Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($\AA^2$)

$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Sn1	0.11179 (3)	0.18480 (2)	0.17387 (1)	0.03449 (6)
C21	−0.1774 (4)	0.3432 (2)	0.19106 (11)	0.0383 (5)
C22	−0.3379 (5)	0.3980 (3)	0.14980 (12)	0.0459 (6)
C23	−0.5257 (5)	0.4968 (3)	0.16355 (14)	0.0556 (7)
C24	−0.5587 (5)	0.5401 (3)	0.21870 (15)	0.0596 (8)
C25	−0.4032 (5)	0.4851 (4)	0.26064 (14)	0.0620 (8)
C26	−0.2146 (5)	0.3888 (3)	0.24675 (12)	0.0509 (7)
C31	0.2718 (4)	0.2272 (3)	0.09503 (10)	0.0391 (5)
C32	0.4591 (5)	0.1327 (3)	0.07781 (12)	0.0513 (7)
C33	0.5683 (6)	0.1580 (4)	0.02733 (14)	0.0664 (9)
C34	0.4920 (7)	0.2776 (5)	−0.00640 (14)	0.0746 (10)
C35	0.3087 (6)	0.3729 (4)	0.00984 (14)	0.0726 (10)
C36	0.1963 (5)	0.3493 (3)	0.06051 (12)	0.0517 (7)
C41	0.0116 (4)	−0.0096 (3)	0.16355 (11)	0.0406 (5)
C42	0.0471 (5)	−0.0770 (3)	0.11286 (14)	0.0596 (8)
C43	−0.0288 (6)	−0.1987 (4)	0.1049 (2)	0.0833 (12)
C44	−0.1408 (7)	−0.2537 (4)	0.1475 (2)	0.0861 (12)
C45	−0.1787 (7)	−0.1891 (4)	0.1979 (2)	0.0849 (12)
C46	−0.1044 (5)	−0.0673 (3)	0.20671 (13)	0.0586 (8)
C20	0.3286 (5)	0.1726 (3)	0.24701 (11)	0.0454 (6)
O1	0.4356 (3)	−0.3130 (2)	0.38508 (7)	0.0405 (4)
C2	0.2137 (4)	−0.3198 (2)	0.37418 (10)	0.0356 (5)
O3	0.0660 (3)	−0.2040 (2)	0.39769 (7)	0.0361 (3)
C4	0.0938 (4)	−0.0749 (2)	0.36913 (10)	0.0369 (5)
C5	0.3302 (4)	−0.0558 (2)	0.37832 (9)	0.0330 (5)
C6	0.4897 (4)	−0.1881 (3)	0.35936 (10)	0.0387 (5)
C7	0.1650 (4)	−0.4526 (3)	0.40146 (10)	0.0386 (5)
C8	0.2917 (5)	−0.5265 (3)	0.44598 (12)	0.0535 (7)
C9	0.2313 (6)	−0.6428 (3)	0.47304 (14)	0.0652 (9)
C10	0.0479 (6)	−0.6861 (3)	0.45558 (14)	0.0617 (8)
C11	−0.0764 (6)	−0.6155 (3)	0.4099 (2)	0.0625 (8)
C12	−0.0193 (5)	−0.4985 (3)	0.38311 (13)	0.0509 (6)
N13	0.3540 (3)	−0.0415 (2)	0.44025 (8)	0.0357 (4)
C14	0.5135 (4)	0.0035 (2)	0.46668 (9)	0.0342 (5)
O15	0.6656 (3)	0.0479 (2)	0.44435 (7)	0.0521 (5)
C16	0.3697 (4)	0.0744 (3)	0.34301 (9)	0.0388 (5)
O17	0.2752 (3)	0.0726 (2)	0.28769 (7)	0.0501 (5)

Table 2. *Selected geometric parameters* (Å, °)

