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1,3-Bis[(naphthalen-2-ylsulfanyl)methyl]-benzene

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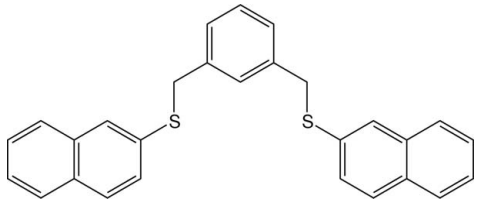
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.124; data-to-parameter ratio = 14.3.

Molecules of the title compound, $C_{28}H_{22}S_2$, are located on a crystallographic mirror plane with one half-molecule in the asymmetric unit. The dihedral angle between the phenyl ring and the naphthyl unit is 83.14 (7)°. In the crystal, molecules are interconnected by C—H···S and C—H···π interactions.

Related literature

For information on pincer compounds, see: Albrecht & Morales-Morales (2009); Arroyo *et al.* (2003); Morales-Morales (2004, 2008, 2009); Morales-Morales & Jensen (2007).



Experimental

Crystal data

$C_{28}H_{22}S_2$
M_r = 422.58
Orthorhombic, *Pnma*
a = 8.651 (2) Å
b = 41.235 (10) Å
c = 6.0517 (14) Å
V = 2158.9 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 298 K
0.48 × 0.42 × 0.07 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: analytical (*SADABS*; Bruker; 2007)
T_{min} = 0.893, T_{max} = 0.979
7907 measured reflections
1994 independent reflections
1345 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.124
S = 1.02
1994 reflections
139 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 and *Cg*2 are the centroids of the C1–C4/C2′/C3′ and C6–C9/C14/C15 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5B···S1^{i}	0.97	2.86	3.806 (4)	164
C1—H1···Cg1^{i}	0.93	2.94	3.867 (4)	173
C13—H13···Cg2^{ii}	0.93	2.76	3.503 (3)	138

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5855).

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supplementary materials

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1,3-Bis[(naphthalen-2-ylsulfanyl)methyl]benzene

Esteban Padilla-Mata, Juan M. German-Acacio, Marco A. García-Eleno, Reyna Reyes-Martínez and David Morales-Morales

Comment

Pincer compounds represent a group of species with very particular and interesting properties among which their high thermal stability and unusual reactivities that confer to the metal complexes they form stand out. It is due, to the characteristics of robustness and thermal stability that pincer compounds have attracted the continuos attention of the chemistry community for multiple applications (Morales-Morales *et al.*, 2004, Morales-Morales *et al.* 2007, Albrecht *et al.*, 2009, Morales-Morales, 2008, Morales-Morales, 2009). In the beginning, the very simple backbone exhibited by these compounds did not anticipate the wide variety of possible functionalization in the main frame of the complex.

Among these species, those including sulfur as donor atom have been scarcely studied (Arroyo *et al.*, 2003), mostly due to the well known tendency of sulfur to kill the activity of homogeneous catalysts. Thus, following our continuous interest in the synthesis of pincer type ligands we report the crystal structure of the potentially pincer sulfur based ligand 1,3-bis((naphthalen-2-ylthio)methyl)benzene.

In the asymmetric unit only half of the molecule of the compound 1,3-bis(naphthalen-2-ylthio)methyl)benzene is found. The other half is generated by a mirror plane. The molecular structure of the title compound is shown in Figure 1. The phenyl and the naphthyl enclose a dihedral angle of 83.14 (7)°. The two naphthyl planes have a dihedral angle of 45.64 (4)°. The sulfur atoms form weak hydrogen bonds (C5—H5···S1). Two C—H···π interactions [C1—H1···*Cg*1 and C13—H13···*Cg*2] further connect the molecules into ribbons running along the *a*-axis

