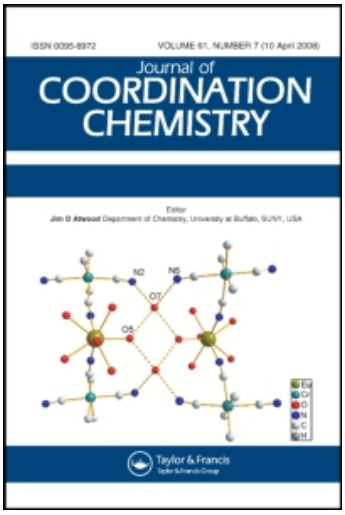

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Metal-Selenium Interactions. Crystal Structures of [Dihalo-{1, 1'-Methylene-Bis(Diphenyl Phosphine Selenide)}] Mercury(II) Complexes

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METAL-SELENIUM INTERACTIONS. CRYSTAL STRUCTURES OF [DIHALO-{1,1'-METHYLENE-BIS(DIPHENYL PHOSPHINE SELENIDE)}] MERCURY(II) COMPLEXES

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Crystal structures of mercury(II) complexes with a bis(tertiary phosphine selenide), namely 1,1'-methylenebis(diphenylphosphine selenide) {$Ph_2P(Se)—CH_2—P(Se)Ph_2$, $dpmSe_2$} are described. Mercury(II) bromide reacts with $dpmSe_2$ in ethanol-benzene to form triclinic crystals of $HgBr_2(dpmSe_2)$ (**1**) and mercury(II) iodide reacts with $dpmSe_2$ in acetone-ethanol to yield monoclinic crystals of $HgI_2(dpmSe_2)$ (**2**) as revealed by X-ray crystallography. In each complex, mercury(II) is bonded to two halogen atoms {bond distances Hg—Br 2.5949(8), 2.5846(8)Å for **1** and Hg—I 2.7052(6), 2.6941(7) Å for **2**} and two selenium atoms {Hg—Se 2.6924(8), 2.6513(8) for **1** and 2.7238(8), 2.7636(8) Å for **2**}. In compounds **1** and **2**, the bond angles about Hg vary from 100.09(3) to 119.50(3)° and 87.47(2) to 121.47(2)° respectively with the smallest and largest bond angles being Se(1)—Hg—Br(2) and Se(2)—Hg—Br(2) for **1** and Se(1)—Hg—Se(2) and I(1)—Hg—I(2) for **2**. Thus $dpmSe_2$ chelates to Hg(II) centres forming distorted tetrahedral geometries.

Keywords: Phosphine selenide; Mercury(II) bromide; Mercury(II) iodide; Tetrahedral; Crystal structure; Methylenebis(diphenylphosphine selenide)

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INTRODUCTION

Complexes of tertiary phosphine chalcogenides with both hard and soft metals have interesting bonding properties, structural novelties and applications in catalysis or extractive metallurgy [1, 2]. In contrast to complexes of phosphine sulphides, only a few tertiary phosphine selenide complexes have been structurally characterised [3 – 5].

Metal-selenide complexes have applications in chemical vapour deposition [6] and we have reported a zinc(II) complex with 1,1'-methylenebis(diphenylphosphine selenide) {$Ph_2P(Se)—CH_2—P(Se)Ph_2$, $dpmSe_2$} [7]. In view of this importance, and also as a part of our interest in metal chalcogenide interactions, [1, 2, 8 – 10] in this paper the crystal structures of two mercury(II) halide complexes with $dpmSe_2$ are reported, these being the first structurally characterised examples of mercury(II) with a bis(tertiary phosphine selenide). Further, the literature reports only one complex of mercury(II) with a tertiary phosphine selenide (*viz*, triphenyl phosphine selenide) which has been structurally characterised [3].

