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1,2-Bis(diphenylthioarsinoyl)ethane

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.043
wR factor = 0.097
Data-to-parameter ratio = 24.1

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

The structure of the title compound, $[As_2S_2(C_2H_4)(C_6H_5)_4]$, which has twofold symmetry, features an As═S bond distance of 2.0674 (13) Å.

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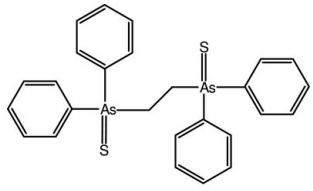
Online 15 May 2004

Comment

The title compound, (I), was prepared for use as a ligand in novel nickel complexes (Smith, 2002) as part of a wider study to prepare synthetic compounds with features similar to those of the active sites of the nickel-containing enzymes: hydrogenase, carbon monoxide dehydrogenase and acetyl-CoA synthase (Smith *et al.*, 2003; Evans & Pickett, 2003).



(I)

The structure of (I) (Fig. 1 and Table 1) lies about a twofold rotation axis which bisects the ethane bond. The As atom is tetrahedrally coordinated, with S—As—C angles lying in the range 111.51 (13)–114.04 (12)° and C—As—C angles lying in the range 105.60 (17)–106.92 (15)°. Bond lengths within the molecule are as expected, with As—C lengths lying in the range 1.924 (4)–1.946 (3) Å and As—S being 2.0674 (13) Å. The torsion angle for the ethane bridge [As—C—C^{i}—As^{i}; symmetry code (i) $1-x, y, \frac{1}{2}-z$] is 156.4 (2)°.

The molecules, separated by normal van der Waals contacts, are arranged so that circular channels run parallel to the crystallographic *a* axis (bounded by four molecules) and rectangular channels run parallel to the *c* axis (bounded by eight molecules), as highlighted in the two views of Fig. 2.

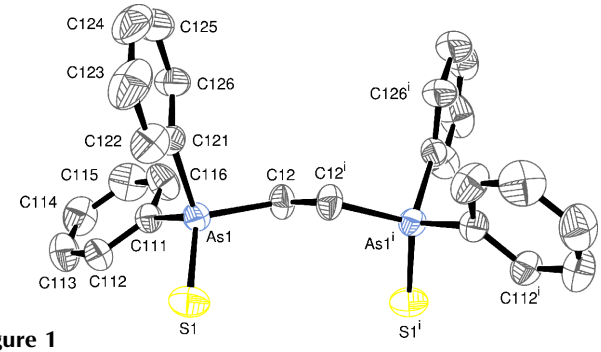


Figure 1
A view of (I). Displacement ellipsoids are drawn at the 50% probability level. Symmetry code (i) $1-x, y, \frac{1}{2}-z$

Experimental

Under an N_2 atmosphere, solid elemental S (0.153 g, 4.77 mmol) was added to a slurry of $[(Ph)_2AsCH_2CH_2As(Ph)_2]$ (1.16 g, 2.39 mmol; Aldrich) in ethanol (50 ml). The mixture was refluxed for 5 h, giving a light-coloured orange–brown solution. Upon cooling and standing overnight, large colourless needles formed that were collected by filtration and dried *in vacuo* (0.21 g, 16%). Expected for $C_{26}H_{24}As_2S_2$: C 56.7, H 4.4, S 11.6%; found: C 56.8, H 4.3, S 12.8%.

Crystal data

$[As_2S_2(C_2H_4)(C_6H_5)_4]$
M_r = 550.43
Monoclinic, $C2/c$
a = 15.976 (3) Å
b = 9.168 (4) Å
c = 17.635 (3) Å
β = 107.213 (13)°
V = 2467.3 (13) Å^3
Z = 4
D_x = 1.482 Mg m^{-3}
Mo–Kα radiation
Cell parameters from 25 reflections
θ = 10–11°
μ = 2.89 mm^{-1}
T = 293 (2) K
Needle, colourless
0.52 × 0.12 × 0.06 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω/θ scans
Absorption correction: ψ scan (*EMPABS*; Sheldrick *et al.*, 1977)
T_{min} = 0.713, T_{max} = 0.841
3937 measured reflections
3573 independent reflections
1815 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 30.0°
$h = -22 \rightarrow 21$
$k = -1 \rightarrow 12$
$l = -1 \rightarrow 24$
3 standard reflections
frequency: 167 min
intensity decay: 13.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.097
S = 1.01
3573 reflections
148 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

