

difference Fourier synthesis 3.6 and $-4.3 \text{ e } \text{Å}^{-3}$. An extinction correction was applied according to the equation $F_c' = F_c(1 - 2.5 \times 10^{-8} F_c^2 / \sin \theta)$. Atomic scattering factors and anomalous-dispersion correction were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Programs used: *SHELX76* (Sheldrick, 1976) for structure refinement and *STRUCTURE TIDY* (Gelato & Parthé, 1987) for structure standardization. Table 1 reports atomic coordinates and isotropic equivalent thermal parameters, while interatomic distances up to $d/\sum r = 1.12$ ($\sum r$ is the sum of the metallic radii for CN 12) are given in Table 2.* From the powder photographs (Fe $K\alpha$ radiation) of the same samples the following unit-cell dimensions were obtained: $a = 11.664$ (4), $b = 9.138$ (4), $c = 8.010$ (4) Å, in good agreement with those obtained by single-crystal diffraction.

Related literature. In Er_5Sb_3 two coordination polyhedra are found around the Sb atoms: a CN 9

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53081 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

truncated trigonal prism and a CN 8 dodecahedron (or bisdisphenoid) with only Er at the vertices as no Sb—Sb contacts occur. The $\beta\text{-Yb}_5\text{Sb}_3$ structure is adopted by several compounds formed by alkaline earths, rare earths or transition metals (e.g. Ti, V, Cr) with P, As, Sb and Bi (Villars & Calvert, 1985). Another phase, Y_5Bi_3 (Wang, Gabe, Calvert & Taylor, 1976), crystallizing in the same space group *Pnma*, has lattice constants very similar to those of $\beta\text{-Yb}_5\text{Sb}_3$ but with the a and c periods interchanged so that these two compounds cannot be considered as isotypic although their structures are very closely related. The similarity and the description in the same group is made possible only by the particular values of the coordinates in the two structures.

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Structure of [1,2-Bis(diphenylphosphino)ethane]di(phenylthio)palladium(II)

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Abstract. $[\text{Pd}(\text{C}_6\text{H}_5\text{S})_2\{(\text{C}_6\text{H}_5)_2\text{PCH}_2\text{CH}_2\text{P}(\text{C}_6\text{H}_5)_2\}]$, $M_r = 723.17$, monoclinic, $C2/c$, $a = 14.320$ (2), $b = 16.383$ (2), $c = 14.295$ (2) Å, $\beta = 93.41$ (2)°, $V = 3347.7$ (9) Å³, $Z = 4$, $D_x = 1.435 \text{ g cm}^{-3}$, Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 7.856 \text{ cm}^{-1}$, $F(000) = 1480$, $T = 296$ (1) K, $R = 0.0269$ for 3084 reflections with $I > 3\sigma(I)$. The molecule possesses a twofold axis passing through Pd and the midpoints of S—S and P—P with these five atoms being essentially coplanar.

Experimental. The title compound was prepared by reaction of PdCl_2 , NaSPh , 1,2-bis(diphenylphosphino)ethane (dppe), and elemental selenium in

DMF solution, and a crystal suitable for the X-ray analysis was grown in a mixed solution of DMF and acetone. Details of the sample preparation and the crystal growth are similar to those for the Co compounds (Wei, Liu, Huang, Huang & Kang, 1989). This synthetic method differs from that of Hayter & Humiec (1964).

Diffraction intensities were collected from a red prismatic crystal $0.20 \times 0.22 \times 0.30 \text{ mm}$, in the ω - 2θ scan mode on an Enraf-Nonius CAD-4 diffractometer using graphite-monochromated Mo $K\alpha$ radiation. 25 reflections ($10 < \theta < 15^\circ$) were used for measuring lattice parameters. 4437 unique reflections measured in the index range $0 < h < 19$, $0 < k < 22$, $-19 < l < 19$, $2\theta_{\text{max}} = 30^\circ$. The intensities of three standard reflections monitored periodically

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Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic mean-square amplitudes of vibration ($\text{\AA}^2 \times 10^2$) for $\text{Pd}(\text{SPh})_2(\text{dppe})$

