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## Structure Reports

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# Methyl 2-(benzenesulfonamido)acetate

Muhammad Nadeem Arshad,<sup>a</sup> Islam Ullah Khan,<sup>a\*</sup>  
Muhammad Zia-ur-Rehman<sup>b</sup> and Muhammad Shafiq<sup>a</sup>

<sup>a</sup>Department of Chemistry, Government College University, Lahore 54000, Pakistan, and <sup>b</sup>Applied Chemistry Research Centre, PCSIR Laboratories Complex, Lahore 54600, Pakistan

Correspondence e-mail: iukhan.gcu@gmail.com

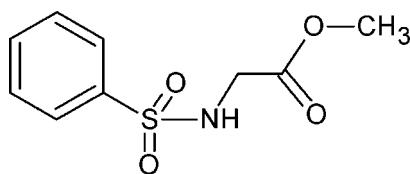
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.036;  $wR$  factor = 0.092; data-to-parameter ratio = 16.2.

The title compound,  $\text{C}_9\text{H}_{11}\text{NO}_4\text{S}$ , is of interest as a precursor to biologically active benzothiazines. The crystal structure is stabilized by intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  interactions.

## Related literature

For the synthesis and biological evaluation of sulfur-containing heterocyclic compounds, see: Zia-ur-Rehman *et al.* (2005, 2006, 2009); Xiao & Timberlake (2000); Martinez *et al.* (2000); Berredjem *et al.* (2000); Lee & Lee (2002). For related literature on sulfonamides, see: Esteve & Bidal (2002); Soledade *et al.* (2006). For related structures, see: Gowda *et al.* (2007a,b,c).



## Experimental

### Crystal data

$\text{C}_9\text{H}_{11}\text{NO}_4\text{S}$   
 $M_r = 229.26$   
Monoclinic,  $P2_1$   
 $a = 9.7268$  (8) Å  
 $b = 5.0781$  (4) Å  
 $c = 10.9286$  (9) Å  
 $\beta = 100.087$  (3)°

$V = 531.46$  (7) Å<sup>3</sup>  
 $Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 0.30$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.23 \times 0.11 \times 0.08$  mm

### Data collection

Bruker APEXII CCD area-detector diffractometer  
Absorption correction: multi-scan (SADABS; Bruker, 2007)  
 $T_{\min} = 0.935$ ,  $T_{\max} = 0.977$   
6159 measured reflections  
2216 independent reflections  
1944 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.092$   
 $S = 1.07$   
2216 reflections  
137 parameters  
1 restraint  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.26$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983), 394 Friedel pairs  
Flack parameter: 0.089 (8)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O3}^{\text{i}}$	0.86	2.28	2.998 (2)	141
$\text{C7}-\text{H7B}\cdots\text{O3}^{\text{ii}}$	0.97	2.55	3.503 (4)	168

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

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: SHELXTL (Sheldrick, 2008) and local programs.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2940).

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**supplementary materials**

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## Methyl 2-(benzenesulfonamido)acetate

M. N. Arshad, I. U. Khan, M. Zia-ur-Rehman and M. Shafiq

### Comment

Sulfonamide is an important functionality found in many naturally occurring as well as synthetic compounds which possess numerous types of biological activities (Soledade *et al.*, 2006; Esteve & Bidal, 2002; Xiao & Timberlake, 2000; Martinez *et al.*, 2000; Berredjem *et al.*, 2000; Lee & Lee, 2002). In the present paper, the structure of the title compound has been determined as a part of our ongoing research on the synthesis and biological evaluation of sulfur containing heterocyclic compounds (Zia-ur-Rehman *et al.*, 2005, 2006, 2009). In the molecule of (**I**) (Fig. 1), bond lengths and bond angles are almost similar to those in related sulfonamide molecules (Gowda *et al.*, 2007*a*, 2007*b*, 2007*c*) and the bond lengths are within normal ranges. In the crystal structure, each molecule is linked to an adjacent one through C7—H7B···O3 contacts giving rise to chains along *b*-axis. Each molecule of the chain is further linked to the one of its neighbouring chain along *a* through intermolecular N—H···O interactions.

