

organic compounds



Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1-(3-Bromo-2-thienyl)ethanone

M. Mahendra,^a H. K. Vivek,^b S. L. Gaonkar,^c B. S. Priya^c
and S. Nanjunda Swamy^{b*}^aDepartment of Studies in Physics, University of Mysore, Manasagangotri, Mysore 570 006, India, ^bDepartment of Biotechnology, Sri Jayachamarajendra College of Engineering, Mysore 570 006, India, and ^cDepartment of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India
Correspondence e-mail: nanju_xrd@yahoo.com

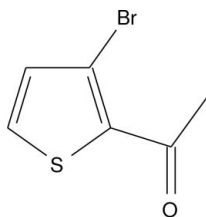
Received 19 August 2010; accepted 28 August 2010

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.027; wR factor = 0.067; data-to-parameter ratio = 17.4.

In the title compound, $\text{C}_6\text{H}_5\text{BrOS}$, the non-H and aromatic H atoms lie on a crystallographic mirror plane. In the crystal, molecules are linked into chains propagating along the c axis by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For the uses of acetyl thiophenes, see: Ashalatha *et al.* (2009); Bando *et al.* (2010); Ito & Furukawa (1990); Lutz *et al.* (2005); Nakayama *et al.* (1989); Pelly *et al.* (2005); Yasuhara *et al.* (2002).



Experimental

Crystal data

 $\text{C}_6\text{H}_5\text{BrOS}$
 $M_r = 205.07$
Orthorhombic, $Cmca$
 $a = 6.8263$ (17) Å
 $b = 13.149$ (4) Å
 $c = 16.007$ (4) Å $V = 1436.8$ (7) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 5.92$ mm⁻¹
 $T = 293$ K
0.25 × 0.21 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
 $T_{\min} = 0.313$, $T_{\max} = 0.384$
12363 measured reflections
973 independent reflections
790 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.067$
 $S = 1.06$
973 reflections56 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.68$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C4}-\text{H4}\cdots\text{O8}^i$	0.93	2.43	3.352 (4)	174

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINTE* (Bruker, 2004); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON*.

SNS is grateful to University Grants Commission (UGC), Government of India, for financial support under the major research project [grant No. 38-220/2009 (SR)]. SNS also expresses his sincere gratitude to J. S. S. Mahavidyapeetha for the encouragement towards this research work. MM thanks the University of Mysore for awarding a project (No. DV3/136/2007-2008/24.09.09).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5166).

References

- Ashalatha, B. V., Narayana, B. & Vijaya Raj, K. K. (2009). *Phosphorus Sulfur Silicon Relat. Elem.* **184**, 1904–1919.
- Bando, S., Satake, S. & Kagano, H. (2010). US Patent No. 7659411 B2.
- Bruker (2004). *APEX2* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Ito, C. & Furukawa, H. (1990). *Chem. Pharm. Bull.* **38**, 1548–1550.
- Lutz, G. B., Thomas, S. & Hans-Hartwig, O. (2005). *Monatsh. Chem.* **136**, 635–653.
- Nakayama, J., Konishi, T., Ishii, A. & Hoshino, M. (1989). *Bull. Chem. Soc. Jpn.* **62**, 2608–2612.
- Pelly, S. C., Parkinson, C. J., van Otterlo, W. A. L. & de Koning, C. B. (2005). *J. Org. Chem.* **70**, 10474–10481.
- Sheldrick, G. M. (2001). *SADABS*. University of Gottingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Yasuhara, A., Suzuki, N. & Sakamoto, T. (2002). *Chem. Pharm. Bull.* **50**, 143–145.

supplementary materials

Acta Cryst. (2010). E66, o2540 [doi:10.1107/S1600536810034677]

