Oxidative addition of (PhSe)₂ and (FcSe)₂ to zerovalent palladium and platinum trialkylphosphine complexes (Fc = ferrocenyl, [Fe(η⁵-C₅H₅)(η⁵-C₅H₄)])

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Abstract

Room temperature reaction of [Pd₂(dba)₃]/PR₃ or [Pt(C₂H₄)(PR₃)₂] (dba = dibenzylideneacetone; R = Et, Bu) with the diselenides (R′Se)₂ (R′ = Ph, Fc) yielded the oxidative addition products *trans*-[M(SeR′)₂(PR₃)₂] (M = Pd, Pt). These have been characterised by multinuclear NMR and UV–Vis spectroscopy, mass spectrometry, and, in the cases of *trans*-[Pt(SePh)₂(PR₃)₂] (R = Et, Bu) and *trans*-[Pt(SeFc)₂(PBu₃)₂], also by X-ray crystallography.
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1. Introduction

The chemistry of selenium-containing ligands is a subject of growing interest [1], as a result of both their increasing accessibility, and the realisation that they may display significantly different properties from their sulfur-containing analogues. The reactions of low-valent palladium and platinum complexes with simple diaryl diselenides have been fairly well studied, with the focus having been on triphenylphosphine derivatives. In 1982, Day and Rauchfuss reported the reaction of [Pt(C₂H₄)(PPh₃)₂] with diphenyl diselenide, which gave *trans*-[Pt(SePh)₂(PPh₃)₂] [2]. Laitinen and Ananikov later showed that the same product was formed when [Pt(PPh₃)₄] was used [3,4], and also that [Pd(PPh₃)₄] gave the dinuclear compound [Pd₂(μ₂-SePh)₂-(SePh)₂(PPh₃)₂] (Eq. (1)). Similar trends were observed in the reaction of *bis*(2-thienyl) diselenide with [M(PPh₃)₄]

(M = Pd, Pt) [5], with the products being [Pd₂(μ₂-SeTh)₂-(SeTh)₂(PPh₃)₂] (Th = 2-thienyl) and *trans*-[Pt(SeTh)₂-(PPh₃)₂]. Laitinen also showed that in the reaction of [Pd(PPh₃)₄] with (ThSe)₂ in dichloromethane, insertion into the Se–C bond is possible giving [PdCl(Th)(PPh₃)₂].

$$\tag{1}$$
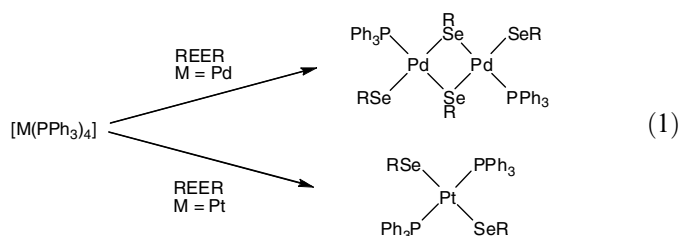
There are some reports of the reactions of ferrocenylselenolates with palladium and platinum complexes [6,7], but the only example of oxidative addition to (FcSe)₂ (Fc = ferrocenyl, [Fe(η⁵-C₅H₅)(η⁵-C₅H₄)]) concerns a ruthenium complex [8]. As part of our ongoing interest in transition metal complexes of redox-active ligands [9], we have therefore investigated the viability of this route for the synthesis of palladium(II) and platinum(II) derivatives.

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2. Results and discussion

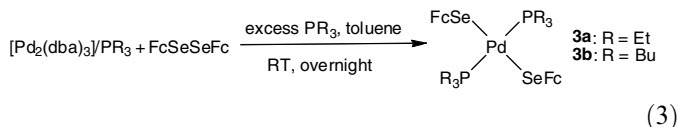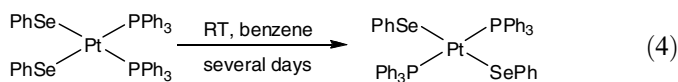Stirring $[Pt(C_2H_4)(PR_3)_2]$ (R = Et, Bu) with $(R'Se)_2$ ($R' = Ph$, Fc) in a mixture of 1,4-dioxane, THF and toluene at room temperature overnight, and subsequent column chromatography, led to the isolation of the compounds **1a,b** (yellow), **2a,b** (orange), as shown in Eq. (2).