Sn1—C20	2.154 (2)	C4—C5	1.529 (3)
Sn1—C21	2.147 (3)	C5—N13	1.464 (3)
Sn1—C31	2.138 (2)	C5—C6	1.529 (3)
Sn1—C41	2.141 (2)	C5—C16	1.537 (3)
O1—C2	1.412 (3)	N13—C14	1.328 (3)
O1—C6	1.430 (3)	C14—O15	1.217 (3)
C2—O3	1.422 (3)	C14—C14^i	1.556 (4)
C2—C7	1.498 (3)	C16—O17	1.418 (3)
O3—C4	1.434 (3)		
C20—Sn1—C21	106.2 (1)	C21—Sn1—C31	113.2 (1)
C20—Sn1—C31	111.7 (1)	C21—Sn1—C41	108.5 (1)
C20—Sn1—C41	111.9 (1)	C31—Sn1—C41	105.5 (1)

D—H···A	D—H	H···A	D···A	D—H···A
N13—H13···O3	0.86	2.51	2.837 (3)	103
N13—H13···O15^i	0.86	2.26	2.681 (2)	110

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

Compound (II) crystallized in the triclinic system and space group $P\bar{1}$ was assumed and confirmed by the analysis. Examination of the structure with *PLATON* (Spek, 1995) showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET*4 and *CELDIM* (Enraf–Nonius, 1992). Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*94. Program(s) used to refine structure: *NRCVAX*94 and *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *PLATON*. Software used to prepare material for publication: *NRCVAX*94, *SHELXL*93 and *WordPerfect*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: AB1356). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cox, P. J., Doidge-Harrison, S. M. S. V., Howie, R. A. & Wardell, J. L. (1991). *J. Crystallogr. Spectrosc. Res.* **21**, 735–739.

Enraf–Nonius (1992). *CAD-4/PC Software.* Version 1.1. Enraf–Nonius, Delft, The Netherlands.

Forrester, A. R., Howie, R. A., Ross, J.-N., Low, J. N. & Wardell J. L. (1991). *Main Group Met. Chem.* **14**, 293–304.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Ross, J.-N., Wardell, J. L., Low, J. N. & Ferguson, G. (1996). *Acta Cryst.* C**52**, 228–231.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Spek, A. L. (1995). *PLATON. Molecular Geometry Program.* Version of July 1995. University of Utrecht, The Netherlands.

Acta Cryst. (1996). C**52**, 1963–1966

catena-Poly[bis(*O*,*O*′-diethyldithiophosphato-*S*)zinc(II)-*μ*-4,4′-bipyridyl-*N*:*N*′]

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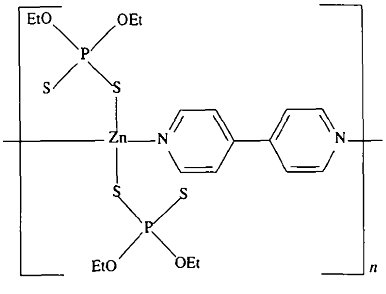
Abstract

The crystal structure of $[Zn\{(C_2H_5O)_2S_2P\}_2(C_{10}H_8N_2)]$ contains polymeric zigzag chains. The asymmetric unit comprises two independent zinc centres having similar distorted-tetrahedral coordination geometries. Each Zn

atom is attached through an S atom to two monodentate dithiophosphate ligands and 4,4′-bipyridyl residues link adjacent Zn atoms in the polymeric chain.

Comment

The asymmetric unit of the title complex, (I), contains two independent zinc centres with their associated ligands. Each Zn atom has a distorted tetrahedral geometry involving two S atoms of two dithiophosphate ligands and two N atoms from two 4,4′-bipyridyl molecules.



(I)

The Zn—S and Zn—N bond lengths in (I) [Zn(1)—S(1) 2.337 (2), Zn(1)—S(4) 2.280 (3), Zn(2)—S(5) 2.276 (3), Zn(2)—S(7) 2.315 (2), and Zn(1)—N(2) 2.061 (5), Zn(1)—N(4) 2.055 (4), Zn(2)—N(1) 2.096 (6) and Zn(2)—N(3) 2.050 (5) Å] at the two metal centres are quite similar, but corresponding bond angles at Zn(1) and Zn(2) show substantial differences (Table 2).

