

## Methyl (4-bromobenzenesulfonamido)-acetate

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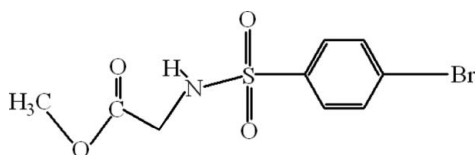
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.040;  $wR$  factor = 0.116; data-to-parameter ratio = 19.6.

The title compound,  $\text{C}_9\text{H}_{10}\text{BrNO}_4\text{S}$ , is an intermediate for the formation of benzothiazines. In the crystal structure, intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules, forming  $R_2^2(10)$  ring motifs, which are linked into a two-dimensional polymeric sheet through intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds.

### Related literature

For general background, see: Arshad *et al.* (2008); Tahir *et al.* (2008). For a related structure, see: Bornaghi *et al.* (2005). For ring motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987).



### Experimental

#### Crystal data

$\text{C}_9\text{H}_{10}\text{BrNO}_4\text{S}$   
 $M_r = 308.15$   
Triclinic,  $P\bar{1}$   
 $a = 6.0451$  (2) Å  
 $b = 7.0369$  (2) Å  
 $c = 13.8695$  (5) Å  
 $\alpha = 83.866$  (2)°  
 $\beta = 81.190$  (1)°

$\gamma = 87.027$  (2)°  
 $V = 579.31$  (3) Å<sup>3</sup>  
 $Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 3.73$  mm<sup>-1</sup>  
 $T = 296$  (2) K  
0.23 × 0.18 × 0.12 mm

#### Data collection

Bruker Kappa APEXII CCD diffractometer  
Absorption correction: multi-scan (SADABS; Bruker, 2005)  
 $T_{\min} = 0.449$ ,  $T_{\max} = 0.639$

13148 measured reflections  
2846 independent reflections  
1893 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.038$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$   
 $wR(F^2) = 0.116$   
 $S = 1.04$   
2846 reflections

145 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.53$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.59$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O2}^i$	0.86	2.41	2.987 (4)	125
$\text{C3}-\text{H3A}\cdots\text{O3}^{ii}$	0.97	2.50	3.410 (4)	156

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

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: ORTEP-3 for Windows (Farrugia, 1997) and PLATON (Spek, 2003); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2573).

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**supplementary materials**

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## Methyl (4-bromobenzenesulfonamido)acetate

M. N. Arshad, M. N. Tahir, I. U. Khan, E. Ahmad and M. Shafiq

### Comment

The glycine methyl ester is the simplest among the amino acids and the *p*-bromobenzenesulfonyl chloride is also commercially available. We have synthesized the title compound, (I), from their condensation. It has been prepared as an intermediate for the formation of benzothiazines, which are of our research interest (Arshad *et al.*, 2008; Tahir *et al.*, 2008).

In the molecule of the title compound, (I), (Fig. 1) the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges, and are comparable with the corresponding values in *N*-(2-nitrophenylsulfonyl)glycine methyl ester, (II), (Bornaghi *et al.*, 2005). Ring A (C4-C9) is, of course, planar, and the Br atom lies slightly out of the ring plane [0.069 (3) Å]. The S1-N1 bond is almost perpendicular to the ring plane, with N1-S1-C4-C5 [-83.4 (3)°] and/or N1/S1/C4/C9 [93.7 (3)°] torsion angles. The (O1/O2/C1-C3) moiety is planar, and it is oriented with respect to ring A at a dihedral angle of 87.74 (3)°.

In the crystal structure, intermolecular N-H...O hydrogen bonds (Table 1) link the molecules to form  $R_2^2(10)$  ring motifs (Bernstein *et al.*, 1995), in which they are linked to form a two dimensional polymeric sheet through intermolecular C-H...O hydrogen bonds (Table 1, Fig. 2).

### Experimental

Glycine methyl ester hydrochloride (0.246 g, 1.95 mmol) was dissolved in water (10 ml) in round bottom flask. The pH of the solution was adjusted to 8–9 using sodium carbonate (1 N), and then 4-bromo benzene sulfonyl chloride (0.5 g, 1.95 mmol) was added. The mixture was stirred for 2 h at room temperature. During the reaction, pH was strictly maintained at 8–9 as HCl produced, which lowers the pH. Colorless solid product obtained was washed, dried and recrystallized from methanol for X-ray analysis (m.p. 393–394 K).