1-(3-Bromo-2-thienyl)ethanone

M. Mahendra, H. K. Vivek, S. L. Gaonkar, B. S. Priya and S. Nanjunda Swamy

Comment

2-Acetyl-3-bromothiophene is one of the well-known bio-active intermediate used for the construction of number of new heterocycles (Lutz *et al.* 2005; Pelly *et al.* 2005). It is used as an intermediate for the synthesis of furo[3,2-a]carbazole alkaloid, furostifoline (Ito *et al.* 1990) and its derivatives, which show broad pharmacological properties (Yasuhara *et al.* 2002). Chalcones of 2-acetyl-3-bromothiophene exhibit promising anti-inflammatory, analgesic and antibacterial activities (Ashalatha *et al.* 2009). Acetyl thiophenes are useful as intermediates for preparing number of pharmaceutical compounds (Bando *et al.* 2010). Acetyl bromothiophenes are also used for the synthesis of number of biologically active pyridazine derivatives (Nakayama *et al.* 1989). With this background, the title compound (I), was synthesized and we report its crystal structure here.

The non-hydrogen and aromatic hydrogen atoms of the title molecule lie on a crystallographic mirror plane (Fig. 1). The molecules are linked into a chain along the *c* axis by intermolecular C—H···O hydrogen bonds (Table 1).

Experimental

A three-necked, round-bottomed flask was charged with CH₂Cl₂ (10 ml) and anhydrous AlCl₃ (2.45 g, 18.4 mmol). The flask was cooled to 273 K. A dropping funnel was charged with freshly distilled acetyl chloride (1.48 g, 19.6 mmol) in CH₂Cl₂ (15 ml), and was added drop wise for a period of 30 min. The reaction mixture was stirred for 1 h at 273 K. The reaction mass was further cooled to 250 K. 3-Bromothiophene (1.00 g, 6.13 mmol) in CH₂Cl₂ (15 mL) was added drop wise for 1 h. The reaction was stirred at 250 K for 30 min and then warmed slowly to room temperature and stirred for 1 h. Then the reaction mixture was quenched on ice water (50 ml). Layers were separated and aqueous layer was repeatedly extracted with CH₂Cl₂ and the combined organic extracts were washed with saturated NaHCO₃ (25 ml), then brine (25 ml) and finally dried over anhydrous Na₂SO₄. Solvent was removed by distillation at atmospheric pressure. The remaining oily mass was distilled under high vacuum (403 K at 10 mbar) to give a pale yellow oil which was crystallized in n-hexane to give 2-acetyl-3-bromothiophene (1.10 g, 88 %) as a yellow solid. Block-shaped yellow single crystals were obtained by slow evaporation of an n-hexane solution.

Refinement

H atoms were placed at idealized positions and allowed to ride on their parent atoms with C—H distances in the range 0.93–0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{carrier atom})$.

Figures

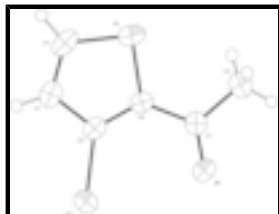


Fig. 1. Molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

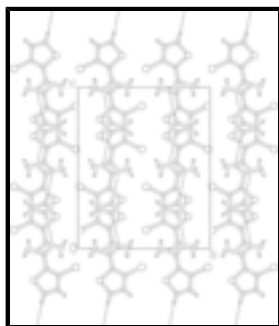


Fig. 2. Packing diagram of (I), viewed down the *a* axis. The dashed lines represent hydrogen bonds.

1-(3-Bromo-2-thienyl)ethanone

Crystal data

C_6H_5BrOS

$M_r = 205.07$

Orthorhombic, *Cmca*

Hall symbol: -C 2bc 2

$a = 6.8263$ (17) Å

$b = 13.149$ (4) Å

$c = 16.007$ (4) Å

$V = 1436.8$ (7) Å³

$Z = 8$

$F(000) = 800$

$D_x = 1.896$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1982 reflections

$\theta = 2.5$ – 28.4°

$\mu = 5.92$ mm⁻¹

$T = 293$ K

Block, yellow

$0.25 \times 0.21 \times 0.20$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

ω and φ scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)