Experimental

To a solution of 2-naphthalenethiol (0.320 g, 2.0 mmol), 0.057 g (2.5 mmol) of NaH in toluene (100 ml) were added. The reaction mixture was stirred at room temperature for 3 h. After this time, 0.264 g (1 mmol) of 1,3-bis(bromomethyl)-benzene were added to yield a colourless solution that was further stirred for 5 h. Then, the solvent was evaporated under vacumm affording 1,3-bis[(naphthalen-2-ylsulfanyl)methyl]benzene (0.24 g) as a microcrystalline white powder (93% based on 1,3-Bis(bromomethyl)benzene). mp: 120–122 °C, MS—EI (m/*z*): 422 (100%) $[M]^+$, ^{1}H-NMR (300 MHz, $CDCl_3$) δ (p.p.m.): 4.05 (s, 4H), 7.06–7.12 (d, 3H), 7.22 (s, 1H), 7.24 (d, 1H), 7.27 (d, 1H), 7.29–7.39 (m, 4H), 7.54–7.70 (m, 2H). 13 C-NMR (757 MHz, CDCl3) δ (p.p.m.): 38.82, 125.79, 126.50, 127.22, 127.72, 127.81, 127.85, 128.36, 128.75, 129.45, 133.72, 133.76, 137.73.

Refinement

H atoms were included in calculated positions (C—H = 0.93 Å for aromatic H, C—H = 0.97 Å for methylene H), and refined using a riding model, with U_{iso}(H) = 1.2U_{eq} of the carrier atom.

Computing details

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

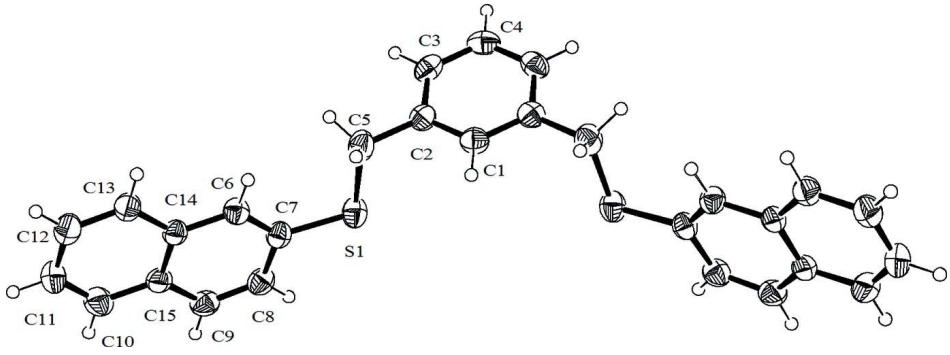

Figure 1

The molecular structure of the title compound with displacement ellipsoids at the 40% probability.

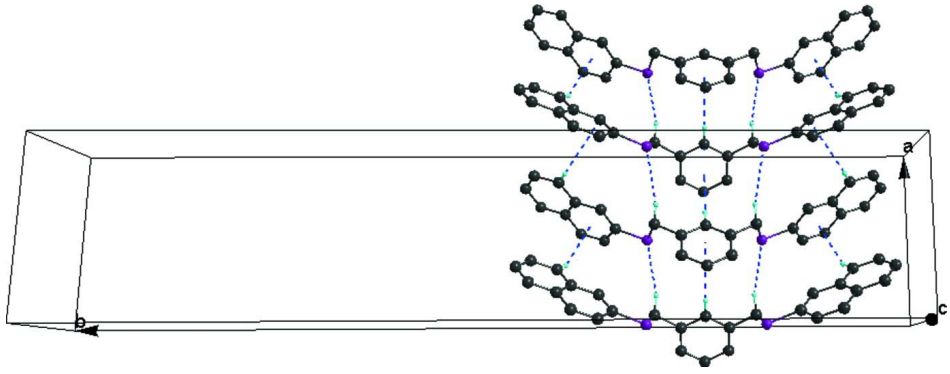

Figure 2

The title compound is linked by C—H···S and C—H···π intermolecular interactions along the *a* axes, the hydrogen atoms for the interactions are drawn.