As1—S1	2.0674 (13)	As1—C121	1.924 (4)
As1—C12	1.946 (3)	C12—C12^{i}	1.511 (7)
As1—C111	1.928 (3)		
C12—As1—S1	111.51 (13)	C121—As1—C12	105.60 (17)
C111—As1—S1	114.04 (12)	C121—As1—C111	106.92 (15)
C121—As1—S1	112.53 (12)	C12^{i}—C12—As1	110.4 (3)
C111—As1—C12	105.64 (14)		
As1—C12—C12^{i}—As1^{i}	156.4 (2)		

Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.

All H atoms were positioned geometrically and allowed to ride on the parent atoms, with C—H distances of 0.93 Å for phenyl H atoms and 0.97 Å for methyl H atoms; isotropic displacement parameters were refined freely.

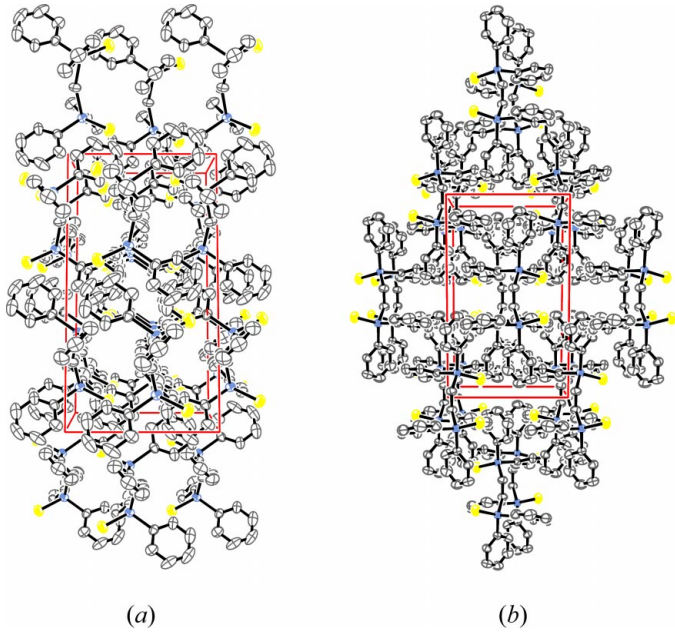


Figure 2
Packing diagrams for (I) showing (*a*) a view in the direction of the crystallographic [100] vector and (*b*) a view in the direction of the crystallographic [001] vector.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1992); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *CAD*-4 (Hursthouse, 1976) and *BAYES* (French & Wilson, 1978); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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References

Enraf–Nonius (1992). *CAD*-4 *EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Evans, D. J. & Pickett, C. J. (2003). *Chem. Soc. Rev.* **32**, 268–275.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
French, S. & Wilson, K. (1978). *Acta Cryst.* A**34**, 517–525.
Hursthouse, M. B. (1976). *CAD*-4. Queen Mary College, London, England.
Sheldrick, G. M., Orpen, A. G., Reichert, B. E. & Raithby, P. R. (1977). *EMPABS*. 4th European Crystallographic Meeting, Oxford, Abstracts, p. 147.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Smith, M. C. (2002). PhD thesis, University of East Anglia, England.
Smith, M. C., Barclay, J. E., Davies, S. C., Hughes, D. L. & Evans, D. J. (2003). *Dalton Trans.* pp. 4147–4151.

supporting information

Acta Cryst. (2004). E**60**, m771–m772 [https://doi.org/10.1107/S1600536804011158]

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Cell parameters from 25 reflections
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Needles, colourless
0.52 × 0.12 × 0.06 mm

