Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Bromothiophene-2-sulfonic acid

Jian-Ping Liu[a,b]* and Yu-Bin Zheng[a]

[a]Department of Polymer Science and Materials, Dalian University of Technology, Dalian 116012, People's Republic of China, and [b]College of Chemistry and Materials Engineering, Wenzhou University, Zhejiang, Wenzhou 325027, People's Republic of China
Correspondence e-mail: liujianping163@163.com

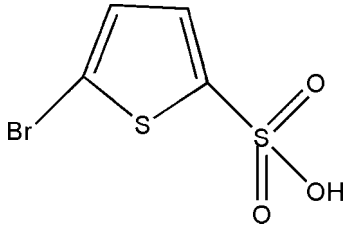
Received 21 July 2007; accepted 30 July 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.050; wR factor = 0.134; data-to-parameter ratio = 14.5.

In the title compound, $C_4H_3BrO_3S_2$, all bond lengths and angles are within normal ranges. The crystal packing exhibits intermolecular O—H···O hydrogen bonds, which link the molecules into chains extended along the c axis.

Related literature

For general background, see Yan *et al.* (2007). For related literature, see: Allen *et al.* (1987); Lin *et al.* (2007). For details of the pharmacological properties of sulfonamide compounds, see: Gayathri *et al.* (2006); Krishnaiah *et al.* (1995); Yu (2006).



Experimental

Crystal data

$C_4H_3BrO_3S_2$
M_r = 243.09
Orthorhombic, *Pccn*
a = 13.7633 (16) Å
b = 14.4540 (17) Å
c = 7.7020 (9) Å
V = 1532.2 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 5.85 mm^{-1}
T = 298 (2) K
0.32 × 0.21 × 0.12 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.241, T_{max} = 0.500
7333 measured reflections
1347 independent reflections
1191 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.134
S = 1.08
1347 reflections
93 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.28 e Å^{-3}
$\Delta\rho_{min}$ = −1.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1^{i}	0.82	2.14	2.920 (5)	158

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge financial support by Dalian University of Technology and Wenzhou University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2285).

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.
Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Gayathri, D., Velmurugan, D., Ravikumar, K., Poornachandran, M. & Raghunathan, R. (2006). *Acta Cryst.* E**62**, o4454–o4455.
Krishnaiah, M., Narayana Raju, K. V., Lu, I.-L., Chen, Y.-S. & Narasinga Rao, S. (1995). *Acta Cryst.* C**51**, 2429–2430.
Lin, S.-M., Zheng, M. & Xiong, J. (2007). *Acta Cryst.* E**63**, o3177.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Yan, X.-W., Hu, M.-L. & Xiong, J. (2007). *Acta Cryst.* E**63**, o678–o679.
Yu, Y.-Y. (2006). *Acta Cryst.* E**62**, o2308–o2309.

 doi:10.1107/S1600536807037208

supplementary materials

Acta Cryst. (2007). E**63**, o3697 [**doi:10.1107/S1600536807037208**]

5-Bromothiophene-2-sulfonic acid

J.-P. Liu and Y.-B. Zheng

Comment

Some sulfonamide compounds exhibit germicidal activities (Gayathri *et al.*, 2006; Krishnaiah *et al.*, 1995; Yu, 2006), and some crystal structures involving sulfonamide groups have been published (Yan *et al.*, 2007; Lin *et al.*, 2007). 5-Bromothiophene-2-sulfonyl chloride is an important starting material used in synthesis of sulfonamide compounds. Recently, during our synthesis of sulfonamide compounds, the hydrolysate of 5-bromothiophene-2-sulfonyl chloride was obtained unexpectedly. Here we report the crystal structure of the title compound, (I).

In (I) (Fig. 1), all bond lengths and angles show normal values (Allen *et al.*, 1987) and are unremarkable when compared with those found in previous reports (Yan *et al.*, 2007; Lin *et al.*, 2007). The crystal packing exhibits intermolecular O—H···O hydrogen bonds (Table 1), which link the molecules into the chains extended along the *c* axis.