1,3-Bis[(naphthalen-2-ylsulfanyl)methyl]benzene

Crystal data

$C_{28}H_{22}S_2$
$M_r = 422.58$
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
$a = 8.651\ (2)$ Å
$b = 41.235\ (10)$ Å
$c = 6.0517\ (14)$ Å
$V = 2158.9\ (9)$ Å^3
$Z = 4$
$F(000) = 888$
$D_x = 1.300$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2418 reflections
$\theta = 3.0–25.1°$
$\mu = 0.26$ mm^{-1}
$T = 298$ K
Plates, colorless
0.48 × 0.42 × 0.07 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.661 pixels mm^{-1}
ω–scans
Absorption correction: analytical (*SADABS*; Bruker; 2007)
T_{min} = 0.893, T_{max} = 0.979
7907 measured reflections
1994 independent reflections
1345 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 25.4°, θ_{min} = 3.0°
h = −10→9
k = −43→48
l = −7→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.124
S = 1.02
1994 reflections
139 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.49764 (9)	0.180146 (17)	0.07221 (12)	0.0516 (3)
C1	0.5547 (5)	0.2500	0.3529 (6)	0.0456 (10)
H1	0.6393	0.2500	0.2581	0.055*
C2	0.4932 (3)	0.22086 (6)	0.4217 (4)	0.0432 (7)
C3	0.3680 (4)	0.22111 (7)	0.5638 (4)	0.0489 (8)
H3	0.3254	0.2017	0.6123	0.059*
C4	0.3065 (5)	0.2500	0.6332 (6)	0.0533 (11)
H4	0.2221	0.2500	0.7285	0.064*
C5	0.5635 (4)	0.18939 (6)	0.3482 (5)	0.0561 (8)
H5A	0.5335	0.1722	0.4484	0.067*
H5B	0.6754	0.1911	0.3498	0.067*
C6	0.6690 (3)	0.12415 (6)	0.1558 (4)	0.0419 (7)
H6	0.7048	0.1337	0.2853	0.050*
C7	0.5702 (3)	0.14102 (6)	0.0228 (4)	0.0417 (7)
C8	0.5192 (3)	0.12636 (7)	−0.1764 (4)	0.0476 (7)
H8	0.4513	0.1376	−0.2677	0.057*

C9	0.5670 (4)	0.09654 (7)	−0.2359 (4)	0.0491 (8)
H9	0.5331	0.0877	−0.3688	0.059*
C10	0.7184 (4)	0.04703 (7)	−0.1541 (5)	0.0560 (8)
H10	0.6865	0.0377	−0.2863	0.067*
C11	0.8133 (4)	0.03010 (7)	−0.0168 (5)	0.0603 (9)
H11	0.8453	0.0093	−0.0553	0.072*
C12	0.8624 (4)	0.04379 (7)	0.1811 (5)	0.0558 (8)
H12	0.9270	0.0321	0.2744	0.067*
C13	0.8166 (3)	0.07420 (6)	0.2392 (5)	0.0474 (7)
H13	0.8505	0.0830	0.3720	0.057*
C14	0.7181 (3)	0.09256 (6)	0.1010 (4)	0.0392 (6)
C15	0.6678 (3)	0.07843 (6)	−0.0998 (4)	0.0426 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0523 (5)	0.0423 (4)	0.0602 (5)	0.0052 (4)	−0.0078 (4)	0.0030 (4)
C1	0.039 (2)	0.052 (3)	0.045 (2)	0.000	0.0039 (18)	0.000
C2	0.0429 (16)	0.0472 (16)	0.0393 (14)	0.0059 (15)	−0.0087 (14)	0.0035 (12)
C3	0.0513 (19)	0.0522 (18)	0.0431 (16)	−0.0068 (16)	−0.0077 (14)	0.0116 (14)
C4	0.046 (3)	0.074 (3)	0.040 (2)	0.000	0.0090 (19)	0.000
C5	0.066 (2)	0.0448 (16)	0.0580 (17)	0.0110 (16)	−0.0093 (16)	0.0050 (15)
C6	0.0420 (17)	0.0402 (15)	0.0435 (14)	−0.0036 (13)	−0.0045 (13)	−0.0030 (13)
C7	0.0382 (16)	0.0395 (15)	0.0473 (16)	−0.0037 (13)	0.0010 (13)	0.0034 (13)
C8	0.0489 (19)	0.0495 (17)	0.0445 (16)	−0.0017 (15)	−0.0083 (14)	0.0061 (14)
C9	0.058 (2)	0.0524 (18)	0.0372 (15)	−0.0095 (15)	−0.0059 (14)	−0.0013 (14)
C10	0.066 (2)	0.0489 (18)	0.0533 (18)	−0.0071 (16)	0.0021 (16)	−0.0089 (16)
C11	0.075 (2)	0.0375 (16)	0.068 (2)	0.0045 (16)	0.0041 (18)	−0.0011 (16)
C12	0.063 (2)	0.0460 (17)	0.0586 (19)	0.0035 (16)	−0.0019 (17)	0.0070 (15)
C13	0.0508 (18)	0.0431 (16)	0.0483 (16)	0.0008 (14)	−0.0011 (14)	0.0020 (14)
C14	0.0413 (16)	0.0360 (14)	0.0404 (15)	−0.0049 (13)	0.0017 (12)	0.0015 (12)
C15	0.0492 (18)	0.0385 (15)	0.0400 (15)	−0.0085 (14)	0.0037 (13)	−0.0004 (12)