Each 4,4′-bipyridyl residue acts as a bridging group linking two Zn atoms to form a polymeric chain (Fig. 1). This contrasts with zinc diisopropyldithiophosphate (Lawton & Kokotailo, 1969), which is dimeric, and zinc diethyldithiophosphate (Ito, Igarashi & Hagihara, 1969), which is polymeric. Both contain two kinds of dithiophosphate ligand, one a chelating group coordinating to a single Zn atom through its two S atoms, the other bridging two Zn atoms. The average Zn—S bond length in the title complex [2.302 (2) Å] is shorter than corresponding values found in both $[Zn\{(C_2H_5O)_2PS_2\}_2]$ and $[Zn_2\{(i\text{-}C_3H_7O)_2PS_2\}_4]$, while the average Zn—N bond length [2.066 (5) Å] is in agreement with comparable distances found in other tetrahedral zinc compounds (Boudreau & Haendler, 1992). The average bond angle around the Zn atom is 109.0 (2)°, with a range of 97.4 (2)–120.1 (2)°. There are two kinds of S—P bond length; for S(2)—P(1), S(3)—P(2), S(6)—P(3) and S(8)—P(4), the average bond length is 1.924 (3) Å, and for S(1)—P(1), S(4)—P(2), S(5)—P(3) and S(7)—P(4), the average is 2.020 (3) Å. These values can be compared with typical double (1.94 Å) and single (2.14 Å) P—S bond lengths. Evidently, coordination to Zn significantly weakens the S—P bonds in the title compound. In both $[Zn\{(C_2H_5O)_2PS_2\}_2]$ and $[Zn_2\{(i\text{-}C_3H_7O)_2PS_2\}_4]$, all P—S bonds are almost the same length because both dithiophosphate S atoms are attached to zinc.

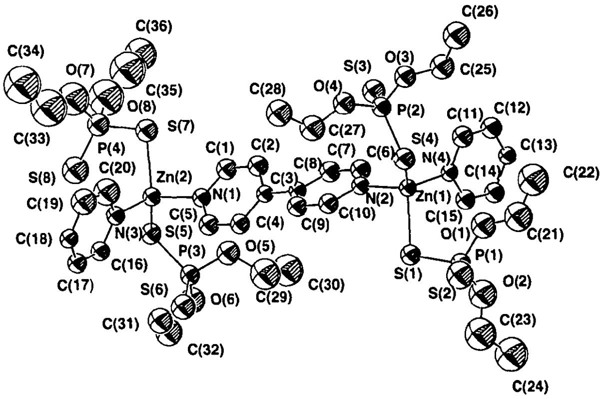


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

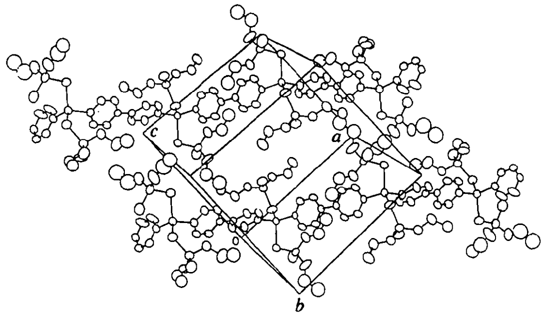


Fig. 2. Packing diagram of the unit cell of the title compound.

Experimental

The title compound was prepared by dissolving equimolar quantities of $[Zn\{(C_2H_5O)_2PS_2\}_2]$ (1 mmol) and 4,4′-bipyridyl (1 mmol) in ethanol (30 ml). A white product precipitated immediately from the reaction mixture. Recrystallization from dimethylformamide solution gave crystals of (I). The structure proposed is consistent with the elemental analysis; calculated: C 36.52, H 4.77, N 4.73%; found: C 37.07, H 5.02, N 4.49%.

