Ring size effects in cyclic fluorophosphites: ligands that span the bonding space between phosphites and PF_3

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Table of Contents

1. Simulated spectra for *cis*-[Mo(CO)$_4$(L5)$_2$] (3a)

For *cis*-[Mo(CO)$_4$L$_2$] complexes, **3a**-**d**, the $^{31}P\{^1H\}$ NMR and $^{19}F\{^1H\}$ NMR spectra were simulated in Mestrenova using calculated *J* values from |K|, |L|, |M| and |N|, determined from Equations 1-4 (see Experimental for calculated values).

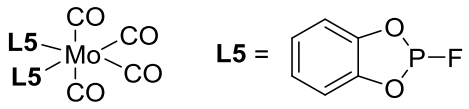
$$|K| = |J_{AA'} + J_{XX'}| \quad (1)$$

$$|L| = |J_{AX} - J_{AX'}| \quad (2)$$

$$|M| = |J_{AA'} - J_{XX'}| \quad (3)$$

$$|N| = |J_{AX} + J_{AX'}| \quad (4)$$

There is a good agreement between the observed and the simulated spectra for all complexes (see Figures S1 and S2 for complex **3a**), although at high resolution the fine structure may indicate that $^4J_{FF'}$ is in fact not equal to zero.[1]



3a

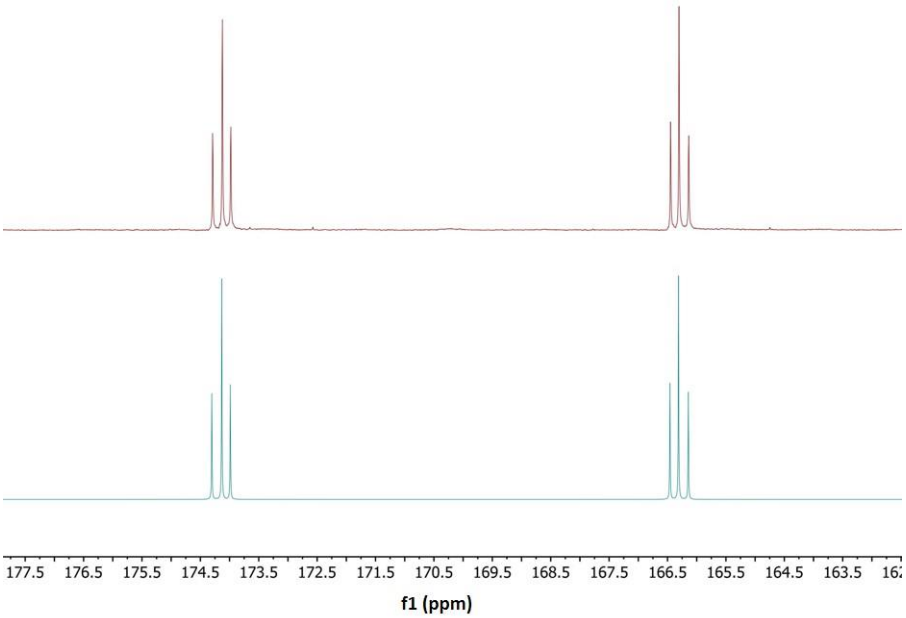


Figure S1 Top spectrum: Observed $^{31}P\{^1H\}$ NMR spectrum of complex **3a**. Bottom spectrum: Simulated $^{31}P\{^1H\}$ NMR spectrum of complex **3a** using calculated *J* values: $^1J_{P,F}$ = 1270.9 Hz, $^2J_{P,P}$ = 50.9 Hz, $^4J_{F,F'}$ = 0 Hz, $^3J_{P,F'}$ = -0.4 Hz.

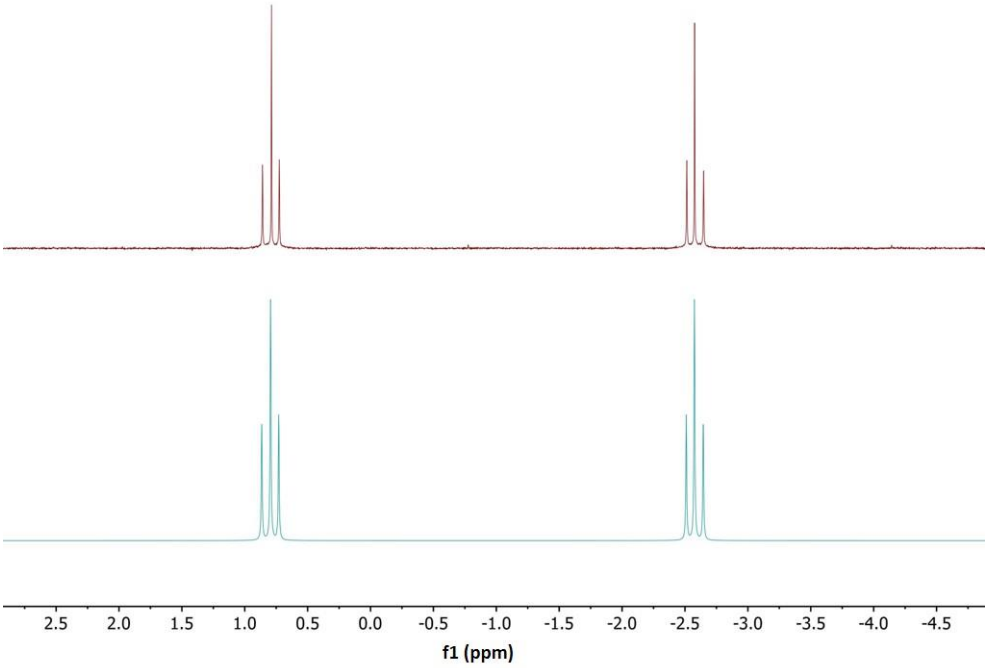


Figure S2 Top spectrum: Observed $^{19}F\{^{1}H\}$ NMR spectrum of complex **3a**. Bottom spectrum: Simulated $^{19}F\{^{1}H\}$ NMR spectrum of complex **3a** using calculated *J* values: $^{1}J_{F,P}$ = 1270.9 Hz, $^{2}J_{P,P}$ = 50.9 Hz, $^{4}J_{F,F'}$ = 0 Hz, $^{3}J_{F',P}$ = -0.4 Hz.

2. Reaction between ligands and [Pt(nbe)$_3$] in a 2:1 stoichiometry

Below are the *in situ* $^{31}P\{^1H\}$, $^{19}F\{^1H\}$ and $^{195}Pt\{^1H\}$ NMR spectra observed upon reaction between ligands **L5**-**8** and **L6a** with [Pt(nbe)$_3$] in a 2:1 stoichiometry.

2.1 Reaction between L5 and [Pt(nbe)$_3$] in a 2:1 stoichiometry

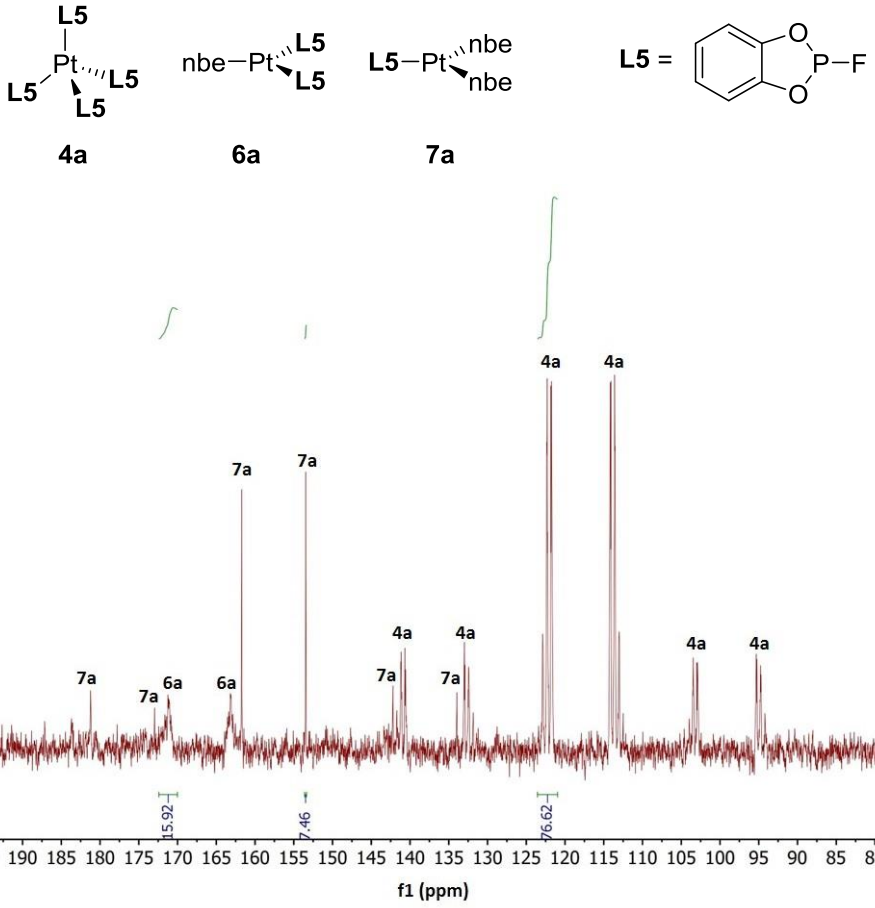


Figure S3 *In situ* $^{31}P\{^1H\}$ NMR spectrum observed when using **L5**. Complexes **4a**, **6a** and **7a** have been labelled.

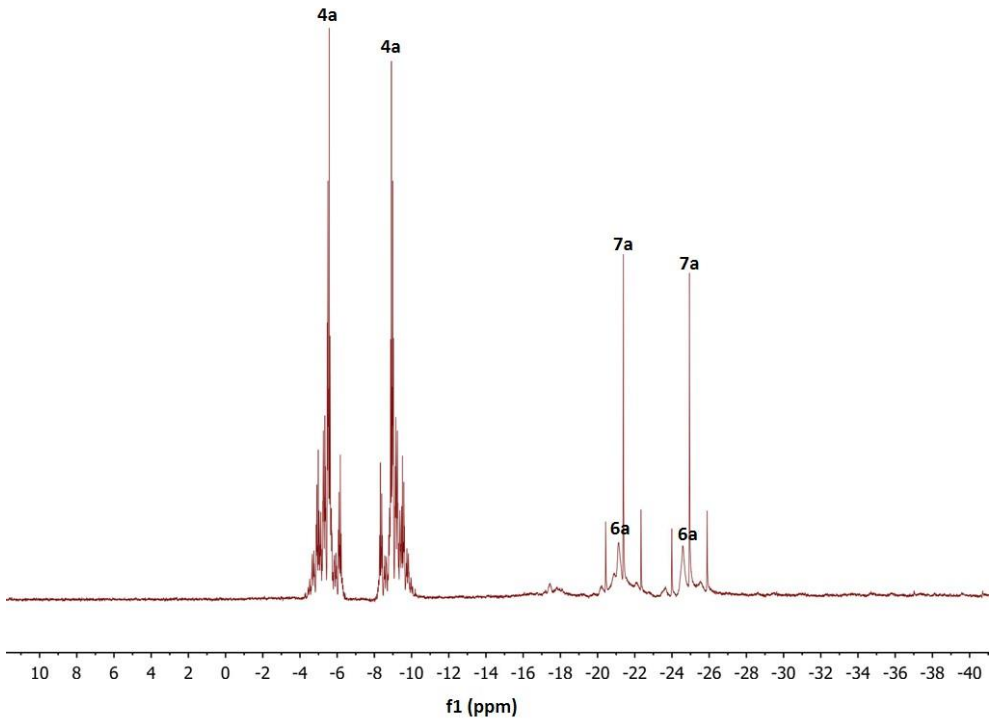


Figure S4 *In situ* $^{19}F\{^{1}H\}$ NMR spectrum observed when using **L5**. Complexes **4a**, **6a** and **7a** have been labelled.

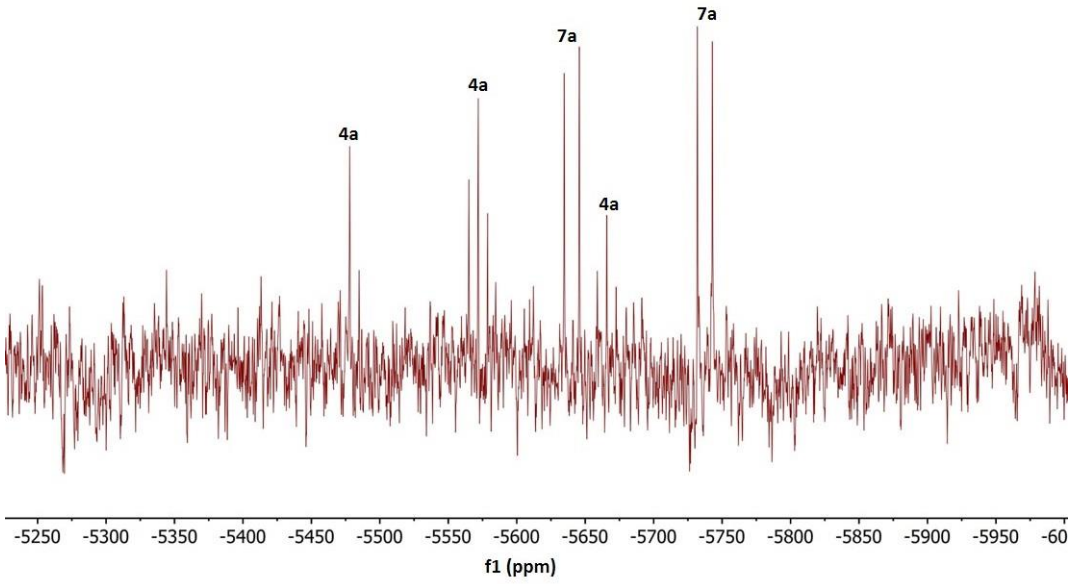
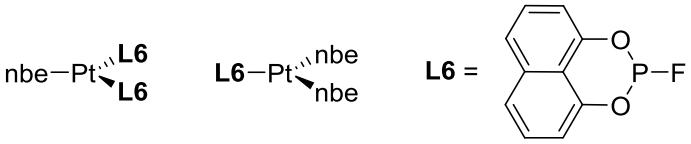


Figure S5 *In situ* $^{195}Pt\{^{1}H\}$ NMR spectrum observed when using **L5**. Complexes **4a** and **7a** have been labelled. Baseline has been corrected. δ_{Pt} for complex **4a** fitted with literature.[2,3]

2.2 Reaction between L6 and [Pt(nbe)$_3$] in a 2:1 stoichiometry



6b **7b**

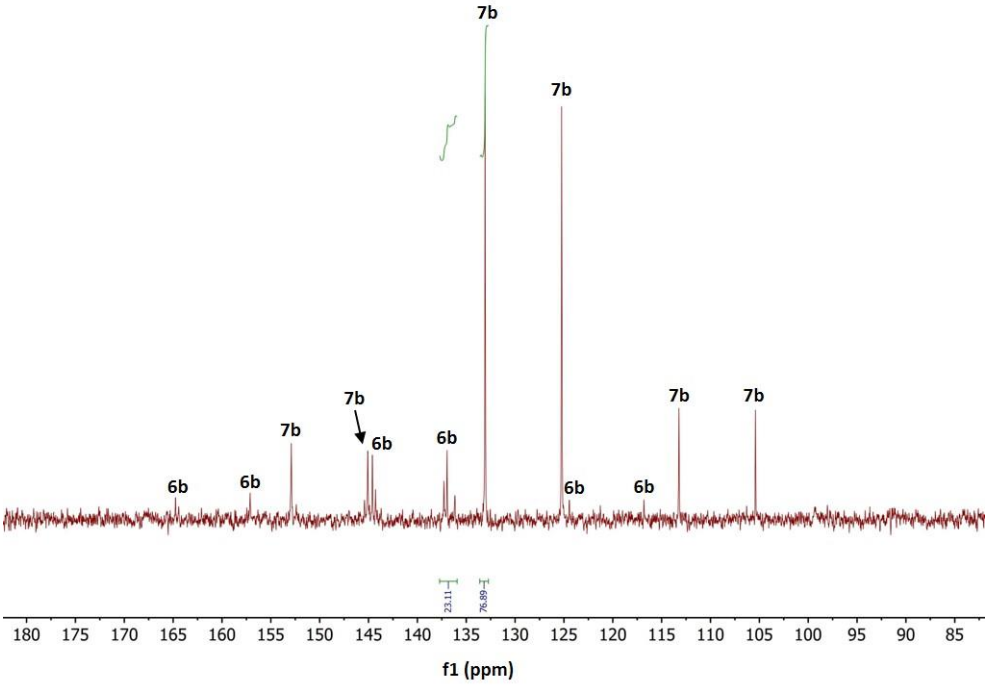


Figure S6 *In situ* $^{31}P\{^{1}H\}$ NMR spectrum of the reaction filtrate observed when using **L6**. Complexes **6b** and **7b** have been labelled.

