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Bis[4-(5-methoxycarbonyl-2-thienyl)phenyl] sulfide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.150
Data-to-parameter ratio = 18.5

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

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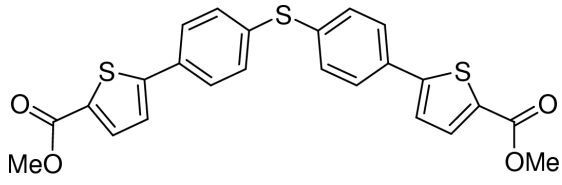
In the title compound, $C_{24}H_{18}O_4S_3$, the central S atom lies on a twofold axis. The asymmetric unit, one half of the molecule, is almost planar, with a dihedral angle between the phenyl and thienyl rings of 10.9 (1)°. The C1—S1—C1′ angle is 103.5 (1)°.

Comment

Supramolecular chemistry based on molecular recognition has added a new dimension to chemistry and stereochemistry and is a fast growing subject. Considered efforts generally directed towards modeling for biological non-covalent binding in chemical systems resulted not only in the synthesis of numerous artificial receptors but also in the development of innovative approaches to the generation of selective non-covalent binders (Haldar *et al.*, 1997). Ray *et al.* (2001) have prepared the title compound, (I), by a very recent method developed for the synthesis of sulfur pivoted cavity-shaped polycyclic thiophene derivatives in three steps from diphenyl sulfide. We have undertaken an X-ray structure determination of (I) in order to establish its chemical structure and conformation.



(I)

In compound (I), the bond lengths and angles show normal values. The values in the phenylthiophene moiety agree with those observed in the related structure previously studied (Joseph *et al.*, 1991). The C1—S1—C1′ angle is 103.5 (1)°.

In the structure of (I) there is only one half of the molecule in the asymmetric unit, and the unit cell contains four molecules. One half of the molecule is related to the other by a twofold axis passing through the S1 atom and is nearly planar. Both the phenyl and thienyl rings are planar, with maximum deviations of 0.009 (3) Å at C3 and 0.009 (2) Å at C7. The mean planes through the phenyl and thienyl rings form a dihedral angle of 10.9 (1)°. The carbomethoxy group is also planar and is twisted by 8.4 (1)° from the plane of the thienyl ring.

Experimental

The title compound, (I), was synthesized from commercially available diphenyl sulfide (Aldrich) by Friedel–Crafts acylation with excess of acetyl chloride and anhydrous aluminium chloride followed by the

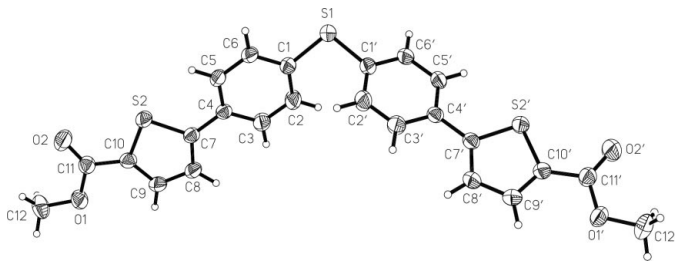


Figure 1
The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

formation of bis-chloroaldehyde (phosphorusoxytrichloride and dimethylformamide) and its condensation with two equivalents of methyl thioglycolate/triethylamine, and concomitant ring closure with 50% potasium hydroxide. Single crystals suitable for X-ray structure determination were obtained by slow evaporation from a benzene solution.

Crystal data

$C_{24}H_{18}O_4S_3$
$M_r = 466.56$
Monoclinic, $C2/c$
$a = 11.8692\ (3)$ Å
$b = 6.0255\ (2)$ Å
$c = 29.7783\ (6)$ Å
$\beta = 94.221\ (1)^\circ$
$V = 2123.9\ (1)$ Å^3
$Z = 4$
$D_x = 1.459$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4461 reflections
$\theta = 1.4$–28.5°
$\mu = 0.38$ mm^{-1}
$T = 293\ (2)$ K
Prism, yellow
0.48 × 0.44 × 0.18 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.839$, $T_{max} = 0.935$
7327 measured reflections
2620 independent reflections
1895 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 28.6^\circ$
$h = -13 \rightarrow 15$
$k = -7 \rightarrow 8$
$l = -36 \rightarrow 39$
Intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.151$
$S = 1.03$
2620 reflections
142 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0803P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.765 (2)	C4—C7	1.464 (3)
S2—C10	1.715 (2)	C7—C8	1.364 (3)
S2—C7	1.718 (2)	C8—C9	1.389 (3)
O1—C12	1.431 (3)	C9—C10	1.354 (3)
C1—C2	1.384 (3)	C10—C11	1.462 (3)
C1—S1—C1^{i}	103.51 (14)	C9—C10—S2	111.44 (18)
C10—S2—C7	91.82 (11)	O2—C11—C10	124.5 (2)
C6—C1—S1	118.83 (17)	O1—C11—C10	111.4 (2)
C8—C7—S2	110.07 (17)		
C1^{i}—S1—C1—C6	143.3 (2)	S2—C10—C11—O2	9.5 (4)
C1^{i}—S1—C1—C2	−41.89 (19)	C9—C10—C11—O1	7.8 (4)
C5—C4—C7—S2	11.9 (3)		

Symmetry code: (i) $-x, y, \frac{3}{2} - z$.

After checking their presence in a difference map, all H atoms were geometrically fixed and allowed to ride on their attached atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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