$T_{\min} = 0.313$, $T_{\max} = 0.384$

12363 measured reflections

973 independent reflections

790 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.041$

$\theta_{\max} = 28.4^\circ$, $\theta_{\min} = 2.5^\circ$

$h = -9 \rightarrow 9$

$k = -17 \rightarrow 17$

$l = -21 \rightarrow 20$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.027$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.067$	H-atom parameters constrained
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0333P)^2 + 1.3829P]$
973 reflections	where $P = (F_o^2 + 2F_c^2)/3$
56 parameters	$(\Delta/\sigma)_{\max} = 0.001$
0 restraints	$\Delta\rho_{\max} = 0.68 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$

Special details

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Br1	0.00000	0.01659 (3)	0.38478 (2)	0.0547 (1)	
S5	0.00000	0.33480 (6)	0.30339 (5)	0.0463 (3)	
O8	0.00000	0.19098 (19)	0.51639 (13)	0.0584 (9)	
C2	0.00000	0.1452 (2)	0.33378 (18)	0.0365 (9)	
C3	0.00000	0.1547 (3)	0.2458 (2)	0.0448 (10)	
C4	0.00000	0.2530 (3)	0.2207 (2)	0.0479 (10)	
C6	0.00000	0.2366 (2)	0.37465 (17)	0.0357 (9)	
C7	0.00000	0.2587 (2)	0.46527 (19)	0.0389 (9)	
C9	0.00000	0.3685 (3)	0.4922 (2)	0.0541 (11)	
H3	0.00000	0.09970	0.20930	0.0540*	
H4	0.00000	0.27350	0.16510	0.0570*	
H9A	0.13140	0.39450	0.49060	0.0810*	0.500
H9B	-0.05010	0.37360	0.54810	0.0810*	0.500
H9C	-0.08130	0.40740	0.45510	0.0810*	0.500

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0910 (3)	0.0316 (2)	0.0415 (2)	0.0000	0.0000	-0.0009 (1)
S5	0.0590 (5)	0.0401 (4)	0.0397 (4)	0.0000	0.0000	0.0117 (3)
O8	0.106 (2)	0.0408 (13)	0.0285 (12)	0.0000	0.0000	0.0023 (10)

supplementary materials

C2	0.0400 (16)	0.0399 (16)	0.0297 (14)	0.0000	0.0000	0.0021 (12)
C3	0.0512 (18)	0.0519 (18)	0.0314 (15)	0.0000	0.0000	-0.0035 (13)
C4	0.0526 (19)	0.063 (2)	0.0281 (14)	0.0000	0.0000	0.0069 (14)
C6	0.0428 (16)	0.0333 (14)	0.0309 (15)	0.0000	0.0000	0.0057 (11)
C7	0.0482 (17)	0.0354 (15)	0.0332 (15)	0.0000	0.0000	-0.0021 (12)
C9	0.079 (2)	0.0385 (16)	0.0448 (19)	0.0000	0.0000	-0.0061 (14)

Geometric parameters (Å, °)