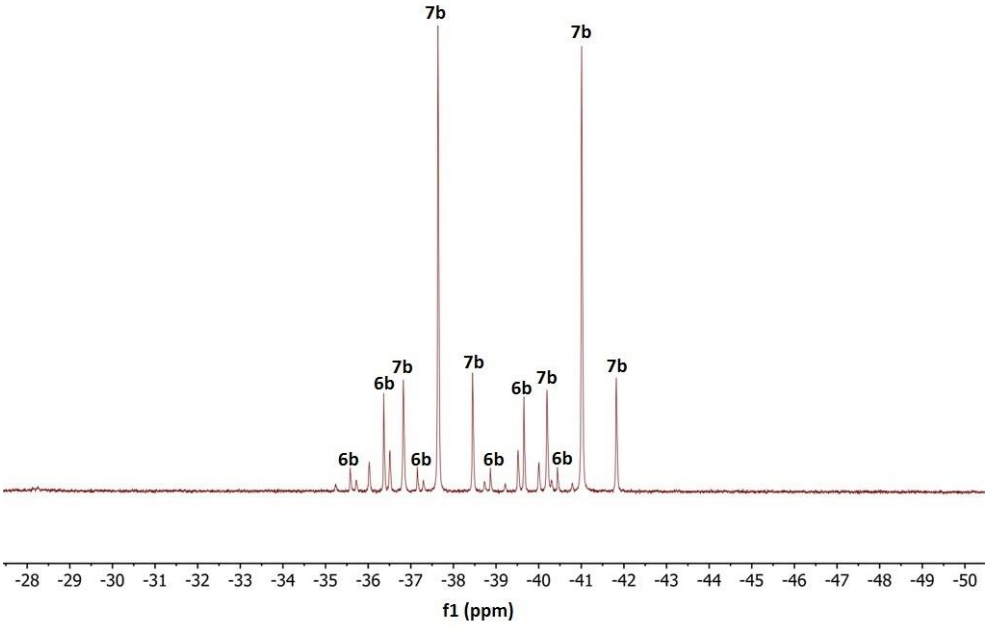


Figure S7 *In situ* $^{19}F\{^{1}H\}$ NMR spectrum observed when using **L6**. Complexes **6b** and **7b** have been labelled.

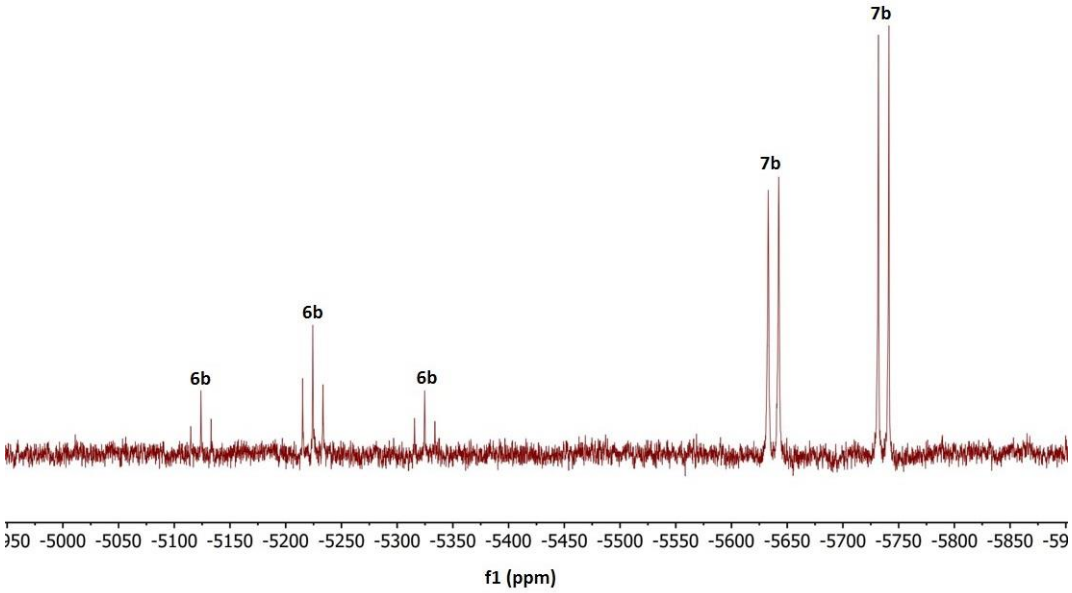


Figure S8 *In situ* $^{195}Pt\{^{1}H\}$ NMR spectrum observed when using **L6**. Complexes **6b** and **7b** have been labelled. Baseline has been corrected.

2.3 Reaction between L7 and [Pt(nbe)$_3$] in a 2:1 stoichiometry

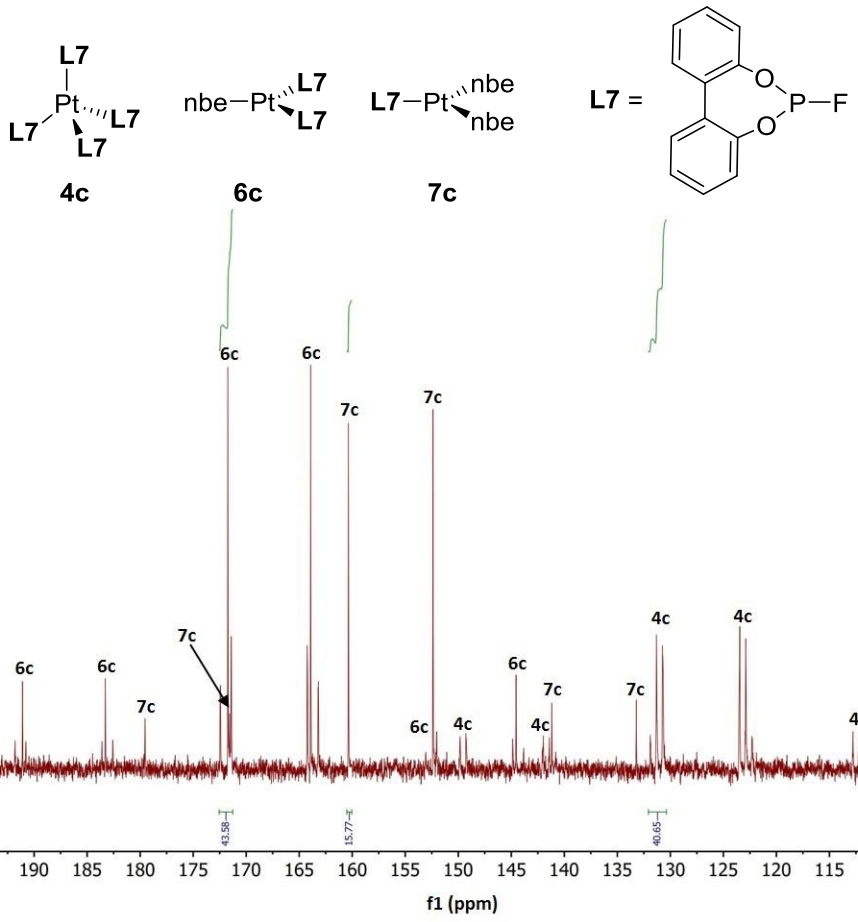


Figure S9 *In situ* $^{31}P\{^{1}H\}$ NMR spectrum observed when using **L7**. Complexes **4c**, **6c** and **7c** have been labelled.

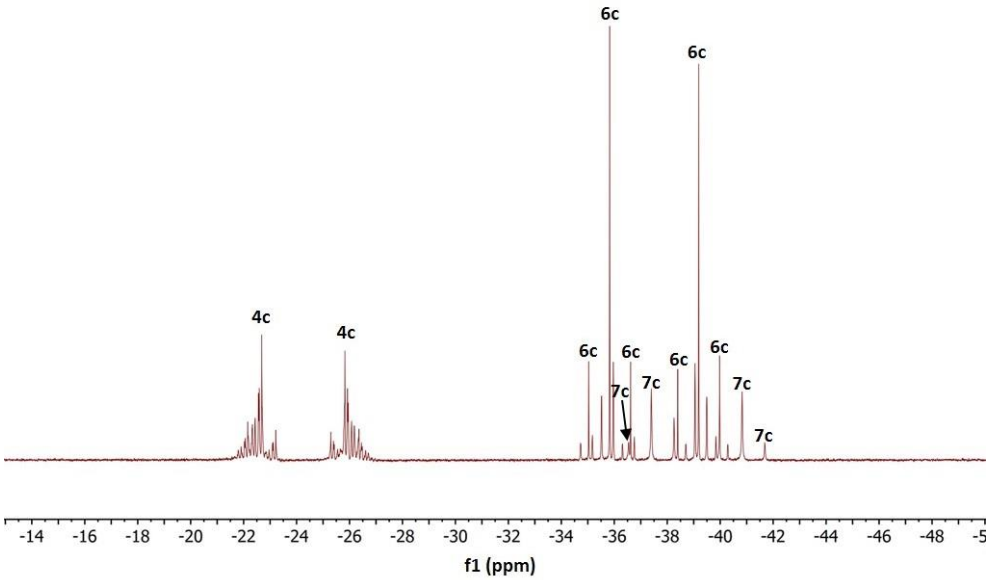
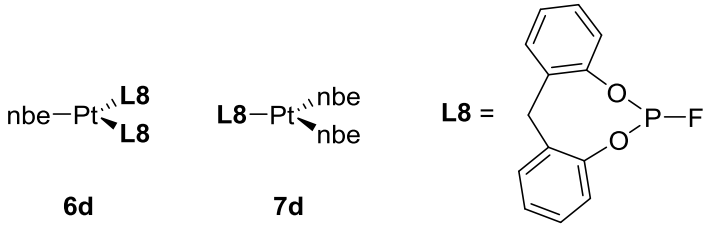


Figure S10 *In situ* $^{19}F\{^{1}H\}$ NMR spectrum observed when using **L7**. Complexes **4c**, **6c** and **7c** have been labelled.

2.4 Reaction between L8 and [Pt(nbe)$_3$] in a 2:1 stoichiometry



6d **7d**

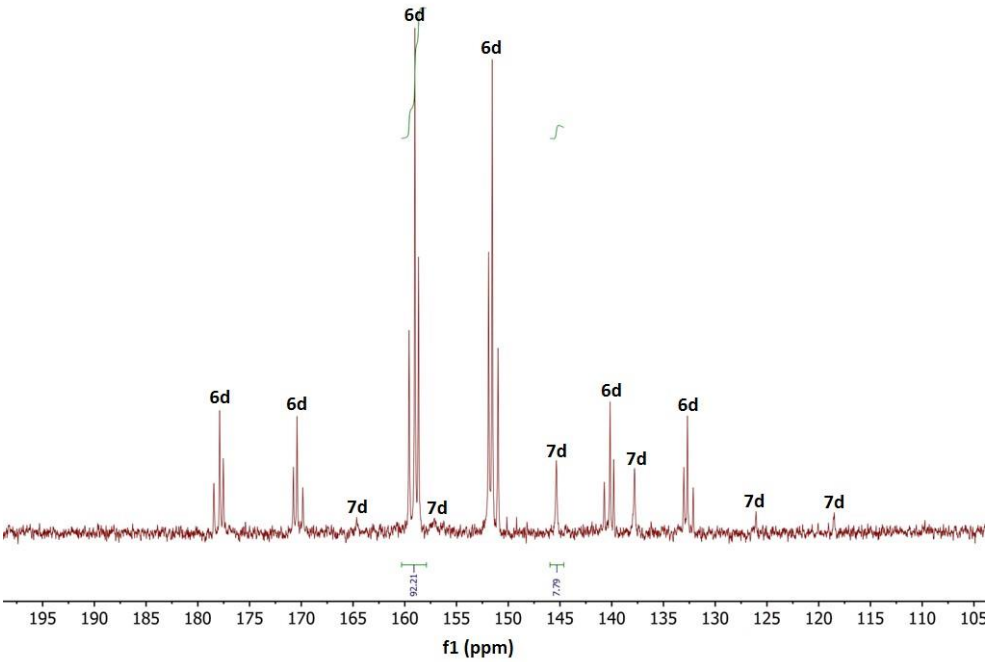


Figure S11 *In situ* $^{31}P\{^{1}H\}$ NMR spectrum observed when using **L8**. Complexes **6d** and **7d** have been labelled.