Experimental

5-Bromothiophene-2-sulfonyl chloride (5 mmol, 1.304 g), 2-aminobenzoic acid (5 mmol, 0.685 g) and *N*,*N*-dimethylpyridin-4-amine (DMAP) (0.5 mmol, 0.061 g) were added to acetone (15 ml) at room temperature with stirring. The reaction was allowed to proceed for 48 h at room temperature, followed by column chromatographic separation. The purified product was dissoved in ethanol–acetone (1:1), and allowed to stand for approximately 15 d until single crystals formed.

Refinement

The atom H3 was located on a difference Fourier map, placed in idealized position (O—H 0.82 Å) and refined as riding, with U_{iso}(H3) = 1.5U_{eq}(O3). C-bound H atoms were positioned geometrically, and were allowed to ride on their parent atoms at distances of C—H 0.93 Å, with U_{iso}(H) = 1.2U_{eq}(C). The highest residual peak [1.28 e Å^{-3}] and deepest hole [−1.21 e Å^{-3}] are situated 0.04 Å at atom S2 and 0.15 Å at atom O3, respectively.

Figures

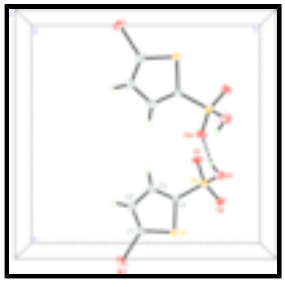


Fig. 1. A portion of the crystal packing of (I), showing the atom numbering and displacement ellipsoids at the 40% probability level [symmetry code: (A) −*x* + 3/2, *y*, *z* + 1/2]. Dashed line denotes intermolecular hydrogen bond.

5-Bromothiophene-2-sulfonic acid

Crystal data

$C_4H_3BrO_3S_2$	F_{000} = 944
M_r = 243.09	D_x = 2.108 Mg m^{-3}
Orthorhombic, *Pccn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ab 2ac	Cell parameters from 2560 reflections
a = 13.7633 (16) Å	θ = 2.8–25.0º
b = 14.4540 (17) Å	μ = 5.85 mm^{-1}
c = 7.7020 (9) Å	*T* = 298 (2) K
V = 1532.2 (3) Å^3	Prism, colourless
Z = 8	0.32 × 0.21 × 0.12 mm

Data collection

Bruker APEX area-detector diffractometer	1347 independent reflections
Radiation source: fine-focus sealed tube	1191 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.035
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	*h* = −16→13
T_{min} = 0.241, T_{max} = 0.500	*k* = −17→17
7333 measured reflections	*l* = −9→9

