

1,10-Bis(phenylsulfanyl)decane

Mohamed Osman Awaleh,*
Antonella Badia and François
BrisseDépartement de Chimie, Université de
Montréal, CP 6128, Succ. Centre-ville,
Montréal, Québec, Canada H3C 3J7Correspondence e-mail:
moawaleh2000@yahoo.fr

Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.050
 wR factor = 0.147
Data-to-parameter ratio = 17.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title structure, $\text{C}_{22}\text{H}_{30}\text{S}_2$, L^{10} , the asymmetric unit contains two independent molecules, both of which lie on crystallographic inversion centers, and the aliphatic chains are in the all-trans conformation. The crystal structure is stabilized by a combination of van der Waals forces and weak $\text{C}-\text{H}\cdots\pi(\text{arene})$ interactions.

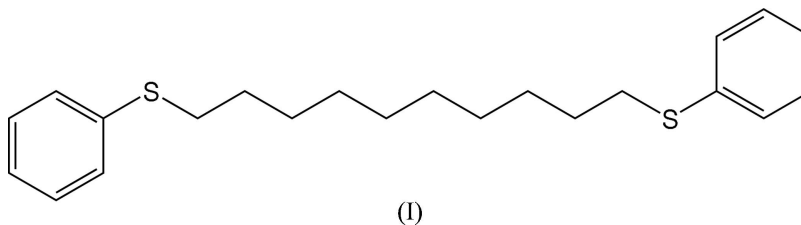
Received 16 June 2005

Accepted 27 June 2005

Online 9 July 2005

Comment

The self-assembly of metal–organic coordination polymers has attracted a great deal of attention because of their potential application as functional materials (Yaghi *et al.*, 1995). Bifunctional organic ligands are usually used as spacers for the building of metal–organic framework materials (MOF) (Carlucci *et al.*, 2002). The S atom is a soft base and has a good ability to coordinate to Ag, which is a soft acid. In addition, the dithiolate building block can afford two coordination sites to a metal center and can be used to construct supramolecular architectures (Black *et al.*, 1995; Bu *et al.*, 2002). When the length of the aliphatic segment between the S atoms is increased, the number of possible conformations and the flexibility of the spacer are augmented. As a result, in metal–organic crystal engineering, when flexible ligands are involved in supramolecular networks, the prediction of the topology of the polymer coordination is more difficult, since there are several other factors affecting the formation of the framework (Withersby *et al.*, 1999, 1997; Noro *et al.*, 2002; Blake *et al.*, 1999). In order to study the effect of the length of the building block on the topology of metal–organic polymer coordination, the title compound, L^{10} , (I), was synthesized and its structure is reported here.



Compound (I) crystallizes with two (essentially equivalent) independent centrosymmetric molecules in the asymmetric unit, one of which is shown in Fig. 1. Each molecule has a center of symmetry at the midpoint of the central C–C bond, namely C35–C35ⁱ and C45–C45ⁱⁱ [symmetry codes: (i) $-x, 1 - y, -z$; (ii) $1 - x, 2 - y, 2 - z$]. The torsion angles in the aliphatic sequence $[\text{S}-(\text{CH}_2)_{10}-\text{S}]$ are all trans, indicating that the molecules are in the fully extended conformation (Table 1). The dihedral angles formed between the aromatic

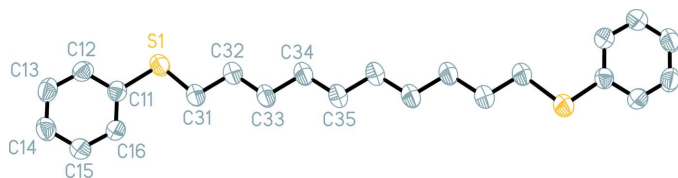


Figure 1
The molecular structure and atomic numbering of one independent molecule of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted. The unlabeled part of the molecule is related by the symmetry transformation $(-x, 1 - y, -z)$.

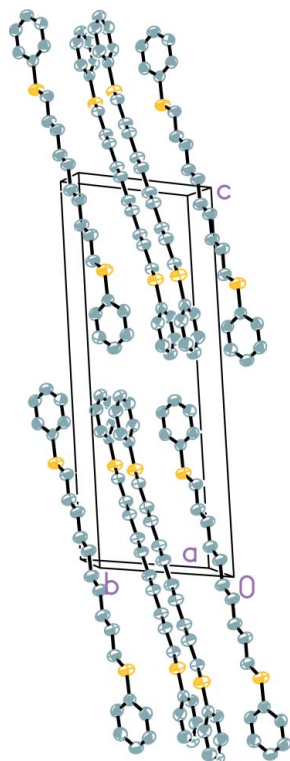


Figure 2
A crystal packing diagram of (I). H atoms have been omitted.

rings and the $[\text{S}-(\text{CH}_2)_{10}-\text{S}]$ sequences are $21.8(2)$ and $23.1(2)^\circ$ for molecules 1 and 2, respectively. The phenyl groups of neighboring molecules form a dihedral angle of $62.8(1)^\circ$. The bond distances and angles in (I) are in the normal ranges (Table 1) (*International Tables for Crystallography*, 1995, Vol. C).

In the crystal structure of (I) (Fig. 2), there are no significant $\pi-\pi$ stacking interactions but, in addition to normal van der Waals interactions, there are some weak $\text{C}-\text{H}\cdots\pi(\text{arene})$ interactions (Table 2).