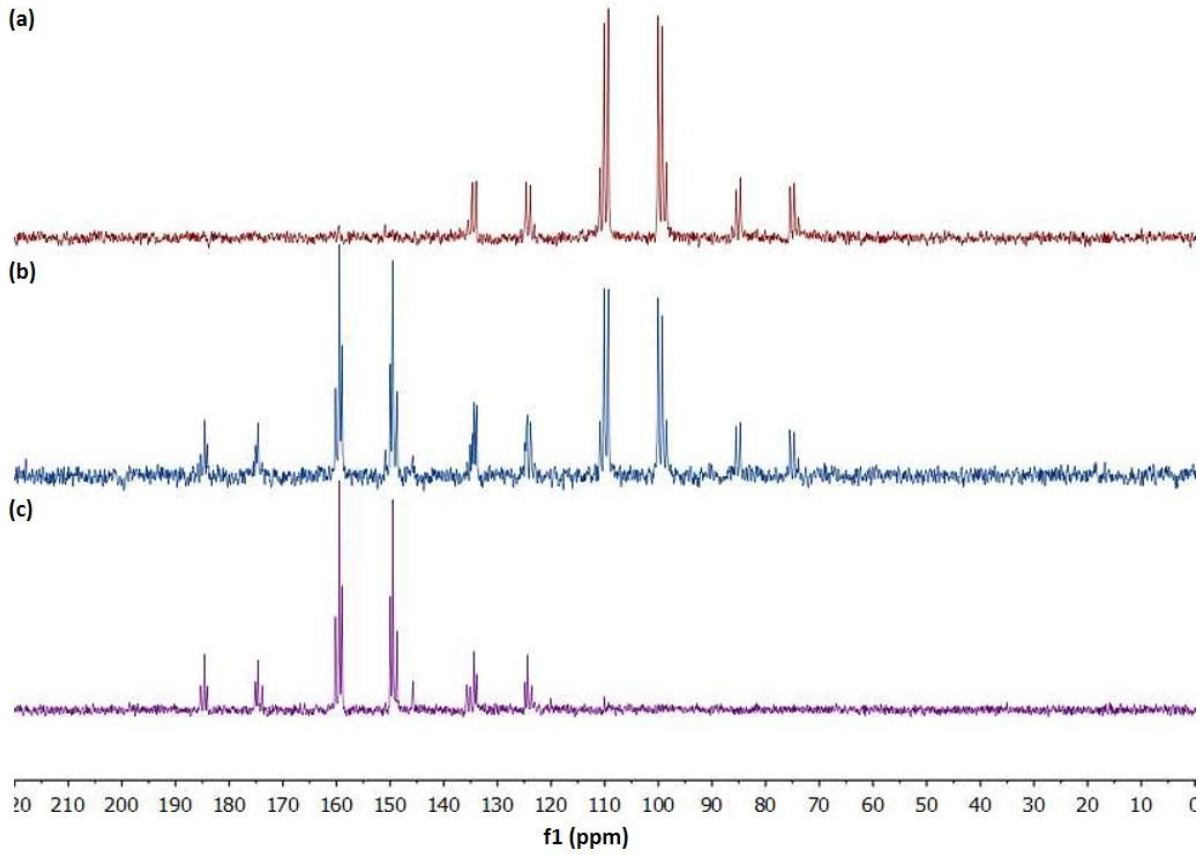


Figure S12 *In situ* $^{31}P\{^{1}H\}$ NMR spectra observed when using **L8** at (a) 5 min (**4d** observed), (b) 30 min (**4d** and **6d** observed) and (c) 1h (**6d** observed).

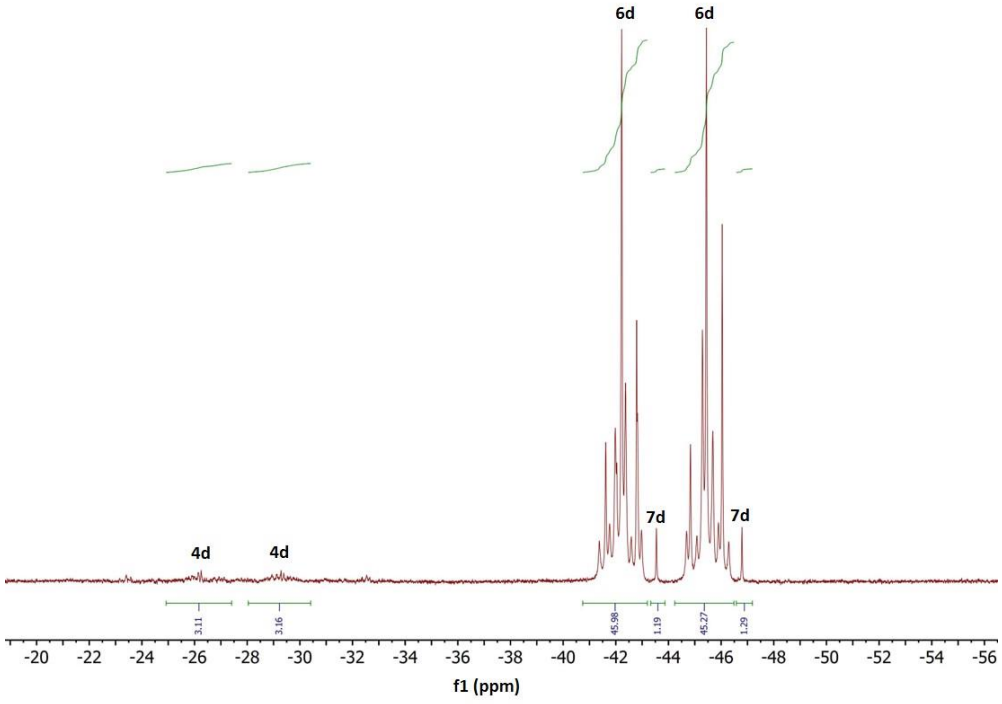


Figure S13 *In situ* $^{19}F\{^{1}H\}$ NMR spectrum observed when using **L8**. Complexes **4d**, **6d** and **7d** have been labelled.

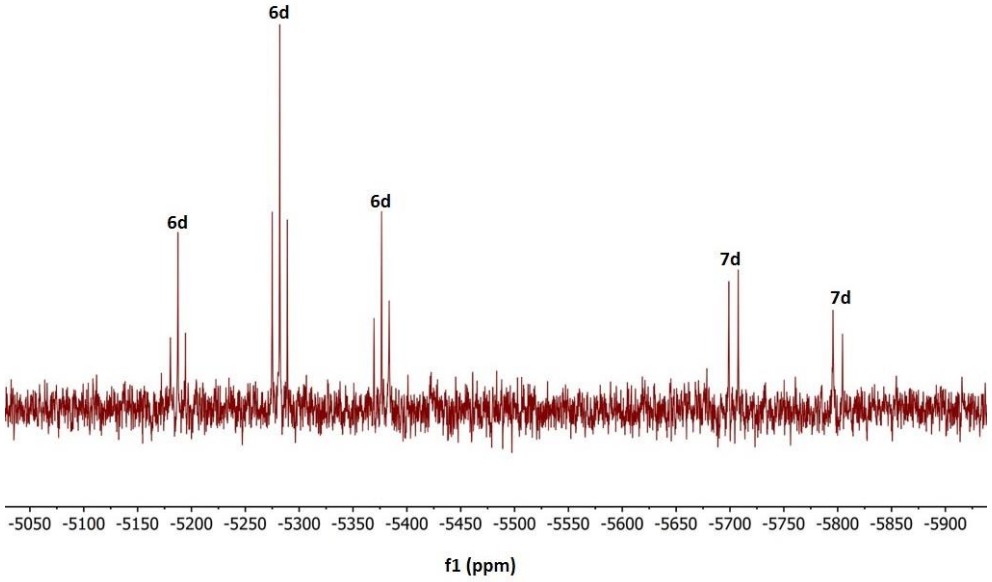
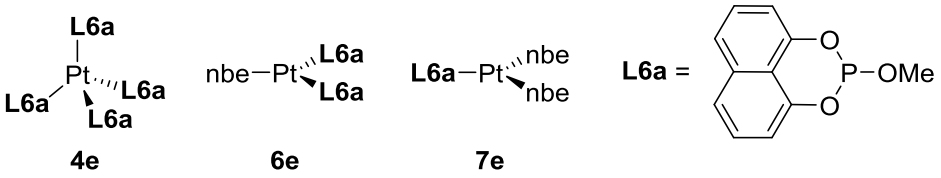


Figure S14 *In situ* $^{195}Pt\{^{1}H\}$ NMR spectrum observed when using **L8**. Complexes **6d** and **7d** have been labelled. Baseline has been corrected.

2.5 Reaction between L6a and [Pt(nbe)$_3$] in a 2:1 stoichiometry



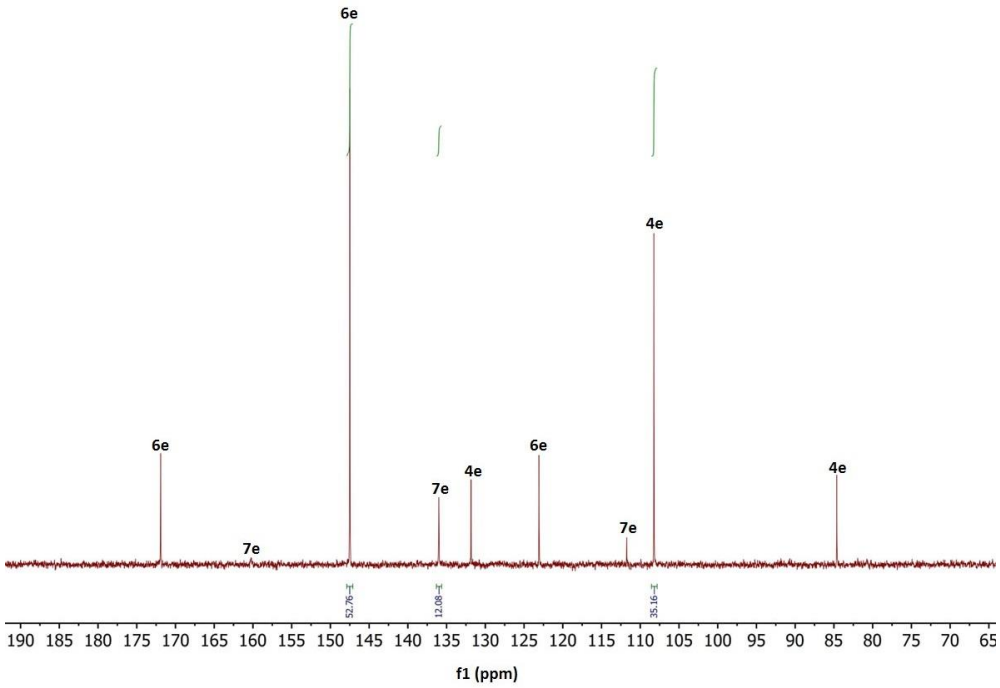


Figure S15 *In situ* $^{31}P\{^{1}H\}$ NMR spectrum observed when using **L6a**. Complexes **4e**, **6e** and **7e** have been labelled.

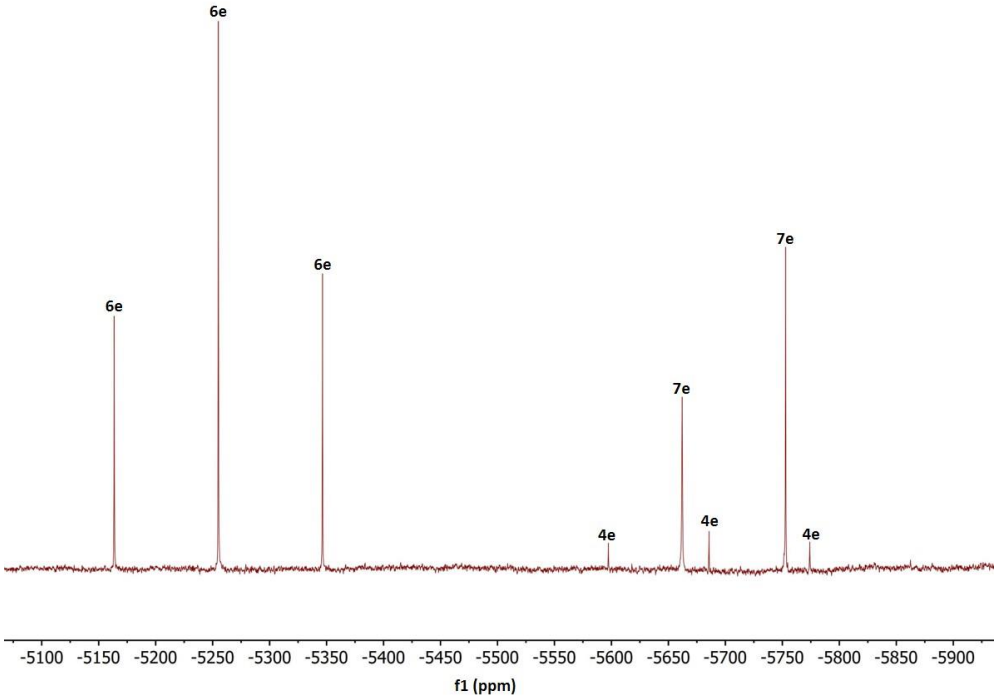


Figure S16 *In situ* $^{195}Pt\{^{1}H\}$ NMR spectrum observed when using **L6a**. Complexes **4e**, **6e** and **7e** have been labelled.

2.6 Simulated spectra for [Pt(L7)$_2$(nbe)] (6c)

For [PtL$_2$(nbe)] complexes, **6b**-**d**, the $^{31}P\{^{1}H\}$ NMR and $^{19}F\{^{1}H\}$ NMR spectra were simulated in Mestrenova using calculated *J* values from |K|, |L|, |M| and |N|, determined from Equations 1-4 (see Experimental for calculated values).

Figure S17 and S18 shows complex **6c** as an example and the good agreement between the observed and the simulated spectra.

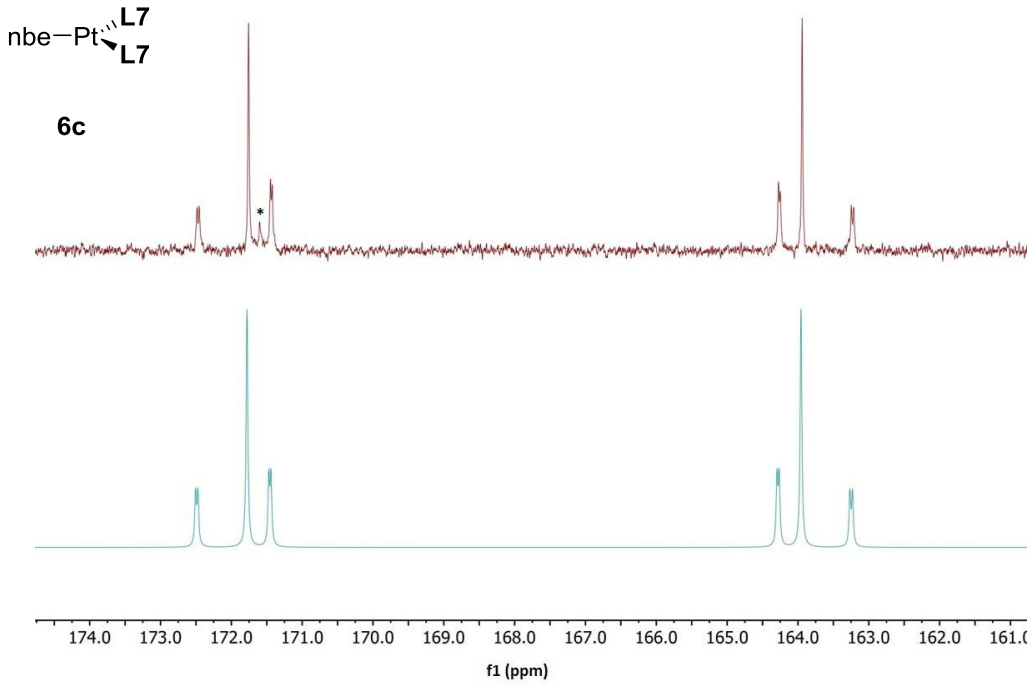


Figure S17 Top spectrum: [PtL_2(nbe)] (**6c**) section of observed $^{31}P\{^1H\}$ NMR spectrum from reaction of 2 equiv. **L7** with [$Pt(nbe)_3$]; (* = complex **7c**). Bottom spectrum: Simulated $^{31}P\{^1H\}$ NMR spectrum of complex **6c** using the *J* calculated values: $^1J_{P,F}$ = 1292.9 Hz, $^2J_{P,P}$ = 162.3 Hz, $^4J_{F,F'}$ = 5.3 Hz, $^3J_{P,F'}$ = -26.1 Hz. ^{195}Pt satellites have been omitted for clarity.