$$
\begin{array}{c}
\text{PhSeSePh} \\
\nearrow \\
[Pt(C_2H_4)(PR_3)_2] \xrightarrow[\text{RT, overnight}]{\text{1,4-dioxane/THF/toluene}} \\
\searrow \\
\text{FcSeSeFc}
\end{array}
$$

1a: R = Et
1b: R = Bu

2a: R = Et
2b: R = Bu

(2)
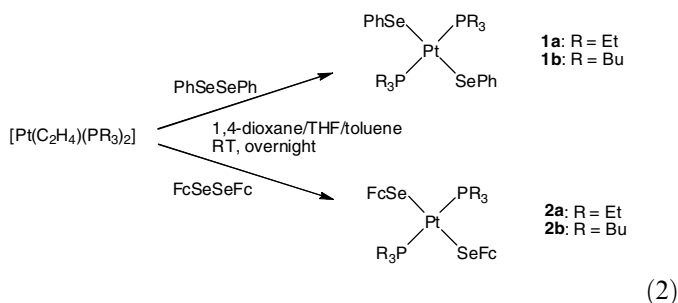
Similarly, treatment of a mixture of $[Pd_2(dba)_3]\cdot dba$ (dba = dibenzylideneacetone) and excess PR_3 with $(FcSe)_2$ in toluene at room temperature overnight, followed by precipitation with hexane and filtration, led to the isolation of the compounds **3a** (red), **3b** (orange), as shown in Eq. (3). Use of a Pd:P ratio of 1:1 yielded a complex mixture of products, rather than the expected dinuclear species $[Pd_2(\mu_2\text{-SeFc})_2(SeFc)_2(PR_3)_2]$. Since *trans*-$[Pd(SePh)_2(P\text{-}Bu_3)_2]$ has already been synthesised [10], reactions with $(PhSe)_2$ were not pursued.

$$
[Pd_2(dba)_3]/PR_3 + FcSeSeFc \xrightarrow[\text{RT, overnight}]{\text{excess } PR_3, \text{ toluene}}
$$

3a: R = Et
3b: R = Bu

(3)

Exclusive isolation of the *trans*-isomers of $[M(SeR')_2(PR_3)_2]$ is attributed to steric effects, as the bulky phosphine substituents are further apart than in the *cis*-isomers. It is possible that the *cis*-isomers are formed initially and subsequently isomerise to the *trans*-geometry. Jain et al. have reported that the reaction of $[Pt(C_2H_4)(PPh_3)_2]$ with diphenyl diselenide produces *cis*-$[Pt(SePh)_2(PPh_3)_2]$, which then isomerises to *trans*-$[Pt(SePh)_2(PPh_3)_2]$ at room temperature in solution (Eq. (4)) [11]. This isomerisation has also been reported for *cis*-$[Pt(SePh)_2(PPh_3)_2]$ generated from the reaction of *cis*-$[PtCl_2(PPh_3)_2]$ with NaSePh [11–13].

$$
\begin{array}{c}
\text{PhSe} \quad \text{PPh}_3 \\
\diagdown \text{Pt} \diagup \\
\text{PhSe} \quad \text{PPh}_3
\end{array}
\xrightarrow[\text{several days}]{\text{RT, benzene}}
\begin{array}{c}
\text{PhSe} \quad \text{PPh}_3 \\
\diagdown \text{Pt} \diagup \\
\text{Ph}_3\text{P} \quad \text{SePh}
\end{array}
$$

(4)

The crystal structures of complexes **1a**, **1b** and **2b** have been determined by X-ray diffraction (see Figs. 1–3). Two stereoisomers (*syn*- and *anti*-) are present in equal amounts in the crystal of **1a**. The other two compounds crystallise solely as the *anti*-isomer. The crystallographic data are presented in Table 1, and selected bond lengths and angles are listed in Table 2.