EXPERIMENTAL

Materials and Techniques Used

The ligand details are the same as reported earlier [7] and mercury(II) bromide and iodide salts were of laboratory grade and used without further purification. Infrared spectra of ligand/complex samples were recorded using KBr pellets in the range 4000 – 200 cm^{-1} on a Pye UnicamSP3-300 infrared spectrophotometer.

Preparation of $[HgBr_2(dpmSe_2)]$ (1)

To a solution of mercury(II) bromide (0.100 g, 0.280 mmol) in 15 cm^3 of acetone was added a solution of $dpmSe_2$ (0.150 g, 0.280 mmol) in 30 cm^3 of acetone. The reaction mixture was stirred for about 5 hours and the colourless precipitates formed were filtered, washed with acetone and finally dried *in vacuo*. Yield (%), 80, m.p. (°C) 276 – 78. Crystals were grown from a benzene-ethanol mixture.

Preparation of $[HgI_2(dpmSe_2)]$ (2)

To a solution of mercury (II) iodide (0.100 g, 0.220 mmol) in a 15 cm^3 of acetone was added a solution of $dpmSe_2$ (0.120 g, 0.220 mmol) in 30 cm^3 of

acetone. The reaction mixture was stirred for about 5 hours and the colourless compound formed after evaporation of the solvent at room temperature. It was recrystallised from acetone. Yield (%),70, m.pt.(°C), 220 – 22. Crystals were grown from acetone-ethanol on slow evaporation.

CRYSTAL STRUCTURE DETERMINATIONS

Compound 1

A colourless prismatic crystal of compound **1** was attached to a thin glass fibre and mounted on a Bruker SMART 1000 CCD diffractometer employing graphite-monochromated MoK_α radiation generated from a sealed tube. Cell constants were obtained from a least-squares refinement against 927 reflections located between 2θ values of 6.44 and 55.68°. Data were collected at 294(2) K with ω scans to 56.60°2θ. The intensities of 146 standard reflections recollected at the end of the experiment showed no significant change during the data collection. An empirical absorption correction determined with SADABS [11] was applied to the data. Data integration and reduction were undertaken with SAINT and XPREP [12], and subsequent computations were carried out with the teXsan [13] graphical user interface. The data reduction included the application Lorentz and polarisation corrections.

The structure was solved in space group $P\bar{1}$ by direct methods with SIR97 [14], extended and refined with SHELXL-97 [15]. Anisotropic displacement parameters were refined for non-hydrogen atoms and a riding atom model was used for the hydrogen atoms included in the model. An ORTEP [16, 17] depiction of the molecule is provided in Figure 1.

Compound 2

A colourless prismatic crystal of **2** was mounted on a glass fibre and used for data collection. Crystal data were collected at 291(2) K, using a Bruker SMART CCD 1000 diffractometer. Graphite monochromated MoK_α radiation ($\lambda = 0.71073$Å) was used throughout. The data were processed with SAINT [12] and corrected for absorption using SADABS [11].

The structure was solved by direct methods using the program SHELXS-97 [18] and refined by full matrix least squares techniques [15] on F^2. Positional and anisotropic atomic displacement parameters were refined for all non-hydrogen atoms. Hydrogen atoms were placed geometrically and positional parameters were refined using a riding model. Isotropic atomic

