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Methyl (4-bromobenzenesulfonamido)-acetate

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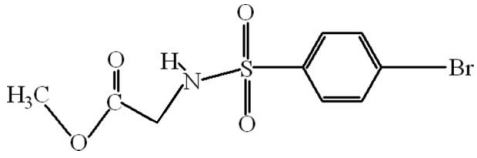
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.040; wR factor = 0.116; data-to-parameter ratio = 19.6.

The title compound, $C_9H_{10}BrNO_4S$, is an intermediate for the formation of benzothiazines. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules, forming $R_2^2(10)$ ring motifs, which are linked into a two-dimensional polymeric sheet through intermolecular C—H···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

$C_9H_{10}BrNO_4S$
M_r = 308.15
Triclinic, $P\overline{1}$
a = 6.0451 (2) Å
b = 7.0369 (2) Å
c = 13.8695 (5) Å
α = 83.866 (2)°
β = 81.190 (1)°
γ = 87.027 (2)°
V = 579.31 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.73 mm^{-1}
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_{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
$\Delta\rho_{max}$ = 0.53 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
N1—H1···O2^{i}	0.86	2.41	2.987 (4)	125
C3—H3A···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).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Arshad, M. N., Tahir, M. N., Khan, I. U., Shafiq, M. & Siddiqui, W. A. (2008). *Acta Cryst.* E**64**, o2045.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bornaghi, L. F., Poulsen, S.-A., Healy, P. C. & White, A. R. (2005). *Acta Cryst.* E**61**, o323–o325.
Bruker (2005). *SADABS*. Bruker AXS Inc. Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc. Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tahir, M. N., Shafiq, M., Khan, I. U., Siddiqui, W. A. & Arshad, M. N. (2008). *Acta Cryst.* E**64**, o557.

supporting information

Acta Cryst. (2008). E**64**, o2380 [doi:10.1107/S1600536808037550]

Methyl (4-bromobenzenesulfonamido)acetate

Muhammad Nadeem Arshad, M. Nawaz Tahir, Islam Ullah Khan, Ejaz Ahmad and Muhammad Shafiq

S1. Comment

The glycine methyl ester is the simplest among the amino acids and the *p*-bromobenzenesulfonyl chloride is also comercially 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).

S2. 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 recrystalized from methanol for X-ray analysis (m.p. 393-394 K).

S3. 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 $U_{iso}(H) = xU_{eq}(C,N)$, where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

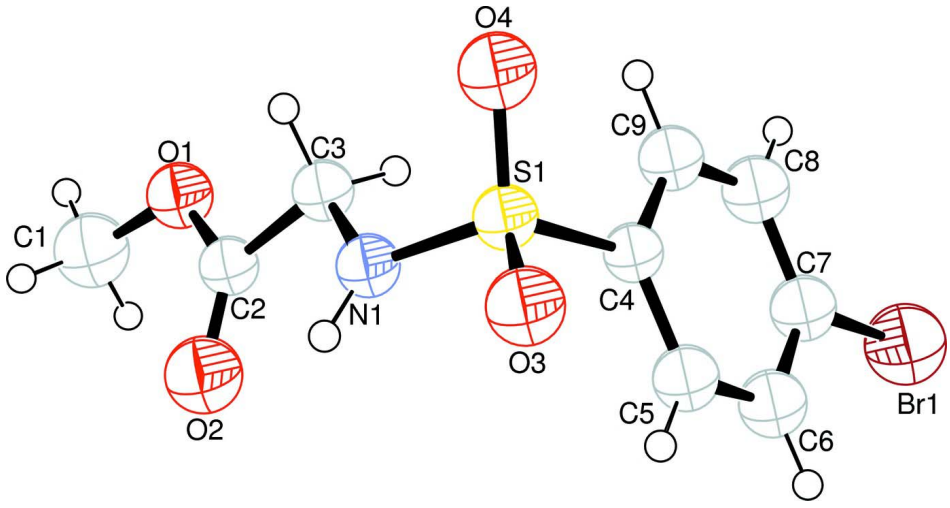

Figure 1

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

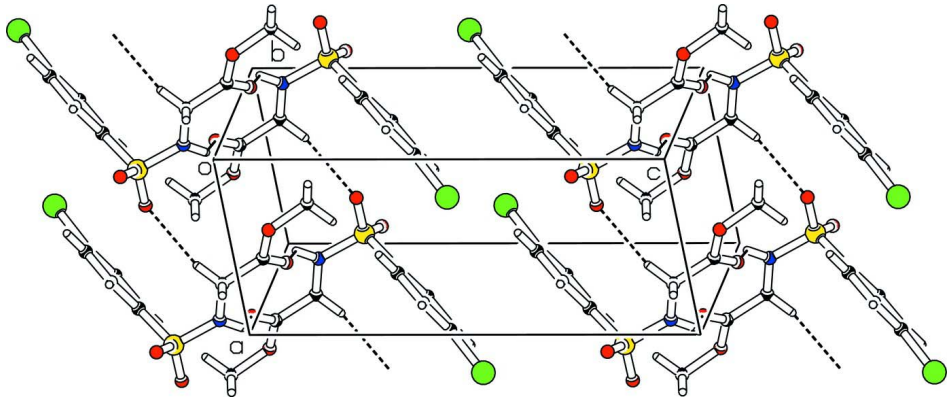

Figure 2

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

Methyl (4-bromobenzenesulfonamido)acetate

Crystal data

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

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.40 pixels mm^{-1}
ω scans
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_{int} = 0.038
θ_{max} = 28.3°, θ_{min} = 1.5°
h = −8→8
k = −9→9
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.116
S = 1.04
2846 reflections
145 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.5395P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 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 (Å^2)

	x	y	z	U_{iso}*/U_{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 (Å²)

	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)
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, º)

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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.41	2.987 (4)	125
C3—H3*A*···O3^{ii}	0.97	2.50	3.410 (4)	156

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