Crystal data

$[Zn(C_4H_{10}O_2PS_2)_2(C_{10}H_8N_2)]$
$M_r = 592.03$
Triclinic
$P\bar{1}$
$a = 15.328\ (7)$ Å
$b = 15.621\ (6)$ Å
$c = 12.427\ (4)$ Å
$\alpha = 103.18\ (3)°$
$\beta = 93.98\ (4)°$
$\gamma = 110.49\ (3)°$
$V = 2677\ (2)$ Å^3
$Z = 4$
$D_x = 1.469$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 20 reflections
$\theta = 1.5$–$6.0°$
$\mu = 1.381$ mm^{-1}
$T = 296$ K
Prism
0.55 × 0.30 × 0.25 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (*TEXSAN*; Molecular Structure Corporation, 1985)
$T_{\min} = 0.7446$, $T_{\max} = 0.9800$
9819 measured reflections
9413 independent reflections
6259 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.08$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 18$
$k = -18 \rightarrow 18$
$l = -14 \rightarrow 14$
3 standard reflections monitored every 250 reflections
intensity decay: 1.8%

Refinement

Refinement on *F*
$R = 0.055$
$wR = 0.070$
$S = 1.68$
6259 reflections
520 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.78$ e Å^{-3}
$\Delta\rho_{\min} = -0.72$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Zn(1)	0.22356 (4)	0.15262 (5)	0.99888 (6)	0.0360 (2)
Zn(2)	−0.23926 (5)	0.37145 (5)	1.57276 (6)	0.0424 (3)
S(1)	0.1139 (1)	0.1071 (1)	0.8360 (1)	0.0519 (6)
S(2)	0.1634 (2)	−0.0456 (2)	0.6327 (2)	0.092 (1)
S(3)	0.3295 (2)	0.1723 (1)	1.2734 (2)	0.0675 (8)
S(4)	0.2635 (1)	0.0271 (1)	1.0108 (2)	0.0580 (7)
S(5)	−0.3005 (1)	0.4795 (1)	1.5451 (2)	0.0534 (7)
S(6)	−0.3709 (2)	0.3499 (2)	1.2749 (2)	0.0778 (9)
S(7)	−0.1355 (1)	0.4068 (2)	1.7356 (1)	0.0659 (8)
S(8)	−0.3290 (2)	0.4434 (2)	1.8080 (2)	0.089 (1)
P(1)	0.1878 (1)	0.0835 (1)	0.7112 (1)	0.0500 (7)
P(2)	0.3125 (1)	0.0502 (1)	1.1744 (2)	0.0488 (7)
P(3)	−0.3022 (1)	0.4717 (1)	1.3806 (2)	0.0499 (7)
P(4)	−0.2103 (1)	0.4376 (2)	1.8556 (2)	0.0652 (8)
O(1)	0.2936 (3)	0.1412 (4)	0.7695 (4)	0.078 (2)
O(2)	0.1763 (5)	0.1465 (5)	0.6326 (5)	0.100 (3)
O(3)	0.4052 (3)	0.0264 (3)	1.1802 (4)	0.058 (2)
O(4)	0.2502 (3)	−0.0380 (3)	1.2151 (4)	0.061 (2)
O(5)	−0.1944 (3)	0.5163 (4)	1.3706 (4)	0.071 (2)
O(6)	−0.3378 (4)	0.5499 (4)	1.3573 (5)	0.083 (3)
O(7)	−0.2133 (5)	0.3728 (7)	1.9311 (7)	0.135 (5)
O(8)	−0.1474 (5)	0.5375 (6)	1.9404 (6)	0.164 (4)
