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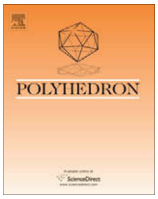

Preparation and compounds of (8-methoxynaphth-1-yl)diphenylphosphine

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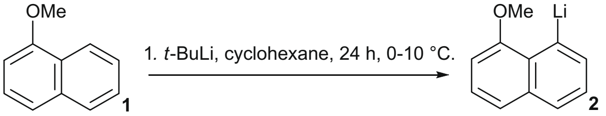
ABSTRACT

Reaction of 8-methoxy-1-naphthyllithium (**2**) with one equivalent of chlorodiphenylphosphine gives the novel (8-methoxynaphth-1-yl)diphenylphosphine (**3**) which was oxidised to the corresponding sulfide (**4**) and selenide (**5**) by reaction with sulfur and selenium, respectively. The P···O peri distance is significantly increased in **4** and **5** at 2.819(3) [2.793(3)] and 2.827(3) [2.806(3)] Å [values in square brackets are for the second independent molecules in each case] relative to the value in **3** of 2.678(2) Å. There are short E···O interactions ie O(1)···S(1) 3.165(3) [3.124(3)], O(1)···Se(1) 3.247(3) [3.200(2)] Å and these may be repulsive and responsible for the increased P···O separation. The use of the naphthalene backbone in the synthesis of a potential hemilabile ligand is demonstrated by the synthesis and X-ray structure of (8-methoxynaphth-1-yl)diethoxyphosphine ruthenium *p*-cymene dichloride (**6**).

Introduction

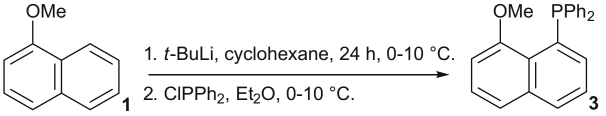
A number of groups have been investigating *peri* substituted naphthalenes in efforts to understand the factors which influence the degree of distortion [1–11]. The majority of the examples involve two heavy atoms at the 1,8 positions but here we demonstrate the facile synthesis of a new P,O system which may be of value as a hemilabile ligand [12–23].

Lithiation of 1-methoxynaphthalene **1** with *t*-butyllithium in anhydrous cyclohexane to produce 8-methoxy-1-naphthyllithium (**2**) was reported by Shirley and Cheng in 1969 (Eq. (1)) [24,25].



(1)

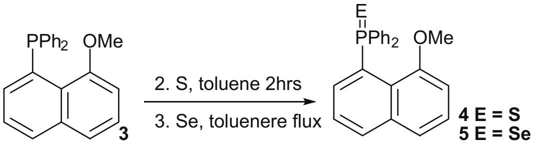
Reaction of 8-methoxy-1-naphthyllithium (**2**) with one equivalent of chlorodiphenylphosphine produced the novel compound (8-methoxynaphth-1-yl)diphenylphosphine (**3**) (Eq. (2)). This compound was characterised by elemental analysis, infra red spectroscopy, ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy and mass spectrometry. The ^{31}P NMR spectrum showed a singlet at $\delta = 2.44$ ppm.



(2)

The molecular structure of **3** (Fig. 1) shows an intramolecular non-bonded *peri*-distance between P(1) and O(1) of 2.678(2) Å. Selected bond lengths and bond angles are shown in Table 1.

In order to assess the impact of oxidation of the phosphorus centre upon the *peri* distance we treated **3** with sulfur or selenium to give P═S and P═Se, respectively (Eq. (3)). The ^{31}P NMR of **4** and **5** display singlets at δ_P 53.08 and δ_P 43.3 ($J_{P=Se(1)}$ 741.4 Hz) whilst for **5** $\delta_{Se} = -150.9$ (d, $J_{Se(1)=P}$ 741.4 Hz) in accord with the formulation as P═S and P═Se, respectively. In **4** $\nu_{P=S}$ is observed at 645 cm^{-1} whilst $\nu_{P=Se}$ in **5** is observed at 494 cm^{-1}.



(3)

The X-ray structures of **4** and **5** (Fig. 2 and Table 2) are isomorphous and both contain two independent molecules in the unit cell. Interestingly, the P···O peri distance is significantly increased in **4** and **5** at 2.819(3) [2.793(3)] and 2.827(3) [2.806(3)] Å [values in square brackets are for the second independent molecules in each case] relative to the value in **3** of 2.678(2) Å. Interestingly, there are short E···O interactions ie O(1)···S(1) 3.165(3) [3.124(3)], O(1)···Se(1) 3.247(3) [3.200(2)] Å and these may be repulsive and responsible for the increased P···O separation.