The asymmetric units of complexes **1a**, **1b** and **2b** contain only half-molecules, so the $PtSe_2P_2$ core is necessarily symmetric in each case: the **1b** and **2b** molecules, as well as
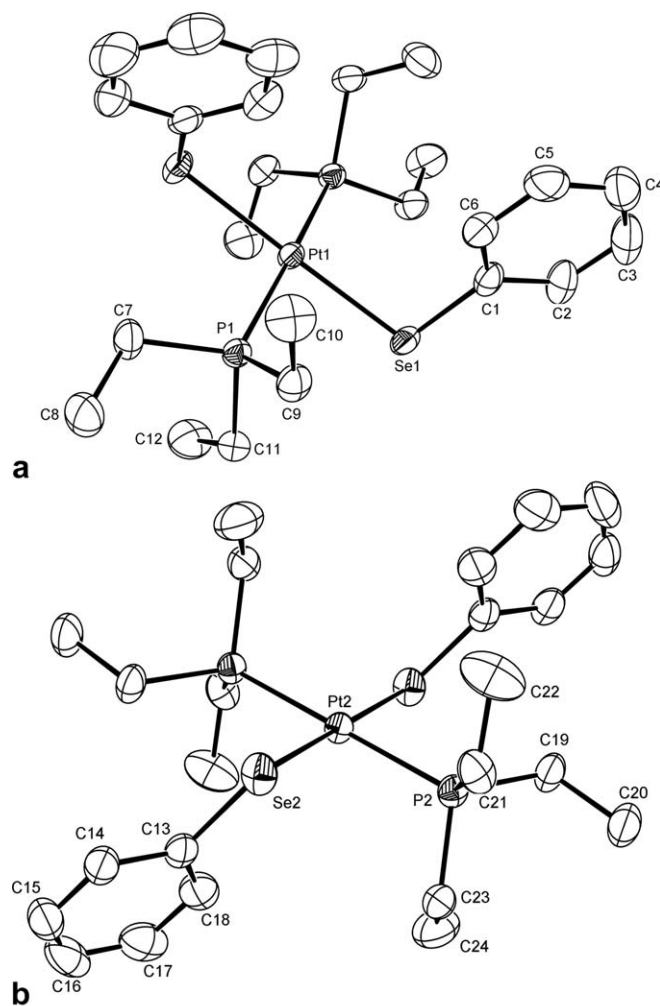
Fig. 1. Molecular structure of **1a**; *syn* (a) and *anti* (b) forms are present in equal amounts in the crystal. Thermal ellipsoids are shown at the 30% probability level and hydrogen atoms are omitted for clarity. Symmetry-independent atoms are labelled.

Fig. 2. Molecular structure of **1b**. Thermal ellipsoids are shown at the 30% probability level and hydrogen atoms are omitted for clarity. Symmetry-independent atoms are labelled.
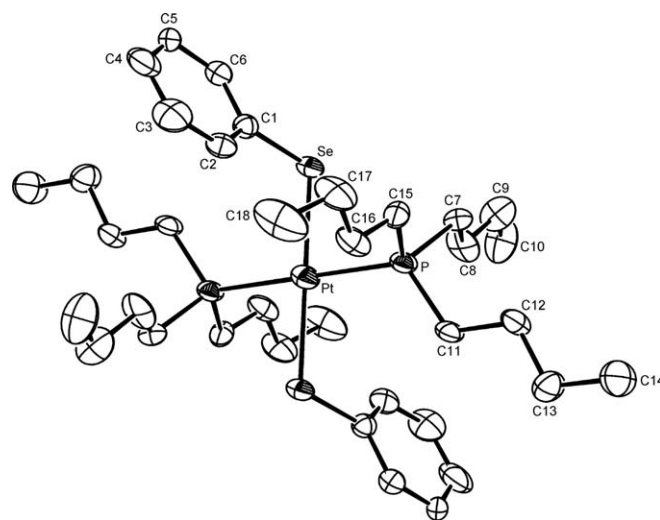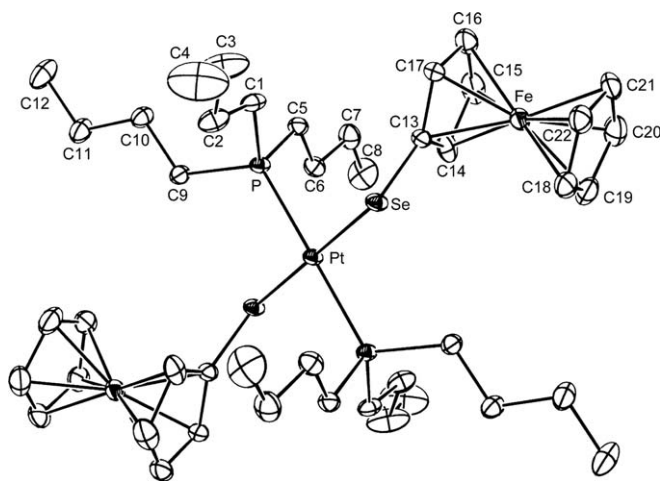
Fig. 3. Molecular structure of **2b**. Thermal ellipsoids are shown at the 30% probability level and hydrogen atoms are omitted for clarity. Symmetry-independent atoms are labelled.

