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4,5-Bis(phenylsulfonylthio)-1,3-dithiolane-2-thione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.014 Å
R factor = 0.066
wR factor = 0.185
Data-to-parameter ratio = 16.0

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

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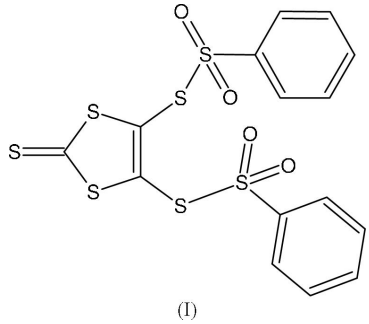
The central 1,3-dithiolane-2-thione fragment of the title compound, $C_{15}H_{10}O_4S_7$, is planar; the conformations of the phenylsulfonylthio substituents in positions 4 and 5 of the dithiolane ring are related by an approximate twofold axis coinciding with the C=S bond of the 1,3-dithiolane-2-thione group. The planes of the phenyl rings form dihedral angles of 25.4 (4) and 31.1 (4)° with the least-squares plane of the central dithiolane ring.

Comment

TTF (tetrathiafulvalene) and BEDT-TTF [bis(ethylenedithio)tetrathiafulvalene] derivatives and their charge-transfer salts have attracted considerable interest because of their high electronic conductivity or superconductivity (Williams *et al.*, 1992). Furthermore, these compounds have received some attention in the third-order nonlinear optical field as a result of their extensively conjugated structure (Huggard & Blau, 1987). In order to obtain materials with high optical and/or electrical properties, and to investigate structure–property relationships, compounds with various groups bonded to sulfur in the 4- and 5-positions of the dithiolane ring are being studied extensively. As a part of these studies, the title compound, (I), the precursor of new TTF and BEDT-TTF derivatives, was prepared.



(I)

The molecular structure of (I), along with the atom-numbering scheme, is shown in Fig. 1. Thione atom S1 is coplanar with the dithiolane ring, and the C1=S1 distance [1.622 (7) Å] is almost identical to that of a typical C=S double bond (1.60 Å; Allen *et al.*, 1987). The C—S bond lengths in the five-membered ring (1.722–1.749 Å), on the other hand, are shorter than a typical C—S single bond (1.82 Å; Allen *et al.*, 1987). These values show the high degree of conjugation in the five-membered ring of the title compound.

The 1,3-dithiolane-2-thione fragment is planar; the conformations of the phenylsulfonylthio substituents are related by

an approximate twofold axis coinciding with the C1═S1 bond. The planes of the C4–C9 and C10–C15 phenyl rings form dihedral angles of 25.4 (4) and 31.1 (4)°, respectively, with the least-squares plane of the central dithiolane ring.

The crystal packing of the title compound does not show any specific interactions. A packing diagram is shown in Fig. 2.

Experimental

The title compound was prepared by the reaction of $(Bu_4N)_2[Zn(dmit)_2]$ (dmit is the 2-thioxo-1,3-dithiolane-4,5-dithiolate dianion, $C_3S_5^{2-}$) and benzenesulfonyl chloride in acetone. Benzenesulfonyl chloride (60 mmol) was added dropwise to a solution of $(Bu_4N)_2[Zn(dmit)_2]$ (15 mmol) in acetone (100 ml). The mixture was stirred for 2 h at room temperature and then dissolved in acetone (100 ml). The resulting orange precipitate was filtered off; the orange filtrate was then left to stand for several days at approximately 277 K, and yellow crystals used for the X-ray structure determination were obtained.