### Experimental

A mixture of benzene sulfonic acid (4.14 g, 23.44 mmoles), glycine methyl ester hydrochloride (2.94 g, 23.44 mmol.) and distilled water (50.0 ml) was stirred for half an hour. pH of the reaction mixture was adjusted to 8.0 with an aqueous sodium carbonate solution. After completion of the reaction, a white solid product was isolated, washed, dried and recrystallized in methanol to get the crystals suitable for X-ray studies; m.p. 332 K.

### Refinement

H atoms were placed in geometric positions (C—H = 0.93-0.97 Å; N—H = 0.86 Å) using a riding model with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$  or  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$ .

### Figures

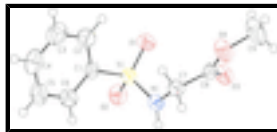


Fig. 1. The molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

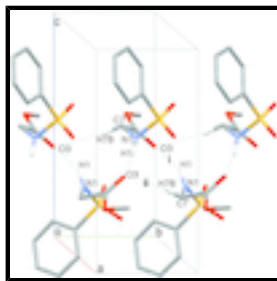


Fig. 2. Part of the crystal structure, showing hydrogen bond interactions (dashed lines). along the [0 0 1] direction. H atoms not involved in hydrogen bonding have been omitted for clarity.

## Methyl 2-(benzenesulfonamido)acetate

### Crystal data

$C_9H_{11}NO_4S$	$F_{000} = 240$
$M_r = 229.26$	$D_x = 1.433 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Melting point: 332 K
Hall symbol: P 2yb	Mo $K\alpha$ radiation
$a = 9.7268 (8) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 5.0781 (4) \text{ \AA}$	Cell parameters from 2710 reflections
$c = 10.9286 (9) \text{ \AA}$	$\theta = 2.6\text{--}26.6^\circ$
$\beta = 100.087 (3)^\circ$	$\mu = 0.30 \text{ mm}^{-1}$
$V = 531.46 (7) \text{ \AA}^3$	$T = 296 \text{ K}$
$Z = 2$	Plates, colourless
	$0.23 \times 0.11 \times 0.08 \text{ mm}$

### Data collection

Bruker APEXII CCD area-detector diffractometer	2216 independent reflections
Radiation source: fine-focus sealed tube	1944 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.028$
$T = 296 \text{ K}$	$\theta_{\text{max}} = 29.3^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 2.6^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2007)	$h = -12 \rightarrow 13$
$T_{\text{min}} = 0.935$ , $T_{\text{max}} = 0.977$	$k = -4 \rightarrow 6$
6159 measured reflections	$l = -11 \rightarrow 15$

### Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.0105P]$
$wR(F^2) = 0.092$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2216 reflections	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
137 parameters	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 394 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.089 (8)

*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>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.61607 (5)	0.09862 (10)	0.21011 (5)	0.03580 (15)
O1	0.52527 (16)	-0.0799 (4)	0.25831 (16)	0.0482 (4)
O2	0.72360 (15)	0.0024 (4)	0.14799 (15)	0.0468 (4)
O3	0.27134 (16)	0.0012 (4)	0.03143 (16)	0.0469 (4)
O4	0.16275 (17)	0.2366 (4)	0.15812 (18)	0.0589 (5)
N1	0.51744 (17)	0.2853 (4)	0.11147 (16)	0.0389 (5)
H1	0.5423	0.3294	0.0426	0.047*
C1	0.6962 (2)	0.2993 (5)	0.3344 (2)	0.0366 (5)
C2	0.6546 (3)	0.2854 (7)	0.4486 (2)	0.0537 (7)
H2	0.5847	0.1687	0.4614	0.064*
C3	0.7181 (3)	0.4473 (8)	0.5441 (2)	0.0669 (9)
H3	0.6913	0.4382	0.6216	0.080*
C4	0.8199 (3)	0.6206 (8)	0.5249 (3)	0.0646 (8)
H4	0.8612	0.7303	0.5891	0.078*
C5	0.8614 (3)	0.6333 (7)	0.4113 (3)	0.0602 (7)
H5	0.9317	0.7494	0.3992	0.072*
C6	0.7990 (2)	0.4739 (5)	0.3148 (2)	0.0481 (6)
H6	0.8259	0.4843	0.2374	0.058*
C7	0.3858 (2)	0.3768 (5)	0.1419 (2)	0.0365 (5)
H7B	0.3610	0.5437	0.1008	0.044*
H7A	0.3966	0.4050	0.2309	0.044*
C8	0.2709 (2)	0.1813 (5)	0.10223 (19)	0.0346 (5)
C9	0.0435 (3)	0.0566 (10)	0.1303 (3)	0.0886 (12)
H9A	0.0132	0.0469	0.0419	0.133*
H9B	-0.0315	0.1204	0.1687	0.133*
H9C	0.0709	-0.1155	0.1619	0.133*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0316 (2)	0.0341 (3)	0.0417 (3)	-0.0008 (3)	0.00653 (19)	0.0014 (3)
O1	0.0446 (9)	0.0426 (10)	0.0565 (10)	-0.0084 (8)	0.0069 (8)	0.0094 (9)

