organic compounds

2-(4-Bromobenzenesulfonamido)acetic acid

Muhammad Nadeem Arshad,[a]* Islam Ullah Khan,[a] Muhammad Shafiq,[a] Muhammad Naeem Khan[b] and Helen Stoeckli-Evans[c]

[a]Materials Chemistry Laboratory, Department of Chemistry, GC University, Lahore, Pakistan, [b]PCSIR, Laboratories Complex, Ferozpur Road, Lahore, Pakistan, and [c]Institute of Physics, Université of Neuchâtel, Rue Emile-Argand 11, CH-2009 Neuchâtel, Switzerland

Correspondence e-mail: mnachemist@hotmail.com

The title compound, $C_8H_8BrNO_4S$, a halogenated sulfonamide, was prepared by basic hydrolysis of the methyl ester. In the crystal, molecules form centrosymmetric hydrogen-bonded dimers *via* the carboxyl groups. These dimers are further linked by N—H···O interactions involving the carbonyl O and amide H atoms, forming a ribbon-like structure propagating in [010]. These ribbons are further linked *via* C—H···O interactions, forming a three-dimensional network.

Related literature

For details of the crystal structure of the methyl ester of the title compound, see: Arshad *et al.* (2008*b*). For related structures, see: Arshad *et al.* (2008*a*); Arshad *et al.* (2009). For related thiazine heterocycles, see: Arshad *et al.* (2008*c*). For hydrogen-bonding patterns, see: Bernstein *et al.* (1995).
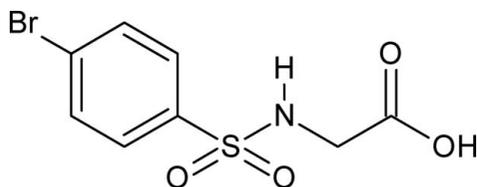
Experimental

Crystal data

$C_8H_8BrNO_4S$	$a = 5.0042$ (4) Å
$M_r = 294.12$	$b = 7.9997$ (6) Å
Triclinic, $P\bar{1}$	$c = 13.2289$ (11) Å
$\alpha = 79.691$ (4)°	Mo $K\alpha$ radiation
$\beta = 88.667$ (5)°	$\mu = 4.18$ mm^{-1}
$\gamma = 81.404$ (4)°	$T = 296$ K
$V = 515.18$ (7) Å^3	$0.28 \times 0.17 \times 0.11$ mm
$Z = 2$	

Data collection

Bruker Kappa APEXII CCD diffractometer	10359 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)	2557 independent reflections
	1243 reflections with $I > 2\sigma(I)$
$T_{min} = 0.612$, $T_{max} = 0.632$	$R_{int} = 0.056$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$	137 parameters
$wR(F^2) = 0.168$	H-atom parameters constrained
$S = 0.95$	$\Delta\rho_{max} = 1.15$ e Å^{-3}
2557 reflections	$\Delta\rho_{min} = -0.38$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···O4[i]	0.82	1.85	2.671 (5)	174
N1—H1···O4[ii]	0.86	2.38	3.124 (5)	146
C2—H2···O1[iii]	0.93	2.53	3.384 (7)	153
C3—H3···O3[iv]	0.93	2.50	3.423 (7)	170

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

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: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2493).

References

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supporting information

2-(4-Bromobenzenesulfonamido)acetic acid

Muhammad Nadeem Arshad, Islam Ullah Khan, Muhammad Shafiq, Muhammad Naeem Khan and Helen Stoeckli-Evans

S1. Comment

The title compound, (I), was prepared by basic hydrolysis of methyl (4-bromobenzenesulfonamido)acetate (II) (Arshad *et al.*, 2008*b*), in a continuation of our studies on the synthesis of thiazine related heterocycles (Arshad *et al.*, 2008*c*). We have previously reported the crystal structures of 2-(benzenesulfonamido)acetic acid (III) (Arshad *et al.*, 2008*a*) and 2-(2-iodobenzenesulfonamido)acetic acid (IV) (Arshad *et al.*, 2009).