Crystal data

$C_{15}H_{10}O_4S_7$	D_x = 1.614 Mg m^{-3}
M_r = 478.65	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 61 reflections
a = 12.3011 (17) Å	θ = 5.0–12.4°
b = 12.3846 (13) Å	μ = 0.82 mm^{-1}
c = 14.187 (2) Å	T = 293 (1) K
β = 114.338 (11)°	Prism, yellow
V = 1969.2 (5) Å^3	0.35 × 0.31 × 0.22 mm
Z = 4	

Data collection

Bruker $P4$ diffractometer	R_{int} = 0.054
ω scans	θ_{max} = 26.0°
Absorption correction: ψ scan (*XSCANS*; Bruker, 1996)	$h = -15 \rightarrow 1$
	$k = -15 \rightarrow 1$
T_{min} = 0.638, T_{max} = 0.835	$l = -16 \rightarrow 17$
4637 measured reflections	3 standard reflections
3768 independent reflections	every 97 reflections
1760 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.02P)^2 + 8.6426P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.066	
$wR(F^2)$ = 0.185	
S = 1.13	$(\Delta/\sigma)_{max}$ <0.001
3768 reflections	$\Delta\rho_{max}$ = 0.45 e Å^{-3}
236 parameters	$\Delta\rho_{min}$ = −0.61 e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXTL*
	Extinction coefficient: 0.0058 (5)

All H atoms, placed in geometrically calculated positions (C—H = 0.93 Å), were refined in the riding-model approximation; their U_{iso}(H) values were set to 1.2 times U_{eq} of their parent atoms.

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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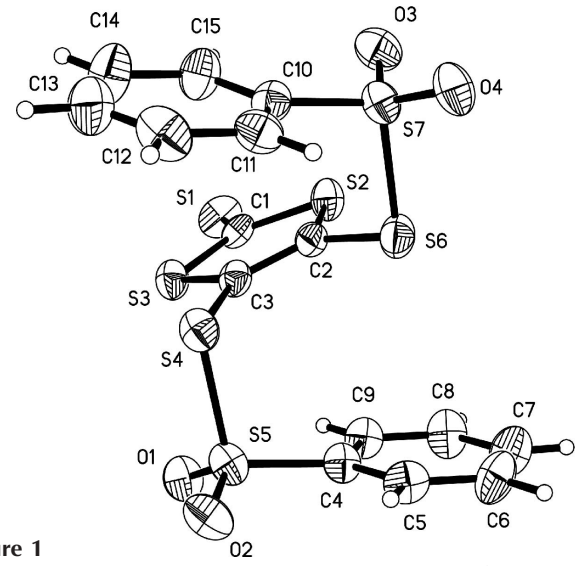

Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

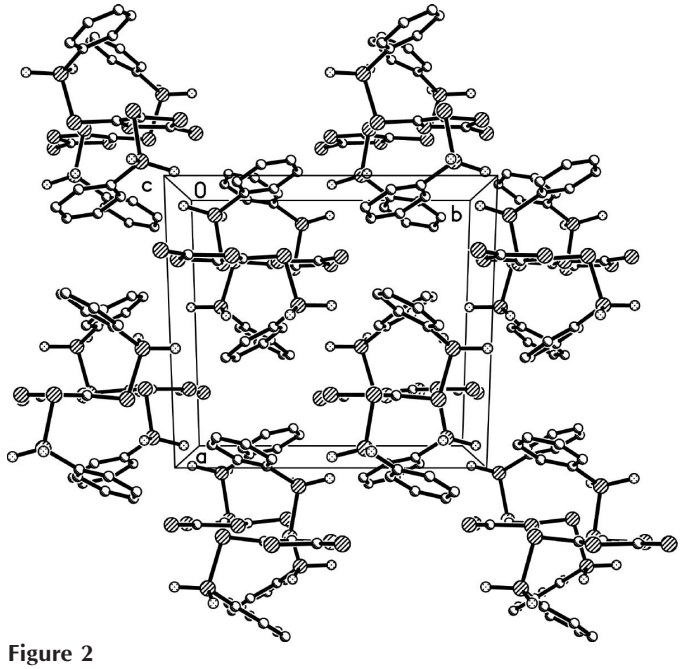

Figure 2
Packing diagram for the title compound viewed along the *c* axis. H atoms have been omitted.

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