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Synthesis and structural characterization of chalcogenido-bridged trinuclear palladium(II) dialkyldithiophosphates, $[Pd_3(\mu_3\text{-}E)_2\{S_2P(OR)_2\}_2(PR'_3)_2]$ (E = S or Se, R = Et, Pr^n, Pr^i, Bu^s and R′ = Ph, Et)

Sanjay Narayan,[a] Vimal K. Jain[a]* and R. J. Butcher[b]

[a] Chemistry Division, Bhabha Atomic Research Centre, Trombay, Mumbai-400 085, India

[b] Department of Chemistry, Howard University, Washington, DC-20059, U.S.A.

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Abstract—Reaction of $[PdCl\{S_2P(OR)_2\}(PR'_3)]$ with sodium sulfide or selenide in acetone afforded neutral trinuclear palladium(II) complexes of the type $[Pd_3(\mu_3\text{-}E)_2\{S_2P(OR)_2\}_2(PR'_3)_2]$ (E = S or Se; R = Et, Pr^n, Pr^i, Bu^s, R′ = Et or Ph) in 24–42% yield. These complexes were characterized by elemental analysis, NMR (^1H, ^{31}P, ^{77}Se) and FAB mass spectral data. The molecular structure of $[Pd_3(\mu_3\text{-}S)_2\{S_2P(OEt)_2\}_2(PPh_3)_2]$ (**1**) was determined by X-ray diffraction. The molecule consists of three distorted square-planar palladium atoms sharing two μ_3-S ligands. One of the palladium atoms is linked with two triphenylphosphine ligands while each of the remaining two palladium atoms is coordinated with chelating dithiophosphate group. © 1998 Elsevier Science Ltd. All rights reserved

Keywords: palladium; chalcogen; dithiophosphate; ^{77}Se NMR; X-ray.

Interest in the chemistry of metal chalcogenide complexes continues to grow [1–4] due to their applications in metal catalyzed hydrodesulfurization [5,6] and in materials science [4,7]. Substitution reactions in palladium(II) dithiolate complexes are quite facile and give a variety of products [8]. The reactions of $[PdCl(S_2CNMe_2)(PEt_3)]$ with thiols in the presence of base have been reported recently to give monomeric palladium thiolate, $[Pd(SR)(S_2CNMe_2)(PEt_3)]$ [9]. We carried out a reaction of $[PdCl\{S_2P(OR)_2\}(PR'_3)]$ with Na_2S/Na_2Se with the hope of isolating binuclear chalcogenido-bridged complexes, similar to Cp*M-Te-MCp* (M = Ti, Zr and Hf) [10]. However, neutral chalcogenido-bridged trinuclear palladium complexes were isolated.

EXPERIMENTAL SECTION

All reactions were carried out in anhydrous solvents under N_2 atmosphere. $[PdCl\{S_2P(OR)_2\}(PR'_3)]$ (R = Et, Pr^n, Pr^i, Bu^s; and R′ = Ph, Et) [11] and Na_2S [12], Na_2Se [12] were prepared by the literature methods. Analysis were performed by the Analytical Chemistry Division of this research centre. The ^1H, $^{31}P\{^1H\}$ and $^{77}Se\{^1H\}$ NMR were recorded in 5 mm NMR tubes as $CDCl_3$ solution on a Varian VXR-300 instrument. Chemical shifts are referenced with internal chloroform peak (δ 7.26 ppm) for ^1H, external 85% H_3PO_4 for $^{31}P\{^1H\}$, Me_2Se for $^{77}Se\{^1H\}$. FAB Mass spectra were recorded on a JEOL SX 102/DA-6000 Mass spectrometer using Argon/Xenon (6 KV, 10 mA) as the FAB gas. m-Nitrobenzyl alcohol was used as the matrix.