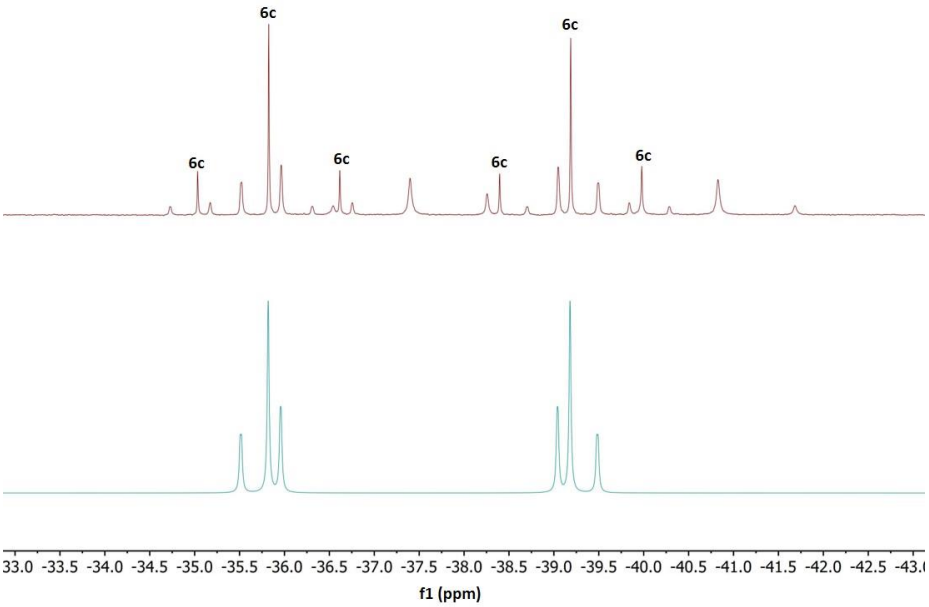


Figure S18 Top spectrum: [PtL_2(nbe)] (**6c**) section of observed $^{19}F\{^1H\}$ NMR spectrum from reaction of 2 equiv. **L7** with [$Pt(nbe)_3$]; contains signals from complex **7c** (see Figure 10). Bottom spectrum: Simulated $^{19}F\{^1H\}$ NMR spectrum of complex **6c** using the *J* calculated values: $^1J_{F,P}$ = 1292.9 Hz, $^2J_{P,P}$ = 162.3 Hz, $^4J_{F,F'}$ = 5.3 Hz, $^3J_{F',P}$ = -26.1 Hz; ^{195}Pt satellite have been omitted for clarity.

3. Spectra observed for [Pt(L7)$_4$] (4c)

Below show examples of the $^{31}P\{^1H\}$, $^{19}F\{^1H\}$ and $^{195}Pt\{^1H\}$ NMR spectra observed for [Pt(**L7**)$_4$] (**4c**).

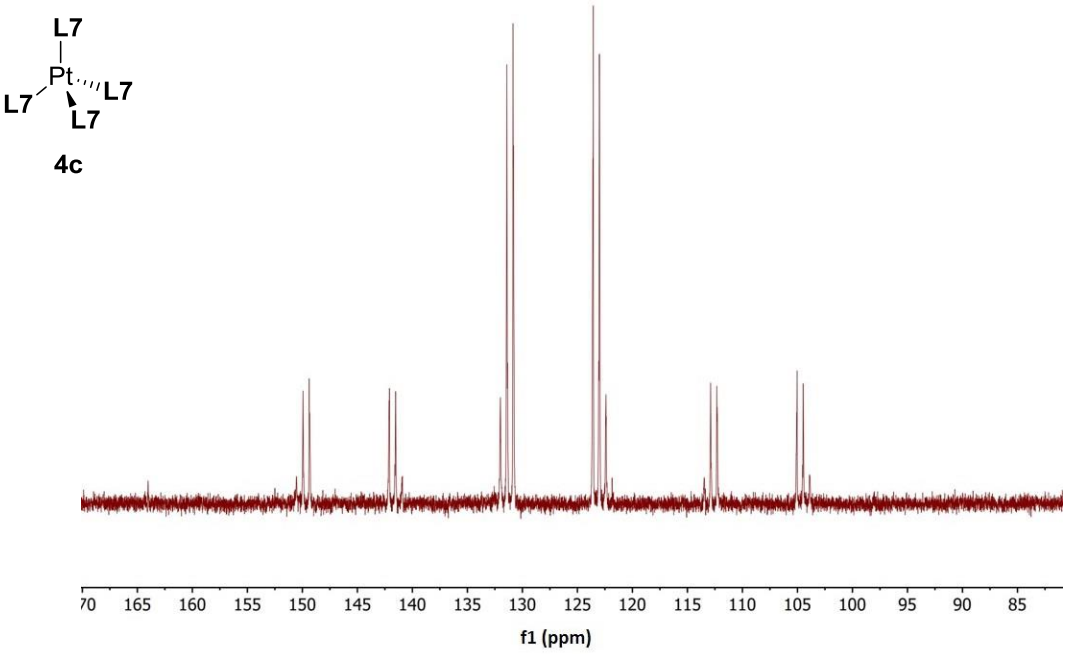


Figure S19 $^{31}P\{^1H\}$ NMR spectrum of [Pt(**L7**)$_4$] (**4c**).

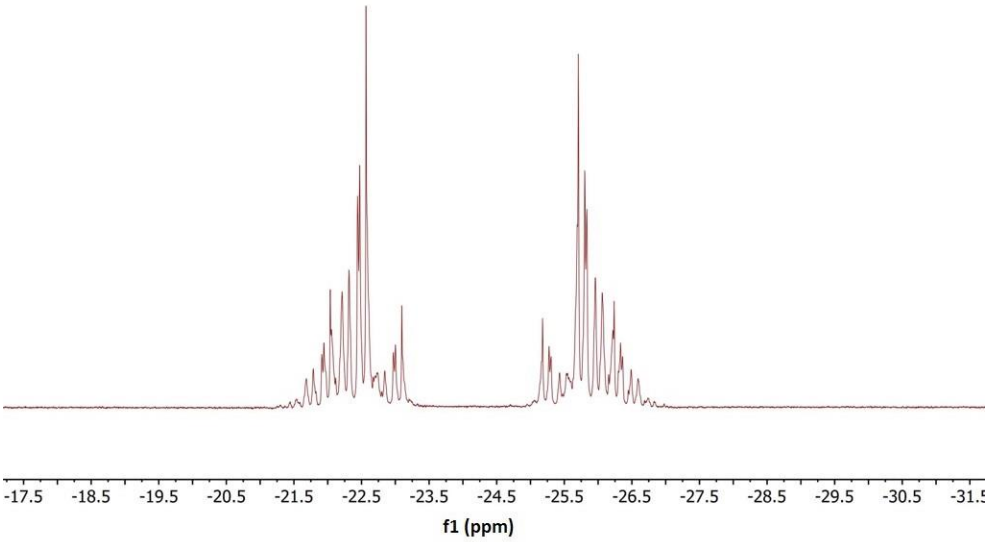


Figure S20 $^{19}F\{^1H\}$ NMR spectrum of [Pt(**L7**)$_4$] (**4c**).

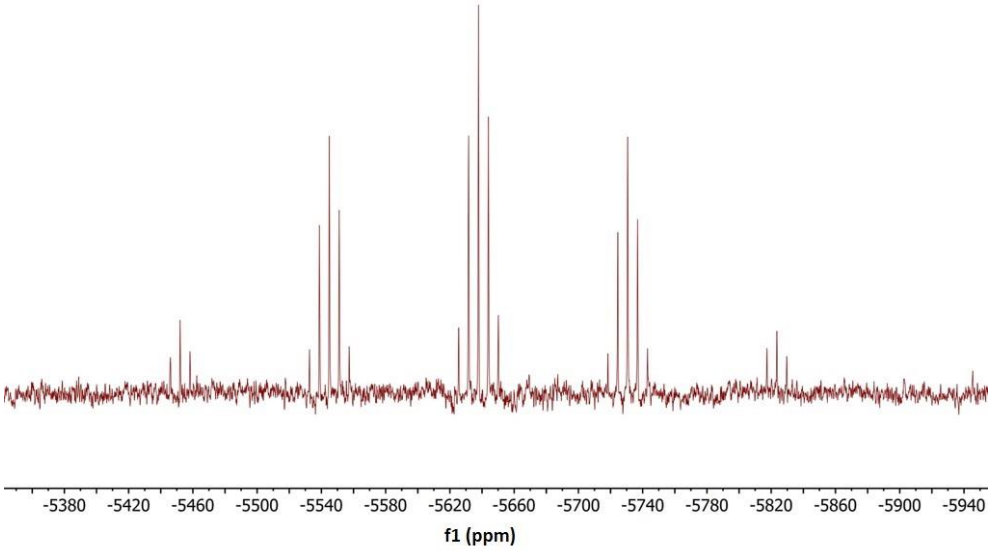
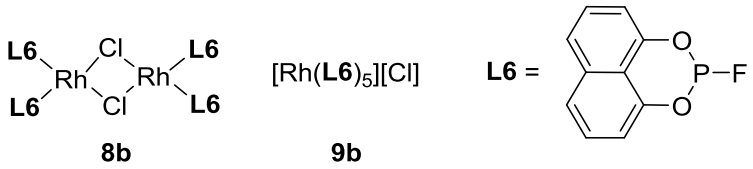


Figure S21 $^{195}Pt\{^{1}H\}$ NMR spectrum of [Pt(**L7**)$_4$] (**4c**).

4. Reaction between ligands and $[RhCl(CO)_2]_2$ in a 4:1 stoichiometry

Below are the $^{31}P\{^1H\}$ and $^{19}F\{^1H\}$ NMR spectra observed upon reaction between ligands **L6**-**8** and **L6a** with $[RhCl(CO)_2]_2$ in a 4:1 stoichiometry.

4.1 Reaction between L6 and $[RhCl(CO)_2]_2$ in a 4:1 stoichiometry



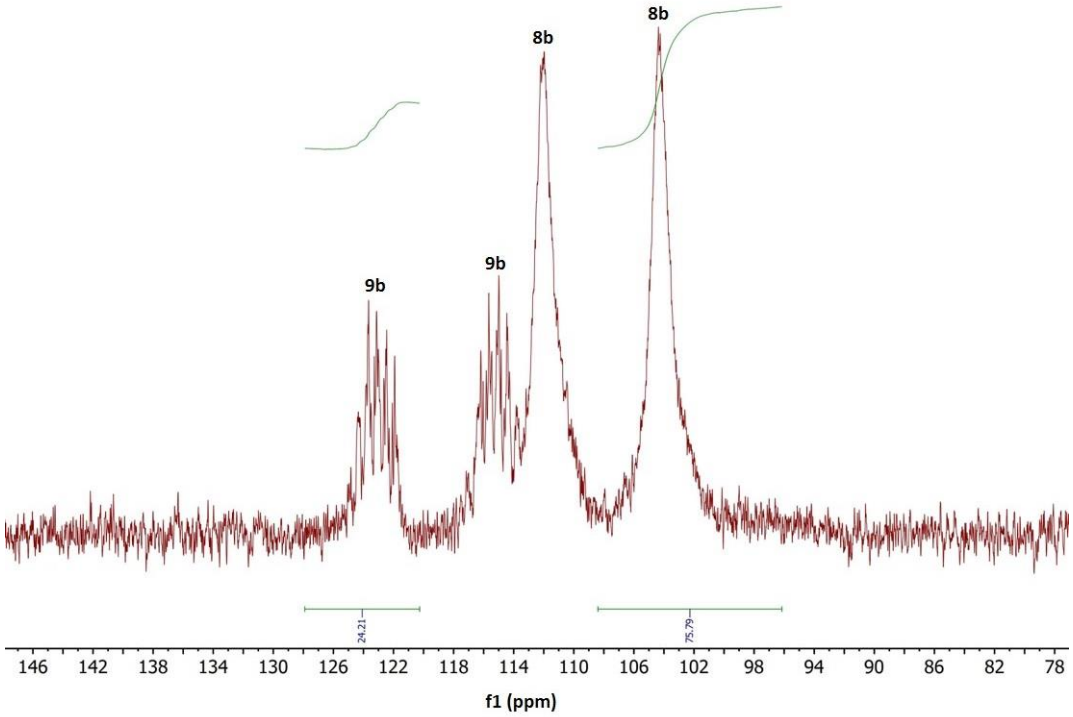


Figure S22 $^{31}P\{^1H\}$ NMR spectrum observed when using **L6**. Complexes **8b** and **9b** have been labelled.

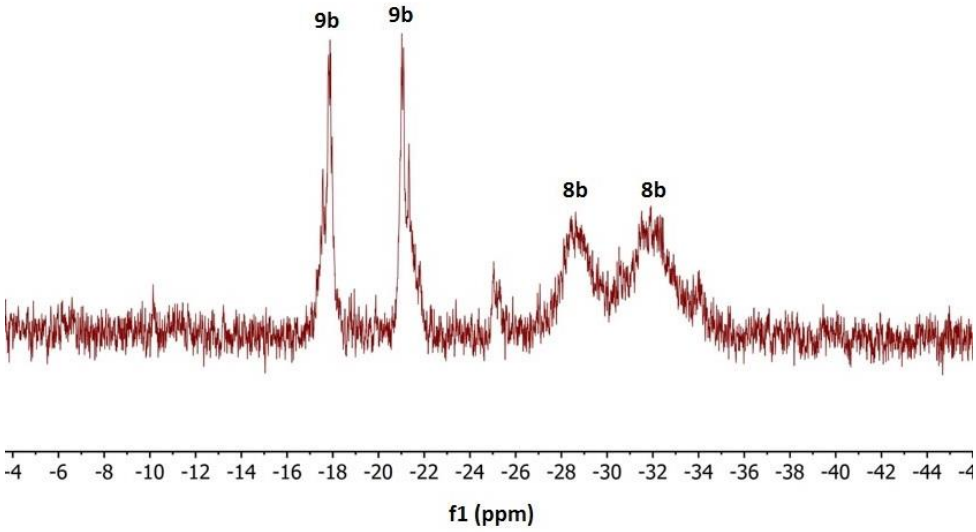


Figure S23 $^{19}F\{^{1}H\}$ NMR spectrum observed when using **L6**. Complexes **8b** and **9b** have been labelled.