In order to test the ability of 1,8 P, O naphthalene derivatives to act as ligands we prepared a simple example of a metal complex **6** according to Eq. (4) without isolation of the ligand. Compound **6** was characterised spectroscopically and by X-ray crystallography (Fig. 3 and Table 3). The complex is essentially octahedral with monodentate P coordination (the Ru···O separation is 3.563(2) Å)

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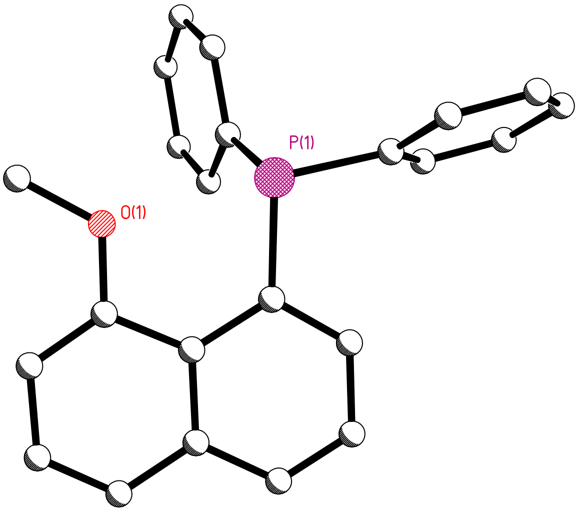
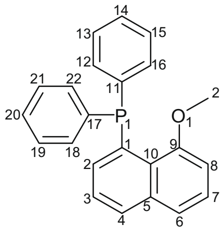


Fig. 1. The crystal structure of (8-methoxynaphth-1-yl)diphenylphosphine (**3**).

Table 1
Selected bond lengths (Å) and angles (°) for (8-methoxynaphth-1-yl)diphenylphosphine (**3**).



3

P(1)···O(1)	2.678(2)
P(1)–C(1)	1.860(3)
O(1)–C(9)	1.362(4)
O(1)–C(23)	1.433(5)
P(1)–C(1)–C(10)	122.8(2)
C(1)–C(10)–C(9)	124.6(3)
O(1)–C(9)–C(10)	114.3(2)
	$\Sigma = 361.7$
Distance from naphthalene mean plane	
P(1)	0.1197(37)
O(1)	0.0332(39)
Torsion angle	
C(6)–C(5)–C(10)–C(1)	179.5(2)
C(4)–C(5)–C(10)–C(9)	−178.1(2)

and like in **4** and **5** the substitution at phosphorus results in an enlarged P···O separation (2.875(3) Å) compared to **3**. This separation coupled with the sum of three internal angles of the C3PO 'ring' gives an indication of the degree of steric strain enforced upon the naphthalene system by the substitution at phosphorus.