Geometric parameters (Å, º)

S1—C7	1.757 (3)	C7—C8	1.419 (4)
S1—C5	1.805 (3)	C8—C9	1.346 (4)
C1—C2	1.378 (3)	C8—H8	0.9300
C1—C2^{i}	1.378 (3)	C9—C15	1.414 (4)
C1—H1	0.9300	C9—H9	0.9300
C2—C3	1.384 (4)	C10—C11	1.361 (4)
C2—C5	1.501 (4)	C10—C15	1.406 (4)
C3—C4	1.371 (3)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.390 (4)
C4—C3^{i}	1.371 (3)	C11—H11	0.9300
C4—H4	0.9300	C12—C13	1.361 (4)
C5—H5A	0.9700	C12—H12	0.9300
C5—H5B	0.9700	C13—C14	1.414 (4)
C6—C7	1.365 (4)	C13—H13	0.9300
C6—C14	1.410 (3)	C14—C15	1.416 (3)

C6—H6	0.9300		
C7—S1—C5	103.79 (13)	C9—C8—C7	121.4 (3)
C2—C1—C2^{i}	121.4 (4)	C9—C8—H8	119.3
C2—C1—H1	119.3	C7—C8—H8	119.3
C2^{i}—C1—H1	119.3	C8—C9—C15	121.1 (3)
C1—C2—C3	118.9 (3)	C8—C9—H9	119.5
C1—C2—C5	120.5 (3)	C15—C9—H9	119.5
C3—C2—C5	120.5 (3)	C11—C10—C15	121.2 (3)
C4—C3—C2	120.1 (3)	C11—C10—H10	119.4
C4—C3—H3	120.0	C15—C10—H10	119.4
C2—C3—H3	120.0	C10—C11—C12	120.1 (3)
C3^{i}—C4—C3	120.7 (4)	C10—C11—H11	119.9
C3^{i}—C4—H4	119.6	C12—C11—H11	119.9
C3—C4—H4	119.6	C13—C12—C11	120.5 (3)
C2—C5—S1	109.20 (19)	C13—C12—H12	119.7
C2—C5—H5A	109.8	C11—C12—H12	119.7
S1—C5—H5A	109.8	C12—C13—C14	121.0 (3)
C2—C5—H5B	109.8	C12—C13—H13	119.5
S1—C5—H5B	109.8	C14—C13—H13	119.5
H5A—C5—H5B	108.3	C6—C14—C13	122.5 (2)
C7—C6—C14	121.4 (2)	C6—C14—C15	119.3 (2)
C7—C6—H6	119.3	C13—C14—C15	118.2 (2)
C14—C6—H6	119.3	C10—C15—C9	122.9 (3)
C6—C7—C8	118.6 (2)	C10—C15—C14	118.9 (3)
C6—C7—S1	126.3 (2)	C9—C15—C14	118.2 (2)
C8—C7—S1	115.1 (2)		

Symmetry code: (i) x, $-y+1/2$, z.

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C1–C4/C2′/C3′ and C6–C9/C14/C15 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*B*···S1^{ii}	0.97	2.86	3.806 (4)	164
C1—H1···*Cg*1^{ii}	0.93	2.94	3.867 (4)	173
C13—H13···*Cg*2^{iii}	0.93	2.76	3.503 (3)	138

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