## supplementary materials

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O2	0.0390 (8)	0.0496 (11)	0.0525 (10)	0.0075 (8)	0.0099 (7)	-0.0064 (8)
O3	0.0415 (9)	0.0443 (10)	0.0559 (10)	-0.0052 (8)	0.0114 (7)	-0.0095 (9)
O4	0.0329 (8)	0.0774 (14)	0.0701 (11)	-0.0025 (9)	0.0194 (8)	-0.0181 (11)
N1	0.0333 (9)	0.0445 (12)	0.0393 (10)	-0.0014 (9)	0.0074 (8)	0.0040 (9)
C1	0.0292 (10)	0.0377 (13)	0.0412 (11)	0.0037 (9)	0.0014 (8)	0.0020 (10)
C2	0.0461 (14)	0.0662 (19)	0.0502 (14)	-0.0020 (14)	0.0123 (11)	-0.0009 (14)
C3	0.0632 (17)	0.091 (3)	0.0467 (15)	0.0050 (18)	0.0092 (13)	-0.0126 (16)
C4	0.0643 (16)	0.064 (2)	0.0587 (15)	0.0052 (18)	-0.0098 (12)	-0.0190 (18)
C5	0.0537 (14)	0.051 (2)	0.0701 (16)	-0.0110 (15)	-0.0044 (12)	-0.0030 (16)
C6	0.0469 (13)	0.0451 (16)	0.0518 (14)	-0.0052 (12)	0.0071 (11)	0.0017 (12)
C7	0.0352 (11)	0.0327 (13)	0.0423 (11)	0.0031 (10)	0.0082 (9)	0.0019 (10)
C8	0.0295 (10)	0.0394 (14)	0.0342 (10)	0.0059 (9)	0.0038 (8)	0.0031 (10)
C9	0.0397 (14)	0.116 (3)	0.114 (3)	-0.0180 (19)	0.0257 (16)	-0.015 (3)