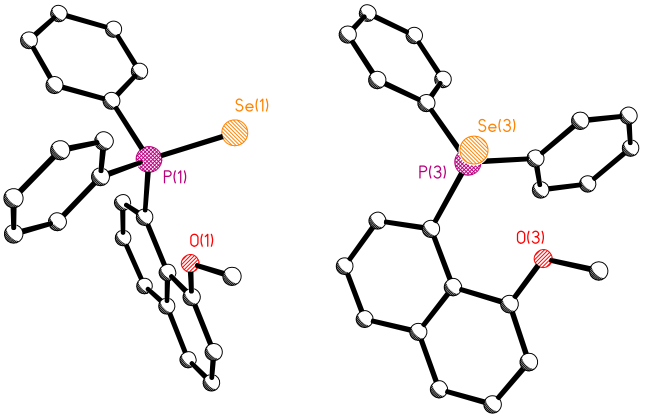


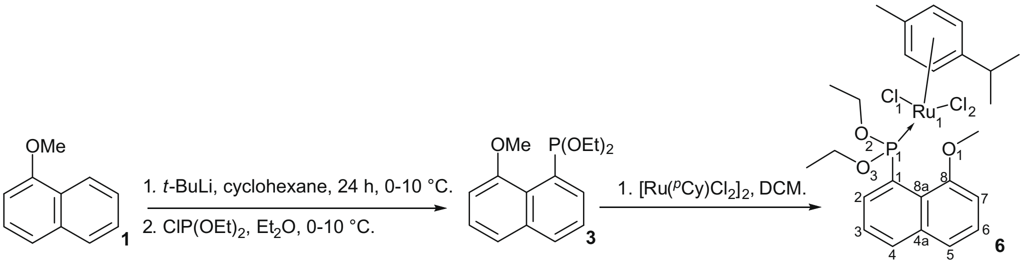
Fig. 2. The crystal structure of the two independent molecules of (8-methoxynaphth-1-yl)diphenylphosphine selenide (**5**), the sulfide (**4**) is isomorphous and is not illustrated.

Experimental

All experiments were carried out under an oxygen- and moisture-free nitrogen atmosphere using standard Schlenk techniques and glassware. Reagents were obtained from commercial sources and used as received. Dry solvents were collected from a MBraun solvent system. Elemental analyses were performed by the University of St. Andrews School of Chemistry Microanalysis Service. Infra-red spectra were recorded as KBr discs in the range 4000–300 cm^{-1} on a Perkin–Elmer System 2000 Fourier transform spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Jeol GSX 270 MHz spectrometer with δ(H) and δ(C) referenced to external tetramethylsilane. ^{31}P and ^{77}Se NMR spectra were recorded on a Jeol GSX 270 MHz spectrometer with δ(P) and δ(Se) referenced to external phosphoric acid and dimethylselenide, respectively. Assignments of ^{13}C and ^{1}H NMR spectra were made with the help of H–H COSY and HSQC experiments. All measurements were performed at 25 °C. All values reported for NMR spectroscopy are in parts per million (ppm). Coupling constants (*J*) are given in Hertz (Hz). Mass spectrometry was performed by the University of St. Andrews Mass Spectrometry Service. Electron impact mass spectrometry (EIMS) and Chemical Ionisation Mass Spectrometry (CIMS) was carried out on a Micromass GCT orthogonal acceleration time of flight mass spectrometer. Electrospray Mass Spectrometry (ESMS) was carried out on a Micromass LCT orthogonal accelerator time of flight mass spectrometer.

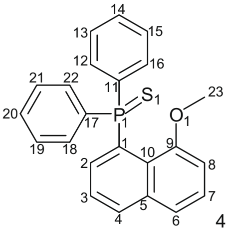
*(8-Methoxynaphth-1-yl)diphenylphosphine (**3**)*

tert-Butyllithium in pentane (22 mL, 1.5 M, 33 mmol, **CARE** this material is pyrophoric and should be handled with care) was added to a solution of 1-methoxynaphthalene (4.75 g, 30 mmol) in dry cyclohexane (60 mL) under a nitrogen atmosphere. The mixture

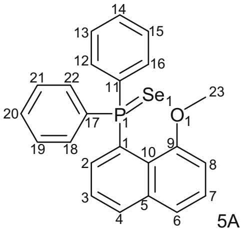


(4)

Table 2
Selected bond lengths (Å) and angles (°) for the independent molecules of (8-methoxynaphth-1-yl)diphenylphosphine sulfide (**4**) and selenide (**5**). A dash implies the deviation from the mean plane is less than the relevant esd ie it is not statistically variant from zero.



4A & 4B



5A & 5B

P(1)···O(1)	2.819(3)	2.793(3)	P(1)···O(1)	2.827(3)	2.806(3)
P(1)–C(1)	1.839(3)	1.832(4)	P(1)–C(1)	1.837(3)	1.837(3)
O(1)–C(9)	1.362(4)	1.353(5)	O(1)–C(9)	1.356(3)	1.354(4)
O(1)···S(1)	3.165(3)	3.124(3)	O(1)···Se(1)	3.247(3)	3.200(2)
O(1)–C(23)	1.427(4)	1.422(4)	O(1)–C(23)	1.427(4)	1.426(4)
S(1)=P(1)	1.9598(14)	1.9589(12)	Se(1)=P(1)	2.1175(9)	2.1157(8)
P(1)–C(1)–C(10)	125.4(2)	126.0(2)	P(1)–C(1)–C(10)	125.6(2)	125.6(2)
C(1)–C(10)–C(9)	125.4(3)	124.7(3)	C(1)–C(10)–C(9)	124.8(2)	125.2(3)
O(1)–C(9)–C(10)	115.5(3)	115.0(4)	O(1)–C(9)–C(10)	116.0(3)	115.1(3)
	Σ = 366.1	Σ = 365.7		Σ = 366.4	Σ = 365.9
Distance from naphthalene mean plane					
P(1)	−0.2149(47)	–	P(1)	–	–
O(1)	0.0381(49)	0.0839(50)	O(1)	0.0794(44)	0.0620(46)
S(1)	−1.9366(54)	1.6333(57)	Se(1)	–	1.8474(51)
Torsion angle					
C(6)–C(5)–C(10)–C(1)	−179.3(3)	176.6(3)	C(6)–C(5)–C(10)–C(1)	−179.8(3)	176.9(3)
C(4)–C(5)–C(10)–C(9)	179.4(3)	177.2(3)	C(4)–C(5)–C(10)–C(9)	178.6(3)	177.3(3)