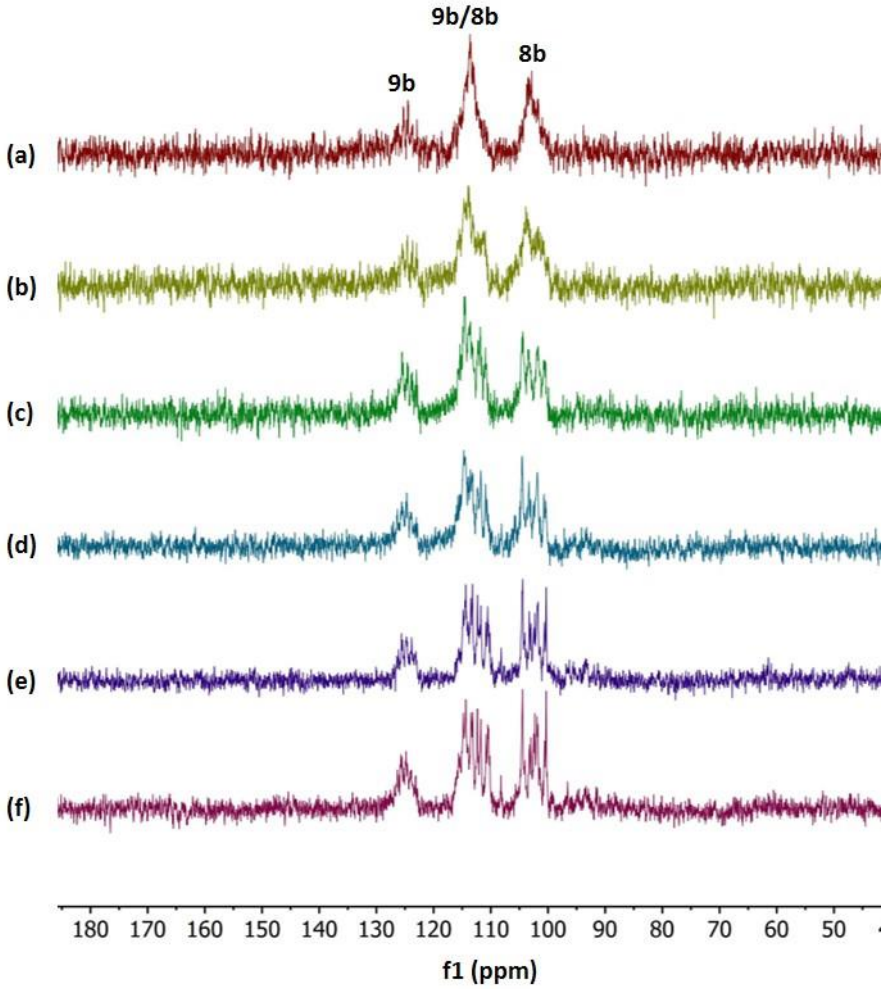
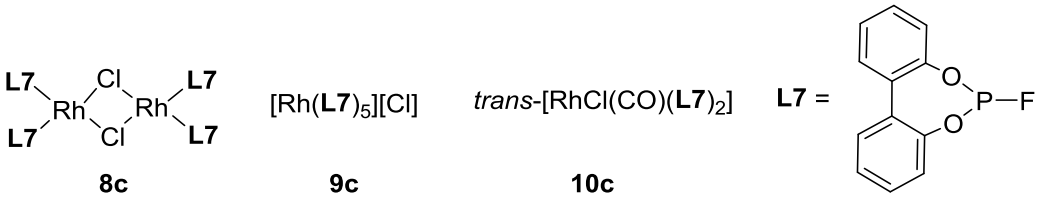


Figure S24 Low temperature $^{31}P\{^{1}H\}$ NMR spectra observed when using **L6**; recorded at (a) 20 °C, (b) 0 °C (c) -20 °C, (d) -40 °C, (e) -60 °C, (f) -80 °C. Complexes **8b** and **9b** have been labelled.

4.2 Reaction between L7 and $[RhCl(CO)_2]_2$ in a 4:1 stoichiometry



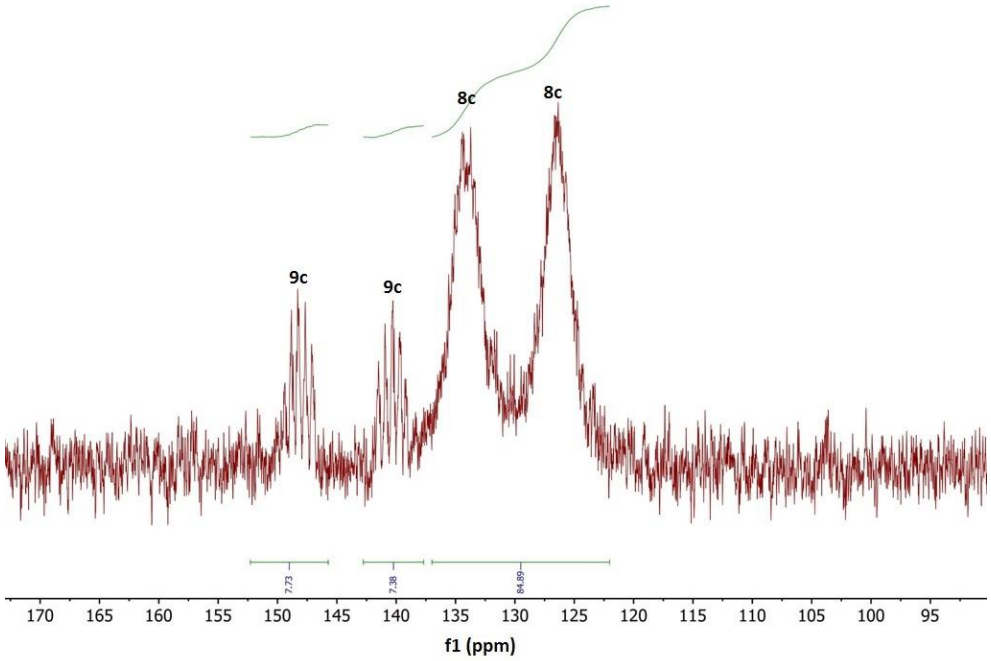


Figure S25 $^{31}P\{^1H\}$ NMR spectrum observed when using **L7**. Complexes **8c** and **9c** have been labelled.

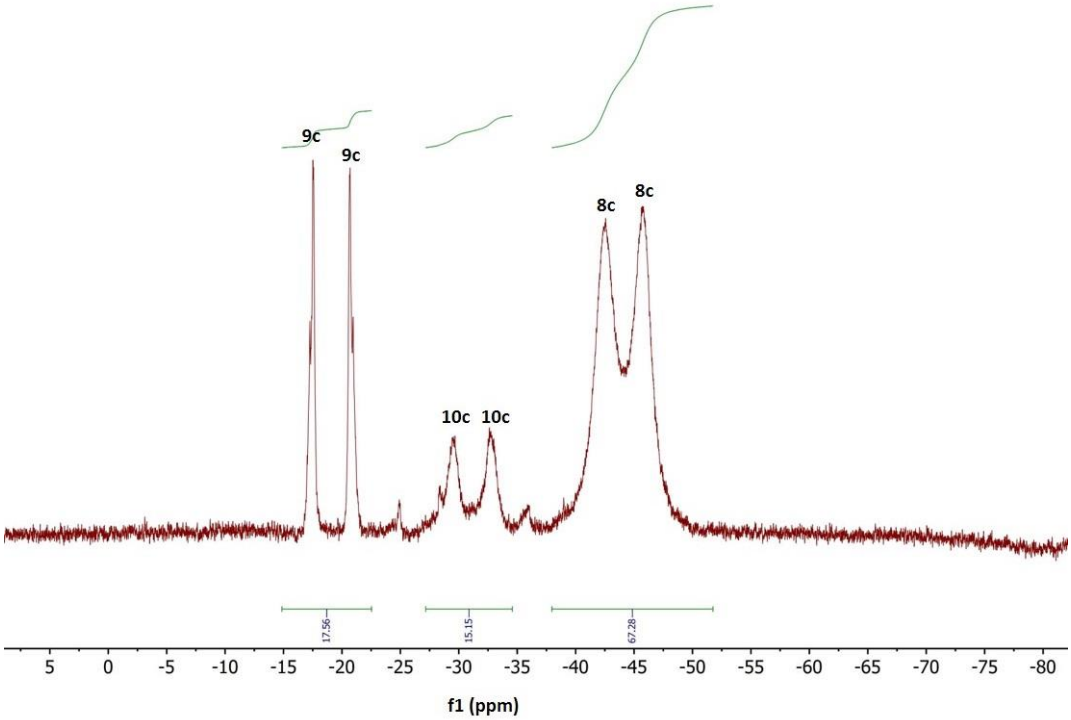
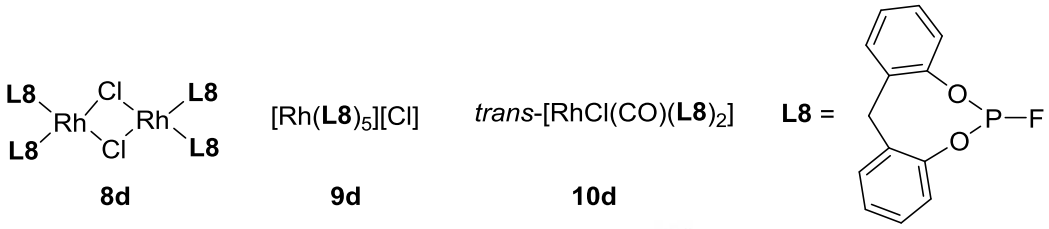


Figure S26 $^{19}F\{^1H\}$ NMR spectrum observed when using **L7**. Complexes **8c**, **9c** and **10c** have been labelled.

4.3 Reaction between L8 and $[RhCl(CO)_2]_2$ in a 4:1 stoichiometry



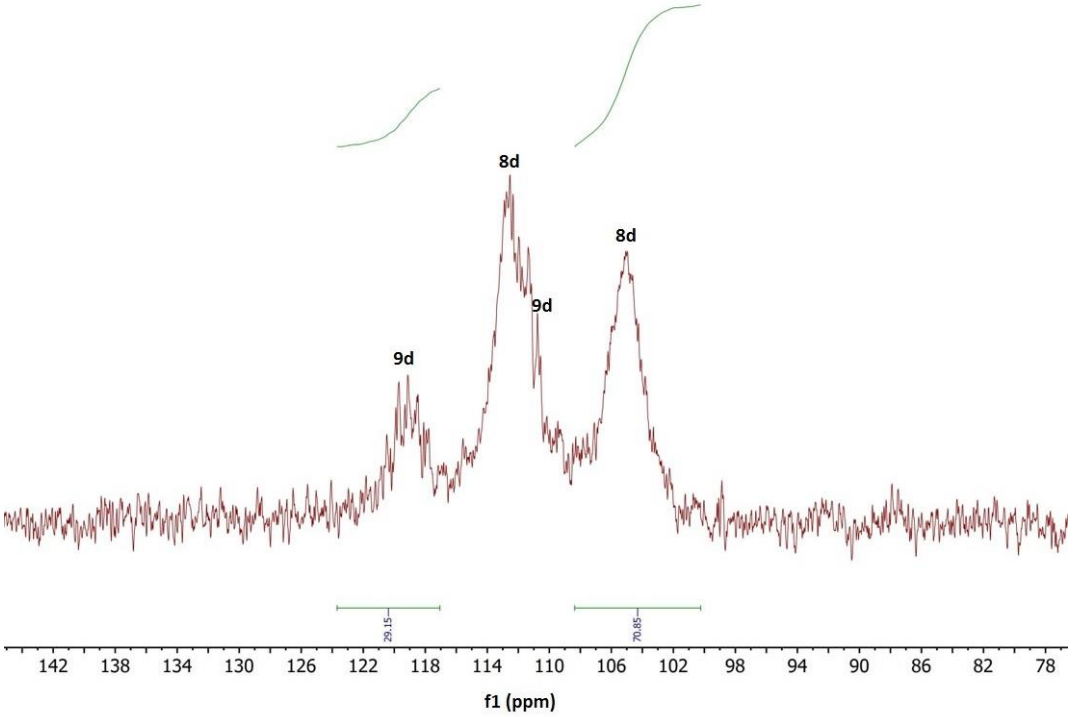


Figure S27 $^{31}P\{^1H\}$ NMR spectrum observed when using **L8**. Complexes **8d** and **9d** have been labelled.

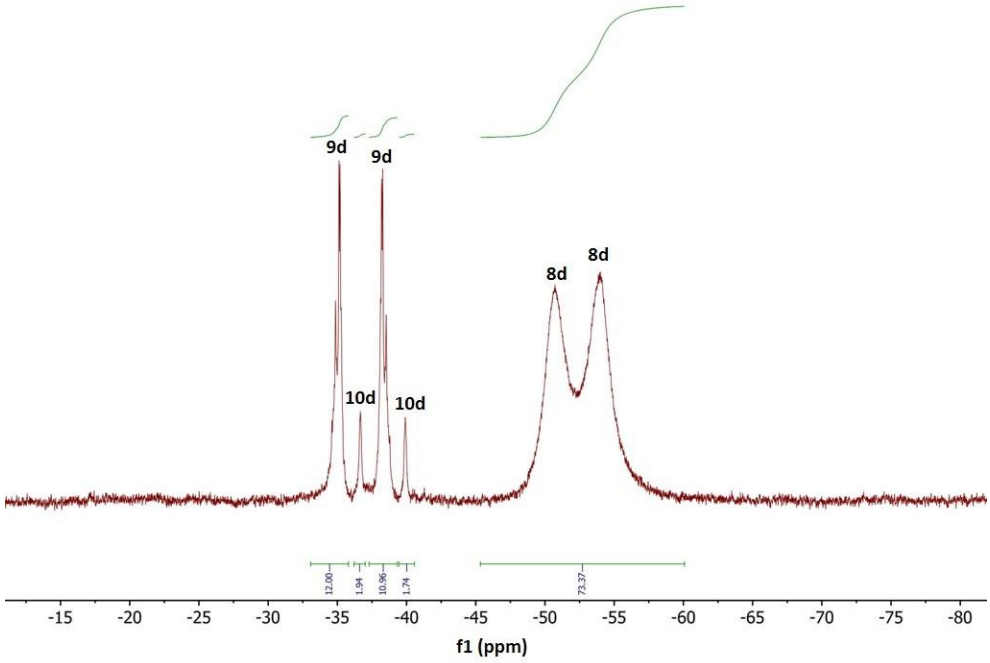


Figure S28 $^{19}F\{^1H\}$ NMR spectrum observed when using **L8**. Complexes **8d**, **9d** and **10d** have been labelled.

4.4 Reaction between L6 and [RhCl(cod)]$_2$ in a 4:1 stoichiometry

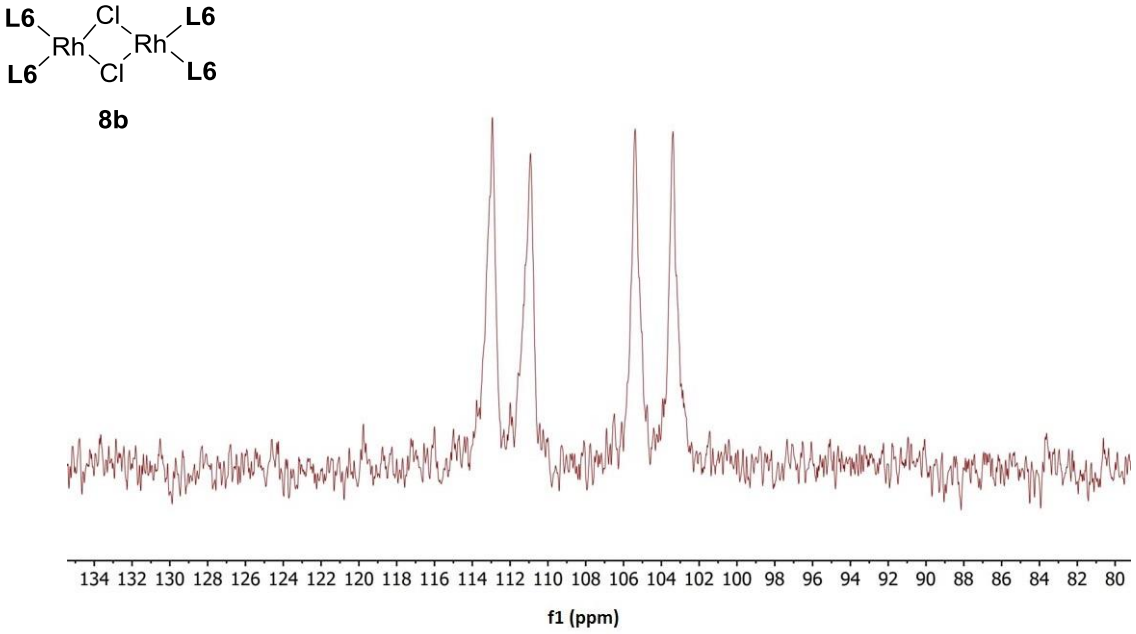


Figure S29 $^{31}P\{^1H\}$ NMR spectrum of [RhCl(**L6**)$_2$]$_2$ (**8b**).