Br1—C2	1.878 (3)	C3—H3	0.93
S5—C4	1.706 (4)	C4—H4	0.93
S5—C6	1.723 (3)	C9—H9A	0.96
O8—C7	1.209 (4)	C9—H9B	0.96
C2—C3	1.414 (4)	C9—H9C	0.96
C2—C6	1.368 (4)	C9—H9A ⁱ	0.96
C3—C4	1.354 (6)	C9—H9B ⁱ	0.96
C6—C7	1.479 (4)	C9—H9C ⁱ	0.96
C7—C9	1.507 (5)		
Br1…O8	3.114 (3)	C3…C3 ^{xi}	3.4158 (11)
Br1…Br1 ⁱⁱ	3.7144 (12)	C3…C3 ^{ix}	3.4158 (11)
Br1…O8 ⁱⁱ	3.155 (3)	C3…C3 ^{vi}	3.4158 (11)
Br1…S5 ⁱⁱⁱ	3.8453 (15)	C4…O8 ^{xvi}	3.352 (4)
Br1…Br1 ^{iv}	3.7144 (12)	C4…O8 ^{xvii}	3.352 (4)
Br1…O8 ^{iv}	3.155 (3)	C4…S5 ^x	3.5993 (17)
Br1…S5 ^v	3.8453 (15)	C4…S5 ^{xi}	3.5993 (17)
S5…C4 ^{vi}	3.5993 (17)	C4…C4 ^x	3.5397 (16)
S5…Br1 ^{vii}	3.8453 (15)	C4…C4 ^{xi}	3.5397 (16)
S5…Br1 ^{viii}	3.8453 (15)	C4…S5 ^{ix}	3.5993 (17)
S5…C4 ^{ix}	3.5993 (17)	C4…S5 ^{vi}	3.5993 (17)
S5…C4 ^x	3.5993 (17)	C4…C4 ^{ix}	3.5397 (16)
S5…C4 ^{xi}	3.5993 (17)	C4…C4 ^{vi}	3.5397 (16)
S5…H9C ⁱ	2.6700	C7…C7 ^{xiv}	3.5970 (17)
S5…H9C	2.6700	C7…C7 ^{xviii}	3.5970 (17)
O8…Br1	3.114 (3)	C7…C7 ^{xix}	3.5970 (17)
O8…C4 ^{xii}	3.352 (4)	C7…C7 ^{xv}	3.5970 (17)
O8…C4 ^{xiii}	3.352 (4)	C9…C9 ^{xx}	3.467 (6)
O8…Br1 ⁱⁱ	3.155 (3)	C9…C9 ^{xxi}	3.467 (6)
O8…Br1 ^{iv}	3.155 (3)	H4…O8 ^{xvi}	2.4300
O8…H4 ^{xii}	2.4300	H4…O8 ^{xvii}	2.4300
O8…H4 ^{xiii}	2.4300	H9A…O8 ^{xviii}	2.7600
O8…H9A ^{xiv}	2.7600	H9A…O8 ^{xv}	2.7600
O8…H9A ^{xv}	2.7600	H9C…S5	2.6700
C3…C3 ^x	3.4158 (11)		

C4—S5—C6	92.36 (16)	C7—C9—H9B	109.00
Br1—C2—C3	120.8 (2)	C7—C9—H9C	109.00
Br1—C2—C6	125.7 (2)	C7—C9—H9A ⁱ	109.00
C3—C2—C6	113.5 (3)	C7—C9—H9B ⁱ	109.00
C2—C3—C4	112.3 (3)	C7—C9—H9C ⁱ	109.00
S5—C4—C3	111.8 (2)	H9A—C9—H9B	109.00
S5—C6—C2	110.0 (2)	H9A—C9—H9C	109.00
S5—C6—C7	120.1 (2)	H9A—C9—H9A ⁱ	138.00
C2—C6—C7	129.9 (2)	H9A—C9—H9B ⁱ	71.00
O8—C7—C6	121.3 (3)	H9B—C9—H9C	109.00
O8—C7—C9	120.8 (3)	H9A ⁱ —C9—H9B	71.00
C6—C7—C9	118.0 (2)	H9B—C9—H9C ⁱ	138.00
C2—C3—H3	124.00	H9B ⁱ —C9—H9C	138.00
C4—C3—H3	124.00	H9C—C9—H9C ⁱ	71.00
S5—C4—H4	124.00	H9A ⁱ —C9—H9B ⁱ	109.00
C3—C4—H4	124.00	H9A ⁱ —C9—H9C ⁱ	109.00
C7—C9—H9A	109.00	H9B ⁱ —C9—H9C ⁱ	109.00
C6—S5—C4—C3	0.00	C3—C2—C6—S5	0.00
C4—S5—C6—C2	0.00	C3—C2—C6—C7	180.00
C4—S5—C6—C7	-180.00	C2—C3—C4—S5	0.00
Br1—C2—C3—C4	-180.00	S5—C6—C7—O8	-180.00
C6—C2—C3—C4	0.00	S5—C6—C7—C9	0.00
Br1—C2—C6—S5	180.00	C2—C6—C7—O8	0.00
Br1—C2—C6—C7	0.00	C2—C6—C7—C9	-180.00