Synthesis

$[Pd_3(\mu_3\text{-}S)_2\{S_2P(OEt)_2\}_2(PPh_3)_2]$ (**1**). To an acetone solution (25 ml) of $[PdCl\{S_2P(OEt)_2\}(PPh_3)]$ (150 mg, 0.255 mmol), solid Na_2S (15 mg, 0.192 mmol) was added and the reaction mixture was stirred at room temperature under N_2 for 48 h. The solvent was

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* Author to whom correspondence should be addressed.

removed *in vacuo*. The residue was extracted with diethylether and filtered. The filtrate was concentrated to 5 ml and hexane (5 ml) was added. This solution on cooling in a freezer for a week gave orange crystals, which were separated by decanting the solvent and recrystallized again from acetone-hexane (Yield: 31 mg, 29%), m.p. 171–173°C (dec.). Anal. Calcd for $C_{44}H_{50}O_4S_6P_4Pd_3$: C, 41.4; H, 4.0. Found: C, 41.0; H, 3.8. ^{1}H NMR in $CDCl_3$: 1.38, 1.47 (each t, 7.1 Hz, 6H each, $OCH_2\mathit{Me}$); 4.26, 4.34 (each dq, J_{PH} 9.6 Hz (d), J_{HH} 7.1 Hz (q), 4H each, $O\mathit{CH}_2$-}; 7.18–7.54 (m, 30H, *Ph*). $^{31}P\{^1H\}$ in $CDCl_3$: 22.8 (s, $\mathit{P}Ph_3$); 103.7 [s, $S_2\mathit{P}(OEt)_2$]. The supernatant on drying *in vacuo* gave a brown paste. The ^{1}H and $^{31}P\{^1H\}$ NMR spectra showed the existence of a mixture of products together with **1**.

$[Pd_3(\mu_3\text{-}S)_2\{S_2P(OPr^n)_2\}_2(PPh_3)_2]$ (**2**). This complex was prepared in an analogous manner to **1** and was recrystallized from acetone-hexane in 41% yield, m.p. 195–197°C (dec.). Anal. Calcd for $C_{48}H_{58}O_4S_6P_4Pd_3$: C, 43.2; H, 4.4. Found: C, 43.1; H, 4.1. ^{1}H in $CDCl_3$: 0.98, 1.04 (each t, 7.1 Hz, 6H, $OCH_2CH_2\mathit{Me}$); 1.76, 1.84 (each sextet, 7.1 Hz, 4H each, $OCH_2\mathit{CH}_2$-): 4.15, 4.23 (each dt, J_{PH} 9.0 Hz (d), J_{HH} 7.1 Hz (t), 4H each, $O\mathit{CH}_2$-); 7.18–7.55 (m, 30H, $P\mathit{Ph}_3$). $^{31}P\{^1H\}$ in $CDCl_3$: 21.6 (s, $\mathit{P}Ph_3$); 103.2 [s, $S_2\mathit{P}(OPr^n)_2$].

$[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OPr^n)_2\}_2(PPh_3)_2]$ (**3**). To an acetone solution of $[PdCl\{S_2P(OPr^n)_2\}(PPh_3)]$ (2.147 g, 3.48 mmol) solid Na_2Se (449 mg, 3.59 mmol) was added. The reaction mixture was stirred at room temperature under a nitrogen atmosphere for 36 h. The solvent was evaporated *in vacuo*. The residue was washed thoroughly with hexane (5 × 10 ml). dried *in vacuo*, dissolved in acetone (20 ml) and passed through a Florisil column. To the solution, hexane (15 ml) was added to give brown crystals which were separated and again recrystallized from a cold acetone-hexane mixture. The yield of maroon crystals thus obtained was 35% (580 mg, based on Pd contents); m.p. 198–202°C (dec.). Anal. Calcd for $C_{48}H_{58}O_4S_4Se_2P_4Pd_3$: C, 40.4; H, 4.4. Found: C, 40.5; H, 3.9. ^{1}H NMR in $CDCl_3$: 0.97, 1.04 (each t, 7.4 Hz, 6H each, $POCH_2CH_2\mathit{Me}$); 1.75, 1.84 (each q, 7.1 Hz, 4H each, $POCH_2\mathit{CH}_2$-); 4.13, 4.21 (each m, 4H each, $PO\mathit{CH}_2$−); 7.16–7.54 (m, 30H, $P\mathit{Ph}_3$). $^{31}P\{^1H\}$ in $CDCl_3$: 13.7 (s, $\mathit{P}Ph_3$) 103.2 [s, $S_2\mathit{P}(OPr^n)_2$]. $^{77}Se\{^1H\}$ NMR in $CDCl_3$: −98.7 (d, $^2J(^{77}Se\text{-}^{31}P) = 48$ Hz). FAB mass: *m/e* 1428 (molecular ion); 1215 $[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OPr^n)_2\}(PPh_3)_2]$; 1166 $[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OPr^n)_2\}_2(PPh_3)]$; 1089 $[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OPr^n)_2\}_2(PPh_2)]$; 953 $[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OPr^n)_2\}(PPh_3)]$.