N(1)	−0.1532 (3)	0.3387 (3)	1.4592 (4)	0.042 (2)
N(2)	0.1443 (3)	0.1935 (3)	1.1125 (4)	0.039 (2)
N(3)	−0.3390 (3)	0.2375 (3)	1.5471 (4)	0.043 (2)
N(4)	0.3268 (3)	0.2810 (3)	1.0049 (4)	0.036 (2)
C(1)	−0.0595 (5)	0.3776 (5)	1.4871 (5)	0.054 (3)
C(2)	0.0009 (4)	0.3525 (5)	1.4216 (5)	0.052 (2)
C(3)	−0.0361 (4)	0.2824 (4)	1.3204 (4)	0.036 (2)
C(4)	−0.1326 (4)	0.2440 (4)	1.2901 (5)	0.048 (2)
C(5)	−0.1884 (4)	0.2730 (5)	1.3611 (5)	0.050 (3)
C(6)	0.1740 (4)	0.2796 (4)	1.1831 (6)	0.050 (2)
C(7)	0.1174 (4)	0.3095 (4)	1.2511 (6)	0.054 (2)
C(8)	0.0266 (4)	0.2514 (4)	1.2498 (5)	0.037 (2)
C(9)	−0.0033 (4)	0.1604 (4)	1.1779 (6)	0.050 (2)
C(10)	0.0565 (4)	0.1354 (4)	1.1126 (5)	0.051 (2)
C(11)	0.4120 (4)	0.3163 (5)	1.0675 (6)	0.057 (3)
C(12)	0.4806 (4)	0.4004 (5)	1.0674 (6)	0.056 (3)
C(13)	0.4625 (4)	0.4515 (4)	1.0007 (5)	0.038 (2)
C(14)	0.3761 (4)	0.4168 (4)	0.9349 (6)	0.050 (2)
C(15)	0.3110 (4)	0.3310 (4)	0.9391 (6)	0.049 (2)
C(16)	−0.4270 (4)	0.2069 (4)	1.4904 (5)	0.046 (2)
C(17)	−0.4908 (4)	0.1156 (4)	1.4711 (5)	0.047 (2)
C(18)	−0.4652 (4)	0.0525 (4)	1.5109 (5)	0.043 (2)
C(19)	−0.3762 (5)	0.0821 (5)	1.5698 (8)	0.085 (4)
C(20)	−0.3165 (5)	0.1741 (6)	1.5856 (8)	0.088 (4)
C(21)	0.3698 (8)	0.1309 (8)	0.7148 (9)	0.103 (3)
C(22)	0.4606 (8)	0.1917 (9)	0.788 (1)	0.121 (4)
C(23)†	0.104 (1)	0.115 (1)	0.542 (2)	0.126 (6)
C(24)	0.1268 (10)	0.179 (1)	0.467 (1)	0.142 (5)
C(25)	0.4900 (6)	0.0914 (6)	1.1568 (7)	0.075 (4)
C(26)	0.5682 (6)	0.0590 (7)	1.1789 (9)	0.089 (4)
C(27)	0.1509 (5)	−0.0561 (7)	1.2179 (8)	0.082 (4)
C(28)	0.1166 (6)	−0.1227 (7)	1.2858 (8)	0.088 (4)
C(29)	−0.1648 (8)	0.5272 (9)	1.2602 (9)	0.113 (6)
C(30)†	−0.116 (1)	0.632 (1)	1.270 (1)	0.122 (6)
C(31)†	−0.4290 (9)	0.5503 (9)	1.378 (1)	0.076 (3)
C(32)†	−0.431 (1)	0.647 (1)	1.380 (2)	0.088 (5)
C(33)	−0.273 (1)	0.292 (1)	1.925 (1)	0.172 (6)
C(34)	−0.282 (1)	0.239 (1)	2.001 (1)	0.175 (6)
C(35)	−0.062 (1)	0.573 (1)	1.970 (1)	0.167 (6)
C(36)	−0.0096 (9)	0.671 (1)	2.020 (1)	0.130 (4)
C(23′)†	0.170 (2)	0.121 (2)	0.512 (3)	0.090 (9)
C(30′)†	−0.119 (2)	0.475 (2)	1.233 (3)	0.093 (9)
C(31′)†	−0.338 (3)	0.631 (3)	1.418 (4)	0.13 (1)
C(32′)†	−0.424 (2)	0.634 (2)	1.454 (2)	0.099 (6)

† Disordered atoms (see below).