The molecular structure of (I), Fig. 1, reveals the bond lengths and angles are similar to those found for compounds (II), (III) and (IV).

The presence of the carboxylic acid group leads to the formation of characteristic O—H⋯O hydrogen-bonded centrosymmetric dimers (Table 1 and Fig. 2). These dimers are linked *via* N1—H1⋯O4 interactions, involving the carbonyl O-atom and the H-atom of the amido group, to form a ribbon-like structure propagating in the [010] direction (Table 1 and Fig. 2). The ribbons are further linked by C—H⋯O interactions to form a 3-D network (Table 1 and Fig. 3).

It is interesting to compare the hydrogen bonding patterns in the three acids; (I), (III) and (IV). The formation of the hydrogen bonded carboxylic acid dimers is the same in all three compounds, i.e. $R_2^2(8)$ (Bernstein *et al.*, 1995). The N—H⋯O hydrogen-bonding involves the sulfonamido groups in (III) and (IV) [$R_2^2(8)$], while in (I) it involves the carbonyl O-atom (O4) and the H-atom of the amido group (Table 1). This leads to a larger hydrogen-bonded ring of the form [$R_2^2(10)$], as shown in Fig. 4.

S2. Experimental

Methyl (4-bromobenzenesulfonamido)acetate(II) (Arshad *et al.*, 2008*b*) (1.0 g, 3.247 mmol) was dissolved in an aqueous sodium hydroxide solution (10%, 10 ml). The resulting solution was refluxed for an hour. The reaction mixture was then cooled to room temperature and acidified with 1 N HCl. A white precipitate was obtained. This was filtered off, washed with distilled water and dried. Crystals were obtained by recrystallization from methanol.

S3. Refinement

The H-atoms were included in calculated positions and treated as riding atoms: O—H = 0.82 Å, N—H = 0.86 Å, C—H = 0.93 - 0.97 Å, with $U_{iso}(H) = k \times U_{eq}$(parent O–, N– or C-atom), where k = 1.5 for OH, and 1.2 for N- and C-bound H-atoms.

The maximum and minimum residual electron density peaks of 1.15 and -0.38 eÅ^{-3}, respectively, were located at 1.10 Å and 0.78 Å, respectively, from atom Br1.
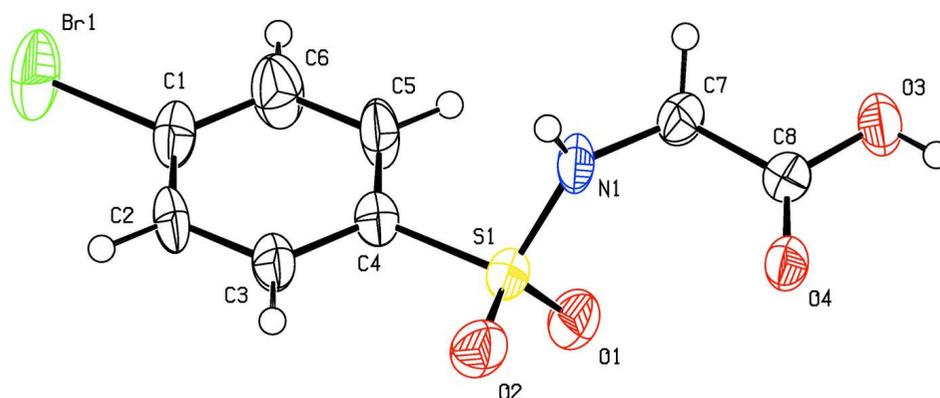
Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.
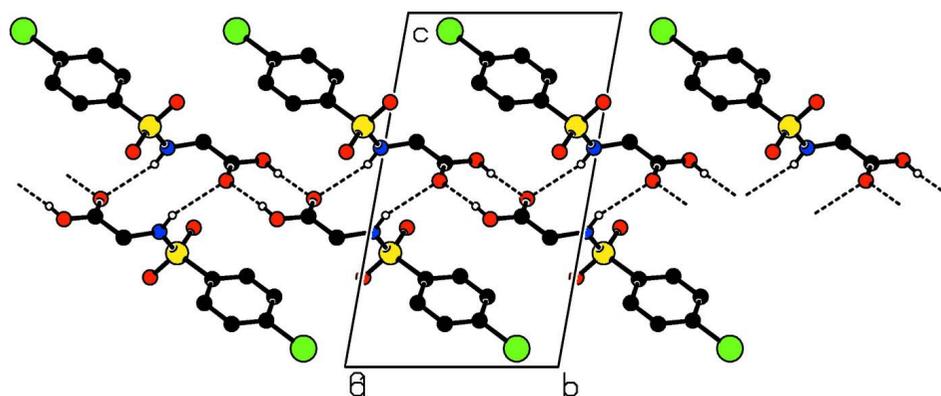
Figure 2