$[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OEt)_2\}_2(PPh_3)_2]$ (**4**). This complex was prepared in an anologous manner to compound **3** and was recrystallized from acetone-hexane mixture in 24% yield, m.p.: 197–198°C (dec.). Anal. Calcd For $C_{44}H_{50}O_4S_4Se_2P_4Pd_3$: C, 38.5; H, 3.7. Found: C, 38.7; H, 3.2. ^{1}H NMR in $CDCl_3$: 1.37, 1.46 (each t, 7.0 Hz, 6H each, $POCH_2\mathit{Me}$); 4.24, 4.33 (each dq, J_{HH} 7 Hz (q), J_{PH} 9.5 Hz (d), 4H each, $PO\mathit{CH}_2$-); 7.15–7.54 (m, 30 H, $P\mathit{Ph}_3$). $^{31}P\{^1H\}$ in $CDCl_3$: 12.1 (s, $\mathit{P}Ph_3$), 101.0 [s, $S_2\mathit{P}(OEt)_2$].

$[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OPr^i)_2\}_2(PPh_3)_2]$ (**5**). This complex was prepared in an analogous manner to **3** and was recrystallized from acetone-hexane in 25% yield, m.p. 224–225°C (dec.). Anal. Calcd for $C_{48}H_{58}O_4S_4Se_2P_4Pd_3$: C, 40.4; H, 4.4. Found: C, 39.5; H, 3.9. ^{1}H NMR in $CDCl_3$: 1.37, 1.47 (each d, 6.2 Hz, 12H each, $POCH\mathit{Me}_2$); 4.98 (m, 4H, $PO\mathit{CH}$-); 7.18–7.52 (m, 30H, $P\mathit{Ph}_3$). $^{31}P\{^1H\}$ in $CDCl_3$: 15.0 (s, $\mathit{P}Ph_3$), 101.1 [s, $S_2\mathit{P}(OPr^i)_2$].

$[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OBu^s)_2\}_2(PPh_3)_2]$ (**6**). This was prepared in an analogous manner to **3**. The complex was recrystallized from diethylether-hexane in 36% yield, m.p. 173–175°C (dec.). Anal. Calcd for $C_{52}H_{66}O_4S_4Se_2P_4Pd_3$: C, 42.1; H, 4.5. Found: C, 42.0; H, 3.7. ^{1}H NMR in $CDCl_3$: 0.95, 1.01 (each t, 7.5 Hz, 6H each, $POCHCH_2\mathit{Me}$); 1.36, 1.47 (each d, 6.2 Hz, 6H each, $POCH\mathit{Me}$); 1.74 (m, 8H, $POCH\mathit{CH}_2$-); 4.76 (m, 4H, $PO\mathit{CH}$-); 7.17–7.57 (m, 30H, $P\mathit{Ph}_3$). $^{31}P\{^1H\}$ in $CDCl_3$: 12.0 (s, $\mathit{P}Ph_3$), 99.0 [s, $S_2\mathit{P}(OBu^s)_2$].