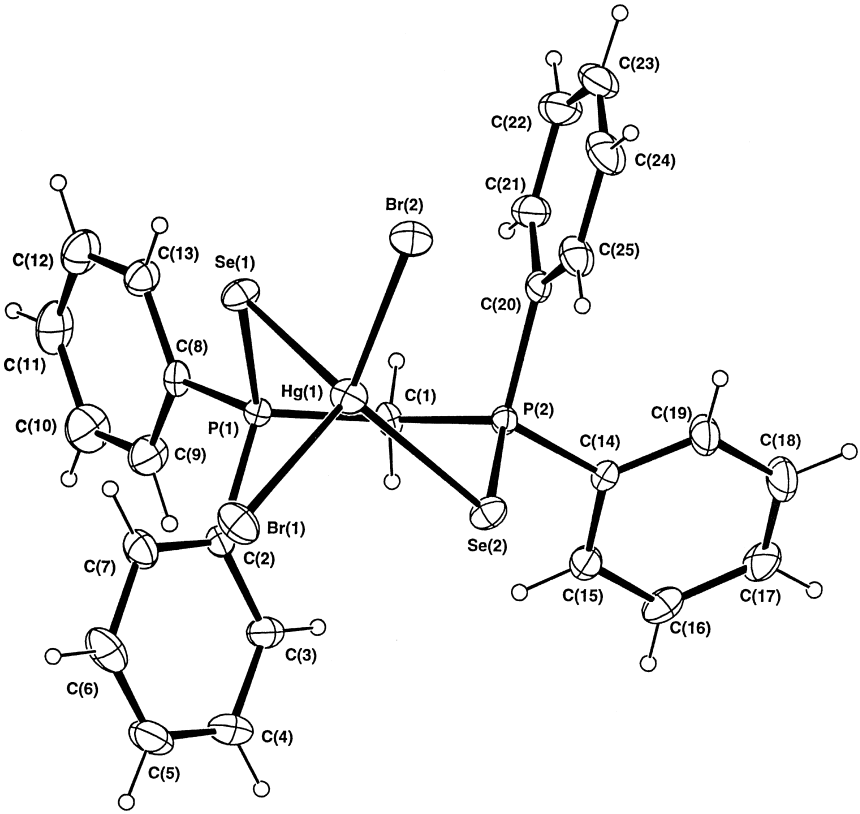

FIGURE 1 The structure of $HgBr_2$ ($dpmse_2$) (**1**) showing the numbering scheme.

displacements for hydrogen atoms were constrained to be 1.2 times that of the atom to which they are attached. Atomic scattering factors from International Tables for X-ray Crystallography [19]. Molecular graphics were taken from PLATON 99 [20]. A summary of the crystal data, experimental details and refinement results is listed in Table I. Atomic positions for the non-hydrogen atoms are listed in Tables II and III.

TABLE I Crystal data and refinement details for $HgBr_2(dpmSe_2)$ **1** and $HgI_2(dpmSe_2)$ **2**

Parameter	**1**	**2**
Empirical formula	$C_{25}H_{22}Br_2P_2Se_2Hg$	$C_{25}H_{22}I_2P_2Se_2Hg$
Molecular weight (M)	902.70	996.68
Wavelength Å	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_{(1)}/n$

TABLE I (Continued)

Parameter	**1**	**2**
Unit cell dimensions		
a/Å	9.729(3)	13.247(1)
b/Å	16.264(5)	13.106(1)
c/Å	8.769(3)	16.636(1)
α/°	95.812(5)	
β /°	90.249(5)	98.849(1)
γ /°	97.721(5)	
Volume Å^3	1367.7(7)	2853.8(3)
Z	2	4
Density(calculated) Mg m^{-3}	2.192	2.320
Absorption Coefficient(μ)/mm^{-1}	11.339	10.232
T(SADABS)$_{min,max}$	0.420, 1.000	0.433,1.000
Crystal size/mm	0.448 × 0.194 × 0.162	0.25 × 0.25 × 0.12
$2\theta_{max}$/°	56.60	56.06
Index ranges	$-12 \le h \le 12$ $-21 \le k \le 21$ $-11 \le l \le 11$	$-17 \le h \le 13$ $-13 \le k \le 17$ $-21 \le l \le 21$
Reflections collected	13605	18370
Independent reflections	6283	6790
R_{int}	0.0282	0.0736
Reflections observed [$I > 2\sigma(I)$]	5343	6790
Parameters (N_{var})	289	289
Goodness of fit on F^2	1.065	0.844
Final R indices [$I > 2\sigma(I)$]	$^*R1 = 0.035$ $wR2 = 0.095$	$R1 = 0.042$ $wR2 = 0.089$
Largest diff. peak and hole/e.Å^{-3}	2.053, −1.350	−1.488, 1.958

