

2-(4-Bromophenyl)-5-(3,4-dimethoxyphenyl)-  
3,4-dimethylthiopheneYingjie Lei,<sup>a,b\*</sup> Jixian Shi,<sup>a</sup> Mei  
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## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(C-C) = 0.004$  Å  
 $R$  factor = 0.040  
 $wR$  factor = 0.105  
Data-to-parameter ratio = 19.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The title compound,  $C_{20}H_{19}BrO_2S$ , was synthesized by the cyclization of 1-(4-bromophenyl)-4-(3,4-dimethoxyphenyl)-2,3-dimethylbutane-1,4-dione with 2,4-bis(4-methoxyphenyl)-2,4-disulfanylene-1,3,2,4-dithiadiphosphetane. The dimethoxyphenyl and bromophenyl rings make dihedral angles of  $28.4(3)$  and  $45.0(3)^\circ$ , respectively, with the thiophene ring. The bond lengths are unexceptional.

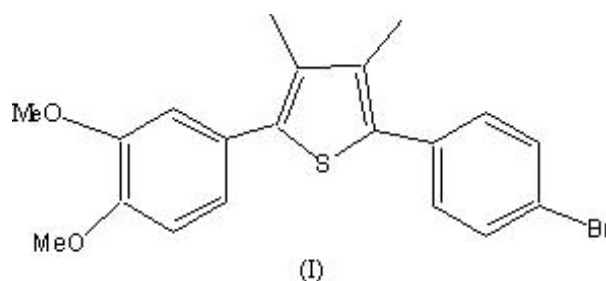
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## Comment

Attention has been attracted to the study of diaryl-substituted heterocycles (Khanna *et al.*, 1997; Penning *et al.*, 1997; Wu-Wong *et al.*, 2001), due to their high biological activity as selective COX-2 inhibitors and antitumor agents (Portevin *et al.*, 2000; Szczepankiewicz *et al.*, 2001). In view of this, we have recently focused on the preparation of diaryl-substituted thiophenes. A new compound, namely 2-(4-bromophenyl)-5-(3,4-dimethoxyphenyl)-3,4-dimethylthiophene, (I), has been synthesized by the cyclization of 1-(4-bromophenyl)-4-(3,4-dimethoxyphenyl)-2,3-dimethylbutane-1,4-dione in the presence of 2,4-bis(4-methoxyphenyl)-2,4-disulfanylene-1,3,2,4-dithiadiphosphetane. An X-ray crystal structure determination of (I) was carried out to elucidate the structure, and the results are presented here.



The molecular structure of (I) is illustrated in Fig. 1. Two benzene rings are linked by a planar thiophene ring. The C5–C10 (*A*) and C13–C18 (*B*) aromatic rings make dihedral angles of  $45.0(3)$  and  $28.4(3)^\circ$ , respectively, with the thiophene ring; rings *A* and *B* are inclined at an angle of  $70.9(3)^\circ$  with respect to one another. The relatively short distances of O1–C15 [ $1.362(3)$  Å], O2–C16 [ $1.361(3)$  Å] and C8–Br1 [ $1.890(3)$  Å] are due to the  $p$ - $\pi$  conjugation of the O atoms or Br atom with the phenyl rings.

## Experimental

2,4-Bis(4-methoxyphenyl)-2,4-disulfanylene-1,3,2,4-dithiadiphosphetane (23 mg, 0.27 mmol) was added to a solution of 1-(4-bromophenyl)-4-(3,4-dimethoxyphenyl)-2,3-dimethylbutane-1,4-

dione (90 mg, 0.22 mmol) in anhydrous dioxane (6 ml). The reaction mixture was stirred at 358 K for about 5 h. The solvent was then removed under reduced pressure and the residue was purified by flash chromatography (ethyl acetate–petroleum ether). A white powder was obtained (yield 68.2%) and single crystals suitable for crystallographic analysis were obtained by slow evaporation of an ethyl acetate solution (m.p. 433–434 K).

Crystal data

$C_{20}H_{19}BrO_2S$   $D_x = 1.484 \text{ Mg m}^{-3}$   
 $M_r = 403.32$  Mo  $K\alpha$  radiation  
 Monoclinic,  $P2_1/c$  Cell parameters from 1893 reflections  
 $a = 16.544 (2) \text{ \AA}$   $\theta = 2.5\text{--}22.3^\circ$   
 $b = 15.0056 (19) \text{ \AA}$   $\mu = 2.40 \text{ mm}^{-1}$   
 $c = 7.3884 (10) \text{ \AA}$   $T = 293 (2) \text{ K}$   
 $\beta = 100.220 (2)^\circ$  Block, colorless  
 $V = 1805.1 (4) \text{ \AA}^3$   $0.20 \times 0.18 \times 0.12 \text{ mm}$   
 $Z = 4$

Data collection

Bruker SMART CCD area-detector diffractometer 4361 independent reflections  
 $\varphi$  and  $\omega$  scans 2503 reflections with  $I > 2\sigma(I)$   
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)  $R_{int} = 0.041$   
 $T_{min} = 0.602, T_{max} = 0.750$   $\theta_{max} = 28.1^\circ$   
 12159 measured reflections  $h = -21 \rightarrow 20$   
 $k = -18 \rightarrow 19$   
 $l = -8 \rightarrow 9$

Refinement

Refinement on  $F^2$  H-atom parameters constrained  
 $R[F^2 > 2\sigma(F^2)] = 0.040$   $w = 1/[\sigma^2(F_o^2) + (0.0489P)^2]$   
 $wR(F^2) = 0.106$  where  $P = (F_o^2 + 2F_c^2)/3$   
 $S = 0.99$   $(\Delta/\sigma)_{max} = 0.001$   
 4361 reflections  $\Delta\rho_{max} = 0.31 \text{ e \AA}^{-3}$   
 221 parameters  $\Delta\rho_{min} = -0.43 \text{ e \AA}^{-3}$

Table 1

Selected bond lengths (Å).

Br1—C8	1.890 (3)	O2—C16	1.361 (3)
O1—C15	1.362 (3)		

All H atoms were positioned geometrically and refined as riding (C—H = 0.93–0.96 Å). For CH and CH<sub>2</sub> groups,  $U_{iso}(H)$  values were set equal to  $1.2U_{eq}(\text{carrier atom})$  and for the methyl groups they were set equal to  $1.5U_{eq}(\text{carrier atom})$ .

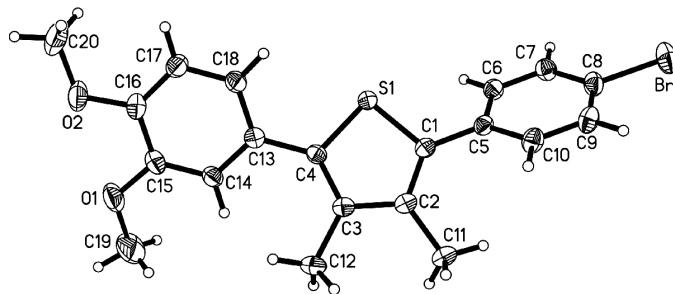


Figure 1 View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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

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