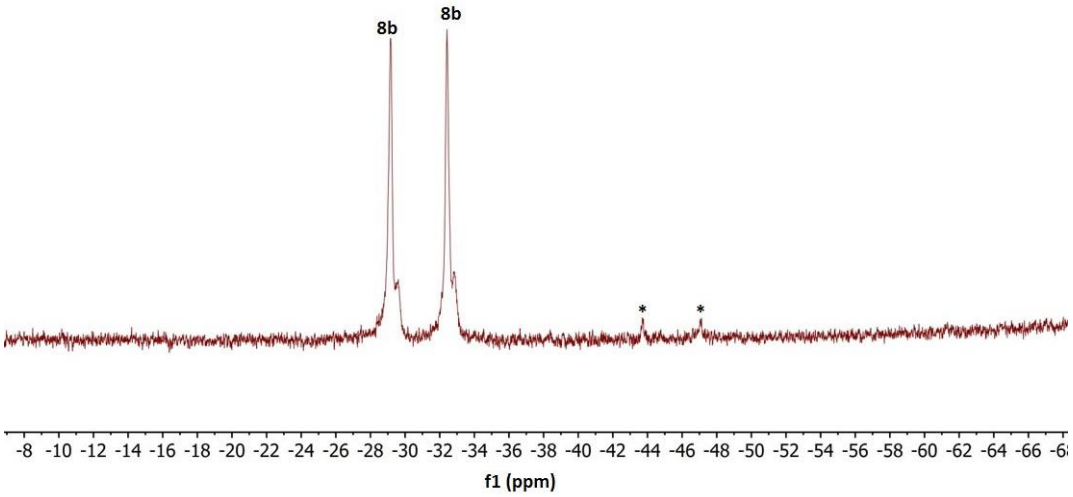


Figure S30 $^{19}F\{^1H\}$ NMR spectrum of [RhCl(**L6**)$_2$]$_2$ (**8b**), ***** = minor unknown contaminant.

4.5 Reaction between L6 and $[Rh(cod)_2][BF_4]$ in a 5:1 stoichiometry

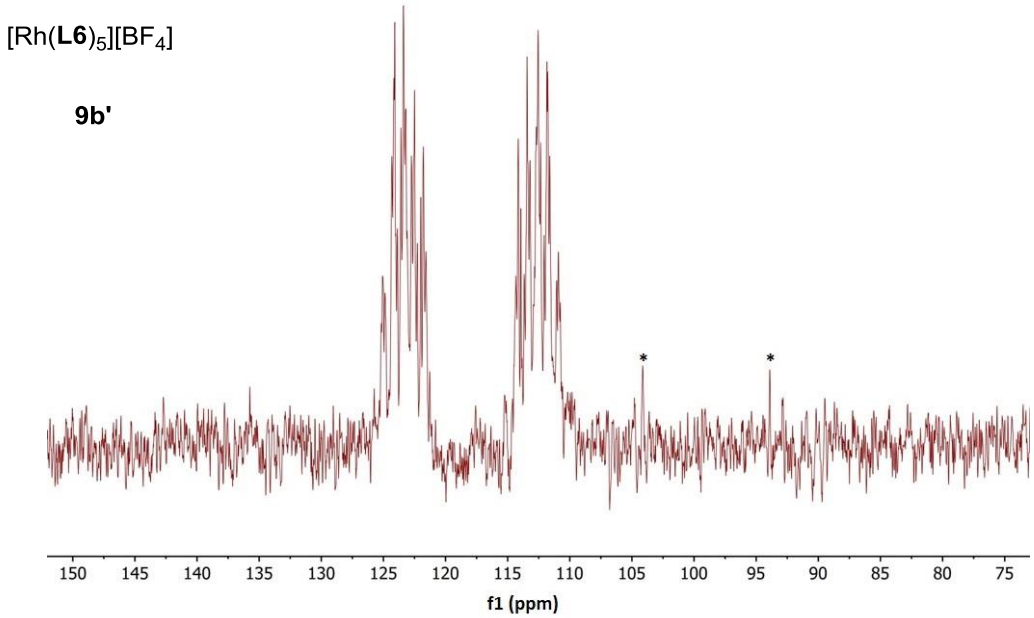


Figure S31 $^{31}P\{^1H\}$ NMR spectrum of $[Rh(\mathbf{L6})_5][BF_4]$ (**9b'**), * = 1% **L6**.

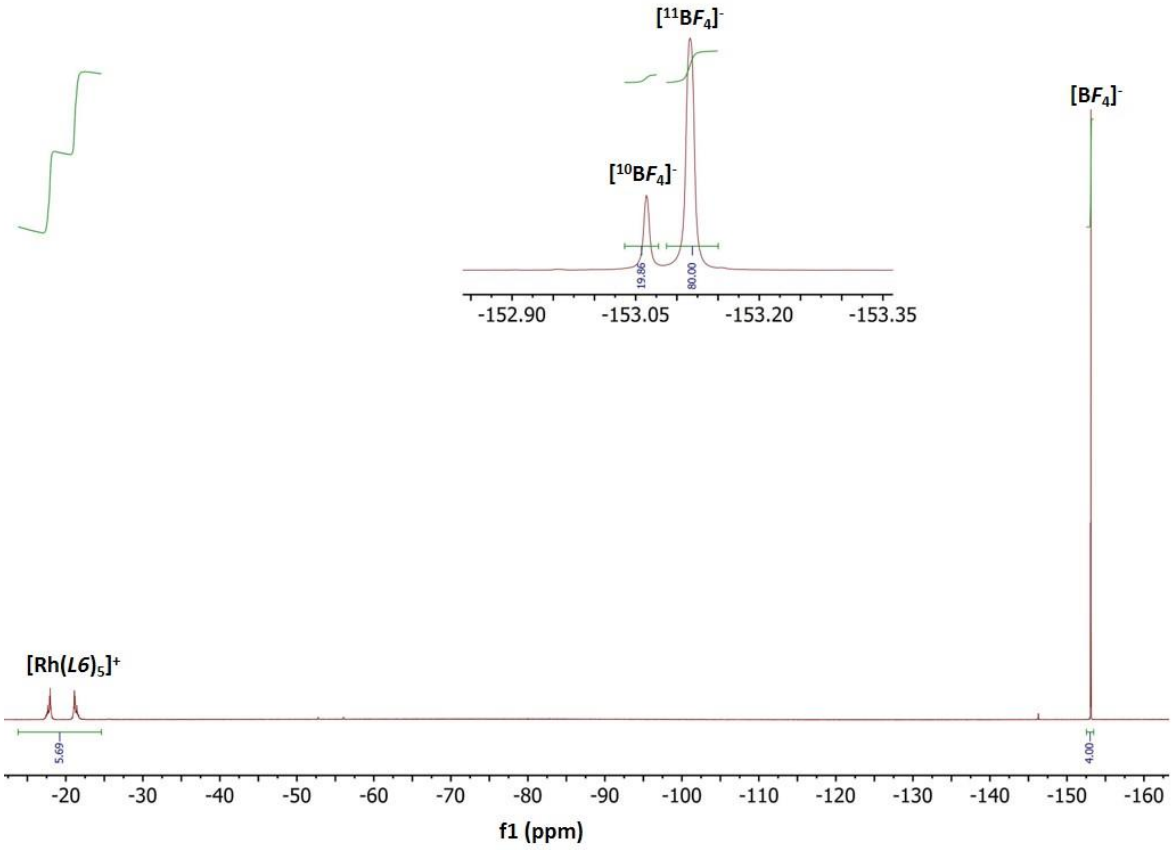


Figure S32 $^{19}F\{^1H\}$ NMR spectrum of $[Rh(\mathbf{L6})_5][BF_4]$ (**9b'**). Two signals in a 5:4 ratio were observed. The $[BF_4]^-$ signal was split into two signals in a 1:4 ratio for the ^{10}B and ^{11}B isotopes respectively.

5. Crystallography

5.1 Crystal structure of [Pt(L6a)$_4$] (4e)

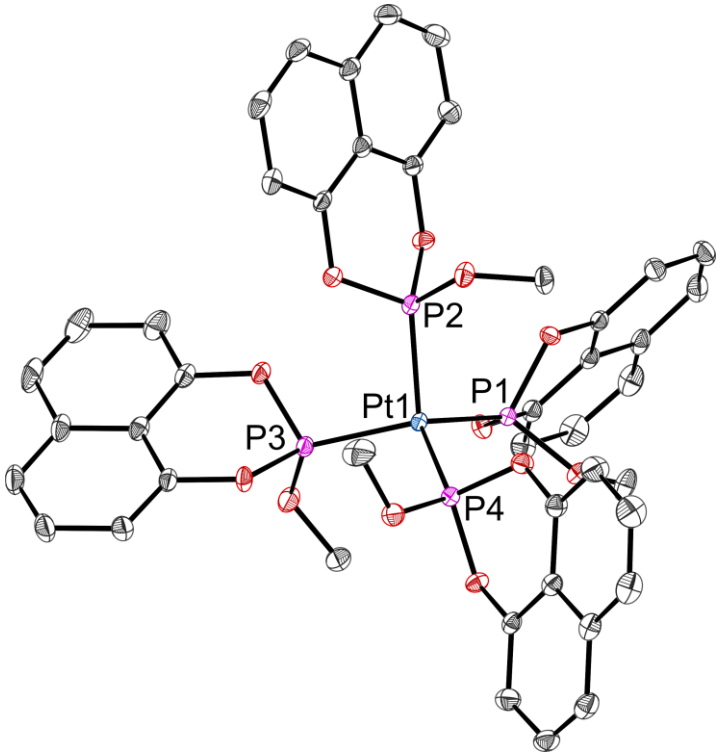


Figure S33 Crystal structure of [Pt(**L6a**)$_4$] (**4e**); thermal ellipsoids at 50% probability. Hydrogens are omitted for clarity. Selected bond lengths (Å) and angles (°): P1-O3 1.6089(13), Pt1-P1 2.2284(5), P1-Pt1-P2 104.822(17), O1-P1-O2 100.86(7).

5.2 Crystallography experimental and data

X-ray diffraction experiments on **3a**-**e** and **4e** were carried out at 100(2) K while **4b** was carried out at 200(2) K, all data were collected on a Bruker APEX II diffractometer using Mo-K_α radiation (λ = 0.71073 Å). Intensities were integrated in SAINT[4] and absorption corrections based on equivalent reflections were applied using SADABS.[5] Structures **3a**, **3c**-**e** and **4** was solved using ShelXT,[6] while **3b** was solved using ShelXS[7] and **4e** using Superflip,[8,9] all of the structures were refined by full matrix least squares against F^2 in ShelXL[7,10] using Olex2.[11] All of the non-hydrogen atoms were refined anisotropically. While all of the hydrogen atoms were located geometrically and refined using a riding model. In the case of **4b** the chlorobenzene solvent molecule was disordered across the 4-fold rotation axis, the occupancies were necessarily fixed at 0.25 and restraints applied to maintain sensible thermal and geometric parameters. Crystal structure and refinement data are given in Table S1. Crystallographic data for compounds **3a**-**e**,**4b** and **4e** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication CCDC 1898118-1898123. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax(+44) 1223 336033, e-mail: deposit@ccdc.cam.ac.uk].

Table S1 Crystal data and structure refinement for testo1.

Identification code	**3a**	**3b**	**3c**	**3d**	**3e**	**4b**	**4e**
Empirical formula	$C_{16}H_8F_2MoO_8P_2$	$C_{24}H_{12}F_2MoO_8P_2$	$C_{28}H_{16}F_2MoO_8P_2$	$C_{30}H_{20}F_2MoO_8P_2$	$C_{26}H_{18}MoO_{10}P_2$	$C_{46}H_{29}ClF_4O_8P_4Pt$	$C_{44}H_{36}O_{12}P_4Pt$
Formula weight	524.10	624.22	676.29	704.34	648.28	1140.11	1075.70
Temperature/K	100(2)	100(2)	100(2)	100(2)	100(2)	200(2)	99.98
Crystal system	orthorhombic	orthorhombic	monoclinic	triclinic	triclinic	tetragonal	triclinic
Space group	*Pnma*	*Fdd*2	$P2_1/c$	*P*-1	*P*-1	*P*4/*n*	*P*-1
a/Å	13.9152(3)	43.6028(6)	17.5271(6)	9.0915(3)	8.2570(2)	15.2985(4)	11.8627(2)
b/Å	20.6660(4)	9.9809(2)	7.6758(3)	12.3578(5)	11.6685(3)	15.2985(4)	12.1538(2)
c/Å	6.43140(10)	10.58170(10)	21.0653(7)	13.8479(5)	14.7840(4)	8.8050(3)	15.4498(3)
α/°	90	90	90	104.240(2)	69.9560(10)	90	83.6712(10)
β/°	90	90	111.373(2)	98.951(3)	76.9430(10)	90	77.7952(10)
γ/°	90	90	90	100.618(3)	83.4250(10)	90	73.4378(10)
Volume/Å^3	1849.49(6)	4605.10(12)	2639.11(17)	1448.86(9)	1302.45(6)	2060.76(13)	2083.86(7)
Z	4	8	4	2	2	2	2
ρ_{calc}g/cm^3	1.882	1.801	1.702	1.614	1.653	1.837	1.714
μ/mm^{-1}	0.946	0.776	0.684	0.627	0.684	3.701	3.586
F(000)	1032.0	2480.0	1352.0	708.0	652.0	1120.0	1068.0
Crystal size/mm^3	0.344 × 0.325 × 0.199	0.294 × 0.288 × 0.126	0.402 × 0.258 × 0.252	0.256 × 0.231 × 0.15	0.297 × 0.279 × 0.216	0.379 × 0.117 × 0.102	0.449 × 0.293 × 0.084
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2θ range for data collection/°	3.942 to 55.988	5.688 to 55.826	3.99 to 55.946	4.664 to 55.98	2.992 to 55.934	3.766 to 55.762	3.502 to 59.148
Index ranges	$-18 \le h \le 18$, $-26 \le k \le 27$, $-8 \le l \le 8$	$-57 \le h \le 57$, $-13 \le k \le 13$, $-13 \le l \le 11$	$-23 \le h \le 22$, $-9 \le k \le 10$, $-27 \le l \le 27$	$-11 \le h \le 11$, $-16 \le k \le 16$, $-18 \le l \le 18$	$-10 \le h \le 10$, $-15 \le k \le 15$, $-19 \le l \le 19$	$-20 \le h \le 19$, $-18 \le k \le 20$, $-11 \le l \le 11$	$-16 \le h \le 16$, $-16 \le k \le 16$, $-21 \le l \le 21$
Reflections collected	15356	10131	23439	26861	24212	18431	45772
R_{int} / R_{sigma}	0.0247 / 0.0151	0.0193 / 0.0170	0.0216 /0.0207	0.0525 / 0.0495	0.0265 / 0.0245	0.0734 / 0.0435	0.0322 / 0.0286
Data/restraints/parameters	2297/0/139	2624/1/168	6334/0/370	6977/0/388	6269/0/354	2476/85/180	11692/0/554
Goodness-of-fit on F^2	1.109	1.112	1.028	1.014	1.022	1.176	1.033
Final R indexes [I>=2σ (I)]	R_1 = 0.0205, wR_2 = 0.0466	R_1 = 0.0145, wR_2 = 0.0375	R_1 = 0.0211, wR_2 = 0.0529	R_1 = 0.0342, wR_2 = 0.0668	R_1 = 0.0238, wR_2 = 0.0558	R_1 = 0.0390, wR_2 = 0.0679	R_1 = 0.0195, wR_2 = 0.0437
Final R indexes [all data]	R_1 = 0.0232, wR_2 = 0.0478	R_1 = 0.0148, wR_2 = 0.0376	R_1 = 0.0249, wR_2 = 0.0546	R_1 = 0.0484, wR_2 = 0.0719	R_1 = 0.0278, wR_2 = 0.0577	R_1 = 0.0587, wR_2 = 0.0724	R_1 = 0.0219, wR_2 = 0.0445
Largest diff. peak/hole / e Å^{-3}	0.56/-0.39	0.25/-0.24	0.38/-0.39	0.47/-0.51	0.49/-0.36	0.64/-0.78	0.95/-0.81
Flack parameter	-	-0.035(11)	-	-	-	-	-