was then stirred at room temperature for 24 h. 8-Lithio-1-methoxynaphthalene precipitated and was filtered off under nitrogen using a sintered glass filter attached to the reaction vessel. The solid was washed with anhydrous cyclohexane (40 mL), washed back into the vessel with anhydrous ether (100 mL) and the suspension cooled to −78 °C in a solid CO_2–acetone bath. To this solution was added chlorodiphenylphosphine (6.62 g, 5.4 mL, 30 mmol). The mixture was stirred for a further 24 h at room temperature. The solvent was removed in vacuo and hexane (40 mL) was added to precipitate out unwanted salts. The solution was filtered under nitrogen and the solvent removed in vacuo. The crude yellow oil obtained was recrystallised from hexane. Yield 4.63 g, (45%); (*Anal.* Calc. for $C_{23}H_{19}OP$: C, 80.70; H, 5.60. Found C, 78.81; H, 5.56%.); ν_{max} (KBr tablet)/cm^{-1}: 3406br s, 3075s, 2610w, 2156w, 1688s, 1584s, 1482w, 1437vs, 1311w, 1181vs, 1127vs, 1067s, 959vs, 857s, 755s, 727vs, 693vs, 553vs, 525s, 491w, 433vs, 329vs; δ_H (270 MHz, $CDCl_3$) 7.76 (1 H, d, *J* 8.2 Hz, nap 4-H), 7.49–7.43 (1 H, m, nap 5-H), 7.41–7.22 (12 H, m, nap 2,3-H, 2 × PPh_2 2-6-H), 6.92–6.85 (1 H, m, nap 6-H), 6.76 (1 H, dd, *J* 1.1 and 7.4 Hz, nap 7-H), 3.53 (3 H, s, $-OCH_3$); δ_C (67.9 MHz,

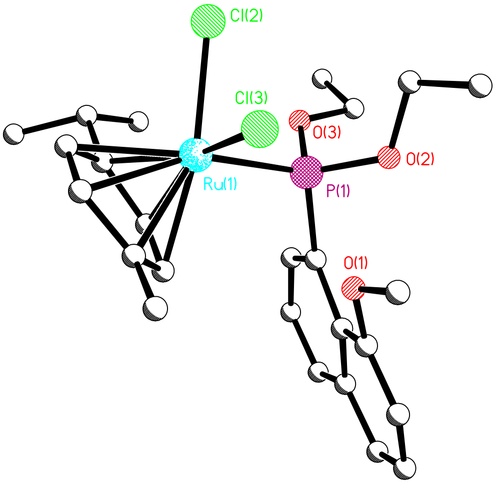
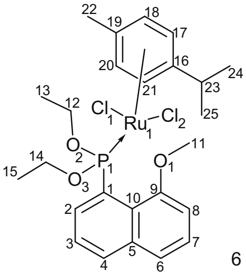


Fig. 3. The crystal structure of (8-methoxynaphth-1-yl)diethoxyphosphine ruthenium *p*-cymene dichloride (**6**).

Table 3
Selected bond lengths (Å) and angles (°) for (8-methoxynaphth-1-yl)diphenylphosphine ruthenium *p*-cymene dichloride (**6**).