the *anti*-stereoisomer in the unit cell of **1a**, are centrosymmetric, whereas the *syn*-isomeric molecule of **1a** possesses two-fold rotational symmetry. The Pt–Se bond lengths are almost the same in the three complexes (Table 2). They are longer than those (avg. 2.418 Å) found with a room temperature structure determination on the related compound *trans*-[Pt(SePh)$_2$(PPh$_3$)$_2$] [11], but they are similar to those (avg. 2.454 Å) found [12] for the same compound at 120 K and are also close to those observed in the thienyl derivative *trans*-[Pt(SeTh)$_2$(PPh$_3$)$_2$] (avg. 2.464 Å) [5] and in the centrosymmetric molecule of the furyl derivative *trans*-[Pt(SeFu)$_2$(PPh$_3$)$_2$] (2.454(1) Å) [13]. The Pt–P bond

lengths of **1a**, **1b** and **2b**, similar to each other (Table 2), are again slightly longer than those found for *trans*-[Pt(SePh)$_2$(PPh$_3$)$_2$] at room temperature (avg. 2.29 Å) [11], but closely agree with those (avg. 2.319 Å) found with the low-temperature study on the same compound [12] as well as with those of the thienyl (avg. 2.315 Å) [5] and furyl (2.306(1) Å) [13] derivatives. The present compounds exhibit values of their Se–Pt–P (acute) angles in the range 86.3–88.4°, with supplementary values for the adjacent obtuse angles in the centrosymmetric species (this is also substantially true for the non-centrosymmetric *syn*-stereoisomer of **1a**). There is broad agreement with the values of the same angles in the above *trans*-[Pt(SePh)$_2$(PPh$_3$)$_2$] compound (85.5° and 94.6°, averages of the two acute and obtuse angles, respectively) [12] and with those of the thienyl (avg. 83.7° and 96.3°) [5] and furyl derivatives, the acute angle in the centrosymmetric core of the latter measuring

Table 2
Selected bond lengths and angles for compounds **1a**, **1b** and **2b**

Compound	**1a** (*syn*)	**1a** (*anti*)	**1b**	**2b**
Bond lengths (Å)				
Pt–Se	2.461(1)	2.452(1)	2.463(3)	2.463(1)
Pt–P	2.311(2)	2.312(2)	2.311(6)	2.316(1)
Se–C	1.906(9)	1.893(9)	1.89(2)	1.902(5)
Bond angles (°)				
Se–Pt–P	86.33(5)	86.29(5)	86.61(16)	88.44(3)
Se–Pt–P′	93.79(5)	93.71(5)	93.39(16)	91.56(3)
P–Pt–P′	172.20(10)	180	180	180
Se–Pt–Se′	178.15(5)	180	180	180
Pt–Se–C	101.8(2)	104.6(3)	104.8(7)	102.74(14)

Table 1
Crystallographic data for compounds **1a**, **1b** and **2b**

Compound	**1a**	**1b**	**2b**
Empirical formula	C$_{24}$H$_{40}$P$_2$PtSe$_2$	C$_{18}$H$_{32}$PPt$_{0.5}$Se	C$_{22}$H$_{36}$FePPt$_{0.5}$Se
Formula weight	743.51	455.91	563.83
Crystal system	Monoclinic	Triclinic	Triclinic
Space group	C2/c	P1̄	P1̄
a (Å)	15.533(2)	8.483(6)	10.577(2)
b (Å)	9.168(1)	10.335(7)	10.645(2)
c (Å)	39.852(3)	12.246(9)	10.866(2)
α (°)	90.00	77.63(6)	99.95(1)
β (°)	97.76(1)	85.64(6)	100.99(1)
γ (°)	90.00	83.43(6)	91.74(1)
V (Å)	5623.2(9)	1040.3(13)	1180.4(4)
Z	8	2	2
F(000)	2880	456	564
D_{calc} (g cm^{-3})	1.756	1.455	1.586
μ (Mo Kα) (mm^{-1})	7.70	5.22	5.20
Temperature (K)	293	293	293
Reflections collected	33 505	5961	5733
Independent reflections [R_{int}]	5744 [0.049]	1610 [0.138]	4451 [0.024]
θ Range (°)	4.17–26.37	4.32–18.84	4.42–26.02
Reflections with $I > 2\sigma(I)$	4641	964	3969
No. of parameters; restraints	271; 0	187; 6	233; 0
R_1; wR_2 [$I > 2\sigma(I)$]	0.0466; 0.0835	0.0717; 0.1648	0.0309; 0.0769
R_1; wR_2 (all data)	0.0585; 0.0897	0.0918; 0.1797	0.0345; 0.0796
GoF	1.126	0.931	1.108

83.74(2)° [13]. It is also notable that the structural parameters for the two stereoisomers of **1a** differ only slightly.