Table S2 Bond lengths for 3a. [1]+X,3/2-Y,+Z

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Mo1	P1[1]	2.3793(4)	O2	C6	1.3961(19)
Mo1	P1	2.3794(4)	O3	C7	1.133(3)
Mo1	C7	2.052(2)	O4	C8	1.137(2)
Mo1	C8[1]	2.0330(17)	O5	C9	1.135(3)
Mo1	C8	2.0330(17)	C1	C2	1.371(2)
Mo1	C9	2.055(2)	C1	C6	1.379(2)
P1	F1	1.5735(10)	C2	C3	1.399(2)
P1	O1	1.6265(12)	C3	C4	1.380(3)
P1	O2	1.6297(12)	C4	C5	1.395(2)
O1	C1	1.4001(19)	C5	C6	1.375(2)

Table S3 Bond angles for 3a. [1]+X,3/2-Y,+Z

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
P1[1]	Mo1	P1	94.65(2)	O1	P1	Mo1	121.03(5)
C7	Mo1	P1[1]	89.75(5)	O1	P1	O2	95.53(6)
C7	Mo1	P1	89.75(5)	O2	P1	Mo1	118.69(4)
C7	Mo1	C9	179.67(9)	C1	O1	P1	109.16(10)
C8[1]	Mo1	P1	176.16(5)	C6	O2	P1	109.03(10)
C8[1]	Mo1	P1[1]	89.19(5)	C2	C1	O1	126.28(15)
C8	Mo1	P1	89.19(5)	C2	C1	C6	122.12(15)
C8	Mo1	P1[1]	176.16(5)	C6	C1	O1	111.57(13)
C8	Mo1	C7	90.15(7)	C1	C2	C3	116.14(16)
C8[1]	Mo1	C7	90.15(7)	C4	C3	C2	121.83(16)
C8[1]	Mo1	C8	86.97(9)	C3	C4	C5	121.33(16)
C8	Mo1	C9	90.08(6)	C6	C5	C4	116.29(16)
C8[1]	Mo1	C9	90.08(6)	C1	C6	O2	111.73(13)
C9	Mo1	P1[1]	90.02(4)	C5	C6	O2	125.96(15)
C9	Mo1	P1	90.02(5)	C5	C6	C1	122.28(15)
F1	P1	Mo1	118.20(4)	O3	C7	Mo1	179.1(2)
F1	P1	O1	99.99(6)	O4	C8	Mo1	178.38(15)
F1	P1	O2	98.68(6)	O5	C9	Mo1	179.1(2)

Table S4 Bond lengths for 3b. [1]-X,1-Y,+Z

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Mo1	P1	2.3846(6)	O4	C12	1.139(3)
Mo1	P1[1]	2.3847(6)	C1	C2	1.368(3)
Mo1	C11[1]	2.042(2)	C1	C6	1.406(3)
Mo1	C11	2.042(2)	C2	C3	1.414(3)
Mo1	C12	2.052(2)	C3	C4	1.367(3)
Mo1	C12[1]	2.052(2)	C4	C5	1.420(3)
P1	F1	1.5832(13)	C5	C6	1.424(3)
P1	O1	1.6041(15)	C5	C10	1.419(3)
P1	O2	1.6008(16)	C6	C7	1.416(3)
O1	C1	1.397(2)	C7	C8	1.358(3)
O2	C7	1.404(2)	C8	C9	1.413(3)
O3	C11	1.134(3)	C9	C10	1.366(3)

Table S5 Bond angles for 3b. [1]-X,1-Y,+Z

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
P1	Mo1	P1[1]	93.35(3)	C7	O2	P1	122.50(13)
C11[1]	Mo1	P1[1]	88.98(7)	O1	C1	C6	119.74(18)
C11	Mo1	P1	88.98(7)	C2	C1	O1	117.87(19)
C11[1]	Mo1	P1	177.56(7)	C2	C1	C6	122.34(19)
C11	Mo1	P1[1]	177.56(7)	C1	C2	C3	118.6(2)
C11	Mo1	C11[1]	88.70(13)	C4	C3	C2	121.18(19)
C11	Mo1	C12	91.15(10)	C3	C4	C5	120.7(2)
C11	Mo1	C12[1]	91.61(10)	C4	C5	C6	118.43(19)
C11[1]	Mo1	C12[1]	91.15(10)	C10	C5	C4	123.21(19)
C11[1]	Mo1	C12	91.61(10)	C10	C5	C6	118.36(19)
C12[1]	Mo1	P1[1]	87.67(7)	C1	C6	C5	118.73(18)
C12	Mo1	P1	87.67(7)	C1	C6	C7	122.62(19)
C12[1]	Mo1	P1	89.68(6)	C7	C6	C5	118.65(19)
C12	Mo1	P1[1]	89.68(6)	O2	C7	C6	119.21(19)
C12	Mo1	C12[1]	176.15(14)	C8	C7	O2	118.43(19)
F1	P1	Mo1	118.96(5)	C8	C7	C6	122.3(2)
F1	P1	O1	99.33(8)	C7	C8	C9	118.6(2)

F1	P1	O2	99.67(8)	C10	C9	C8	121.5(2)
O1	P1	Mo1	114.53(6)	C9	C10	C5	120.6(2)
O2	P1	Mo1	117.90(6)	O3	C11	Mo1	179.0(3)
O2	P1	O1	103.52(8)	O4	C12	Mo1	178.0(2)
C1	O1	P1	123.43(14)				

Table S6 Bond lengths for 3c.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Mo1	P1	2.3891(4)	C3	C4	1.384(2)
Mo1	P2	2.3892(4)	C4	C5	1.384(2)
Mo1	C25	2.0326(16)	C5	C6	1.400(2)
Mo1	C26	2.0532(16)	C6	C7	1.478(2)
Mo1	C27	2.0507(16)	C7	C8	1.400(2)
Mo1	C28	2.0466(16)	C7	C12	1.396(2)
P1	F1	1.5744(10)	C8	C9	1.386(2)
P1	O1	1.5999(11)	C9	C10	1.385(3)
P1	O2	1.6124(11)	C10	C11	1.386(3)
P2	F2	1.5764(9)	C11	C12	1.385(2)
P2	O3	1.6035(11)	C13	C14	1.384(2)
P2	O4	1.6118(11)	C13	C18	1.394(2)
O1	C1	1.4081(17)	C14	C15	1.389(2)
O2	C12	1.4040(19)	C15	C16	1.391(2)
O3	C13	1.4092(18)	C16	C17	1.383(2)
O4	C24	1.4055(18)	C17	C18	1.399(2)
O5	C25	1.139(2)	C18	C19	1.480(2)
O6	C26	1.1333(19)	C19	C20	1.401(2)
O7	C27	1.136(2)	C19	C24	1.391(2)
O8	C28	1.137(2)	C20	C21	1.386(2)
C1	C2	1.379(2)	C21	C22	1.388(2)
C1	C6	1.389(2)	C22	C23	1.389(2)
C2	C3	1.388(2)	C23	C24	1.385(2)

Table S7 Bond angles for 3d.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
P2	Mo1	P1	89.700(14)	C1	C6	C5	116.56(15)
C25	Mo1	P1	91.32(4)	C1	C6	C7	122.56(14)
C25	Mo1	P2	89.71(4)	C5	C6	C7	120.87(14)
C25	Mo1	C26	87.92(6)	C8	C7	C6	121.11(14)
C25	Mo1	C27	177.64(6)	C12	C7	C6	121.74(14)
C25	Mo1	C28	91.22(6)	C12	C7	C8	117.12(15)
C26	Mo1	P1	176.57(4)	C9	C8	C7	120.97(16)
C26	Mo1	P2	93.65(4)	C10	C9	C8	120.45(16)
C27	Mo1	P1	89.45(4)	C9	C10	C11	119.94(16)
C27	Mo1	P2	88.06(4)	C12	C11	C10	119.03(16)
C27	Mo1	C26	91.44(6)	C7	C12	O2	119.01(14)
C28	Mo1	P1	86.52(4)	C11	C12	O2	118.30(14)
C28	Mo1	P2	176.13(4)	C11	C12	C7	122.49(15)
C28	Mo1	C26	90.15(6)	C14	C13	O3	117.40(13)
C28	Mo1	C27	91.06(6)	C14	C13	C18	122.44(14)
F1	P1	Mo1	116.66(4)	C18	C13	O3	119.90(13)
F1	P1	O1	101.77(6)	C13	C14	C15	119.10(15)
F1	P1	O2	94.83(6)	C14	C15	C16	119.92(15)
O1	P1	Mo1	114.16(4)	C17	C16	C15	120.02(15)
O1	P1	O2	102.39(6)	C16	C17	C18	121.42(15)
O2	P1	Mo1	123.39(4)	C13	C18	C17	117.11(14)
F2	P2	Mo1	116.06(4)	C13	C18	C19	122.21(14)
F2	P2	O3	101.44(6)	C17	C18	C19	120.67(14)
F2	P2	O4	93.86(5)	C20	C19	C18	121.14(14)
O3	P2	Mo1	115.96(4)	C24	C19	C18	121.71(13)
O3	P2	O4	102.51(6)	C24	C19	C20	117.04(14)
O4	P2	Mo1	122.98(4)	C21	C20	C19	121.13(15)
C1	O1	P1	123.21(10)	C20	C21	C22	120.15(15)
C12	O2	P1	119.02(10)	C21	C22	C23	120.10(15)
C13	O3	P2	122.65(9)	C24	C23	C22	118.76(15)
C24	O4	P2	119.04(9)	C19	C24	O4	118.80(13)
C2	C1	O1	117.11(14)	C23	C24	O4	118.18(14)
C2	C1	C6	122.90(14)	C23	C24	C19	122.82(14)
C6	C1	O1	119.76(13)	O5	C25	Mo1	178.16(13)

C1	C2	C3	119.18(15)	O6	C26	Mo1	177.26(13)
C4	C3	C2	119.65(15)	O7	C27	Mo1	177.93(14)
C5	C4	C3	120.18(15)	O8	C28	Mo1	177.28(14)
C4	C5	C6	121.52(15)				

Table S8 Bond lengths for 3d.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Mo1	P2	2.3968(6)	C6	C5	1.396(3)
Mo1	P1	2.4037(6)	C6	C7	1.512(3)
Mo1	C28	2.032(2)	C12	C11	1.382(3)
Mo1	C27	2.038(3)	C8	C9	1.400(3)
Mo1	C30	2.015(3)	C8	C7	1.518(3)
Mo1	C29	2.063(3)	C5	C4	1.383(3)
P2	F2	1.5741(14)	C19	C14	1.388(3)
P2	O3	1.5943(16)	C19	C20	1.514(3)
P2	O4	1.6018(17)	C19	C18	1.396(3)
P1	F1	1.5758(14)	C14	C15	1.376(3)
P1	O2	1.6006(16)	C21	C26	1.393(3)
P1	O1	1.6065(16)	C21	C20	1.513(3)
O2	C13	1.408(3)	C21	C22	1.390(3)
O1	C1	1.414(3)	C9	C10	1.384(4)
O3	C14	1.414(3)	C26	C25	1.381(3)
O4	C26	1.408(3)	C2	C3	1.389(3)
O6	C28	1.136(3)	C15	C16	1.387(4)
O8	C30	1.140(3)	C11	C10	1.378(4)
O7	C29	1.139(3)	C25	C24	1.379(3)
O5	C27	1.132(3)	C22	C23	1.384(3)
C13	C12	1.382(3)	C18	C17	1.381(4)
C13	C8	1.390(3)	C23	C24	1.387(3)
C1	C6	1.389(3)	C3	C4	1.380(4)
C1	C2	1.380(3)	C16	C17	1.381(4)

Table S9 Bond angles for 3d.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
P2	Mo1	P1	87.83(2)	C1	C6	C7	121.5(2)
C28	Mo1	P2	91.75(7)	C5	C6	C7	121.4(2)
C28	Mo1	P1	176.79(7)	C11	C12	C13	119.3(2)
C28	Mo1	C27	89.27(10)	C13	C8	C9	116.6(2)
C28	Mo1	C29	86.40(10)	C13	C8	C7	124.0(2)
C27	Mo1	P2	89.93(8)	C9	C8	C7	119.4(2)
C27	Mo1	P1	87.54(7)	C4	C5	C6	120.8(2)
C27	Mo1	C29	175.67(10)	O5	C27	Mo1	179.8(3)
C30	Mo1	P2	177.59(7)	C14	C19	C20	122.2(2)
C30	Mo1	P1	89.77(7)	C14	C19	C18	116.7(2)
C30	Mo1	C28	90.63(10)	C18	C19	C20	121.0(2)
C30	Mo1	C27	89.76(10)	C19	C14	O3	120.0(2)
C30	Mo1	C29	90.56(10)	C15	C14	O3	117.1(2)
C29	Mo1	P2	89.93(7)	C15	C14	C19	122.8(2)
C29	Mo1	P1	96.78(7)	C26	C21	C20	121.5(2)
F2	P2	Mo1	117.80(6)	C22	C21	C26	116.8(2)
F2	P2	O3	99.76(8)	C22	C21	C20	121.6(2)
F2	P2	O4	99.67(9)	C10	C9	C8	121.4(2)
O3	P2	Mo1	116.39(6)	C21	C26	O4	120.0(2)
O3	P2	O4	106.42(9)	C25	C26	O4	117.4(2)
O4	P2	Mo1	114.50(7)	C25	C26	C21	122.6(2)
F1	P1	Mo1	115.71(6)	C21	C20	C19	110.7(2)
F1	P1	O2	94.75(8)	O8	C30	Mo1	178.7(2)
F1	P1	O1	99.45(8)	C1	C2	C3	118.1(2)
O2	P1	Mo1	124.92(6)	O7	C29	Mo1	174.5(2)
O2	P1	O1	102.40(9)	C14	C15	C16	119.2(3)
O1	P1	Mo1	115.13(6)	C10	C11	C12	119.9(2)
C13	O2	P1	122.40(14)	C24	C25	C26	119.2(2)
C1	O1	P1	121.12(14)	C23	C22	C21	121.6(2)
C14	O3	P2	127.35(14)	C17	C18	C19	121.3(3)
C26	O4	P2	126.25(15)	C6	C7	C8	116.2(2)
C12	C13	O2	116.2(2)	C22	C23	C24	119.9(2)
C12	C13	C8	122.6(2)	C11	C10	C9	120.2(2)
C8	C13	O2	121.1(2)	C25	C24	C23	119.9(2)