TABLE II Final atomic coordinates for the bromide complex with equivalent isotropic thermal parameters

	x/a	y/b	z/c	U_{eq}(Å^2)
Hg(1)	0.76872(2)	0.756616(14)	−0.14740(2)	0.04948(8)
Br(1)	0.61421(7)	0.83942(5)	−0.29915(7)	0.06198(17)
Br(2)	0.91625(7)	0.67203(4)	−0.33228(7)	0.05493(15)
Se(1)	0.62799(6)	0.63231(3)	−0.00713(6)	0.04847(14)
Se(2)	0.89359(6)	0.86537(3)	0.07236(6)	0.04651(13)
P(1)	0.59199(12)	0.70684(7)	0.20138(14)	0.0336(2)
P(2)	0.91206(12)	0.78010(8)	0.24224(13)	0.0332(2)
C(1)	0.7465(5)	0.7378(3)	0.3230(5)	0.0380(10)
C(2)	0.5161(5)	0.7997(3)	0.1710(6)	0.0390(10)
C(3)	0.5390(7)	0.8718(4)	0.2741(8)	0.0570(15)
C(4)	0.4737(8)	0.9400(4)	0.2478(10)	0.075(2)
C(5)	0.3851(8)	0.9359(5)	0.1257(10)	0.073(2)
C(6)	0.3628(8)	0.8656(5)	0.0246(9)	0.075(2)
C(7)	0.4281(6)	0.7957(4)	0.0460(7)	0.0555(14)
C(8)	0.4758(5)	0.6452(3)	0.3196(6)	0.0401(11)
C(9)	0.4022(7)	0.6849(5)	0.4350(8)	0.0650(17)

TABLE II (Continued)

	x/a	y/b	z/c	$U_{eq}(Å^2)$
C(10)	0.3157(8)	0.6376(6)	0.5257(9)	0.081(2)
C(11)	0.2995(7)	0.5521(5)	0.5024(9)	0.076(2)
C(12)	0.3690(8)	0.5129(5)	0.3900(9)	0.0699(19)
C(13)	0.4568(6)	0.5595(4)	0.2985(7)	0.0531(13)
C(14)	1.0089(5)	0.8363(3)	0.4040(6)	0.0370(10)
C(15)	0.9461(6)	0.8862(4)	0.5143(7)	0.0492(13)
C(16)	1.0202(7)	0.9285(4)	0.6381(7)	0.0586(15)
C(17)	1.1588(8)	0.9219(4)	0.6554(8)	0.0640(17)
C(18)	1.2204(7)	0.8749(5)	0.5504(10)	0.085(3)
C(19)	1.1482(6)	0.8311(5)	0.4227(8)	0.0652(18)
C(20)	0.9996(5)	0.6933(3)	0.1744(6)	0.0389(10)
C(21)	0.9949(7)	0.6249(4)	0.2586(7)	0.0546(14)
C(22)	1.0651(8)	0.5588(4)	0.2088(9)	0.072(2)
C(23)	1.1421(8)	0.5619(5)	0.0782(10)	0.079(2)
C(24)	1.1470(7)	0.6286(5)	−0.0054(9)	0.074(2)
C(25)	1.0771(6)	0.6949(4)	0.0415(7)	0.0571(15)