$[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OPr^n)_2\}_2(PEt_3)_2]$ (**7**). This complex was prepared in an analogous manner to **3,** and was recrystallized by cooling a diethylether-hexane mixture in a freezer for a week (yield 42%) m.p. 163–165°C. Anal. Calcd for $C_{24}H_{58}O_4S_4Se_2P_4Pd_3$: C, 25.3; H, 5.1. Found: C, 24.5; H, 4.9. ^{1}H NMR in $CDCl_3$: 0.94 (dt. J_{HH} 7.5 Hz (t), J_{PH} 22 Hz (d), 18H, $PCH_2\mathit{Me}$); 1.22, 1.28 (each t, 7.5 Hz, 6H each, $POCH_2CH_2\mathit{Me}$); 1.71 (m, 12H, $P\mathit{CH}_2$-); 1.92 (m, 8H, $POCH_2\mathit{CH}_2$-); 3.97, 4.13 (each dt, J_{HH} 7 Hz (t), J_{PH} 9 Hz (d), 4H each, $PO\mathit{CH}_2$-); $^{31}P\{^1H\}$ in $CDCl_3$: 11.4 (s, $\mathit{P}Et_3$), 101.7 [s, $S_2\mathit{P}(OPr^n)_2$]. FAB mass: *m/e* 1140 (molecular ion).

X-ray crystallography

All measurements were made at 20±2°C on a Rigaku AFC 6S diffractomer using graphite monochromated Mo–K_α radiations ($\lambda = 0.71073$ Å). Crystal data and data collection details are given in Table 1. The data were corrected for Lorentz and polarization effects. Semi-empirical absorptions correction was based on Ψ scan of several reflections (maximum and minimum transmission factors: 0.57 and 0.26, respectively). The structure was solved by a combination of the Patterson and direct methods and refined by full-matrix least-squares on F^2. Neutral atom factors were taken from Cromer and Waber [14]. Anamolous dispersion effects were included in F_{calc} [15]. All calculations were performed using the TEXSAN [16] crystallographic software package of the Molecular Structure Corporation. The ORTEP [17] plot of the molecule is shown in Fig. 1.

RESULTS AND DISCUSSION

Treatment of $[PdCl\{S_2P(OR)_2\}(PR'_3)]$ with sodium sulfide or selenide in acetone gave neutral chal-

Table 1. Crystal data and structure refinement for $[Pd_3(\mu\text{-}S)_2\{S_2P(OEt)_2\}_2(PPh_3)_2]$

Empirical formula	$C_{44}H_{50}O_4P_4Pd_3S_6$
Formula weight	1278.28
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	$P\bar{1}$
a (Å)	11.988(4)
b (Å)	13.523(5)
c (Å)	18.953(8)
α (°)	77.18
β (°)	82.18
γ (°)	69.370(10)
V (Å)^3	2798(2)
Z	2
D_{calc} (Mg m^{-3})	1.517
Absorption coefficient (mm^{-1})	1.328
F(000)	1280
Crystal size	0.39 × 0.25 × 0.12 mm
θ range for data collection (°)	2.65 to 25.00
Index ranges	$0 \leqslant h \leqslant 14, -15 \leqslant k \leqslant 16, -22 \leqslant l \leqslant 22$
Reflection collected	10 343
Independent reflections	9832(R_{int} = 0.0591)
Absorption correction	SHELXA
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	9823/0/556
Goodness-of-fit on F^2	1.014
Final R indices [$I > 2(I)$]	$R_1 = 0.0644$, $wR_2 = 0.0994$
R indices (all data)	$R_1 = 0.1608$, $wR_2 = 0.1354$
Extinction coefficient	0.0000(2)
Largest diff. peak and hole (e Å^{-3})	0.583 and −0.585