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Graphite monochromator
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Absorption correction: ψ scan (EMPABS; Sheldrick et al., 1977)
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3573 independent reflections
1815 reflections with $I > 2\sigma(I)'$
$R_{int} = 0.021$
$\theta_{max} = 30.0°$, $\theta_{min} = 1.5°$
$h = -22\rightarrow21$
$k = -1\rightarrow12$
$l = -1\rightarrow24$
3 standard reflections every 167 min
intensity decay: 13.2%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.097$
$S = 1.01$
3573 reflections
148 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 constrained
$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
As1	0.37035 (2)	0.58646 (4)	0.14995 (2)	0.03721 (13)
S1	0.40216 (7)	0.77551 (12)	0.10081 (6)	0.0570 (3)
C111	0.2570 (2)	0.5915 (4)	0.16775 (19)	0.0390 (8)
C112	0.1942 (2)	0.6818 (5)	0.1219 (2)	0.0473 (10)
H112	0.2078	0.7442	0.0856	0.041 (10)*
C113	0.1104 (3)	0.6804 (6)	0.1295 (3)	0.0629 (13)
H113	0.0678	0.7419	0.0981	0.072 (14)*
C114	0.0901 (3)	0.5901 (6)	0.1823 (3)	0.0664 (13)
H114	0.0335	0.5887	0.1866	0.075 (14)*
C115	0.1530 (3)	0.5010 (6)	0.2293 (3)	0.0695 (14)
H115	0.1393	0.4402	0.2662	0.067 (14)*
C116	0.2362 (3)	0.5010 (5)	0.2223 (2)	0.0585 (12)
H116	0.2787	0.4400	0.2542	0.060 (13)*
C121	0.3719 (2)	0.4171 (4)	0.0860 (2)	0.0409 (9)
C122	0.3939 (3)	0.4326 (6)	0.0164 (2)	0.0576 (12)
H122	0.4073	0.5243	0.0006	0.064 (14)*
C123	0.3960 (3)	0.3125 (7)	−0.0295 (3)	0.0819 (17)
H123	0.4114	0.3228	−0.0761	0.087 (16)*
C124	0.3756 (3)	0.1787 (7)	−0.0071 (4)	0.0835 (18)
H124	0.3765	0.0979	−0.0387	0.108 (19)*
C125	0.3540 (3)	0.1624 (6)	0.0614 (4)	0.0762 (15)
H125	0.3407	0.0703	0.0767	0.060 (14)*
C126	0.3516 (3)	0.2815 (5)	0.1081 (3)	0.0552 (11)
H126	0.3361	0.2700	0.1547	0.080 (16)*
C12	0.4538 (2)	0.5458 (5)	0.2530 (2)	0.0460 (10)
H12A	0.4485	0.6192	0.2909	0.067 (14)*
H12B	0.4409	0.4516	0.2719	0.062 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1	0.0383 (2)	0.0374 (2)	0.0347 (2)	0.0010 (2)	0.00898 (14)	0.0018 (2)
S1	0.0718 (7)	0.0420 (6)	0.0622 (7)	−0.0013 (6)	0.0276 (6)	0.0087 (6)
C111	0.0395 (19)	0.044 (2)	0.0336 (18)	0.001 (2)	0.0108 (15)	−0.0002 (19)
C112	0.046 (2)	0.051 (3)	0.043 (2)	0.005 (2)	0.0104 (18)	0.007 (2)
C113	0.044 (3)	0.079 (4)	0.061 (3)	0.010 (3)	0.010 (2)	0.003 (3)
C114	0.045 (3)	0.087 (4)	0.073 (3)	0.003 (3)	0.027 (2)	−0.011 (3)
C115	0.075 (3)	0.081 (4)	0.067 (3)	−0.005 (3)	0.044 (3)	0.008 (3)
C116	0.054 (3)	0.070 (3)	0.057 (3)	0.012 (3)	0.024 (2)	0.020 (3)
C121	0.0349 (19)	0.040 (2)	0.044 (2)	0.000 (2)	0.0056 (16)	−0.007 (2)

C122	0.053 (3)	0.066 (3)	0.059 (3)	−0.006 (2)	0.025 (2)	−0.008 (3)
C123	0.070 (3)	0.109 (5)	0.071 (3)	−0.005 (3)	0.027 (3)	−0.045 (4)
C124	0.058 (3)	0.077 (4)	0.105 (5)	0.006 (3)	0.007 (3)	−0.051 (4)
C125	0.063 (3)	0.046 (3)	0.111 (5)	0.004 (3)	0.011 (3)	−0.008 (3)
C126	0.057 (3)	0.038 (3)	0.068 (3)	0.002 (2)	0.016 (2)	−0.003 (2)
C12	0.041 (2)	0.059 (3)	0.0325 (19)	−0.0008 (19)	0.0027 (17)	0.005 (2)

Geometric parameters (Å, º)

