

William T. A. Harrison,<sup>a\*</sup>  
H. S. Yathirajan,<sup>b</sup>  
B. V. Ashalatha,<sup>c</sup> S. Bindya<sup>d</sup> and  
B. Narayana<sup>c</sup>

<sup>a</sup>Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, <sup>b</sup>Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, <sup>c</sup>Department of Chemistry, Mangalore University, Mangalagangotri 574 199, India, and <sup>d</sup>Department of Chemistry, Sri Jayachamarajendra College of Engineering, Mysore 570 006, India

Correspondence e-mail:  
w.harrison@abdn.ac.uk

#### Key indicators

Single-crystal X-ray study  
 $T = 120$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.023  
 $wR$  factor = 0.056  
Data-to-parameter ratio = 16.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## (2E)-1-(3-Bromo-2-thienyl)-3-(4-methoxyphenyl)prop-2-en-1-one

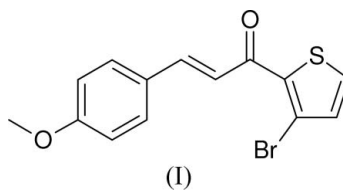
The molecules of the title compound,  $\text{C}_{14}\text{H}_{11}\text{BrO}_2\text{S}$ , display some distorted geometrical values that may be ascribed to an  $\text{H}\cdots\text{Br}$  close contact. In the crystal structure, the molecules form translation-symmetry-generated infinite chains by way of a  $\text{C}-\text{H}\cdots\text{O}$  interaction.

Received 22 August 2006

Accepted 22 August 2006

#### Comment

Chalcones and their heterocyclic derivatives show numerous biological effects (Opletalova & Sedivy, 1999). As part of our ongoing studies of these types of chalcones (Harrison *et al.*, 2006; Yathirajan *et al.*, 2006), the synthesis and structure of the title compound, (I) (Fig. 1), are presented here.



The bond lengths and angles in (I) mostly fall within their expected ranges (Cambridge Structural Database, Version 5.27; Allen, 2002). The terminal C14 methyl group is almost coplanar with its adjacent C8–C13 benzene ring mean plane [deviation of C14 = 0.015 (5) Å]. The dihedral angle between the C8–C13 benzene ring and C1–C4/S1 thiophene ring is 19.58 (9)°. The C5=O1 carbonyl group is also twisted with respect to the heterocycle, as reflected in the S1–C4–C5–O1 and C3–C4–C5–O1 torsion angles of –15.9 (3) and 164.0 (3)°, respectively.

The C3–C4–C5 angle of 135.8 (2)° is far more obtuse than the S1–C4–C5 angle of 114.52 (18)°, possibly as a result of a close intramolecular contact between Br1 and H6 (attached to C6): the separation of these atoms in (I) is 2.73 Å compared to the expected Bondi (1964) van der Waals separation of 3.05 Å. We presume that this represents a steric repulsion between Br and H rather than a  $\text{C}-\text{H}\cdots\text{Br}$  'bond'. The difference between the C4–C3–Br1 and C2–C3–Br1 bond angles [126.74 (18) and 119.08 (18)°, respectively] might also reflect this repulsive contact. Similar angular distortions have been seen in other 4-bromothiophenes such as 4-(4-bromo-5-methylthiophen-2-yl)pyridine (Xu *et al.*, 2005) and 3,4'-dibromo-2,2'-bithiophene (Antolini *et al.*, 1997).

The crystal packing in (I) is consolidated by  $\text{C1}-\text{H1}\cdots\text{O2}^i$  interactions (Table 1) that link the molecules into chains propagating in [001]. A slightly short  $\text{Br1}\cdots\text{O2}^{ii}$  [symmetry code: (ii)  $1-x, \frac{1}{2}+y, 1-z$ ] contact of 3.2184 (18) Å arises; the expected Bondi separation is 3.37 Å.

## Experimental

3-Bromo-2-acetylthiophene (10 g, 0.048 mol) in methanol (50 ml) was mixed with 4-methoxybenzaldehyde (6.52 g, 0.048 mol) and the mixture was treated with 10 ml of 30% potassium hydroxide solution at 278 K. The reaction mixture was then brought to room temperature and stirred for 3 h. The precipitated solid was filtered and washed with water, dried and recrystallized from acetone to yield light yellow crystals of (I) (yield 80%; m.p. 383 K).