### Refinement

H atoms were positioned geometrically, with N-H = 0.86 Å (for NH) and C-H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H, respectively, and constrained to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C,N})$ , where  $x = 1.5$  for methyl H and  $x = 1.2$  for all other H atoms.

### Figures

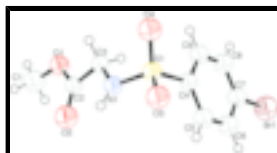


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme.

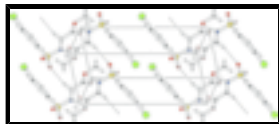


Fig. 2. A partial packing diagram. Hydrogen bonds are shown as dashed lines.

## Methyl (4-bromobenzenesulfonamido)acetate

### Crystal data

$C_9H_{10}BrNO_4S$	$Z = 2$
$M_r = 308.15$	$F_{000} = 308$
Triclinic, $PT$	$D_x = 1.767 \text{ Mg m}^{-3}$
Hall symbol: $-P 1$	Mo $K\alpha$ radiation
$a = 6.0451 (2) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 7.0369 (2) \text{ \AA}$	Cell parameters from 2846 reflections
$c = 13.8695 (5) \text{ \AA}$	$\theta = 1.5\text{--}28.3^\circ$
$\alpha = 83.866 (2)^\circ$	$\mu = 3.73 \text{ mm}^{-1}$
$\beta = 81.190 (1)^\circ$	$T = 296 (2) \text{ K}$
$\gamma = 87.027 (2)^\circ$	Prism, colorless
$V = 579.31 (3) \text{ \AA}^3$	$0.23 \times 0.18 \times 0.12 \text{ mm}$

### Data collection

Bruker Kappa APEXII CCD diffractometer	2846 independent reflections
Radiation source: fine-focus sealed tube	1893 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.038$
Detector resolution: $7.40 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 28.3^\circ$
$T = 296(2) \text{ K}$	$\theta_{\text{min}} = 1.5^\circ$
$\omega$ scans	$h = -8 \rightarrow 8$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -9 \rightarrow 9$
$T_{\text{min}} = 0.449$ , $T_{\text{max}} = 0.639$	$l = -18 \rightarrow 18$
13148 measured reflections	

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.116$	$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.5395P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
2846 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
145 parameters	$\Delta\rho_{\text{max}} = 0.53 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.59 \text{ e \AA}^{-3}$

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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.31108 (8)	0.18201 (7)	0.47759 (3)	0.0859 (2)
S1	-0.15899 (12)	0.80316 (11)	0.19808 (6)	0.0428 (2)
O1	0.4939 (4)	0.7999 (3)	-0.06466 (17)	0.0535 (6)
O2	0.2102 (5)	0.6116 (4)	-0.06458 (19)	0.0694 (8)
O3	-0.3871 (3)	0.7563 (4)	0.20253 (19)	0.0589 (6)
O4	-0.1032 (4)	0.9893 (3)	0.21559 (19)	0.0602 (6)
N1	-0.0358 (4)	0.7668 (4)	0.08966 (18)	0.0426 (6)
H1	-0.1060	0.7168	0.0497	0.051*
C1	0.6104 (7)	0.7313 (6)	-0.1532 (3)	0.0720 (12)
H1A	0.7505	0.7935	-0.1714	0.108*
H1B	0.5207	0.7589	-0.2048	0.108*
H1C	0.6379	0.5957	-0.1425	0.108*
C2	0.2962 (5)	0.7298 (4)	-0.0293 (2)	0.0411 (7)
C3	0.1966 (5)	0.8203 (5)	0.0605 (2)	0.0427 (7)
H3A	0.2818	0.7791	0.1133	0.051*
H3B	0.2032	0.9583	0.0477	0.051*
C4	-0.0381 (5)	0.6363 (4)	0.2804 (2)	0.0385 (6)
C5	-0.1158 (6)	0.4529 (5)	0.2982 (2)	0.0482 (8)
H5	-0.2363	0.4206	0.2696	0.058*
C6	-0.0151 (6)	0.3180 (5)	0.3582 (2)	0.0534 (8)
H6	-0.0672	0.1944	0.3709	0.064*
C7	0.1645 (6)	0.3690 (6)	0.3992 (2)	0.0528 (9)
C8	0.2420 (6)	0.5515 (6)	0.3828 (3)	0.0549 (9)
H8	0.3625	0.5834	0.4115	0.066*
C9	0.1398 (5)	0.6868 (5)	0.3233 (2)	0.0484 (8)
H9	0.1898	0.8111	0.3121	0.058*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.0822 (3)	0.1005 (4)	0.0689 (3)	0.0267 (3)	-0.0172 (2)	0.0133 (2)