The NMR spectroscopic data (Tables 3 and 4) are in accord with the proposed structures, assuming rapid interconversion in solution of *syn-* and *anti-*isomers (via inversion at selenium or rotation about the metal–selenium bond), as is usual for monodentate selenolates. Unsurprisingly, the ^{77}Se NMR chemical shift, and the coupling constants $^1J(^{195}Pt–^{77}Se)$, $^2J(^{77}Se–^{31}P)$ are predominantly determined by the nature of the selenium substituent, with the phosphine having a much smaller effect. Neglecting the

coupling to ^{195}Pt, the ^{77}Se resonances of compounds **1a,b**, **2a,b** and **3a,b** are triplets, as a result of coupling to two chemically and magnetically equivalent ^{31}P nuclei.

Compounds **1a,b**, **2a,b** and **3a,b** have been further characterised by FAB mass spectrometry and UV–Vis spectroscopy, and the data are shown in Table 5. The molecular ion was observed in each case. In contrast to what we have observed in the case of neutral ferrocenylselenide ligands [9], there is little difference between the UV–Vis spectra, and hence the colours, of the analogous palladium and platinum ferrocenylselenolates. Experimental and theoreti-

Table 3
^{1}H and ^{13}C NMR spectroscopic data for **1a,b**, **2a,b**, **3a,b** in d_6-benzene solution

Compound		1a	1b	2a	2b	3a	3b
$\delta(^1H)$	CH_3	0.90 (18 H, dt)[a]	0.84 (18 H, t)[b]	1.00 (18 H, dt)[a]	0.96 (18 H, t)[b]	0.99 (18 H, dt)[a]	1.07 (18 H, t)[b]
	CH_2P	1.94–2.05 (12 H, m)	1.20–1.30 (12 H, m)	1.83–1.97 (12 H, m)	1.36–1.48 (12 H, m)	1.80–1.89 (12 H, m)	1.37–1.50 (12 H, m)
	$CH_3CH_2CH_2$		1.42–1.53 (12 H, m)		1.48–1.60 (12 H, m)		1.46–1.58 (12 H, m)
	CH_2CH_2P		2.04–2.14 (12 H, m)		1.97–2.06 (12 H, m)		1.91–1.99 (12 H, m)
	C_6H_5 (o/p)	6.94–7.07 (6 H, m)	6.93–7.08 (6 H, m)				
	C_6H_5 (m)	7.97–8.02 (4 H, m)	7.99–8.03 (4 H, m)				
	C_5H_4			3.96–3.99 (4 H, m)	3.98–4.02 (4 H, m)	3.97–4.00 (4 H, m)	3.98–4.01 (4 H, m)
				4.48–4.51 (4 H, m)	4.52–4.55 (4 H, m)	4.48–4.51 (4 H, m)	4.51–4.54 (4 H, m)
	C_5H_5			4.19 (10 H, s)	4.24 (10 H, s)	4.22 (10 H, s)	4.25 (10 H, s)
$\delta(^{13}C)$	CH_3	8.2[c]	14.0	8.8[c]	14.3	8.9[c]	14.2
	CH_2P	14.5[d]	23.6[d]	15.3[d]	23.0[d]	15.9[d]	24.0[d]
	$CH_3CH_2CH_2$		27.0		27.2		27.6
	CH_2CH_2P		24.6[e]		24.8[e]		25.0[e]
	i-C_6H_5	133.7[g]	134.1[g]				
	o-C_6H_5	139.4[h]	139.7[h]				
	m-C_6H_5	127.9	128.1				
	p-C_6H_5	123.4	123.6				
	C_5H_4			78.9[i]	78.1[i]	79.4[i]	79.5[i]
				75.3	75.7	75.5	76.1
				67.6	68.3	67.5	67.9
	C_5H_5			70.4	70.4	70.2	70.5

