Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[4-(Benzyloxy)phenyl]-5-(3,4-dimethoxyphenyl)-3,4-dimethylthiophene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.057
wR factor = 0.178
Data-to-parameter ratio = 13.1

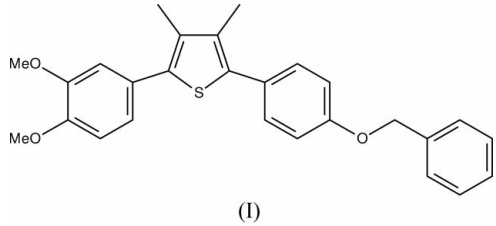
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 September 2004
Accepted 4 October 2004
Online 9 October 2004

The title compound, $C_{27}H_{26}O_3S$, was synthesized by the cyclization of 1-[4-(benzyloxy)phenyl]-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 bond lengths are unexceptional and the C atoms of the benzyloxy group are disordered.

Comment

Diaryl-substituted heterocycles have shown promising biological activities as selective COX-2 inhibitors (Portevin *et al.*, 2000) and antitumor agents (Szczepankiewicz *et al.*, 2001). In particular, celecoxib (SC-58635), rofecoxib and A-105972 have been marketed for the treatment of inflammatory disorders and cancer cells. In view of this potential, we have recently focused our attention on the preparation of diaryl-substituted thiophenes. A new compound, namely 2-[4-(benzyloxy)phenyl]-5-(3,4-dimethoxyphenyl)-3,4-dimethylthiophene, (I), has been synthesized by the cyclization of 1-[4-(benzyloxy)phenyl]-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.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The bond lengths are within the normal published ranges (Allen *et al.*, 1987). The C atoms of the benzyloxy group are disordered over two positions. We denote the four rings as follows: *A*, C1–C6; *B*, S1/C7–C10; *C*, C11–C16; *D*, C18–C23. The dihedral angles between these rings are 29.6 (2) (*A*/*B*), 21.4 (4) (*A*/*D*), 45.0 (2) (*B*/*C*) and 87.2 (4)° (*C*/*D*).

Experimental

The title compound was synthesized as follows: 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-(benzyloxy)phenyl]-4-(3,4-dimethoxyphenyl)-2,3-dimethylbutane-1,4-dione (200 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 chromato-

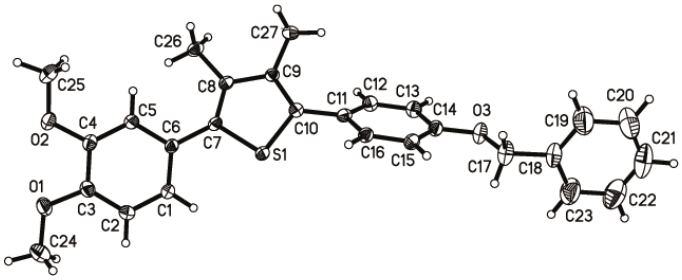


Figure 1
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. The minor disorder component is not shown.

graphy (ethyl acetate–petroleum ether). A white powder was obtained (yield 85.2%) and single crystals suitable for crystallographic analysis were obtained by slow evaporation of an ethyl acetate solution (m.p. 445–447 K). ^{1}H NMR (DMSO, p.p.m.): δ 7.47–6.94 (*m*, 12H), 5.14 (*s*, 2H), 3.78 (*s*, 6H), 2.17 (*s*, 3H), 2.15 (*s*, 3H).

Crystal data

$C_{27}H_{26}O_3S$
M_r = 430.55
Monoclinic, $P2_1/c$
a = 20.779 (8) Å
b = 15.097 (6) Å
c = 7.317 (3) Å
β = 96.624 (7)°
V = 2280.0 (16) Å^3
Z = 4
D_x = 1.254 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 874 reflections
θ = 3.2–24.6°
μ = 0.17 mm^{-1}
T = 293 (2) K
Block, colorless
0.18 × 0.16 × 0.12 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.960, T_{max} = 0.980
11 551 measured reflections
4011 independent reflections
2389 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 25.0°
$h = -24 \rightarrow 20$
$k = -17 \rightarrow 14$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.178
S = 1.07
4011 reflections
306 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0845P)^2 + 0.7221P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

All H atoms were positioned geometrically and refined as riding (C—H = 0.93–0.97 Å). For CH and CH_2 groups, U_{iso}(H) values were set equal to $1.2U_{eq}$(carrier atom), and for the methyl groups they were set equal to $1.5U_{eq}$(carrier atom). Atoms C17—C23 are disordered over two sites with occupancies of 0.61 (2) and 0.39 (2).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge support from the Basic Research Project (#2002CCA01500) of the MOST, and from the National Young Scholar Award of the NSFC (#30125043).

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