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1,6-Bis(phenylsulfanyl)hexane

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The title compound, $C_{16}H_{18}S_2$ (or L^6), crystallizes with two half-molecules in the asymmetric unit and each independent molecule lies about a crystallographic center of symmetry. The aliphatic segment of this ligand is in an all-*trans* conformation.

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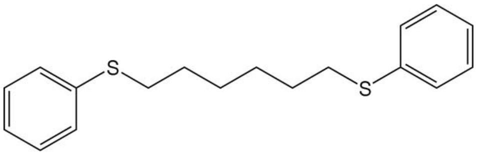
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.036
wR factor = 0.068
Data-to-parameter ratio = 17.0

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

Comment

Linear bifunctional ligands are usually used as building blocks for the construction of metal-organic framework materials (MOF) (Carlucci *et al.*, 2002). Sulfur, being a soft base, has good ability to coordinate to an Ag atom which is a soft acid. As the dithiolate ligand affords two coordination sites to metal centers, supramolecular architectures may be achieved (Black *et al.*, 1995; Bu *et al.*, 2002). This flexible ligand was synthesized in order to help understand the formation of supramolecular networks, which is affected by many factors, such as the type of solvents, the counter-anions, the metal-to-ligand ratio, the metal coordination and the ligand flexibility [see, for example, Withersby *et al.* (1997, 1999), Noro *et al.* (2002), Blake *et al.* (1999)]. We have recently determined the stuctures of the analagous compounds L^{10} and L^2 (Awaleh *et al.*, 2005*a*,*b*). We report here the structural characterization of 1,6-bis(phenylsulfanyl)hexane (L^6).



(I)

L^6 crystallizes with two half-molecules in the asymmetric unit. A view of one of the molecules is shown in Fig. 1. Each of the two molecules has a center of symmetry at the mid-point of the central C—C bond, *viz*. C19—C19^{i} [symmetry code: (i)

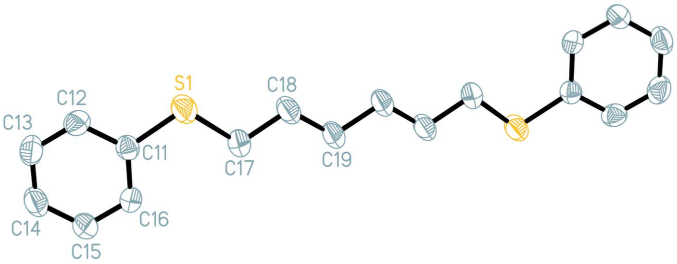


Figure 1
View of one molecule of L^6, showing the atom-numbering scheme. Probability displacement ellipsoids are shown at the 50% level. The H atoms have been omitted. The unlabeled part of the molecule is related by the symmetry code ($-x$, $-y + 1$, $-z$).

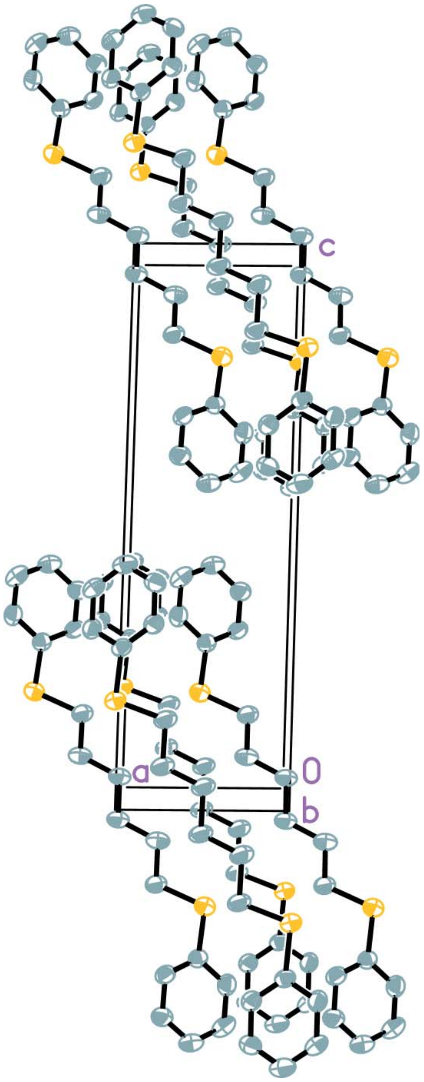


Figure 2
The crystal packing of L^6, viewed along the b axis. H atoms have been omitted.