TABLE III Final atomic coordinates ($\times 10^4$) for the iodide complex with equivalent isotropic thermal parameters ($Å^2 \times 10^2$)

	x/a	y/b	z/c	U_{eq}
Hg(1)	6000(1)	584(1)	3063(1)	55(1)
I(1)	7994(1)	1072(1)	3122(1)	59(1)
I(2)	5412(1)	−1326(1)	3372(1)	85(1)
Se(1)	4801(1)	1968(1)	3692(1)	44(1)
Se(2)	4788(1)	886(1)	1575(1)	44(1)
P(1)	5351(1)	3298(1)	3141(1)	32(1)
P(2)	5119(1)	2442(1)	1335(1)	32(1)
C(1)	5869(4)	3103(5)	2195(4)	32(1)
C(11)	6358(5)	3903(5)	3826(4)	39(2)
C(12)	7339(5)	4008(5)	3666(5)	47(2)
C(13)	8056(6)	4508(6)	4235(6)	65(2)
C(14)	7794(7)	4892(6)	4934(6)	65(2)
C(15)	6816(7)	4778(7)	5090(5)	66(2)
C(16)	6112(6)	4267(6)	4548(5)	55(2)
C(21)	4345(5)	4248(5)	2941(4)	35(2)
C(22)	3326(5)	3949(5)	2744(4)	40(2)
C(23)	2580(5)	4668(6)	2577(4)	51(2)
C(24)	2817(6)	5685(6)	2593(5)	53(2)
C(25)	3820(6)	5989(6)	2775(5)	52(2)
C(26)	4581(5)	5270(5)	2951(4)	42(2)
C(31)	3987(5)	3179(5)	994(4)	38(2)
C(32)	4096(6)	4207(6)	813(5)	53(2)
C(33)	3230(8)	4782(7)	510(5)	70(3)
C(34)	2308(8)	4325(9)	403(6)	80(3)
C(35)	2187(6)	3308(8)	582(5)	72(3)
C(36)	3041(5)	2736(6)	885(4)	48(2)
C(41)	5872(5)	2518(5)	520(4)	38(2)
C(42)	6923(5)	2539(5)	641(4)	46(2)
C(43)	7442(6)	2520(6)	−12(5)	54(2)
C(44)	6936(7)	2459(6)	−778(5)	66(2)
C(45)	5886(7)	2417(7)	−927(5)	70(3)
C(46)	5347(6)	2455(6)	−265(4)	54(2)

RESULTS AND DISCUSSION

Mercury(II) halides react with $Ph_2P(Se)—(CH_2)—P(Se)Ph_2$ according to the following equation

$$HgX_2 + Ph_2P(Se)—(CH_2)—P(Se)Ph_2 \longrightarrow HgX_2\{Ph_2P(Se)—(CH_2)—P(Se)Ph_2\} (X = Br, I)$$

Due to the low sensitivity of heavier Se donor atom, it is problematic to establish using IR spectroscopy whether Hg-Se bonds are weak or normal. Though IR spectra of ligand and complexes are different, there are no diagnostic frequencies except $\nu P=Se$ which can help in understanding the strengths of metal-ligand bonds. The $\nu P=Se$ modes for ligand and the complexes are not significantly different.

Crystal and Molecular Structures

The atom numbering schemes for $HgBr_2(dpmSe_2)$ **1** and $HgI_2(dpmSe_2)$ **2** are shown in Figures 1 and 2 respectively. While Table I lists crystal data, experimental details and refinement results, and Table IV contains important bond lengths and angles. The basic unit is $[HgX_2(dpmSe_2)]$ (X = Br or I) with no evidence for intermolecular interaction.

