

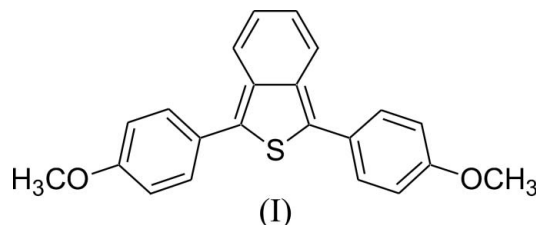
## 1,3-Bis(4-methoxyphenyl)-2-benzothiophene

K. Palani,<sup>a</sup> P. Amaladass,<sup>b</sup>  
A. K. Mohanakrishnan<sup>b</sup> and  
M. N. Ponnuswamy<sup>a\*</sup><sup>a</sup>Department of Crystallography and Biophysics,  
University of Madras, Guindy Campus, Chennai  
600 025, India, and <sup>b</sup>Department of Organic  
Chemistry, University of Madras, Guindy  
Campus, Chennai 600 025, IndiaCorrespondence e-mail:  
mnpsy2004@yahoo.com

## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.058  
 $wR$  factor = 0.235  
Data-to-parameter ratio = 22.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.In the title compound,  $\text{C}_{22}\text{H}_{18}\text{O}_2\text{S}$ , the two benzene rings are twisted away from the thiophene ring by 34.99 (9) and 41.57 (9)°.  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\pi$  hydrogen bonds are observed in the crystal structure.Received 21 November 2005  
Accepted 28 November 2005  
Online 7 December 2005

## Comment

Most Schiff bases and their thiophene derivatives possess pharmacological activities such as antibacterial, anticancer, anti-inflammatory and antitoxic properties (Gewald *et al.*, 1966). The diaryl-substituted heterocyclic molecules act as selective COX-2 inhibitors (Portevin *et al.*, 2000) and anti-tumor agents (Szczepankiewicz *et al.*, 2001). In view of this importance, the crystal structure of the title compound, (I), has been determined and the results are presented here.

A *ZORTEP* (Zsolnai, 1997) plot of the molecule is shown in Fig. 1. The bond lengths and bond angles (Table 1) in the thiophene ring are comparable with those reported for 4-{5-[3,4-dimethyl-5-(3,4,5-trimethoxyphenyl)thiophen-2-yl]-2-methoxyphenyl}morpholine (Shi *et al.*, 2004). The C9–C14 and C16–C21 benzene rings are oriented at angles of 34.99 (9) and 41.57 (9)°, respectively, with respect to the thiophene ring. The dihedral angle between the C9–C14 and C16–C21 benzene rings is 71.2 (1)°. Both the methoxy groups are coplanar with the attached rings. The crystal packing is stabilized by  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\pi$  type hydrogen bonds (Table 2 and Fig. 2).

## Experimental

4-Methoxymagnesium bromide was prepared from 4-bromoanisole (23 mmol) and Mg (25 mmol). 4-Methoxymagnesium bromide was added to a solution of 3-(4-methoxyphenyl)isobenzofuran-1(3H)-one (20.8 mmol) at 273 K. The reaction mixture was stirred at room temperature for 5 h and then poured into an ice-cooled aqueous  $\text{NH}_4\text{Cl}$  solution, extracted with  $\text{CH}_2\text{Cl}_2$  (50 ml) and dried over  $\text{Na}_2\text{SO}_4$ . The reaction mixture was treated with Lawesson's reagent (10.4 mmol) and stirred at room temperature for 5 h. The solvent was removed and the residue was gently heated on a steam bath with ethanol. The product was purified by column chromatography (neutral alumina, hexane) to afford compound (I) as a yellow

powder. Single crystals of (I) were obtained by recrystallization from boiling hexane.

Crystal data

C<sub>22</sub>H<sub>18</sub>O<sub>2</sub>S  
*M<sub>r</sub>* = 346.42  
 Orthorhombic, *Pcab*  
*a* = 7.508 (6) Å  
*b* = 16.493 (9) Å  
*c* = 27.952 (9) Å  
*V* = 3461 (4) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 1.330 Mg m<sup>-3</sup>

Mo Kα radiation  
 Cell parameters from 25 reflections  
 $\theta = 1.5\text{--}30.0^\circ$   
 $\mu = 0.20\text{ mm}^{-1}$   
*T* = 293 (2) K  
 Block, yellow  
 0.24 × 0.22 × 0.19 mm

Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 5044 measured reflections  
 5044 independent reflections  
 3579 reflections with *I* > 2σ(*I*)

$\theta_{\text{max}} = 30.0^\circ$   
*h* = 0 → 10  
*k* = 0 → 23  
*l* = 0 → 39  
 3 standard reflections  
 frequency: 60 min  
 intensity decay: none

Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.058  
*wR* (*F*<sup>2</sup>) = 0.235  
*S* = 1.04  
 5044 reflections  
 228 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1453P)^2 + 1.73P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.65\text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.49\text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

S1—C1	1.710 (3)	O1—C15	1.422 (4)
S1—C8	1.712 (3)	O2—C19	1.367 (3)
O1—C12	1.378 (3)	O2—C22	1.420 (4)
C1—S1—C8	94.5 (2)	C14—C9—C10	116.9 (2)
C12—O1—C15	117.3 (2)	C21—C16—C17	117.7 (2)
C19—O2—C22	116.7 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

*Cg*<sub>1</sub> and *Cg*<sub>2</sub> are the centroids of the C16–C21 and C9–C14 benzene rings, respectively.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C15—H15A...O2 <sup>i</sup>	0.96	2.52	3.385 (5)	149
C11—H11... <i>Cg</i> <sub>1</sub> <sup>ii</sup>	0.93	2.73	3.595 (4)	155
C14—H14... <i>Cg</i> <sub>2</sub> <sup>iii</sup>	0.93	2.78	3.550 (4)	141
C18—H18... <i>Cg</i> <sub>2</sub> <sup>iv</sup>	0.93	2.88	3.662 (4)	143

Symmetry codes: (i)  $-x + \frac{3}{2}, y + \frac{1}{2}, -z + 1$ ; (ii)  $-x + 2, -y, -z + 1$ ; (iii)  $x - \frac{1}{2}, -y + \frac{1}{2}, z$ ; (iv)  $-x + 1, -y, -z + 1$ .

H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H = 0.93 or 0.96 Å and *U*<sub>iso</sub>(H) = 1.2–1.5*U*<sub>eq</sub>(C).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

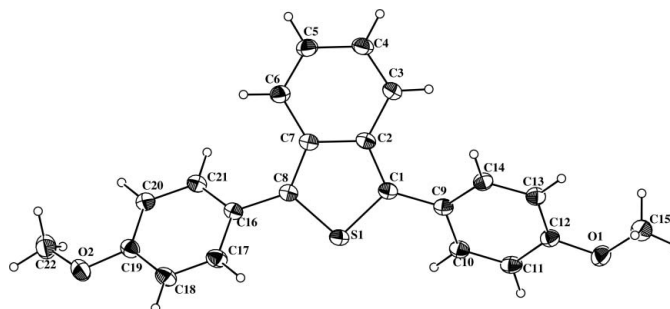


Figure 1  
 The molecular structure of (I), showing 30% probability displacement ellipsoids.

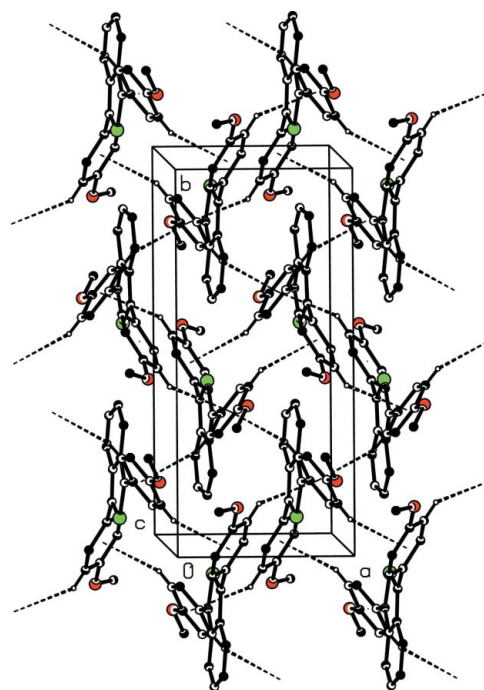


Figure 2

The crystal packing of (I), viewed approximately down the *c* axis. Dashed lines indicate C—H...π interactions. Most H atoms have been omitted.

KP thanks the University Grants Commission (UGC) Herbal Science programme for financial support under the ‘University with Potential for Excellence’ scheme. The UGC and the Department of Science & Technology (DST) are gratefully acknowledged for financial support to the Department of Crystallography and Biophysics under the UGC-SAP and DST-FIST programmes.

References

Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.  
 Gewald, K., Schinke, E. & Botcher, H. (1966). *Chem. Ber.* **99**, 99–100.  
 Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.  
 Portevin, B., Tordjman, C., Pastoureau, P., Bonnet, J. & De Nanteuil, G. (2000). *J. Med. Chem.* **43**, 4582–4593.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Shi, J.-X., Hu, Q.-P., Lei, Y.-J. & Reiner, J. (2004). *Acta Cryst.* **E60**, o1810–o1811.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Szczepankiewicz, B. G., Liu, G., Jac, H.-S., Tasker, A. & Gunawardana, I. W. (2001). *J. Med. Chem.* **44**, 4416–1492.

Zsolnai, L. (1997). *ZORTEP*. Univeristy of Heidelberg, Germany.