Table 2. *Selected geometric parameters (Å, °)*

Zn(1)—S(1)	2.337 (2)	S(1)—P(1)	2.024 (3)
Zn(1)—S(4)	2.280 (3)	S(2)—P(1)	1.919 (3)
Zn(1)—N(2)	2.061 (5)	S(3)—P(2)	1.937 (3)
Zn(1)—N(4)	2.055 (4)	S(4)—P(2)	2.023 (3)
Zn(2)—S(5)	2.276 (3)	S(5)—P(3)	2.017 (3)
Zn(2)—S(7)	2.315 (2)	S(6)—P(3)	1.929 (2)
Zn(2)—N(1)	2.096 (6)	S(7)—P(4)	2.017 (3)
Zn(2)—N(3)	2.050 (5)	S(8)—P(4)	1.913 (4)
S(1)—Zn(1)—S(4)	108.76 (8)	S(3)—P(2)—O(4)	114.0 (2)
S(1)—Zn(1)—N(2)	98.1 (1)	S(4)—P(2)—O(3)	107.4 (2)
S(1)—Zn(1)—N(4)	108.1 (2)	S(4)—P(2)—O(4)	108.1 (2)
S(4)—Zn(1)—N(2)	117.8 (2)	O(3)—P(2)—O(4)	95.1 (3)
S(4)—Zn(1)—N(4)	120.1 (2)	S(5)—P(3)—S(6)	117.7 (1)
N(2)—Zn(1)—N(4)	101.5 (2)	S(5)—P(3)—O(5)	103.8 (2)
S(5)—Zn(2)—S(7)	119.34 (8)	S(5)—P(3)—O(6)	106.9 (3)
S(5)—Zn(2)—N(1)	116.4 (2)	S(6)—P(3)—O(5)	114.3 (2)
S(5)—Zn(2)—N(3)	113.6 (2)	S(6)—P(3)—O(6)	111.5 (2)
S(7)—Zn(2)—N(1)	97.4 (2)	O(5)—P(3)—O(6)	101.1 (3)
S(7)—Zn(2)—N(3)	108.6 (2)	S(7)—P(4)—S(8)	117.3 (1)
N(1)—Zn(2)—N(3)	98.5 (2)	S(7)—P(4)—O(7)	106.8 (4)
Zn(1)—S(1)—P(1)	103.7 (1)	S(7)—P(4)—O(8)	108.5 (3)
Zn(1)—S(4)—P(2)	106.3 (1)	S(8)—P(4)—O(7)	115.4 (3)
Zn(2)—S(5)—P(3)	104.3 (1)	S(8)—P(4)—O(8)	107.5 (4)
Zn(2)—S(7)—P(4)	102.4 (1)	O(7)—P(4)—O(8)	99.9 (5)
S(1)—P(1)—S(2)	118.1 (1)	Zn(2)—N(1)—C(1)	120.5 (4)
S(1)—P(1)—O(1)	103.3 (2)	Zn(2)—N(1)—C(5)	122.4 (4)
S(1)—P(1)—O(2)	105.7 (3)	Zn(1)—N(2)—C(6)	122.8 (4)
S(2)—P(1)—O(1)	113.4 (3)	Zn(1)—N(2)—C(10)	120.5 (4)
S(2)—P(1)—O(2)	113.9 (2)	Zn(2)—N(3)—C(16)	124.7 (5)
O(1)—P(1)—O(2)	100.5 (3)	Zn(2)—N(3)—C(20)	119.2 (4)
S(3)—P(2)—S(4)	116.8 (1)	Zn(1)—N(4)—C(11)	124.5 (5)
S(3)—P(2)—O(3)	113.2 (2)	Zn(1)—N(4)—C(15)	119.0 (3)

The title structure was solved by Patterson methods and refined by full-matrix least squares. Atoms C(23), C(30), C(31) and C(32) are each disordered over two sites having relative occupancies 0.65/0.35 for C(32) and 0.70/0.30 for the other disordered atoms. These atoms, together with atoms C(21)–C(24) and C(30)–C(36), were assigned isotropic displacement parameters. For other non-H atoms, anisotropic displacement parameters were refined.