### Crystal data

$C_{14}H_{11}BrO_2S$	$Z = 2$
$M_r = 323.20$	$D_x = 1.711 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 4.0025 (1) \text{ \AA}$	$\mu = 3.43 \text{ mm}^{-1}$
$b = 10.7048 (3) \text{ \AA}$	$T = 120 (2) \text{ K}$
$c = 14.6451 (5) \text{ \AA}$	Slab, yellow
$\beta = 91.789 (2)^\circ$	$0.34 \times 0.18 \times 0.07 \text{ mm}$
$V = 627.18 (3) \text{ \AA}^3$	

### Data collection

Nonius KappaCCD diffractometer	7197 measured reflections
$\varphi$ and $\omega$ scans	2705 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2003)	2555 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.388$ , $T_{\max} = 0.795$	$R_{\text{int}} = 0.024$
	$\theta_{\max} = 27.5^\circ$

### Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\max} = 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.023$	$\Delta\rho_{\max} = 0.70 \text{ e \AA}^{-3}$
$wR(F^2) = 0.056$	$\Delta\rho_{\min} = -0.55 \text{ e \AA}^{-3}$
$S = 1.08$	Extinction correction: <i>SHELXL97</i>
2705 reflections	Extinction coefficient: 0.016 (2)
165 parameters	Absolute structure: Flack (1983), 1198 Friedel pairs
H-atom parameters constrained	Flack parameter: 0.013 (7)
$w = 1/[\sigma^2(F_o^2) + 0.0736P]$ where $P = (F_o^2 + 2F_c^2)/3$	

**Table 1**

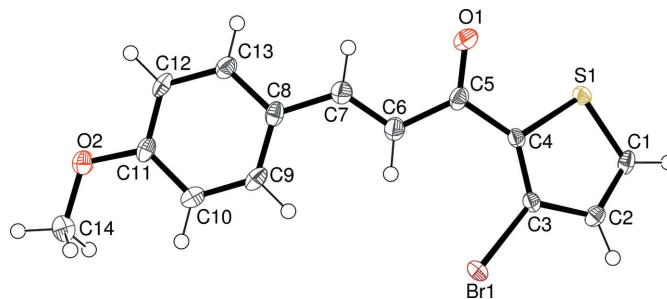
Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C1-H1\cdots O2^i$	0.95	2.54	3.457 (3)	162

Symmetry code: (i)  $x, y, z - 1$ .

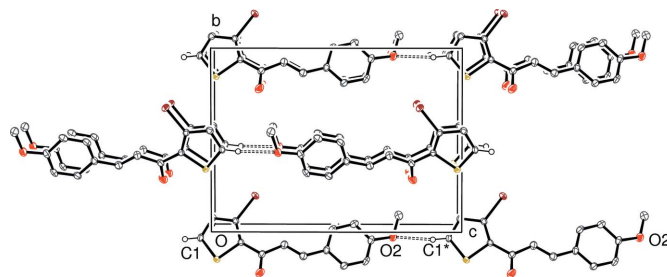
The H atoms were placed in idealized locations ( $C-H = 0.95-0.99 \text{ \AA}$ ) and refined as riding, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{methyl C})$ . The methyl group was rotated about its  $C-O$  bond to best fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997), and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.



**Figure 1**

View of (I) showing 50% displacement ellipsoids and arbitrary spheres for the H atoms.



**Figure 2**

Unit-cell packing in (I), with all H atoms except H1 omitted for clarity and  $C-H\cdots O$  interactions indicated by dashed lines. Atoms with an asterisk (\*) are generated by the symmetry operation  $(x, y, z + 1)$ .

We thank the EPSRC National Crystallography Service (University of Southampton) for data collection.

## References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Antolini, L., Goldoni, F., Iarossi, D., Mucci, A. & Schenetti, L. (1997). *J. Chem. Soc. Perkin Trans. 1*, pp. 1957–1961.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Harrison, W. T. A., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Vijaya Raj, K. K. (2006). *Acta Cryst.* **E62**, o1578–o1579.
- Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Opletalova, V. & Sedivy, D. (1999). *Ceska Slov. Farm.* **48**, 252–255.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Xu, F., Chen, Z., Zhang, F., Wang, R.-J. & Zhao, F. (2005). *Acta Cryst.* **E61**, o1672–o1673.
- Yathirajan, H. S., Sarojini, B. K., Bindya, S., Narayana, B. & Bolte, M. (2006). *Acta Cryst.* **E62**, o4046–o4047.