Mercury(II) binds to two Se atoms of the $dpmSe_2$ ligand and two bromine atoms in compound **1** (or iodine atoms in compound **2**) and in each case $dpmSe_2$ chelates to Hg(II) forming a distorted tetrahedral geometry. In compound **1**, the Br(1)—Hg(1)—Br(2) bond angle 110.58(3)° and the corresponding I(1)—Hg(1)—I(2) bond angle in compound **2** expands to 121.47(2)° due to the bulky iodine atoms. The bond angle Se(1)—Hg(1)—Se(2) is 87.47(2)° in **2** and the same angle in **1** is 106.56(3)°. It is interesting to compare the corresponding angles in the zinc compound $[ZnI_2(dpmSe_2)]$ (compound **3**)[7] which are I(1)—Zn(1)—I(2), 114.69(3)° and Se(1)—Zn(1)—Se(2), 104.57(3)°. Thus variations in the size of the metal and the anion, both affect significantly the X—M—X (X = halogen; M = metal) and the Se—M—Se bond angles. The angles at Se, namely, M—Se—P lie in the close range M—Se(1)—P(1) *ca* 96° and M—Se(2)—P(2) *ca* 98 – 101° (compounds **1 – 3**). The angles at P or C atoms of the metallacyclic rings {M(1), Se(1), P(1), C, P(2), Se(2)} are similar in all three cases: **1**, Se(1)—P(1)—C(1), 113.77(17), Se(2)—P(2)—C(1), 114.58(15); P(1)—C(1)—P(2) 120.9(3); **2** Se(1)—P(1)—C(1), 116.8(2), Se(2)—P(2)—C(1), 114.1(2), P(1)—C(1)—C(2), 120.6(3); **3** Se(1)—P(1)—C(13), 114.54(16), Se(2)—P(2)—C(13), 112.59(17); P(1)—C(13)—P(2) 119.7(3).

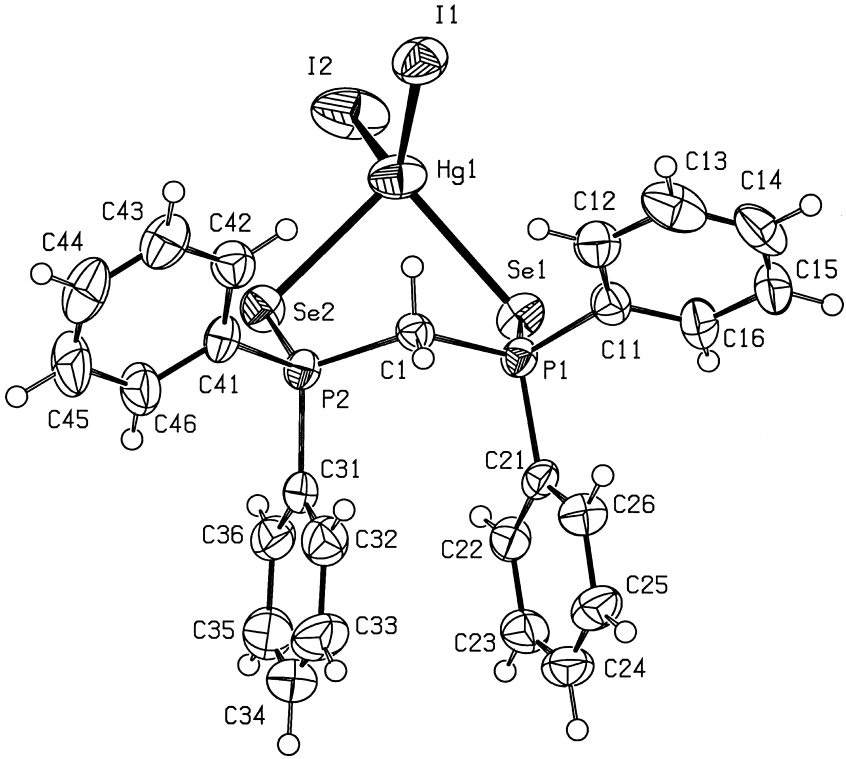

FIGURE 2 The structure of HgI_2 ($dpmSe_2$) (**2**) showing the numbering scheme.

