Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,2-Bis(diphenylthioarsinoyl)ethane

Sian C. Davies,* Matt C. Smith and David J. Evans

Department of Biological Chemistry, John Innes Centre, Norwich Research Park, Colney Lane, Norwich NR4 7UH, England

Correspondence e-mail: sianc.davies@bbsrc.ac.uk

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

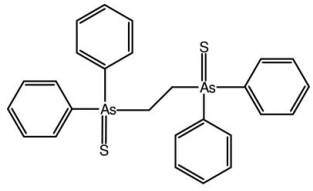
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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.

Received 2 April 2004
Accepted 7 May 2004
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.

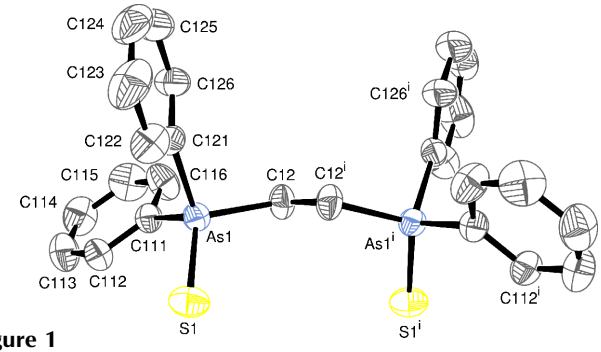


Figure 1
A view of (I). Displacement ellipsoids are drawn at the 50% probability level. Symmetry code (i) $1-x, y, \frac{1}{2}-z$

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.

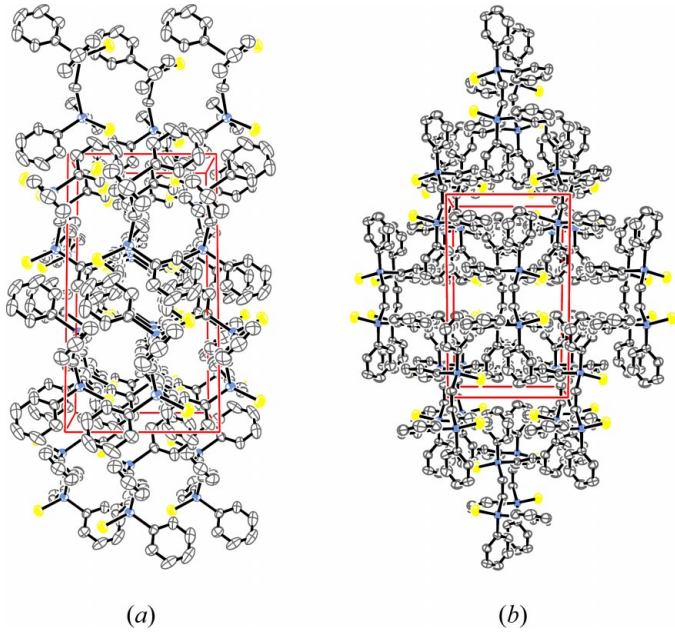


Figure 2
Packing diagrams for (I) showing (*a*) a view in the direction of the crystallographic [100] vector and (*b*) a view in the direction of the crystallographic [001] vector.

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.

The Biotechnology and Biological Sciences Research Council is thanked for funding.

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.