O6	C28	Mo1	178.9(2)	C4	C3	C2	120.4(2)
C6	C1	O1	117.8(2)	C17	C16	C15	119.5(3)
C2	C1	O1	118.9(2)	C3	C4	C5	120.4(2)
C2	C1	C6	123.3(2)	C16	C17	C18	120.4(2)
C1	C6	C5	117.1(2)				

Table S10 Bond lengths for 3e.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Mo1	P2	2.4163(4)	C5	C10	1.411(2)
Mo1	P1	2.4270(4)	C5	C6	1.356(2)
Mo1	C4	2.0258(17)	C16	C21	1.409(2)
Mo1	C3	2.0500(18)	C16	C17	1.361(2)
Mo1	C2	2.0165(18)	C22	C21	1.413(2)
Mo1	C1	2.0341(18)	C22	C23	1.359(2)
P2	O9	1.6085(12)	C21	C20	1.424(2)
P2	O8	1.6218(12)	C23	C24	1.411(2)
P2	O10	1.6034(12)	C10	C11	1.408(2)
P1	O5	1.6069(12)	C10	C9	1.423(2)
P1	O6	1.6213(12)	C17	C18	1.413(3)
P1	O7	1.6002(13)	C11	C12	1.364(2)
O9	C22	1.4016(19)	C24	C25	1.361(3)
O8	C16	1.4014(19)	C20	C19	1.418(3)
O5	C5	1.4019(19)	C20	C25	1.412(3)
O10	C26	1.436(2)	C8	C9	1.412(3)
O6	C11	1.402(2)	C8	C7	1.362(3)
O7	C15	1.435(2)	C6	C7	1.411(3)
O4	C4	1.134(2)	C9	C14	1.413(3)
O1	C1	1.140(2)	C19	C18	1.360(3)
O3	C3	1.137(2)	C14	C13	1.359(3)
O2	C2	1.139(2)	C13	C12	1.418(3)

Table S11 Bond angles for 3e.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
P2	Mo1	P1	92.391(15)	C17	C16	O8	118.22(16)
C4	Mo1	P2	178.45(5)	C17	C16	C21	122.30(16)
C4	Mo1	P1	89.03(5)	O9	C22	C21	118.85(15)
C4	Mo1	C3	88.73(7)	C23	C22	O9	118.66(15)
C4	Mo1	C1	91.58(7)	C23	C22	C21	122.44(15)
C3	Mo1	P2	91.81(5)	O4	C4	Mo1	178.66(15)
C3	Mo1	P1	92.96(5)	C16	C21	C22	123.19(15)
C2	Mo1	P2	88.88(5)	C16	C21	C20	118.68(16)
C2	Mo1	P1	177.97(5)	C22	C21	C20	118.10(16)
C2	Mo1	C4	89.72(7)	C22	C23	C24	118.71(17)
C2	Mo1	C3	85.42(7)	C5	C10	C9	118.53(16)
C2	Mo1	C1	90.92(7)	C11	C10	C5	122.66(15)
C1	Mo1	P2	87.79(5)	C11	C10	C9	118.81(16)
C1	Mo1	P1	90.71(5)	C16	C17	C18	118.49(18)
C1	Mo1	C3	176.33(7)	O6	C11	C10	119.49(15)
O9	P2	Mo1	116.66(4)	C12	C11	O6	118.42(16)
O9	P2	O8	102.55(6)	C12	C11	C10	122.07(17)
O8	P2	Mo1	112.58(4)	C25	C24	C23	121.14(17)
O10	P2	Mo1	122.75(5)	C19	C20	C21	118.37(17)
O10	P2	O9	96.96(6)	C25	C20	C21	118.72(16)
O10	P2	O8	102.34(6)	C25	C20	C19	122.91(16)
O5	P1	Mo1	116.46(5)	C7	C8	C9	120.69(17)
O5	P1	O6	101.63(6)	C5	C6	C7	119.07(17)
O6	P1	Mo1	114.19(5)	O3	C3	Mo1	174.87(16)
O7	P1	Mo1	120.86(5)	C8	C9	C10	118.59(17)
O7	P1	O5	97.20(7)	C8	C9	C14	123.04(17)
O7	P1	O6	103.52(7)	C14	C9	C10	118.37(17)
C22	O9	P2	119.04(10)	C18	C19	C20	120.64(17)
C16	O8	P2	119.34(10)	C19	C18	C17	121.52(17)
C5	O5	P1	119.25(10)	C24	C25	C20	120.89(16)
C26	O10	P2	120.95(12)	O2	C2	Mo1	177.34(16)
C11	O6	P1	119.55(11)	C13	C14	C9	121.05(18)
C15	O7	P1	119.95(12)	O1	C1	Mo1	178.28(15)

O5	C5	C10	118.97(15)	C14	C13	C12	121.04(18)
C6	C5	O5	118.95(15)	C8	C7	C6	121.07(18)
C6	C5	C10	122.04(16)	C11	C12	C13	118.64(19)
O8	C16	C21	119.44(14)				

Table S12 Bond lengths for 4b. [1]1/2-X,3/2-Y,+Z; [2]-1/2+Y,1-X,-Z; [3]1-Y,1/2+X,-Z

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Pt1	P1	2.2303(12)	C6	C5	1.428(7)
Pt1	P1[1]	2.2303(12)	C2	C3	1.405(7)
Pt1	P1[2]	2.2303(12)	C9	C8	1.408(8)
Pt1	P1[3]	2.2303(12)	C9	C10	1.363(8)
P1	F1	1.575(3)	C3	C4	1.339(9)
P1	O1	1.596(3)	C5	C10	1.405(8)
P1	O2	1.604(3)	C5	C4	1.418(7)
Cl1	C11	1.700(12)	C11	C12	1.3900
O1	C1	1.406(6)	C11	C16	1.3900
O2	C7	1.412(6)	C12	C13	1.3900
C1	C6	1.405(7)	C13	C14	1.3900
C1	C2	1.354(7)	C14	C15	1.3900
C7	C6	1.401(7)	C15	C16	1.3900
C7	C8	1.348(7)			

Table S13 Bond angles for 4b. [1]-1/2+Y,1-X,-Z; [2]1/2-X,3/2-Y,+Z; [3]1-Y,1/2+X,-Z

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
P1	Pt1	P1[1]	107.61(3)	C1	C6	C5	118.0(5)
P1[1]	Pt1	P1[2]	107.61(3)	C7	C6	C1	123.3(5)
P1	Pt1	P1[2]	113.26(6)	C7	C6	C5	118.7(5)
P1[2]	Pt1	P1[3]	107.61(3)	C1	C2	C3	117.9(6)
P1	Pt1	P1[3]	107.61(3)	C10	C9	C8	120.8(5)
P1[1]	Pt1	P1[3]	113.25(6)	C4	C3	C2	122.0(6)
F1	P1	Pt1	118.74(12)	C10	C5	C6	117.9(6)
F1	P1	O1	99.47(17)	C10	C5	C4	124.3(6)
F1	P1	O2	98.34(18)	C4	C5	C6	117.8(6)

O1	P1	Pt1	116.97(13)	C7	C8	C9	118.9(5)
O1	P1	O2	102.61(18)	C9	C10	C5	121.2(6)
O2	P1	Pt1	117.37(13)	C3	C4	C5	121.1(6)
C1	O1	P1	121.9(3)	C12	C11	Cl1	119.7(7)
C7	O2	P1	121.3(3)	C12	C11	C16	120.0
C6	C1	O1	119.1(4)	C16	C11	Cl1	120.3(7)
C2	C1	O1	117.8(5)	C13	C12	C11	120.0
C2	C1	C6	123.1(5)	C12	C13	C14	120.0
C6	C7	O2	118.6(4)	C13	C14	C15	120.0
C8	C7	O2	118.9(5)	C16	C15	C14	120.0
C8	C7	C6	122.5(5)	C15	C16	C11	120.0

Table S14 Bond lengths for 4e.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.367(3)	C28	C29	1.416(3)
C1	C6	1.413(3)	C29	C30	1.364(3)
C1	O1	1.397(2)	C29	O8	1.391(2)
C2	C3	1.414(3)	C30	C31	1.411(3)
C3	C4	1.363(3)	C31	C32	1.369(4)
C4	C5	1.421(3)	C33	O9	1.436(2)
C5	C6	1.423(3)	C34	C35	1.368(3)
C5	C10	1.414(3)	C34	C39	1.413(3)
C6	C7	1.416(3)	C34	O10	1.400(2)
C7	C8	1.366(3)	C35	C36	1.417(3)
C7	O2	1.395(2)	C36	C37	1.361(3)
C8	C9	1.411(3)	C37	C38	1.417(3)
C9	C10	1.370(3)	C38	C39	1.425(3)
C11	O3	1.438(2)	C38	C43	1.413(3)
C12	C13	1.367(3)	C39	C40	1.412(3)
C12	C17	1.414(3)	C40	C41	1.361(3)
C12	O4	1.390(2)	C40	O11	1.392(2)
C13	C14	1.414(3)	C41	C42	1.413(3)
C14	C15	1.370(3)	C42	C43	1.363(3)
C15	C16	1.417(3)	C44	O12	1.440(2)

C16	C17	1.426(3)	O1	P1	1.6250(14)
C16	C21	1.419(3)	O2	P1	1.6337(13)
C17	C18	1.422(3)	O3	P1	1.6089(13)
C18	C19	1.364(3)	O4	P2	1.6338(14)
C18	O5	1.395(2)	O5	P2	1.6228(14)
C19	C20	1.411(3)	O6	P2	1.6111(15)
C20	C21	1.370(3)	O7	P3	1.6357(14)
C22	O6	1.440(2)	O8	P3	1.6179(14)
C23	C24	1.364(3)	O9	P3	1.6020(15)
C23	C28	1.416(3)	O10	P4	1.6253(13)
C23	O7	1.394(2)	O11	P4	1.6340(15)
C24	C25	1.410(3)	O12	P4	1.6039(14)
C25	C26	1.363(3)	P1	Pt1	2.2284(5)
C26	C27	1.421(3)	P2	Pt1	2.2386(5)
C27	C28	1.420(3)	P3	Pt1	2.2416(5)
C27	C32	1.414(3)	P4	Pt1	2.2413(5)

Table S15 Bond angles for 4e.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C6	122.04(18)	C34	C35	C36	118.8(2)
C2	C1	O1	118.55(18)	C37	C36	C35	121.52(19)
O1	C1	C6	119.40(17)	C36	C37	C38	120.51(19)
C1	C2	C3	118.5(2)	C37	C38	C39	118.54(19)
C4	C3	C2	121.5(2)	C43	C38	C37	122.73(19)
C3	C4	C5	120.65(19)	C43	C38	C39	118.73(19)
C4	C5	C6	118.32(19)	C34	C39	C38	119.08(17)
C10	C5	C4	122.96(19)	C40	C39	C34	122.51(17)
C10	C5	C6	118.71(19)	C40	C39	C38	118.40(18)
C1	C6	C5	118.91(18)	C41	C40	C39	122.02(19)
C1	C6	C7	122.36(17)	C41	C40	O11	118.60(18)
C7	C6	C5	118.73(18)	O11	C40	C39	119.35(17)
C8	C7	C6	121.74(18)	C40	C41	C42	119.1(2)
C8	C7	O2	118.64(17)	C43	C42	C41	121.0(2)
O2	C7	C6	119.61(16)	C42	C43	C38	120.8(2)

C7	C8	C9	119.0(2)	C1	O1	P1	119.76(12)
C10	C9	C8	121.2(2)	C7	O2	P1	119.59(12)
C9	C10	C5	120.60(19)	C11	O3	P1	120.71(13)
C13	C12	C17	121.82(18)	C12	O4	P2	119.30(12)
C13	C12	O4	118.49(17)	C18	O5	P2	119.73(12)
O4	C12	C17	119.63(16)	C22	O6	P2	118.19(13)
C12	C13	C14	119.18(19)	C23	O7	P3	120.55(12)
C15	C14	C13	120.88(19)	C29	O8	P3	121.12(12)
C14	C15	C16	120.79(19)	C33	O9	P3	122.44(14)
C15	C16	C17	118.66(18)	C34	O10	P4	119.92(12)
C15	C16	C21	122.98(19)	C40	O11	P4	120.54(12)
C21	C16	C17	118.32(19)	C44	O12	P4	118.00(13)
C12	C17	C16	118.67(18)	O1	P1	O2	100.86(7)
C12	C17	C18	122.31(17)	O1	P1	Pt1	117.57(5)
C18	C17	C16	119.01(17)	O2	P1	Pt1	113.39(5)
C19	C18	C17	121.53(18)	O3	P1	O1	96.14(7)
C19	C18	O5	119.07(17)	O3	P1	O2	101.14(7)
O5	C18	C17	119.35(16)	O3	P1	Pt1	123.93(5)
C18	C19	C20	119.11(19)	O4	P2	Pt1	113.57(5)
C21	C20	C19	121.40(19)	O5	P2	O4	101.15(7)
C20	C21	C16	120.60(19)	O5	P2	Pt1	119.15(5)
C24	C23	C28	121.46(18)	O6	P2	O4	101.31(7)
C24	C23	O7	118.85(17)	O6	P2	O5	96.29(7)
O7	C23	C28	119.63(17)	O6	P2	Pt1	121.77(5)
C23	C24	C25	119.3(2)	O7	P3	Pt1	115.69(5)
C26	C25	C24	121.1(2)	O8	P3	O7	100.41(7)
C25	C26	C27	120.7(2)	O8	P3	Pt1	113.56(6)
C28	C27	C26	118.43(19)	O9	P3	O7	100.90(8)
C32	C27	C26	122.9(2)	O9	P3	O8	97.85(8)
C32	C27	C28	118.6(2)	O9	P3	Pt1	124.54(6)
C23	C28	C27	119.00(18)	O10	P4	O11	101.16(7)
C23	C28	C29	122.26(18)	O10	P4	Pt1	120.90(5)
C29	C28	C27	118.69(18)	O11	P4	Pt1	111.36(5)
C30	C29	C28	122.02(19)	O12	P4	O10	96.59(7)
C30	C29	O8	119.02(19)	O12	P4	O11	101.62(8)

O8	C29	C28	118.89(17)	O12	P4	Pt1	121.73(6)
C29	C30	C31	118.7(2)	P1	Pt1	P2	104.822(17)
C32	C31	C30	121.3(2)	P1	Pt1	P3	113.362(18)
C31	C32	C27	120.6(2)	P1	Pt1	P4	112.554(17)
C35	C34	C39	121.50(18)	P2	Pt1	P3	104.625(18)
C35	C34	O10	118.68(18)	P2	Pt1	P4	107.783(18)
O10	C34	C39	119.76(16)	P4	Pt1	P3	112.868(17)

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