	x	y	z	B*
Pd	10000	2499 (1)	-2500	277 (1)
S	9922 (1)	3416 (1)	-3764 (1)	338 (1)
P	9904 (1)	1490 (1)	-1430 (1)	326 (1)
C	10203 (2)	516 (2)	-1992 (2)	445 (5)
C(11)	10523 (2)	4329 (1)	-3457 (2)	326 (4)
C(12)	11367 (2)	4349 (2)	-2935 (2)	433 (6)
C(13)	11818 (2)	5090 (2)	-2759 (2)	536 (7)
C(14)	11449 (3)	5798 (2)	-3119 (2)	620 (7)
C(15)	10620 (3)	5783 (2)	-3636 (3)	634 (8)
C(16)	10148 (2)	5056 (2)	-3801 (2)	466 (6)
C(21)	8727 (2)	1368 (2)	-1058 (2)	351 (4)
C(22)	8068 (2)	1963 (2)	-1293 (2)	422 (5)
C(23)	7173 (2)	1911 (2)	-965 (2)	542 (7)
C(24)	6942 (2)	1282 (2)	-406 (2)	568 (6)
C(25)	7573 (2)	686 (2)	-180 (2)	632 (8)
C(26)	8475 (2)	716 (2)	-506 (2)	520 (6)
C(31)	10651 (2)	1547 (2)	-357 (2)	396 (5)
C(32)	10300 (2)	1746 (2)	489 (2)	597 (8)
C(33)	10905 (3)	1844 (3)	1273 (3)	810 (10)
C(34)	11817 (3)	1733 (3)	1240 (3)	840 (10)
C(35)	12190 (2)	1535 (2)	417 (4)	850 (10)
C(36)	11608 (2)	1449 (2)	-403 (3)	617 (8)

* Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{1}{3}[\alpha^2 B(1,1) + b^2 B(2,2) + c^2 B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table 2. Selected atomic distances (\AA) and bond angles ($^\circ$) for the complex $\text{Pd}(\text{SPh})_2(\text{dppe})$

Pd—S	2.3486 (7)	P—C	1.848 (3)
Pd—P	2.2612 (7)	P—C(21)	1.809 (2)
S—C	1.768 (2)	P—C(31)	1.819 (3)
S—Pd—S	100.44 (3)	Pd—S—C	110.47 (8)
S—Pd—P	170.50 (2)	Pd—P—C	108.2 (1)
S—Pd—P	87.08 (2)	Pd—P—C(21)	112.00 (9)
P—Pd—P	86.08 (3)	Pd—P—C(31)	118.51 (9)

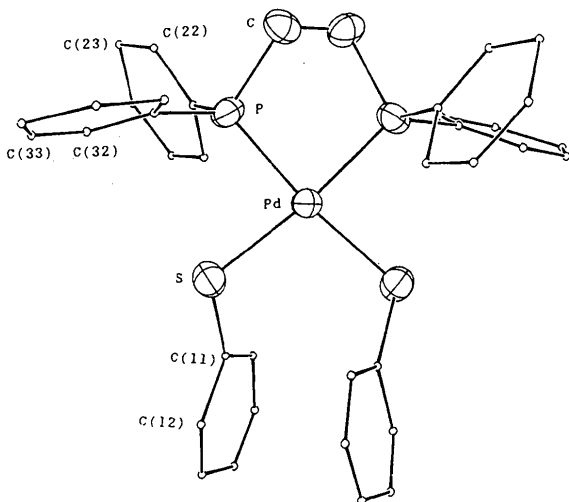


Fig. 1. Molecular structure and atomic labelling scheme for $\text{Pd}(\text{SPh})_2(\text{dppe})$.

varied slightly over the whole course of the data collection (between 0.991 and 1.045). The intensities of the reflections were corrected for fluctuation of the monitored reflections, for Lp factor and for empirical absorption (between 0.945 and 1.000), but no extinction correction was made. The structure was solved by direct methods. 3084 reflections with $I > 3\sigma(I)$ were used for structure solution and refinement. Calculations were performed on a VAX 11/785 computer with the SDP program package (Frenz, 1978). Full-matrix least-squares refinement with anisotropic thermal parameters for all non-H atoms and isotropic for all H atoms led to convergence with $R = 0.0269$ and $wR = 0.0345$. Function minimized was $\sum w(|F_o| - |F_c|)^2$, $w^{-1} = 1 + \{[F_o^2 + 2\sigma(F_o)|F_o|]^{1/2} - |F_c|\}^2 + (0.02F_o)^2$. The largest Δ/σ value in the final cycle was 0.50 while the extreme peaks in the difference electron density map were 0.35 and -0.33 e\AA^{-3} . Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The goodness of fit (S) was 1.043. Final positional parameters are given in Table 1, and selected atomic distances and bond angles are listed in Table 2.* The atomic labelling scheme and molecular structure are shown in Fig. 1.

Related literature. Crystal structures of related compounds have been studied: dppe (Pelizzi & Pelizzi, 1979), $\text{Pd}(\text{dppe})(\text{NSC})(\text{SCN})$ (Palenik, Mathew, Steffen & Beran, 1975), $\text{PdCl}_2(\text{dppe})\cdot\text{CH}_2\text{Cl}_2$ (Steffen & Palenik, 1976) and $\text{Co}_2(\text{dppe})(\text{SPh})_4$ (Wei, Liu, Huang, Huang & Kang, 1989). The title compound has been studied by IR and UV spectroscopy (Roundhill, 1980).

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