A view along the *a* axis of the crystal packing of compound (I), with O—H···O and N—H···O hydrogen bonds drawn as dashed lines [see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity].
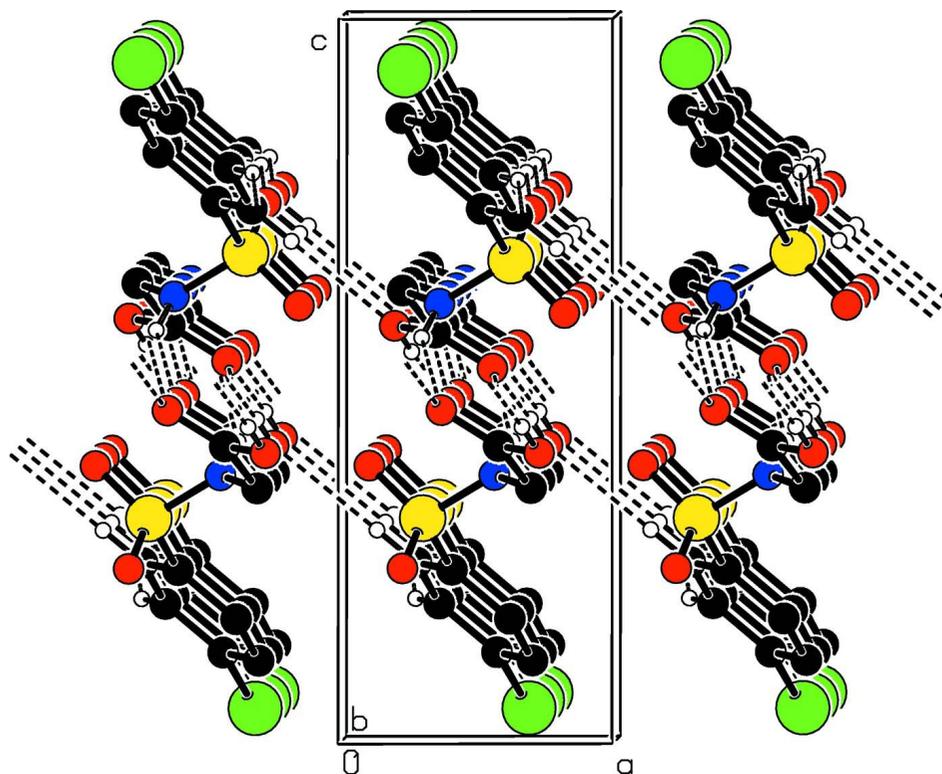
Figure 3

A view along the *b* axis of the crystal packing of (I), with O—H···O, N—H···O and C—H···O hydrogen bonds drawn as dashed lines [see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity].

Figure 4

A view of the hydrogen bonding patterns in the three acid compounds: (I), (III) and (IV). The hydrogen bonds are shown as pale-blue lines.