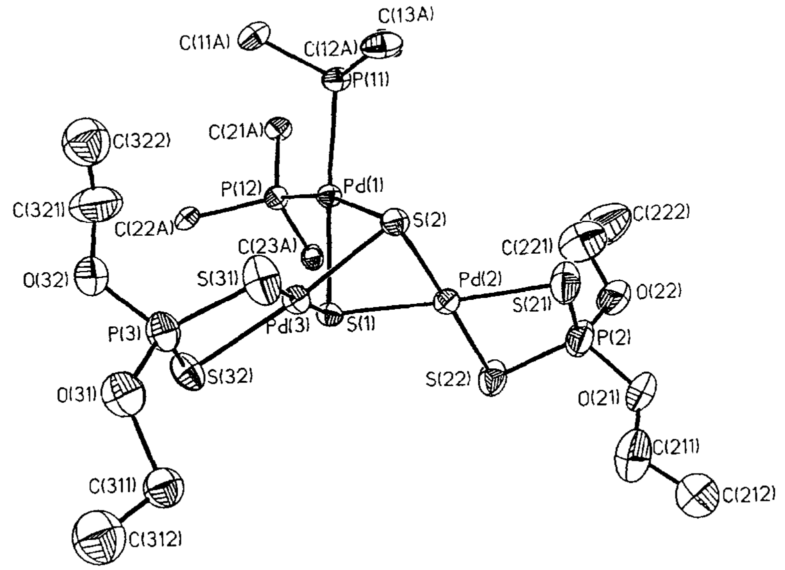
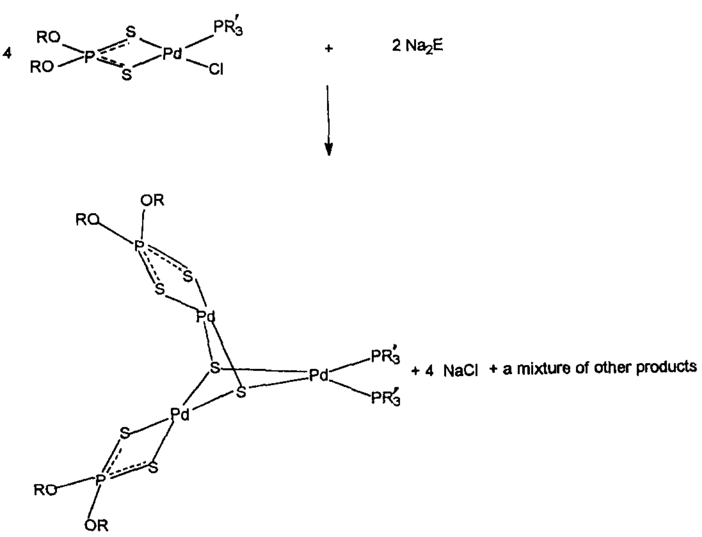


Fig. 1. Molecular structure of $[Pd_3(\mu_3\text{-}S)_2\{S_2P(OEt)_2\}_2(PPh_3)_2]$. Phenyl groups of PPh_3 are omitted for the sake of clarity.

ogenido-bridged trinuclear palladium(II) complexes of the type $[Pd_3(\mu_3\text{-}E)_2\{S_2P(OR)_2\}_2(PR'_3)_2]$ (E = S, Se; R = Et, Pr^n, Pr^i, Bu^s and R′ = Et, Ph) (Scheme 1) in 24–42% yield. The ^{1}H NMR showed two sets of OR proton resonances in 1 : 1 ratio indicating non-equivalence of the alkoxy groups on the dithi-



	R	R′	E
1	Et	Ph	S
2	Pr^n	Ph	S
3	Pr^n	Ph	Se
4	Et	Ph	Se
5	Pr^i	Ph	Se
6	Bu^s	Ph	Se
7	Pr^n	Et	Se

Scheme 1.

ophosphate ligand. The $^{31}P\{^1H\}$ NMR spectra showed singlets for the triphenylphosphine and dialkyldithiophosphate moiety. The ^{31}P resonance for triphenylphosphine is more shielded for the selenido-bridged complexes than the corresponding sulfido-bridged derivatives. The $^{77}Se\{^1H\}$ NMR spectrum (^{77}Se $I = 1/2$, natural abundance 7.58%) of **3** showed a doublet at −98.7 ppm with $^2J(^{77}Se\text{-}^{31}P)_{trans}$ of 48 Hz. FAB mass spectra of $[Pd_3(\mu_3\text{-}Se)_2\{S_2P(OPr^n)_2\}_2(PR'_3)_2]$ (R′ = Et, Ph) showed molecular ion peaks. Other prominent ions observed in the spectra are due to the species formed either by loss of a dithiophosphate ligand or triphenylphosphine ligand. The isotopic patterns for these species are in accord with the pattern calculated for $Pd_3Se_2S_4$ fragment.