6

P(1)···O(1)	2.875(3)	Ru(1)–P(1)	2.3272(12)
P(1)–C(1)	1.852(5)	Ru(1)–Cl(2)	2.4177(10)
O(1)–C(9)	1.382(7)	Ru(1)–Cl(3)	2.4081(15)
O(1)–C(23)		Ru···Centroid	1.722(1)
P(1)–C(1)–C(10)	128.5(4)	Cl(2)–Ru(1)–Cl(3)	86.39(4)
C(1)–C(10)–C(9)	124.6(5)	Cl(2)–Ru(1)–P(1)	88.49(3)
O(1)–C(9)–C(10)	115.0(4)	Cl(3)–Ru(1)–P(1)	93.43(5)
	Σ = 368.1		
Distance from naphthalene mean plane			
P(1)	−0.1455(65)		
O(1)	0.2516(70)		
Torsion angle			
C(6)–C(5)–C(10)–C(1)	174.0(4)		
C(4)–C(5)–C(10)–C(9)	173.7(4)		

$CDCl_3$) 134.0(d, *J* 19.7 Hz), 133.0(d, *J* 3.1 Hz), 129.2(s), 128.7(s), 128.6(d, *J* 7.3 Hz), 126.3(s), 126.0(s), 121.5(s), 106.3(s), 54.7(s); δ_P (109 MHz, $CDCl_3$) 2.81; *m/z* (ES$^+$) 343.12 ([M+H]$^+$, 50%).

$C_{23}H_{19}OP$, formula weight = 342.38, triclinic, space group $P\bar{1}$, $a = 8.8248(8)$, $b = 10.7137(10)$, $c = 11.1950(10)$ Å, $\alpha = 102.816(3)$, $\beta = 112.641(3)$, $\gamma = 103.520(3)°$, $U = 890.62(14)$ Å^3, $Z = 2$, $D_{Calc} = 1.277$ Mg m^{-3}, $\mu = 0.161$ mm^{-1}, $F(0\,0\,0) = 360$. Of 7630 measured data, 3138 were unique ($R_{int} = 0.047$) and 2463 observed ($I > 2\sigma(I)$]) to give $R_1 = 0.0631$ and $wR_2 = 0.1355$. CCDC 752700.

*(8-Methoxynaphth-1-yl)diphenylphosphine sulfide (**4**)*

To a solution of (8-methoxynaphth-1-yl)diphenylphosphine (0.317 g, 0.927 mmol) in toluene (10 mL) powdered sulfur (0.03 g, 0.927 mmol) was added in small batches at 0 °C. The mixture was stirred for ½ h at 0 °C and another 2 h at ambient temperature. The resulting cloudy solution was filtered, the solvent was removed in vacuo and the remaining oil vigorously stirred with hexane (10 mL) over night, which resulted in a thick suspension. The solid product was collected by filtration and recrystallised from dichloromethane/pentane. Yield 0.236 g, (68%); (Anal. Calc. for $C_{23}H_{19}OPS$: C, 73.78; H, 5.12. Found C, 73.10; H, 4.57%.); ν_{max} (KBr tablet)/cm^{-1}: 3053s, 2929s, 2829s, 2268w, 1960w, 1893s, 1826w, 1753w, 1683w, 1639w, 1613s, 1563vs, 1502vs, 1457vs, 1434vs, 1367vs, 1328vs, 1264vs, 1211s, 1186s, 1152s, 1116vs, 1051s, 1023s, 995vs, 911vs, 847w, 819vs, 763vs, 746vs, 716vs, 690vs, 654vs, 634vs, 595s, 553w, 517vs, 483s, 450s, 416vs; δ_H (270 MHz, $CDCl_3$) 7.96–7.88 (1 H, m, nap 4-H), 7.88–7.71 (4 H, m, 2 × P*Ph*$_2$ 2,6-H), 7.51–7.34 (8 H, m, nap 5,6-H, 2 × P*Ph*$_2$ 3-5-H), 7.34–7.22 (2 H, m, nap 2,3-H), 6.76 (1 H, dd, *J* 1.1 and 7.3 Hz, nap 7-H), 3.24 (3 H, s, –OC*H*$_3$); δ_C (67.9 MHz, $CDCl_3$) 135.4(d, *J* 11.4 Hz), 132.9(d, *J* 3.1 Hz), 131.6(d, *J* 10.4 Hz), 130.5(d, *J* 3.2 Hz), 128.2(d, *J* 12.4 Hz), 127.2(s), 125.1(d, *J* 14.6 Hz), 121.3(s), 107.1(s), 53.0(s); δ_P (109 MHz, $CDCl_3$) 53.08; *m/z* (ES$^+$) 343.07 ([M–S]$^+$, 100%).