2-(4-Bromobenzenesulfonamido)acetic acid
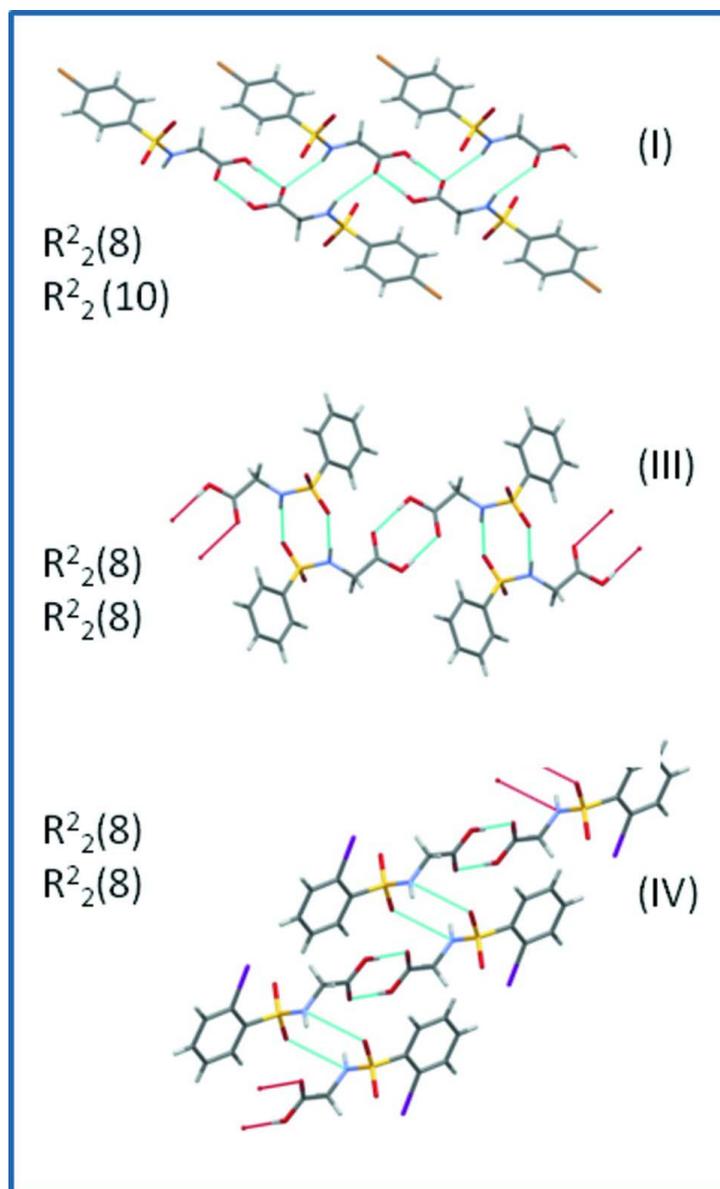
Crystal data

$C_8H_8BrNO_4S$	$\gamma = 81.404\ (4)°$
$M_r = 294.12$	$V = 515.18\ (7)$ Å^3
Triclinic, $P\bar{1}$	$Z = 2$
Hall symbol: -P 1	$F(000) = 292$
$a = 5.0042\ (4)$ Å	$D_x = 1.896$ Mg m^{-3}
$b = 7.9997\ (6)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$c = 13.2289\ (11)$ Å	Cell parameters from 1869 reflections
$\alpha = 79.691\ (4)°$	$\theta = 2.2–21.8°$
$\beta = 88.667\ (5)°$	$\mu = 4.18$ mm^{-1}

T = 296 K	0.28 × 0.17 × 0.11 mm
Needle, colorless	

Data collection

Bruker Kappa APEXII CCD diffractometer	10359 measured reflections
Radiation source: fine-focus sealed tube	2557 independent reflections
Graphite monochromator	1243 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.056
Absorption correction: multi-scan	θ_{max} = 28.3°, θ_{min} = 2.6°
(*SADABS*; Bruker, 2007)	$h = -6 \rightarrow 6$
T_{min} = 0.612, T_{max} = 0.632	$k = -10 \rightarrow 10$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)]$ = 0.058	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.168	
S = 0.95	H-atom parameters constrained
2557 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0872P)^2 + 0.2177P]$
137 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max}$ = 1.15 e Å⁻³
	$\Delta\rho_{min}$ = −0.38 e Å⁻³