$-x, 1 - y, -z$] and C29—C29^{ii} [symmetry code: (ii) $1 - x, -y, 2 - z$]. The torsion angles in the aliphatic segment of L^6 are all *trans*, indicating that the molecules are in the fully extended conformation (Table 1). The dihedral angles between the aromatic groups and the corresponding S–$(CH_2)_6$–S planes are 19.4 (2)° for both molecules. The phenyl groups of neighbouring molecules form a 61.5 (1)° dihedral angle. The bond distances and angles in L^6 are in the normal range (Table 1). The crystal packing of L^6 is depicted in Fig. 2. There are no significant π-stacking interactions in the crystal structure.

Experimental

The title compound, L^6, was synthesized according to a published procedure (Hartley *et al.*, 1979). L^6 was found to be pure from NMR in acetone-d_6 (^{1}H). The compound was obtained as a crystalline powder from which platelet-shaped crystals were gathered. Several crystals were examined, but only one was of barely suitable quality for X-ray analysis (yield: 87%). Analysis found: C 71.43, H 7.32%; calculated for $C_{18}H_{22}S_2$: C 71.47, H 7.33%. ^{1}H NMR (acetone-d$_6$): δ 1.46 [*qt*, 4H, –S–CH$_2$—CH$_2$–(CH$_2$)$_2$–CH$_2$–CH$_2$–S–], 1.63 [*qt*, 4H, –S–CH$_2$–(CH$_2$)–CH$_2$–CH$_2$–(CH$_2$)–CH$_2$–S–], 2.95 [*t*, 4H, –S–(CH$_2$)–CH$_2$–CH$_2$–CH$_2$–CH$_2$–(CH$_2$)–S–], 7.14–7.34 (*m*, 10H, C$_6$H$_5$–).

Crystal data

$C_{18}H_{22}S_2$
$M_r = 302.48$
Triclinic, $P\overline{1}$
$a = 5.627$ (2) Å
$b = 7.862$ (3) Å
$c = 18.486$ (6) Å
$\alpha = 94.55$ (3)°
$\beta = 91.36$ (3)°
$\gamma = 90.46$ (3)°
$V = 815.0$ (5) Å^3

$Z = 2$
$D_x = 1.233$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 20.0$–25.0°
$\mu = 2.84$ mm^{-1}
$T = 298$ (2) K
Platelet, colorless
0.23 × 0.15 × 0.02 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: Gaussian
$T_{\min} = 0.560$, $T_{\max} = 0.940$
25176 measured reflections
3086 independent reflections
1270 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.059$
$\theta_{\max} = 69.8$°
$h = -6 \rightarrow 6$
$k = -9 \rightarrow 9$
$l = -22 \rightarrow 22$
5 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.068$
$S = 0.66$
3086 reflections
181 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0003P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C11	1.764 (3)	S2—C27	1.798 (3)
S1—C17	1.802 (3)	C19—C19^{i}	1.524 (6)
S2—C21	1.766 (3)	C29—C29^{ii}	1.512 (6)
C11—S1—C17	104.78 (17)	C21—S2—C27	105.51 (17)
S1—C17—C18—C19	−179.4 (2)	S2—C27—C28—C29	179.7 (2)
C17—C18—C19—C19^{i}	179.9 (4)	C27—C28—C29—C29^{ii}	−179.7 (4)

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x+1, -y, -z+2$.

The poor quality of the crystal and the fact that only 41% of the measured reflections have $I > 2\sigma(I)$ account for the low S value. H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and were included in the refinement in the riding-model approximation; their displacement parameters were set at 1.2$U_{\rm eq}$ of the parent C atoms. A final verification of possible voids was performed using the VOID routine of the *PLATON* program (Spek, 2003).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: local Program; 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: *UdMX* (Maris, 2004).

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