In compound **1**, the Hg—Se bond distances {2.6924(8), 2.6513(8)Å} are shorter than those in compound **2** {2.7238(8), 2.7636(8)Å} and both these in turn are longer than that in chlorobridged $\{HgCl_2(Ph_3PSe)\}_2$ {Hg—Se = 2.527(3) Å, compound **4**} [3]. These distances are much smaller than the sum of van der Walls radii {3.40 Å} and also sum of radii of Hg^{2+} and Se^{2-} 3.00 Å) [21]. As expected, the Lewis acidity of mercury appears to increase with an increase in the electronegativity of halogen atoms **4**(Cl) > **1**(Br) > **2**(I) and thus the corresponding P—Se bond distances vary in the reverse order **2**(I) < **1**(Br) < 4(Cl). These distances are longer than in the free ligand Ph_3PSe {d(P—Se) = 2.106(1)Å} [22] and in turn all these coordinated and uncoordinated P—Se distances are smaller than the sum of covalent radii of P and Se atoms {2.27Å} [21]. Obviously, the double bond character of the P—Se bond in the free ligand is reduced in the coordinated ligand. Other significant bond distances of the metallacyclic ring, namely P(1)—C(1) and P(2)—C(1), are longer than P—C_{Ph} bond distances and this

TABLE IV Bond lengths (Å) and bond angles (°) for compounds **1** and **2**

1	*Bond lengths*	**2**	*Bond lengths*
Hg(1)—Br(1)	2.5949(8)	Hg(1)—I(1)	2.7052(6))
Hg(1)—Br(2)	2.5846(8)	Hg(1)—I(2)	2.6941(7)
Hg(1)—Se(1)	2.6924(8)	Hg(1)—Se(1)	2.7238(8)
Hg(1)—Se(2)	2.6513(8)	Hg(1)—Se(2)	2.7636(8)
P(1)—Se(1)	2.1460(14)	P(1)—Se(1)	2.1485(18)
P(2)—Se(2)	2.1581(14)	P(2)—Se(2)	2.1373(18)
P(1)—C(1)	1.823(5)	P(1)—C(1)	1.828(6)
P(2)—C(1)	1.839(5)	P(2)—C(1)	1.830(6)
P(1)—C(2)	1.809(5)	P(1)—C(11)	1.800(7)
P(1)—C(8)	1.809(5)	P(1)—C(21)	1.817(6)
P(2)—C(14)	1.797(5)	P(2)—C(31)	1.800(7)
P(2)—C(20)	1.797(5)	P(2)—C(41)	1.805(6)
	Bond angles		*Bond angles*
Hg(1)—Se(1)—P(1)	96.38(4)	Hg(1)—Se(1)—P(1)	96.93(5)
Hg(1)—Se(2)—P(2)	98.18(4)	Hg(1)—Se(2)—P(2)	101.31(5)
Se(1)—Hg(1)—Br(2)	100.09(3)	Se(1)—Hg(1)—I(2)	110.05(2)
Se(2)—Hg(1)—Br(2)	119.50(3)	Se(2)—Hg(1)—I(2)	99.11(2)
Se(1)—Hg(1)—Se(2)	106.56(3)	Se(1)—Hg(1)—Se(2)	87.47(2)
Se(2)—Hg(1)—Br(1)	105.82(3)	Se(2)—Hg(1)—I(1)	115.35(2)
Se(1)—Hg(1)—Br(1)	114.54(3)	Se(1)—Hg(1)—I(1)	116.91(2)
Br(2)—Hg(1)—Br(2)	110.58(3)	I(2)—Hg(1)—I(1)	121.47(2)
P(1)—C(1)—C(2)	120.9(3)	P(1)—C(1)—P(2)	120.6(3)
Se(1)—P(1)—C(1)	113.77(17)	Se(1)—P(1)—C(1)	116.8(2)
Se()—P(2)—C(1)	114.58(15)	Se(2)—P(2)—C(1)	114.1(2)

is attributed to the "flexible" electron density on phenyl rings in the vicinity of the P—C bond which tends to compensate for electron density imbalances caused by bonding to metal centres *via* Se donor atoms.

Supplementary Data

Full lists of crystallographic data are available from the authors upon request.

Acknowledgements

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