$C_{23}H_{19}OPS$, formula weight = 374.44, triclinic, space group $P\bar{1}$, $a = 9.7972(12)$, $b = 11.7887(18)$, $c = 17.681(3)$ Å, $\alpha = 86.043(13)$, $\beta = 77.168(12)$, $\gamma = 71.227(10)°$, $U = 1885.1(5)$ Å^3, $Z = 2$, $D_{Calc} = 1.318$ Mg m^{-3}, $\mu = 0.265$ mm^{-1}, $F(0\,0\,0) = 784$. Of 19 723 measured data, 6507 were unique ($R_{int} = 0.073$) and 5702 observed ($I > 2\sigma(I)$]) to give $R_1 = 0.0788$ and $wR_2 = 0.1353$. CCDC 752701.

*(8-Methoxynaphth-1-yl)diphenylphosphine selenide (**5**)*

A solution of (8-methoxynaphth-1-yl)diphenylphosphine (7.363 g, 21.5 mmol) and Se (1.70 g, 21.5 mmol) were heated under reflux in toluene (20 mL) for 2 h. The resulting cloudy solution was filtered, the solvent was removed in vacuo and the remaining oil vigorously stirred with hexane (10 mL) over night, which resulted in a thick suspension. The solid product was collected by filtration and recrystallised from dichloromethane/pentane. Yield 7.17 g, (79%); (Anal. Calc. for $C_{23}H_{19}OPSe$: C, 65.57; H, 4.55. Found C, 64.67; H, 4.07%.); ν_{max} (KBr tablet)/cm^{-1}: 2930br s, 1968w, 1898w, 1825w, 1776w, 1674w, 1587w, 1572w, 1476s, 1433vs, 1386w, 1334w, 1308s, 1273w, 1180s, 1157s, 1107vs, 1067w, 1023s, 994w, 953w, 892vs, 779w, 744vs, 718s, 703vs, 686vs, 613w, 590w, 558vs, 494s, 468w, 445w, 392w, 361s, 323w; δ_H (270 MHz, $CDCl_3$) 7.95–7.70 (5 H, m, nap 4-H, 2 × P*Ph*$_2$ 2,6-H), 7.55–7.34 (8 H, m, nap 5,6-H, 2 × P*Ph*$_2$ 3-5-H), 7.34–7.15 (2 H, m, nap 2,3-H), 6.69 (1 H, dd, *J* 2.4 and 6.5 Hz, nap 7-H), 3.19 (3 H, s, –OC*H*$_3$); δ_C (67.9 MHz, $CDCl_3$) 135.5(d, *J* 11.5 Hz), 133.2(d, *J* 4.3 Hz), 132.5(d, *J* 10.0 Hz), 131.0(d, *J* 2.5 Hz), 128.6(d, *J* 12.5 Hz), 127.6(s), 125.4(d, *J* 14.3 Hz), 121.6(s), 107.5(s), 53.0(s); δ_P (109 MHz, $CDCl_3$) 43.32 ($J_{P=Se(1)}$ 741.4 Hz); δ_{Se} (51.5 MHz, $CDCl_3$) −150.9 (d, $J_{Se(1)=P}$ 741.4 Hz); *m/z* (CI$^+$) 423.04 ([M+H]$^+$, 100%).

$C_{23}H_{19}OPSe$, formula weight = 421.34, triclinic, space group $P\bar{1}$, $a = 9.8572(17)$, $b = 11.841(3)$, $c = 17.750(5)$ Å, $\alpha = 85.944(19)$, $\beta = 77.638(18)$, $\gamma = 71.454(16)°$, $U = 1918.6(8)$ Å^3, $Z = 4$, $D_{Calc} = 1.459$ Mg m^{-3}, $\mu = 2.049$ mm^{-1}, $F(0\,0\,0) = 856$. Of 21 068 measured data, 6645 were unique ($R_{int} = 0.037$) and 6211 observed ($I > 2\sigma(I)$]) to give $R_1 = 0.0386$ and $wR_2 = 0.1372$. CCDC 752702.