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
Br1	0.70371 (17)	0.78972 (8)	0.05167 (5)	0.0774 (4)
S1	0.3354 (2)	0.11701 (17)	0.32101 (10)	0.0365 (4)
O1	0.2650 (7)	0.0106 (5)	0.2528 (3)	0.0486 (10)
O2	0.1387 (7)	0.1738 (5)	0.3930 (3)	0.0492 (10)
O3	0.7775 (7)	−0.4424 (5)	0.4169 (3)	0.0505 (10)
H3O	0.7174	−0.5221	0.4536	0.076*
O4	0.4156 (7)	−0.2871 (4)	0.4743 (3)	0.0427 (9)
N1	0.5998 (8)	0.0162 (5)	0.3816 (3)	0.0389 (10)
H1	0.6632	0.0630	0.4278	0.047*
C1	0.5927 (12)	0.5917 (7)	0.1309 (4)	0.0456 (14)
C2	0.3808 (12)	0.6089 (7)	0.1979 (5)	0.0495 (15)
H2	0.2914	0.7172	0.2039	0.059*
C3	0.3022 (11)	0.4635 (7)	0.2560 (5)	0.0453 (14)
H3	0.1575	0.4737	0.3010	0.054*

C4	0.4368 (10)	0.3039 (7)	0.2478 (4)	0.0355 (12)
C5	0.6490 (11)	0.2867 (7)	0.1801 (4)	0.0489 (15)
H5	0.7386	0.1784	0.1742	0.059*
C6	0.7271 (13)	0.4320 (8)	0.1211 (5)	0.0584 (17)
H6	0.8695	0.4221	0.0751	0.070*
C7	0.7389 (10)	−0.1470 (6)	0.3652 (4)	0.0410 (13)
H7A	0.9274	−0.1559	0.3839	0.049*
H7B	0.7316	−0.1519	0.2925	0.049*
C8	0.6253 (10)	−0.2978 (7)	0.4249 (4)	0.0368 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1144 (7)	0.0372 (4)	0.0728 (6)	−0.0176 (4)	0.0098 (4)	0.0154 (3)
S1	0.0376 (7)	0.0264 (7)	0.0458 (8)	−0.0085 (5)	0.0029 (5)	−0.0043 (6)
O1	0.053 (2)	0.037 (2)	0.060 (3)	−0.0124 (18)	−0.0073 (18)	−0.0143 (19)
O2	0.045 (2)	0.046 (2)	0.058 (2)	−0.0118 (18)	0.0163 (18)	−0.011 (2)
O3	0.050 (2)	0.027 (2)	0.069 (3)	0.0001 (18)	0.0167 (19)	−0.001 (2)
O4	0.0398 (19)	0.027 (2)	0.059 (2)	−0.0058 (16)	0.0127 (17)	−0.0008 (17)
N1	0.050 (2)	0.019 (2)	0.046 (3)	−0.0062 (19)	−0.002 (2)	−0.001 (2)
C1	0.064 (4)	0.026 (3)	0.044 (3)	−0.009 (3)	−0.004 (3)	0.002 (3)
C2	0.059 (3)	0.017 (3)	0.068 (4)	0.003 (3)	0.000 (3)	−0.006 (3)
C3	0.045 (3)	0.025 (3)	0.065 (4)	−0.002 (2)	0.008 (3)	−0.007 (3)
C4	0.041 (3)	0.027 (3)	0.038 (3)	−0.003 (2)	−0.001 (2)	−0.005 (2)
C5	0.064 (4)	0.022 (3)	0.054 (4)	0.001 (3)	0.013 (3)	0.004 (3)
C6	0.073 (4)	0.040 (4)	0.054 (4)	−0.001 (3)	0.021 (3)	0.006 (3)
C7	0.039 (3)	0.032 (3)	0.049 (3)	−0.010 (2)	0.009 (2)	0.001 (3)
C8	0.035 (3)	0.033 (3)	0.042 (3)	−0.007 (2)	0.000 (2)	−0.003 (2)