## supplementary materials

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S1	0.0313 (4)	0.0443 (5)	0.0512 (5)	-0.0016 (3)	-0.0027 (3)	-0.0019 (3)
O1	0.0403 (12)	0.0621 (15)	0.0588 (14)	-0.0191 (10)	0.0057 (10)	-0.0201 (12)
O2	0.0744 (17)	0.0703 (18)	0.0649 (16)	-0.0429 (14)	0.0103 (13)	-0.0240 (14)
O3	0.0288 (11)	0.0740 (17)	0.0704 (16)	-0.0032 (10)	-0.0048 (10)	0.0059 (13)
O4	0.0603 (15)	0.0420 (14)	0.0770 (18)	0.0016 (11)	-0.0034 (13)	-0.0112 (12)
N1	0.0335 (13)	0.0531 (16)	0.0423 (14)	-0.0158 (11)	-0.0078 (10)	0.0004 (12)
C1	0.060 (2)	0.078 (3)	0.076 (3)	-0.016 (2)	0.020 (2)	-0.031 (2)
C2	0.0409 (16)	0.0361 (16)	0.0465 (17)	-0.0116 (12)	-0.0062 (13)	-0.0003 (13)
C3	0.0363 (16)	0.0476 (18)	0.0453 (17)	-0.0140 (13)	-0.0052 (12)	-0.0053 (14)
C4	0.0320 (14)	0.0470 (17)	0.0359 (15)	-0.0035 (12)	-0.0008 (11)	-0.0067 (13)
C5	0.0478 (18)	0.052 (2)	0.0467 (18)	-0.0114 (15)	-0.0110 (14)	-0.0048 (15)
C6	0.061 (2)	0.050 (2)	0.0488 (19)	-0.0070 (16)	-0.0077 (16)	-0.0017 (15)
C7	0.0484 (19)	0.071 (2)	0.0348 (16)	0.0117 (17)	0.0007 (14)	-0.0016 (15)
C8	0.0398 (17)	0.076 (3)	0.050 (2)	-0.0057 (16)	-0.0110 (14)	-0.0062 (18)
C9	0.0386 (17)	0.055 (2)	0.0524 (19)	-0.0110 (14)	-0.0041 (14)	-0.0102 (16)

### *Geometric parameters (Å, °)*

Br1—C7	1.889 (3)	C3—N1	1.458 (4)
S1—O4	1.424 (2)	C3—H3A	0.9700
S1—O3	1.425 (2)	C3—H3B	0.9700
S1—N1	1.613 (3)	C4—C5	1.381 (4)
S1—C4	1.759 (3)	C4—C9	1.385 (4)
O1—C2	1.320 (3)	C5—C6	1.374 (5)
O1—C1	1.436 (4)	C5—H5	0.9300
O2—C2	1.188 (4)	C6—C7	1.381 (5)
N1—H1	0.8600	C6—H6	0.9300
C1—H1A	0.9600	C7—C8	1.374 (5)
C1—H1B	0.9600	C8—C9	1.377 (5)
C1—H1C	0.9600	C8—H8	0.9300
C2—C3	1.489 (4)	C9—H9	0.9300
O4—S1—O3	120.49 (15)	N1—C3—H3B	109.6
O4—S1—N1	106.96 (14)	C2—C3—H3B	109.6
O3—S1—N1	106.53 (14)	H3A—C3—H3B	108.1
O4—S1—C4	107.89 (15)	C5—C4—C9	120.5 (3)
O3—S1—C4	107.66 (14)	C5—C4—S1	119.4 (2)
N1—S1—C4	106.55 (14)	C9—C4—S1	120.1 (2)
C2—O1—C1	117.5 (3)	C6—C5—C4	120.1 (3)
C3—N1—S1	118.9 (2)	C6—C5—H5	120.0
C3—N1—H1	120.6	C4—C5—H5	120.0
S1—N1—H1	120.6	C5—C6—C7	118.9 (3)
O1—C1—H1A	109.5	C5—C6—H6	120.6
O1—C1—H1B	109.5	C7—C6—H6	120.6
H1A—C1—H1B	109.5	C8—C7—C6	121.6 (3)
O1—C1—H1C	109.5	C8—C7—Br1	119.2 (3)
H1A—C1—H1C	109.5	C6—C7—Br1	119.2 (3)
H1B—C1—H1C	109.5	C7—C8—C9	119.4 (3)
O2—C2—O1	124.5 (3)	C7—C8—H8	120.3
O2—C2—C3	125.0 (3)	C9—C8—H8	120.3