All calculations were performed on a MicroVAX II computer using the *TEXSAN* (Molecular Structure Corporation, 1985) package.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Boudreau, S. M. & Haendler, H. M. (1992). *Acta Cryst.* C**48**, 615–618.
Ito, T., Igarashi, T. & Hagihara, H. (1969). *Acta Cryst.* B**25**, 2303–2309.
Lawton, S. L. & Kokotailo, G. T. (1969). *Inorg. Chem.* **8**, 2410–2421.
Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Acta Cryst. (1996). C**52**, 1966–1968

[1,1′-Bis(diphenylphosphonato-*O*)-ferrocene]tetrachlorotin

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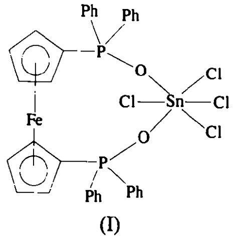
Abstract

The structure of the title compound, [$SnCl_4(C_{34}H_{28}FeO_2P_2)$], shows the 1,1′-bis(diphenylphosphino)ferrocene ligand connected to an octahedral Sn atom through the formation of P—O bonds. The molecule has near twofold rotational symmetry through the line joining the Fe and Sn atoms. The cyclopentadienyl (Cp) rings are perfectly eclipsed but the two C_{Cp}—P bonds are at an angle of 72° about the centroids of the Cp rings.

Comment

Complexes of the type (dppf)*M*Cl_2 [dppf = 1,1′-bis(diphenylphosphino)ferrocene; *M* = Pt, Pd, Ni], where dppf acts as a bidentate ligand, have been reported together with their X-ray structures (Clemente, Pilloni, Corain, Longato & Camellini, 1986; Casellato, Ajo, Valle, Corain, Longato & Graziani, 1988). Alternatively, (dppf)bis(chlorogold) is an example of a complex where dppf acts either as a monodentate ligand or as a bridge connecting the two Au atoms (Hill *et al.*, 1989). Recently, diphenylphosphinomethane (dppm) and (+)-(*R*,*R*)-1,2-bis(methylphenylphosphino)benzene (P*2) have been reported to give monodentate [$SnCl_4(dppm)_2$] and bidentate [$PhSnCl_3(P^*2)$], respectively, when reacted with phenyltin trichloride (Dakternieks, Zhu & Tiekink, 1994). We have studied the reaction of dppf with $SnCl_4$ and found that there is oxidation at the P centres resulting in the formation of the title compound, [$Fe(5-C_5H_4PPh_2O)_2SnCl_4$], (I).



(I)

A displacement ellipsoid plot of the title molecule together with the numbering scheme is shown in Fig. 1. The Sn atom has octahedral coordination with the O1 atom *cis* with respect to the O2 atom. Atoms Cl1 and Cl2 are *trans* to atoms O2 and O1, respectively. The Sn—Cl1 and Sn—Cl2 distances are longer than the other two Sn—Cl distances and this may be attributed to the *trans* effect of oxygen. These variations in Sn—Cl bond length and a Cl3—Sn—Cl4 bond angle of 168.38 (3)° result in the geometry around the Sn atom deviating slightly from ideal octahedral geometry. The geometry about both P atoms is tetrahedral; the average P—C_{Cp} and P—C_{phenyl} bond lengths of 1.779 (3)

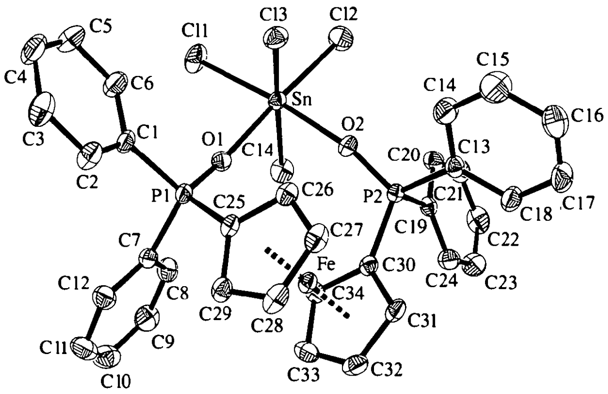


Fig. 1. A 30% probability displacement ellipsoid plot of the title molecule showing the numbering scheme.

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