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$w = 1/[\sigma^2(F_o^2) + (0.0718P)^2 + 4.907P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.134	$(\Delta/\sigma)_{max} < 0.001$
S = 1.08	$\Delta\rho_{max}$ = 1.28 e Å^{-3}
1347 reflections	$\Delta\rho_{min}$ = −1.21 e Å^{-3}
93 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0100 (13)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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	1.17226 (5)	0.61516 (5)	1.03375 (11)	0.0594 (4)
S1	1.06565 (9)	0.80207 (10)	0.9827 (2)	0.0412 (4)
S2	0.88696 (9)	0.91154 (8)	0.88437 (17)	0.0346 (4)
O1	0.8001 (3)	0.8886 (3)	0.7904 (6)	0.0457 (10)
O2	0.9573 (3)	0.9723 (2)	0.8092 (5)	0.0395 (9)
O3	0.8528 (3)	0.9585 (3)	1.0626 (5)	0.0459 (10)
H3	0.8073	0.9291	1.1034	0.069*
C1	1.0605 (4)	0.6842 (4)	0.9861 (7)	0.0366 (12)
C2	0.9721 (4)	0.6498 (4)	0.9457 (7)	0.0405 (12)
H2	0.9574	0.5870	0.9417	0.049*
C3	0.9050 (4)	0.7209 (4)	0.9103 (7)	0.0377 (11)
H3A	0.8404	0.7105	0.8809	0.045*
C4	0.9452 (3)	0.8064 (3)	0.9237 (6)	0.0296 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0396 (5)	0.0554 (5)	0.0834 (6)	0.0193 (3)	−0.0019 (3)	0.0086 (3)
S1	0.0220 (7)	0.0379 (8)	0.0637 (9)	−0.0003 (5)	−0.0064 (5)	0.0044 (6)
S2	0.0294 (7)	0.0336 (7)	0.0408 (7)	−0.0015 (5)	−0.0021 (5)	0.0069 (5)
O1	0.0268 (19)	0.051 (2)	0.059 (3)	−0.0032 (16)	−0.0157 (18)	0.0120 (19)
O2	0.0332 (19)	0.0395 (19)	0.046 (2)	−0.0060 (15)	0.0035 (16)	0.0103 (16)
O3	0.038 (2)	0.048 (2)	0.052 (2)	0.0015 (18)	0.0066 (18)	−0.0026 (19)
C1	0.034 (3)	0.035 (3)	0.040 (3)	0.009 (2)	0.001 (2)	0.001 (2)
C2	0.042 (3)	0.032 (3)	0.048 (3)	0.001 (2)	−0.001 (2)	−0.002 (2)
C3	0.029 (3)	0.037 (3)	0.047 (3)	−0.004 (2)	−0.005 (2)	−0.001 (2)
C4	0.021 (2)	0.033 (3)	0.035 (3)	−0.0014 (18)	−0.0023 (18)	0.003 (2)

Geometric parameters (Å, °)

Br1—C1	1.870 (5)	O3—H3	0.8200
S1—C1	1.705 (5)	C1—C2	1.351 (8)
S1—C4	1.720 (5)	C2—C3	1.409 (7)

S2—O2	1.429 (4)	C2—H2	0.9300
S2—O1	1.437 (4)	C3—C4	1.358 (7)
S2—O3	1.602 (4)	C3—H3A	0.9300
S2—C4	1.745 (5)		
C1—S1—C4	90.0 (3)	S1—C1—Br1	120.1 (3)
O2—S2—O1	120.1 (2)	C1—C2—C3	111.5 (5)
O2—S2—O3	106.6 (2)	C1—C2—H2	124.3
O1—S2—O3	106.6 (2)	C3—C2—H2	124.3
O2—S2—C4	107.1 (2)	C4—C3—C2	112.5 (5)
O1—S2—C4	105.6 (2)	C4—C3—H3A	123.8
O3—S2—C4	110.8 (2)	C2—C3—H3A	123.8
S2—O3—H3	109.5	C3—C4—S1	112.3 (4)
C2—C1—S1	113.7 (4)	C3—C4—S2	126.3 (4)
C2—C1—Br1	126.1 (4)	S1—C4—S2	121.4 (3)
C4—S1—C1—C2	−0.3 (4)	C1—S1—C4—S2	−178.4 (3)
C4—S1—C1—Br1	177.3 (3)	O2—S2—C4—C3	−144.9 (5)
S1—C1—C2—C3	0.0 (6)	O1—S2—C4—C3	−15.8 (5)
Br1—C1—C2—C3	−177.5 (4)	O3—S2—C4—C3	99.3 (5)
C1—C2—C3—C4	0.5 (7)	O2—S2—C4—S1	34.0 (4)
C2—C3—C4—S1	−0.8 (6)	O1—S2—C4—S1	163.1 (3)
C2—C3—C4—S2	178.2 (4)	O3—S2—C4—S1	−81.8 (3)
C1—S1—C4—C3	0.6 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1^{i}	0.82	2.14	2.920 (5)	158

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

Fig. 1