[a] $^3J(^1H–^1H) = 7.6$ Hz (**1a, 2a, 3a**), $^3J(^{31}P–^1H) = 16.0$ Hz (**1a, 2a**), 16.4 Hz (**3a**).
[b] $^3J(^1H–^1H) = 7.2$ Hz (**1b, 3b**), 7.6 Hz (**2b**).
[c] $^3J(^{195}Pt–^{13}C) = 19$ Hz (**1a**), 21 Hz (**2a**), not resolved (**3a**).
[d] virtual triplet, avg. $J(^{31}P–^{13}C) = 18$ Hz (**1a**), 17 Hz (**1b, 2a, 2b**), 14 Hz (**3a**), 13 Hz (**3b**).
[e] virtual triplet, avg. $J(^{31}P–^{13}C) = 7$ Hz (**1b, 2b, 3b**).
[g] $^2J(^{195}Pt–^{13}C) = 38$ Hz (**1a**), 37 Hz (**1b**).
[h] $^3J(^{195}Pt–^{13}C) = 26$ Hz (**1a**), not resolved (**1b**).
[i] $^3J(^{31}P–^{13}C) = $ not resolved (**2a**), 2 Hz (**2b, 3a**), 3 Hz (**3b**).

Table 4
^{31}P and ^{77}Se NMR spectroscopic data for **1a,b**, **2a,b** and **3a,b** in d_6-benzene solution

	Compound	1a	1b	2a	2b	3a	3b
^{31}P	δ (ppm)	19.7	12.6	19.4	11.7	22.4	14.5
	$^1J(^{195}Pt–^{31}P)$ (Hz)	2479	2469	2610	2605	–	–
^{77}Se	δ (ppm)	78	82	−47	−38	−20	−12
	$^1J(^{195}Pt–^{77}Se)$ (Hz)	106	98	225	235	–	–
	$^2J(^{77}Se–^{31}P)$ (Hz)	7	6	11	12	6	8

Table 5
Mass spectral and UV–Vis data for compounds **1a,b**, **2a,b** and **3a,b**

Compound		1a	1b	2a	2b	3a	3b
Mass spectrum[a]	$[M]^+$	745 (30)	913 (15)	961 (10)	1129 (10)	872 (3)	1040 (3)
	$[M-SeR']^+$	588 (100)	756 (40)	696 (20)	864 (60)	607 (25)	775 (40)
	$[Pd/Pt(PR_3)_2]^+$		599 (100)		599 (100)		510 (45)
UV–Vis[b]		215 (60 000)		215 (54 000)		215 (41 200)	215 (35 000)
		225 (42 500)	230 (47 000)	225 (49 000)	225 (41 500)	220 (33 700)	225 (23 600)
		250 (38 000)	250 (34 000)	255 (45 000)	255 (34 700)	250 (26 000)	250 (27 600)
		295 (32 500)	295 (31 200)	310sh (19 600)	315sh (15 900)	335 (18 500)	335 (60 000)
		350sh (4400)	345sh (5800)			395sh (5000)	400sh (12 300)
				465sh (2120)	460sh (2650)	470sh (2300)	470sh (8700)

[a] m/z (%); recorded using FAB; figures are for isotopomers containing ^{106}Pd or ^{195}Pt, ^{80}Se, ^{56}Fe, ^{12}C, ^{1}H; expected isotope patterns were observed.

[b] λ_{max} (nm); ε (cm^{-1} M^{-1}) in parentheses; recorded in CH$_2$Cl$_2$ solution (2×10^{-5} M); sh = shoulder.

cal investigations of the related complexes [MCl-(ECHR'CHR'NMe$_2$)(PR$_3$)] (M = Pd or Pt; E = S, Se or Te; R' = H or Me; R$_3$ = Me$_2$Ph, etc.) [14,15] have shown that their highest wavelength absorption bands are ligand (E) to ligand (PR$_3$) charge transfer in character, and a similar assignment may be appropriate here. We now intend to investigate the electrochemistry of these complexes, in the expectation that in **2a,b** and **3a,b** there will be electronic communication between the two ferrocenyl groups.