Symmetry codes: (i) $-x, y, z$; (ii) $x, -y, -z+1$; (iii) $-x, y-1/2, -z+1/2$; (iv) $-x, -y, -z+1$; (v) $x, y-1/2, -z+1/2$; (vi) $x+1/2, y, -z+1/2$; (vii) $-x, y+1/2, -z+1/2$; (viii) $x, y+1/2, -z+1/2$; (ix) $x-1/2, y, -z+1/2$; (x) $-x-1/2, y, -z+1/2$; (xi) $-x+1/2, y, -z+1/2$; (xii) $-x, -y+1/2, z+1/2$; (xiii) $x, -y+1/2, z+1/2$; (xiv) $x-1/2, -y+1/2, -z+1$; (xv) $-x+1/2, -y+1/2, -z+1$; (xvi) $-x, -y+1/2, z-1/2$; (xvii) $x, -y+1/2, z-1/2$; (xviii) $x+1/2, -y+1/2, -z+1$; (xix) $-x-1/2, -y+1/2, -z+1$; (xx) $x, -y+1, -z+1$; (xxi) $-x, -y+1, -z+1$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4 [⋯] O8 ^{xvi}	0.93	2.43	3.352 (4)	174

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

Fig. 1

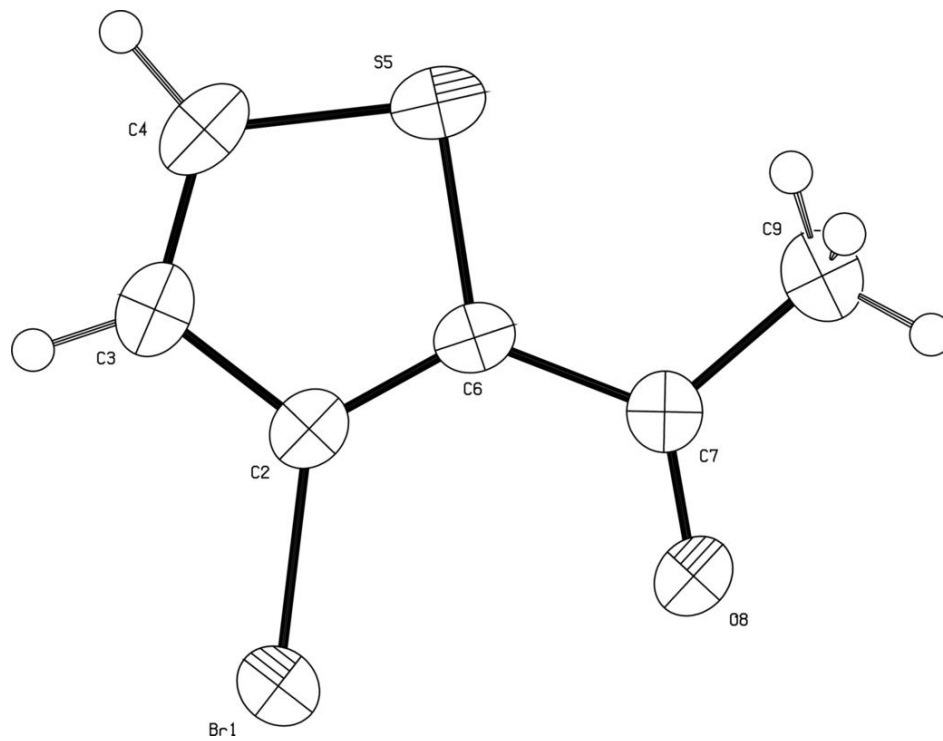


Fig. 2