*Ruthenium complex **6***

tert-Butyllithium (1.7 M) in pentane (19.4 mL, 33 mmol, **CARE,** pyrophoric handle with caution) was added to a solution of 1-methoxynaphthalene (4.75 g, 30 mmol) in dry cyclohexane (60 mL). The solution was stirred at room temperature for 24 h. 8-Lithio-1-methoxynaphthalene precipitated and was filtered off under nitrogen using a sintered glass filter attached to the reaction vessel. The solid was washed with dry cyclohexane (40 mL), washed back into the reaction vessel with dry diethyl ether (100 mL) and the suspension was cooled to −78 °C. Chlorodiethoxyphosphine (30 mmol, 4.3 mL) was added dropwise to the suspension which was stirred overnight. The solvent was removed in vacuo and hexane (100 mL) was added to precipitate out unwanted salts. The solution was filtered under nitrogen and the solvent removed in vacuo. The crude ligand has $\delta_P = 7.3$ ppm. To a schlenk tube containing ligand (8-methoxynaphthyl-1-yl)diethoxyphosphine (0.299 g, 1.08 mmol) and $[Ru(^{p}cym)Cl_2]_2$ (0.329 g, 0.54 mmol) was added dichloromethane (5 mL). The reaction was stirred for 2 h. Removal of the solvent and recrystallisation of the red oil from dichloromethane/pentane gave red crystals. Yield 1.02 g (24%); (*Anal.* Calc. for $C_{25}H_{33}O_3PRuCl_2$: C, 51.40; H, 5.70. Found: C, 51.70; H, 5.49%.); ν_{max} (KBr tablet)/cm^{-1}: 3442br, 3032s, 2961s, 2925s, 2896s, 2824s, 2433w, 1943w, 1742w, 1615w, 1561s, 1502s, 1461vs, 1367vs, 1323s, 1261vs, 1201s, 1148s, 1116vs, 1092s, 1023vs, 941vs, 861s, 823s, 776vs, 760vs, 734s, 634s, 604w, 545vs, 519vs, 465s, 424s, 288s; δ_H (270 MHz, $CDCl_3$) 8.07-8.00 (1 H, m, nap 4-H), 7.93 (1 H, d, *J* 8.1 Hz, nap 2-H), 7.89 (1 H, d, *J* 8.2 Hz, nap 5-H), 7.59–7.53 (1 H, m, nap 3-H), 7.47–7.37 (4 H, m, nap 6,7-H, 2 × Ar-*H*), 6.95–6.89 (2 H, m, 2 x Ar-*H*), 4.16–4.07 (4 H, m, –P(OC*H*$_2$CH$_3$)$_2$), 3.97 (3 H, s, –OC*H*$_3$), 2.85 (1 H, q, *J* 6.9 Hz, Ar-C*H*(CH$_3$)$_2$), 1.84 (3 H, s, Ar-C*H*$_3$), 1.25–1.15 (6 H, m, –P(OCH$_2$C*H*$_3$)$_2$), 1.10 (6 H, d, *J* 7.0 Hz, Ar-CH(C*H*$_3$)$_2$); δ_C (67.9 MHz, $CDCl_3$) 136.0(d, *J* 6.2 Hz), 132.4(s), 131.0(d, *J* 8.3 Hz), 127.1(s), 124.7(d, *J* 10.4 Hz), 121.2(s), 106.9(s), 63.9(–P(O*C*H$_2$CH$_3$)$_2$), 57.9(–O*C*H$_3$), 30.1(Ar-*C*H(CH$_3$)$_2$), 21.7(Ar-CH(*C*H$_3$)$_2$), 17.9(Ar-*C*H$_3$), 16.1(–P(OCH$_2$*C*H$_3$)$_2$); δ_P (109 MHz, $CDCl_3$) 138.1; *m/z* (ES$^+$) 549.10 ([M–Cl]$^+$, 100%).

$C_{25}H_{33}O_3PCl_2Ru$, formula weight = 584.48, triclinic, space group $P\bar{1}$, $a = 9.916(4)$, $b = 10.349(4)$, $c = 13.298(7)$ Å, $\alpha = 88.26(3)$, $\beta = 89.46(3)$, $\gamma = 68.07(2)°$, $U = 1265.3(10)$ Å^3, $Z = 2$, $D_{Calc} = 1.534$ Mg m^{-3}, $\mu = 0.918$ mm^{-1}, $F(0\,0\,0) = 600$. Of 14 990 measured data, 4401 were unique ($R_{int} = 0.041$) and 4279 observed ($I > 2\sigma(I)$]) to give $R_1 = 0.0482$ and $wR_2 = 0.1398$. CCDC 752703.

Supplementary data

CCDC 752700–752703 contains the supplementary crystallographic data for **3**–**6**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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