3. Experimental

All reactions were performed using standard Schlenk techniques under an atmosphere of dry argon. 1,4-Dioxane and toluene were refluxed over sodium and distilled under argon before use; THF was refluxed over potassium and distilled under argon before use. ^{1}H and ^{13}C NMR spectra were recorded using a Bruker AC400 with tetramethylsilane as internal standard. ^{31}P NMR spectra were recorded on the same instrument with 85% phosphoric acid as external standard. ^{77}Se NMR spectra were recorded on a Bruker WM250 with dimethylselenide as external standard. UV–Vis spectra were recorded on a Unicam UV300 spectrometer. Mass spectra were recorded by the EPSRC mass spectrometry centre using fast atom bombardment (FAB). [PtCl$_2$(PEt$_3$)$_2$] [16], [PtCl$_2$(PBu$_3$)$_2$] [17], [Pd$_2$(dba)$_3$]·dba [18], (PhSe)$_2$ [19] and (FcSe)$_2$ [20] were synthesised by literature procedures (slightly adapted in some cases); PEt$_3$ (Strem) and PBu$_3$ (Aldrich) were used as supplied.

3.1. Synthesis of compounds 1a,b and 2a,b

Experiments were carried out according to the following general procedure. The preparation of [Pt(C$_2$H$_4$)(PR$_3$)$_2$] is based on the method described in the literature [21]. Under argon [PtCl$_2$(PR$_3$)$_2$] (0.20 g (R = Et) or 0.27 g (R = Bu), 0.4 mmol) was taken up in 1,4-dioxane (20 mL) and the solution degassed. An ethene atmosphere was introduced, and the solution treated dropwise with NaC$_{10}$H$_8$ (∼0.12 M solution in THF), until the green colour held for approximately 2–3 min before clearing. The ethene atmosphere was replaced with argon, and a solution of

the diselenide ((PhSe)$_2$, 0.13 g, or (FcSe)$_2$, 0.21 g; 0.4 mmol) in degassed toluene (8 mL) was added. After stirring at room temperature overnight, concentration *in vacuo* gave a yellow or orange solid, which was purified by column chromatography on alumina with toluene/hexane elution. Collection of the coloured band and recrystallisation at −10 °C from toluene/hexane gave **1a** (yellow, 0.11 g, 38% yield), **1b** (yellow, 0.14 g, 38% yield), **2a** (orange, 0.12 g, 32% yield) and **2b** (orange, 0.16 g, 35% yield). Yields are based on [PtCl$_2$(PR$_3$)$_2$]; conversion to [Pt(C$_2$H$_4$)(PR$_3$)$_2$] may not be 100%. Spectroscopic and analytical data are summarised in Tables 3–5.

3.2. Synthesis of compounds 3a,b

Under argon [Pd$_2$(dba)$_3$]·dba (0.06 g, 0.05 mmol) was taken up in dry toluene (5 mL). The resulting purple solution was treated with PR$_3$ (0.1 mL) and the reaction mixture allowed to stir for 5 min, after which time it was brown/yellow in colour. (FcSe)$_2$ (0.05 g, 0.1 mmol) was then added. After stirring at room temperature overnight, concentration *in vacuo* followed by treatment with hexane resulted in the precipitation of brown/orange solids. Filtration followed by recrystallisation from toluene/hexane at −10 °C gave **3a** (orange, 0.05 g, 57% yield) and **3b** (red, 0.03 g, 33% yield). Spectroscopic and analytical data are summarised in Tables 3–5.

3.3. X-ray crystallography

Data collections were performed at room temperature with an Oxford Diffraction Xcalibur 3 CCD diffractometer and using graphite-monochromated Mo Kα radiation (λ = 0.71069 Å). Intensity data sets were corrected for absorption by a multi-scan procedure [22]. Crystals of **1b** diffracted poorly, yielding relatively low-angle reflections. The structures were solved by direct methods, with SIR-97 [23], and refined with SHELXL-97 [24]. In the final refinement cycles, all non-hydrogen atoms were assigned anisotropic temperature factors and the hydrogens were in geometrically generated positions, riding, each with a temperature factor linked to the isotropic equivalent of that of the

respective carrier atom. Small corrections for extinction were applied to the data of **1a** and **2b**. For graphics ORTEP-3 was employed [25]. Crystal data, data collection parameters and analysis statistics are summarised in Table 1. Crystallographic data (excluding structure factors) for the structures of **1a**, **1b** and **2b** have been deposited with the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk; www: http://www.ccdc.cam.ac.uk), and are available on request quoting the corresponding deposition numbers CCDC 608084, 608085 and 608086 (in the above order).

Acknowledgements

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