Geometric parameters (Å, º)

Br1—C1	1.886 (5)	C2—C3	1.379 (7)
S1—O1	1.429 (4)	C2—H2	0.9300
S1—O2	1.436 (4)	C3—C4	1.374 (7)
S1—N1	1.594 (4)	C3—H3	0.9300
S1—C4	1.765 (5)	C4—C5	1.380 (7)
O3—C8	1.306 (6)	C5—C6	1.382 (7)
O3—H3O	0.8200	C5—H5	0.9300
O4—C8	1.223 (6)	C6—H6	0.9300
N1—C7	1.436 (6)	C7—C8	1.499 (6)
N1—H1	0.8600	C7—H7A	0.9700
C1—C2	1.373 (8)	C7—H7B	0.9700
C1—C6	1.379 (8)		
O1—S1—O2	119.3 (2)	C3—C4—C5	120.5 (5)
O1—S1—N1	106.7 (2)	C3—C4—S1	120.6 (4)
O2—S1—N1	109.3 (2)	C5—C4—S1	118.9 (4)
O1—S1—C4	108.9 (2)	C4—C5—C6	119.4 (5)

O2—S1—C4	106.5 (2)	C4—C5—H5	120.3
N1—S1—C4	105.3 (2)	C6—C5—H5	120.3
C8—O3—H3O	109.5	C1—C6—C5	119.6 (5)
C7—N1—S1	124.8 (4)	C1—C6—H6	120.2
C7—N1—H1	117.6	C5—C6—H6	120.2
S1—N1—H1	117.5	N1—C7—C8	113.8 (4)
C2—C1—C6	121.0 (5)	N1—C7—H7A	108.8
C2—C1—Br1	119.6 (4)	C8—C7—H7A	108.8
C6—C1—Br1	119.4 (4)	N1—C7—H7B	108.8
C1—C2—C3	119.2 (5)	C8—C7—H7B	108.8
C1—C2—H2	120.4	H7A—C7—H7B	107.7
C3—C2—H2	120.4	O4—C8—O3	124.3 (5)
C4—C3—C2	120.2 (5)	O4—C8—C7	124.4 (5)
C4—C3—H3	119.9	O3—C8—C7	111.4 (4)
C2—C3—H3	119.9		
O1—S1—N1—C7	2.2 (4)	O1—S1—C4—C5	−57.6 (5)
O2—S1—N1—C7	132.5 (4)	O2—S1—C4—C5	172.6 (4)
C4—S1—N1—C7	−113.5 (4)	N1—S1—C4—C5	56.6 (5)
C6—C1—C2—C3	0.0 (9)	C3—C4—C5—C6	0.6 (9)
Br1—C1—C2—C3	−179.5 (4)	S1—C4—C5—C6	178.9 (5)
C1—C2—C3—C4	0.8 (9)	C2—C1—C6—C5	−0.5 (10)
C2—C3—C4—C5	−1.1 (8)	Br1—C1—C6—C5	179.1 (5)
C2—C3—C4—S1	−179.4 (4)	C4—C5—C6—C1	0.2 (9)
O1—S1—C4—C3	120.7 (5)	S1—N1—C7—C8	−85.5 (5)
O2—S1—C4—C3	−9.1 (5)	N1—C7—C8—O4	8.2 (8)
N1—S1—C4—C3	−125.2 (5)	N1—C7—C8—O3	−172.4 (4)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···O4[i]	0.82	1.85	2.671 (5)	174
N1—H1···O4[ii]	0.86	2.38	3.124 (5)	146
C2—H2···O1[iii]	0.93	2.53	3.384 (7)	153
C3—H3···O3[iv]	0.93	2.50	3.423 (7)	170
C3—H3···O2	0.93	2.50	2.884 (7)	105

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