### *Geometric parameters (Å, °)*

S1—O1	1.4290 (16)	C3—C4	1.369 (4)
S1—O2	1.4291 (15)	C3—H3	0.9300
S1—N1	1.618 (2)	C4—C5	1.372 (4)
S1—C1	1.767 (2)	C4—H4	0.9300
O3—C8	1.198 (3)	C5—C6	1.383 (4)
O4—C8	1.336 (2)	C5—H5	0.9300
O4—C9	1.466 (4)	C6—H6	0.9300
N1—C7	1.454 (3)	C7—C8	1.501 (3)
N1—H1	0.8600	C7—H7B	0.9700
C1—C2	1.380 (3)	C7—H7A	0.9700
C1—C6	1.381 (3)	C9—H9A	0.9600
C2—C3	1.386 (4)	C9—H9B	0.9600
C2—H2	0.9300	C9—H9C	0.9600
O1—S1—O2	120.61 (11)	C4—C5—C6	120.2 (3)
O1—S1—N1	106.53 (9)	C4—C5—H5	119.9
O2—S1—N1	106.36 (9)	C6—C5—H5	119.9
O1—S1—C1	107.48 (10)	C1—C6—C5	119.4 (2)
O2—S1—C1	107.55 (10)	C1—C6—H6	120.3
N1—S1—C1	107.73 (10)	C5—C6—H6	120.3
C8—O4—C9	115.6 (2)	N1—C7—C8	111.31 (18)
C7—N1—S1	118.54 (14)	N1—C7—H7B	109.4
C7—N1—H1	120.7	C8—C7—H7B	109.4
S1—N1—H1	120.7	N1—C7—H7A	109.4
C2—C1—C6	120.6 (2)	C8—C7—H7A	109.4
C2—C1—S1	120.4 (2)	H7B—C7—H7A	108.0
C6—C1—S1	119.01 (17)	O3—C8—O4	123.2 (2)
C1—C2—C3	119.2 (3)	O3—C8—C7	127.20 (19)
C1—C2—H2	120.4	O4—C8—C7	109.60 (19)
C3—C2—H2	120.4	O4—C9—H9A	109.5
C4—C3—C2	120.3 (3)	O4—C9—H9B	109.5
C4—C3—H3	119.8	H9A—C9—H9B	109.5
C2—C3—H3	119.8	O4—C9—H9C	109.5
C3—C4—C5	120.3 (3)	H9A—C9—H9C	109.5

C3—C4—H4	119.9	H9B—C9—H9C	109.5
C5—C4—H4	119.9		
O1—S1—N1—C7	40.2 (2)	C1—C2—C3—C4	0.6 (5)
O2—S1—N1—C7	170.05 (17)	C2—C3—C4—C5	-0.8 (5)
C1—S1—N1—C7	-74.87 (19)	C3—C4—C5—C6	1.0 (5)
O1—S1—C1—C2	-7.1 (2)	C2—C1—C6—C5	0.8 (4)
O2—S1—C1—C2	-138.4 (2)	S1—C1—C6—C5	179.5 (2)
N1—S1—C1—C2	107.3 (2)	C4—C5—C6—C1	-1.0 (4)
O1—S1—C1—C6	174.20 (18)	S1—N1—C7—C8	-86.9 (2)
O2—S1—C1—C6	42.9 (2)	C9—O4—C8—O3	1.7 (3)
N1—S1—C1—C6	-71.3 (2)	C9—O4—C8—C7	-178.5 (2)
C6—C1—C2—C3	-0.6 (4)	N1—C7—C8—O3	-15.8 (3)
S1—C1—C2—C3	-179.2 (2)	N1—C7—C8—O4	164.42 (18)

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

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1 $\cdots$ O3 <sup>i</sup>	0.86	2.28	2.998 (2)	141
C7—H7B $\cdots$ O3 <sup>ii</sup>	0.97	2.55	3.503 (4)	168

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

Fig. 1

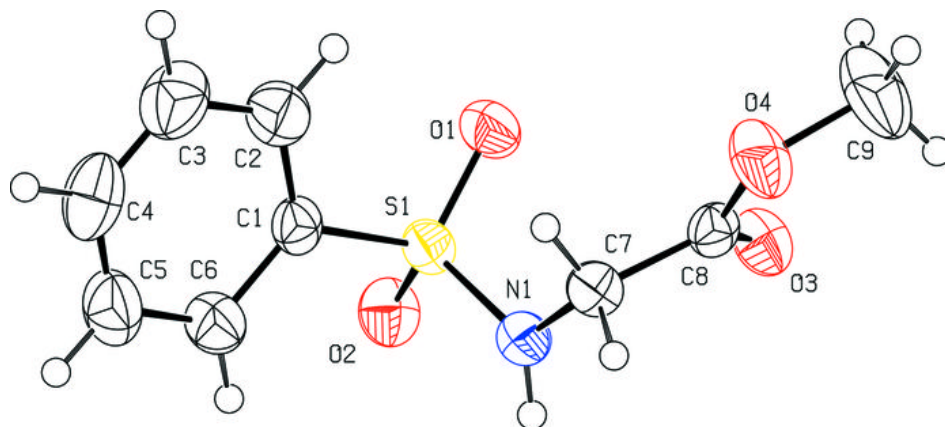




Fig. 2