As1—S1	2.0674 (13)	C115—C116	1.371 (5)
As1—C12	1.946 (3)	C115—H115	0.9300
As1—C111	1.928 (3)	C116—H116	0.9300
As1—C121	1.924 (4)	C121—C126	1.372 (5)
C12—C12^{i}	1.511 (7)	C121—C122	1.380 (5)
C12—H12A	0.9700	C122—C123	1.373 (6)
C12—H12B	0.9700	C122—H122	0.9300
C111—C112	1.366 (5)	C123—C124	1.357 (7)
C111—C116	1.383 (5)	C123—H123	0.9300
C112—C113	1.384 (5)	C124—C125	1.359 (7)
C112—H112	0.9300	C124—H124	0.9300
C113—C114	1.356 (6)	C125—C126	1.376 (6)
C113—H113	0.9300	C125—H125	0.9300
C114—C115	1.368 (6)	C126—H126	0.9300
C114—H114	0.9300		

C12—As1—S1	111.51 (13)	C114—C115—C116	120.2 (4)
C111—As1—S1	114.04 (12)	C114—C115—H115	119.9
C121—As1—S1	112.53 (12)	C116—C115—H115	119.9
C111—As1—C12	105.64 (14)	C115—C116—C111	120.1 (4)
C121—As1—C12	105.60 (17)	C115—C116—H116	119.9
C121—As1—C111	106.92 (15)	C111—C116—H116	119.9
C12^{i}—C12—As1	110.4 (3)	C126—C121—C122	119.4 (4)
As1—C12—H12A	109.6	C126—C121—As1	121.3 (3)
As1—C12—H12B	109.6	C122—C121—As1	119.3 (3)
C12^{i}—C12—H12A	109.6	C123—C122—C121	119.9 (5)
C12^{i}—C12—H12B	109.6	C123—C122—H122	120.1
H12A—C12—H12B	108.1	C121—C122—H122	120.1
C112—C111—C116	119.4 (4)	C124—C123—C122	120.3 (5)
C112—C111—As1	118.6 (3)	C124—C123—H123	119.9
C116—C111—As1	121.9 (3)	C122—C123—H123	119.9
C111—C112—C113	119.9 (4)	C123—C124—C125	120.3 (5)
C111—C112—H112	120.1	C123—C124—H124	119.9
C113—C112—H112	120.1	C125—C124—H124	119.9
C114—C113—C112	120.5 (4)	C124—C125—C126	120.3 (5)
C114—C113—H113	119.8	C124—C125—H125	119.8
C112—C113—H113	119.8	C126—C125—H125	119.8
C113—C114—C115	119.9 (4)	C121—C126—C125	119.9 (4)
C113—C114—H114	120.0	C121—C126—H126	120.1

C115—C114—H114	120.0	C125—C126—H126	120.1
C121—As1—C111—C112	−99.4 (3)	S1—As1—C121—C126	−179.2 (3)
C12—As1—C111—C112	148.4 (3)	C111—As1—C121—C122	126.7 (3)
S1—As1—C111—C112	25.6 (3)	C12—As1—C121—C122	−121.1 (3)
C121—As1—C111—C116	77.0 (3)	S1—As1—C121—C122	0.7 (3)
C12—As1—C111—C116	−35.2 (4)	C126—C121—C122—C123	−0.6 (6)
S1—As1—C111—C116	−158.0 (3)	As1—C121—C122—C123	179.5 (3)
C116—C111—C112—C113	−1.0 (6)	C121—C122—C123—C124	0.7 (7)
As1—C111—C112—C113	175.5 (3)	C122—C123—C124—C125	−0.7 (8)
C111—C112—C113—C114	0.1 (7)	C123—C124—C125—C126	0.7 (8)
C112—C113—C114—C115	0.9 (7)	C122—C121—C126—C125	0.6 (6)
C113—C114—C115—C116	−1.1 (7)	As1—C121—C126—C125	−179.5 (3)
C114—C115—C116—C111	0.2 (7)	C124—C125—C126—C121	−0.6 (7)
C112—C111—C116—C115	0.8 (6)	C121—As1—C12—C12^{i}	69.40 (15)
As1—C111—C116—C115	−175.6 (3)	C111—As1—C12—C12^{i}	−177.52 (13)
C111—As1—C121—C126	−53.2 (3)	S1—As1—C12—C12^{i}	−53.11 (13)
C12—As1—C121—C126	58.9 (3)	As1—C12—C12^{i}—As1^{i}	156.4 (2)

Symmetry code: (i) −*x*+1, *y*, −*z*+1/2.