Experimental

1,10-Bis(phenylthio)decane, L^{10} , (I), was synthesized according to the published procedure of Hartley *et al.* (1979). Compound (I) was obtained as a crystalline powder from which a platelet crystal suitable for X-ray analysis was isolated (yield 91%). Analysis, found: C 73.47, H 8.59%; calculated for $\text{C}_{22}\text{H}_{30}\text{S}_2$: C 73.68, H 8.43%. ^1H NMR (acetone- d_6 , δ , p.p.m.): 1.28 [t, 4H, $-\text{S}-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-(\text{CH}_2)_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{S}-$], 1.30 [qt, 4H, $-\text{S}-\text{CH}_2-$

$\text{CH}_2-\text{CH}_2-(\text{CH}_2)-\text{CH}_2-\text{CH}_2-(\text{CH}_2)-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{S}-$], 1.45 [qt, 4H, $-\text{S}-\text{CH}_2-\text{CH}_2-(\text{CH}_2)-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-(\text{CH}_2)-\text{CH}_2-\text{CH}_2-\text{S}-$], 1.64 [qt, 4H, $-\text{S}-\text{CH}_2-(\text{CH}_2)-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-(\text{CH}_2)-\text{CH}_2-\text{S}-$], 2.97 [t, 4H, $-\text{S}-(\text{CH}_2)-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-$], 7.16–7.35 (m, 10H, C_6H_5-).

Crystal data

$\text{C}_{22}\text{H}_{30}\text{S}_2$
 $M_r = 358.58$
 Triclinic, $P\bar{1}$
 $a = 5.570(2) \text{ \AA}$
 $b = 7.943(3) \text{ \AA}$
 $c = 22.953(7) \text{ \AA}$
 $\alpha = 82.82(3)^\circ$
 $\beta = 85.05(3)^\circ$
 $\gamma = 89.55(3)^\circ$
 $V = 1003.8(6) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.186 \text{ Mg m}^{-3}$
 Cu $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 20.0\text{--}25.0^\circ$
 $\mu = 2.38 \text{ mm}^{-1}$
 $T = 298(2) \text{ K}$
 Platelet, colorless
 $0.25 \times 0.11 \times 0.05 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: Gaussian, from crystal shape (ABSORP in NRCVAX; Gabe *et al.* 1989)
 $T_{\text{min}} = 0.528$, $T_{\text{max}} = 0.868$
 24003 measured reflections
 3799 independent reflections

2520 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$
 $\theta_{\text{max}} = 69.9^\circ$
 $h = -6 \rightarrow 6$
 $k = -9 \rightarrow 9$
 $l = -28 \rightarrow 28$
 4 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.147$
 $S = 0.91$
 3799 reflections
 217 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0908P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.47 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

S1–C11	1.764 (2)	S2–C41	1.790 (3)
S1–C31	1.796 (3)	C35–C35 ⁱ	1.513 (4)
S2–C21	1.759 (2)	C45–C45 ⁱⁱ	1.511 (4)
C11–S1–C31	104.59 (11)	C21–S2–C41	105.20 (12)
S1–C31–C32–C33	179.33 (19)	S2–C41–C42–C43	178.6 (2)
C31–C32–C33–C34	179.8 (2)	C41–C42–C43–C44	178.7 (2)
C32–C33–C34–C35	−179.9 (2)	C42–C43–C44–C45	178.9 (2)
C33–C34–C35–C35 ⁱ	179.8 (3)	C43–C44–C45–C45 ⁱⁱ	179.4 (3)

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

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$\text{Cg}2$ is the centroid of the C21–C26 ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
C12–H12 $\cdots\text{Cg}2^{\text{iii}}$	0.93	2.88	3.621 (3)	137
C15–H15 $\cdots\text{Cg}2^{\text{iv}}$	0.93	2.91	3.638 (3)	136

Symmetry codes: (iii) $1 - x, 1 - y, 1 - z$; (iv) $-x, 2 - y, 1 - z$.

H atoms were positioned geometrically ($\text{C}-\text{H}$ distances of $0.93\text{--}0.97 \text{ \AA}$) and were included in the refinement in the riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. 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).

This work was supported by the Natural Sciences and Engineering Research Council of Canada (FB). MOA thanks the Programme Canadien de Bourse de la Francophonie and the Organisation Internationale de la Francophonie for a graduate scholarship.

References

- Black, J. R., Champness, N. R., Levason, W. & Reid, G. (1995). *J. Chem. Soc. Dalton Trans.* pp. 3439–3445.
- Blake, A. J., Champness, N. R., Hubberstey, P., Li, W. S., Withersby, M. A. & Schroder, M. (1999). *Coord. Chem. Rev.* **183**, 117–138.
- Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bu, X. H., Chen, W., Hou, W. F., Du, M., Zhang, R. H. & Brisse, F. (2002). *Inorg. Chem.* **41**, 3477–3482.
- Carlucci, L., Ciani, G., Proserpio, D. M. & Rizzato, S. (2002). *CrystEngComm*, **4**, 413–425.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
- Hartley, F. R., Murray, S. G., Levason, W., Soutter, H. E. & McAuliffe, C. A. (1979). *Inorg. Chim. Acta*, **35**, 265–277.
- Maris, T. (2004). *UdMX*. Université de Montréal, Canada.
- Noro, S. I., Kitaura, R., Kondo, M., Kitagawa, S., Ishii, T., Matsuzaka, H. & Yamashita, M. (2002). *J. Am. Chem. Soc.* **124**, 2568–2583.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–11.
- Withersby, M. A., Blake, A. J., Champness, N. R., Cooke, P. A., Hubberstey, P., Li, W. S. & Schroder, M. (1999). *Inorg. Chem.* **38**, 2259–2266.
- Withersby, M. A., Blake, A. J., Champness, N. R., Hubberstey, P., Li, W. S. & Schroder, M. (1997). *Angew. Chem. Int. Ed.* **36**, 2327–2329.
- Yaghi, O. M., Li, G. & Li, H. (1995). *Nature (London)*, **378**, 703–706.