H7A	119.1
O3—S1—C8	109.91 (5)	C6—C7—H7A	119.1
O2—S1—C8	109.02 (5)	C9—C8—C13	121.11 (10)
N2—S1—C8	106.82 (5)	C9—C8—S1	119.93 (8)
C1—O1—H1O1	108.2 (18)	C13—C8—S1	118.85 (8)
C7—N1—N2	115.19 (9)	C10—C9—C8	118.81 (10)
N1—N2—S1	114.34 (7)	C10—C9—H9A	120.6
N1—N2—H1N2	113.8 (13)	C8—C9—H9A	120.6
S1—N2—H1N2	110.0 (13)	C9—C10—C11	121.39 (10)
O1—C1—C2	118.13 (11)	C9—C10—H10A	119.3
O1—C1—C6	121.98 (10)	C11—C10—H10A	119.3
C2—C1—C6	119.89 (12)	C10—C11—C12	118.51 (10)
C3—C2—C1	120.33 (12)	C10—C11—C14	120.42 (11)
C3—C2—H2A	119.8	C12—C11—C14	121.07 (11)
C1—C2—H2A	119.8	C13—C12—C11	121.06 (10)

C4—C3—C2	119.57 (11)	C13—C12—H12A	119.5
C4—C3—H3A	120.2	C11—C12—H12A	119.5
C2—C3—H3A	120.2	C12—C13—C8	119.09 (10)
C5—C4—C3	120.94 (12)	C12—C13—H13A	120.5
C5—C4—Br1	118.48 (10)	C8—C13—H13A	120.5
C3—C4—Br1	120.58 (9)	C11—C14—H14A	109.5
C4—C5—C6	120.05 (12)	C11—C14—H14B	109.5
C4—C5—H5A	120.0	H14A—C14—H14B	109.5
C6—C5—H5A	120.0	C11—C14—H14C	109.5
C5—C6—C1	119.22 (11)	H14A—C14—H14C	109.5
C5—C6—C7	117.93 (10)	H14B—C14—H14C	109.5
C7—N1—N2—S1	−166.98 (8)	C5—C6—C7—N1	173.17 (11)
O3—S1—N2—N1	177.80 (7)	C1—C6—C7—N1	−7.24 (17)
O2—S1—N2—N1	−54.59 (9)	O3—S1—C8—C9	155.48 (9)
C8—S1—N2—N1	61.63 (8)	O2—S1—C8—C9	21.84 (11)
O1—C1—C2—C3	−179.66 (12)	N2—S1—C8—C9	−92.44 (10)
C6—C1—C2—C3	0.54 (19)	O3—S1—C8—C13	−28.24 (10)
C1—C2—C3—C4	−0.2 (2)	O2—S1—C8—C13	−161.87 (9)
C2—C3—C4—C5	−0.25 (19)	N2—S1—C8—C13	83.85 (9)
C2—C3—C4—Br1	178.81 (10)	C13—C8—C9—C10	−1.12 (18)
C3—C4—C5—C6	0.31 (18)	S1—C8—C9—C10	175.08 (9)
Br1—C4—C5—C6	−178.78 (9)	C8—C9—C10—C11	−0.07 (19)
C4—C5—C6—C1	0.06 (17)	C9—C10—C11—C12	1.54 (18)
C4—C5—C6—C7	179.67 (11)	C9—C10—C11—C14	−177.72 (12)
O1—C1—C6—C5	179.73 (11)	C10—C11—C12—C13	−1.87 (18)
C2—C1—C6—C5	−0.48 (17)	C14—C11—C12—C13	177.39 (11)
O1—C1—C6—C7	0.14 (17)	C11—C12—C13—C8	0.73 (18)
C2—C1—C6—C7	179.92 (11)	C9—C8—C13—C12	0.80 (17)
N2—N1—C7—C6	−178.58 (10)	S1—C8—C13—C12	−175.44 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···O2^{i}	0.851 (19)	2.002 (19)	2.8476 (13)	172.4 (19)
O1—H1*O*1···N1	0.84 (3)	1.94 (3)	2.6740 (14)	146 (3)
C7—H7*A*···O1^{i}	0.93	2.60	3.3793 (15)	142
C10—H10*A*···Br1^{ii}	0.93	2.91	3.8082 (12)	164
C12—H12*A*···O3^{iii}	0.93	2.49	3.3691 (15)	158

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) −*x*+2, *y*−1/2, −*z*+3/2; (iii) −*x*+1, *y*−1/2, −*z*+3/2.