The structure of **1**, established by X-ray study, shows that this is a sulfido-bridged trinuclear palladium(II) complex (Fig. 1). Molecule consists of three distorted square-planar palladium atoms sharing two μ_3-S ligands. The molecular geometry may alternatively be viewed of a trigonal bipyramidal Pd_3S_2 core with one palladium atom linked with two triphenylphosphine and each of the remaining two palladium atoms is coordinated with chelating dithiophosphate group. The central palladium atoms in the triangle are not equilateral, the two Pd—Pd distances (av. 3.0446(14) Å) are shorter than the third one (3.217(2) Å). Although **1** is the first neutral trinuclear sulfido-bridged complex, the Pd—Pd distances are in accord with the Pd—Pd bond lengths reported in $[Pd_3(\mu\text{-}S)_2(PMe_3)_6][BPh_4]_2$ [18]. However, the metal–metal distances in other ionic chalcogenido-bridged trinuclear complexes, $[Pt_3(\mu_3\text{-}Te)_2(PEt_3)_6][PF_6]_2$ [19], $[Pt_3(\mu_3\text{-}Te)_2(dppe)_3][BPh_4]_2$ [20], $[Ni_3(\mu_3\text{-}E)_3(PR_3)_6]^{2+}$ (E = S, Se) [21], are equidistant.

The dithiophosphate ligands are symmetrically chelated to the palladium atoms, the $Pd—S_{(dithio)}$ distances are essentially the same (i.e. av. 2.383 Å) and are in good agreement with the values reported for chelated dithio palladium complexes [22,23]. The P—S bond lengths (av. 1.982(5) Å) are intermediate between the double bond (1.94 Å) and single bond (2.09 Å) values confirming the partial double bond character, as expected from symmetrically chelated dithiophosphate ligand. The angles S(21)—Pd(2)—S(22) (84.16(11)°), S(31)—Pd(3)—S(32) (83.64(11)°), S(1)—Pd(2)—S(2) (78.96(9)°), S(1)—Pd(3)—S(2) (78.50(9)°) and S(1)—Pd(1)—S(2) (76.87(9)°) are

Table 2. Selected bond distances (Å) and angles (deg) for $[Pd_3(\mu_3\text{-}S)_2\{S_2P(OEt)_2\}_2(PPh_3)_2]$