O1—C2—C3	110.4 (2)	C8—C9—C4	119.5 (3)
N1—C3—C2	110.1 (2)	C8—C9—H9	120.2
N1—C3—H3A	109.6	C4—C9—H9	120.2
C2—C3—H3A	109.6		
C1—O1—C2—O2	1.4 (5)	C4—C5—C6—C7	-0.4 (5)
C1—O1—C2—C3	-178.3 (3)	C5—C6—C7—C8	1.1 (5)
O2—C2—C3—N1	-9.3 (5)	C5—C6—C7—Br1	-177.6 (3)
O1—C2—C3—N1	170.5 (3)	C6—C7—C8—C9	-0.5 (5)
O4—S1—C4—C5	162.0 (2)	Br1—C7—C8—C9	178.1 (2)
O3—S1—C4—C5	30.5 (3)	C7—C8—C9—C4	-0.6 (5)
N1—S1—C4—C5	-83.4 (3)	C5—C4—C9—C8	1.2 (5)
O4—S1—C4—C9	-20.8 (3)	S1—C4—C9—C8	-175.9 (2)
O3—S1—C4—C9	-152.3 (3)	C2—C3—N1—S1	165.0 (2)
N1—S1—C4—C9	93.7 (3)	O4—S1—N1—C3	44.2 (3)
C9—C4—C5—C6	-0.7 (5)	O3—S1—N1—C3	174.3 (2)
S1—C4—C5—C6	176.5 (3)	C4—S1—N1—C3	-71.0 (3)

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 $\cdots$ O2 <sup>i</sup>	0.86	2.41	2.987 (4)	125
C3—H3A $\cdots$ O3 <sup>ii</sup>	0.97	2.50	3.410 (4)	156

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Fig. 1

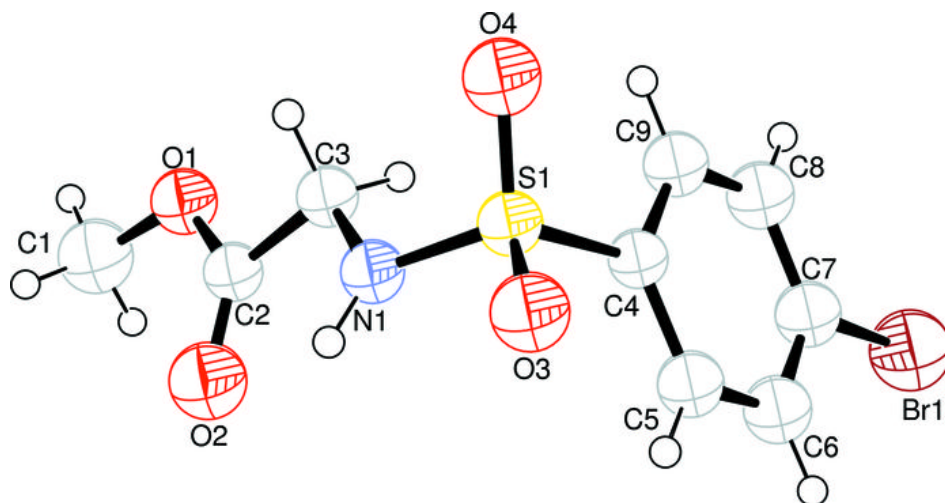




Fig. 2