	Distances		
Pd(1)—S(1)	2.348(3)	Pd(3)—S(32)	2.370(3)
Pd(1)—S(2)	2.337(3)	S(31)—P(3)	1.980(5)
Pd(1)—P(11)	2.292(3)	S(32)—P(3)	1.973(4)
Pd(1)—P(12)	2.328(3)	P(3)—O(31)	1.572(8)
Pd(2)—S(1)	2.301(3)	P(3)—O(32)	1.575(9)
Pd(2)—S(2)	2.280(3)	Pd(1)—Pd(2)	3.0930(14)
Pd(2)—S(21)	2.384(3)	Pd(1)—Pd(3)	2.9962(14)
Pd(2)—S(22)	2.386(3)	Pd(2)—Pd(3)	3.217(2)
S(21)—P(2)	1.985(5)	P(11)—C(11A)	1.794(10)
S(22)—P(2)	1.989(5)	P(11)—C(12A)	1.833(10)
P(2)—O(21)	1.573(9)	P(11)—C(13A)	1.815(11)
P(2)—O(22)	1.584(10)	P(12)—C(21A)	1.826(10)
Pd(3)—S(1)	2.304(3)	P(12)—C(22A)	1.834(10)
Pd(3)—S(2)	2.300(3)	P(12)—C(23A)	1.815(10)
Pd(3)—S(31)	2.392(2)		
	Angles		
S(1)—Pd(1)—P(11)	168.68(10)	Pd(3)—Pd(1)—P(11)	121.19(8)
S(2)—Pd(1)—P(11)	92.04(10)	Pd(3)—Pd(1)—P(12)	123.38(7)
S(1)—Pd(1)—S(2)	76.87(9)	Pd(2)—Pd(1)—S(1)	47.65(7)
S(1)—Pd(1)—P(12)	90.21(9)	Pd(2)—Pd(1)—S(2)	47.16(7)
P(11)—Pd(1)—P(12)	100.98(10)	Pd(3)—Pd(1)—S(1)	49.26(7)
S(2)—Pd(1)—P(12)	166.72(9)	Pd(3)—Pd(1)—S(2)	49.21(7)
Pd(2)—Pd(1)—P(11)	125.24(8)	Pd(2)—Pd(1)—Pd(3)	63.75(4)
Pd(2)—Pd(1)—P(12)	121.02(7)		
Pd(1)—S(1)—Pd(2)	83.40(9)	Pd(1)—S(2)—Pd(2)	84.10(9)
Pd(1)—S(1)—Pd(3)	80.19(8)	Pd(1)—S(2)—Pd(3)	80.49(9)
Pd(2)—S(1)—Pd(3)	88.61(9)	Pd(2)—S(2)—Pd(3)	89.22(9)
Pd(1)—Pd(2)—Pd(3)	56.66(3)	Pd(1)—Pd(3)—Pd(2)	59.59(3)
Pd(1)—Pd(2)—S(1)	48.95(6)	Pd(1)—Pd(3)—S(1)	50.55(6)
Pd(1)—Pd(2)—S(2)	48.74(7)	Pd(1)—Pd(3)—S(2)	50.30(7)
Pd(3)—Pd(2)—S(1)	45.73(6)	Pd(2)—Pd(3)—S(1)	45.66(7)
Pd(3)—Pd(2)—S(2)	45.64(7)	Pd(2)—Pd(3)—S(2)	45.13(6)
S(1)—Pd(2)—S(2)	78.96(9)	S(1)—Pd(3)—S(2)	78.50(9)
S(1)—Pd(2)—S(21)	176.47(11)	S(1)—Pd(3)—S(31)	178.02(11)
S(1)—Pd(2)—S(22)	99.29(10)	S(1)—Pd(3)—S(32)	98.26(10)
S(2)—Pd(2)—S(21)	97.61(11)	S(2)—Pd(3)—S(31)	99.68(11)
S(2)—Pd(2)—S(22)	177.92(11)	S(2)—Pd(3)—S(32)	172.62(11)
S(21)—Pd(2)—S(22)	84.16(11)	S(31)—Pd(3)—S(32)	83.64(11)
Pd(2)—S(21)—P(2)	83.5(2)	Pd(3)—S(31)—P(3)	83.39(14)
Pd(2)—S(22)—P(2)	83.37(14)	Pd(3)—S(32)—P(3)	84.08(14)
S(21)—P(2)—S(22)	107.1(2)	S(31)—P(3)—S(32)	106.9(2)
S(21)—P(2)—O(21)	114.4(4)	S(31)—P(3)—O(31)	115.6(4)
S(22)—P(2)—O(21)	114.0(4)	S(32)—P(3)—O(31)	113.0(4)
S(21)—P(2)—O(22)	113.4(4)	S(31)—P(3)—O(32)	112.5(4)
S(22)—P(2)—O(22)	113.6(4)	S(32)—P(3)—O(32)	114.2(4)

compressed owing to the formation of four-membered chelate rings. Consequently other angles have opened. The Pd—P [9,23,24], Pd—$S_{(\text{sulfido})}$ [18] are in good agreement with the reported values. It may be noted that the two of the palladium-sulfido bonds for the palladium linked to triphenylphosphines are longer (0.04 Å) than the other Pd—S bonds where palladium atoms are bound to the dialkyldithiophosphate groups. Selected bond lengths and angles are given in Table 2.

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