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Differences in the η^1-ligating properties of 2,4,6-tritertiarybutyl-phosphabenzene, $PC_5H_2Bu^t_3$ and 2,4,6-tritertiarybutyl-1,3,5-triphosphabenzene, $P_3C_3Bu^t_3$

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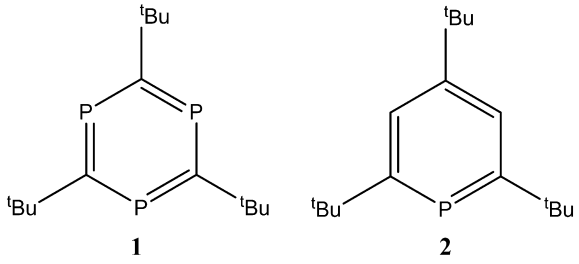
ABSTRACT

Several η^1-complexes of 2,4,6-tritertiarybutylphosphabenzene, $PC_5H_2Bu^t_3$, have been synthesised and structurally characterised including *trans*-[$PtCl_2(PEt_3)(\eta^1$-$PC_5Bu^t_3)$], *cis*-[$PdCl(C_{10}H_6CHMe(NMe_2)(\eta^1$-$PC_5H_2Bu^t_3)$] and [$AuCl(\eta^1$-$PC_5H_2Bu^t_3)$]. NMR spectroscopic evidence is presented for the partial isomerisation in solution of the 2,4,6-tritertiarybutyl-1,3,5-triphosphabenzene complexes *trans*-[$PtCl_2(PR_3)(\eta^1$-$P_3C_3Bu^t_3)$], ($PR_3 = PMe_3$ and PMe_2Ph), to the corresponding *cis*-isomers.

1. Introduction

Although previous studies have shown that the 2,4,6-tritertiarybutyl-1,3,5-triphosphabenzene ring **1** readily acts as a 6π-electron donor towards metal centres [1–3], the only reported examples of η^1-bonded metal complexes are *trans*-[$PtCl_2(PR_3)(\eta^1$-$P_3C_3Bu^t_3)$] ($PR_3 = PMe_3$, PEt_3, PMe_2Ph and $PMePh_2$) [4,5]. We therefore sought to investigate the relative σ-bonding properties of the structurally related 2,4,6-tritertiarybutylphosphabenzene, $PC_5Bu^t_3$ **2** and **1**, in view of current interest in the catalytic potential of these types of sp^2-hybridised phosphorus-containing rings and related phosphabarrelenes [6–14].

Recently, we showed for the first time that both **1** and **2** could be protonated, alkylated and silylated at phosphorus using appropriate electrophilic reagents having halogenated carborane anions [15], despite the extremely weakly basic and poor nucleophilic nature of both ring systems [10,15–17]. We now report on the unexpected and strikingly different σ-ligating ability of the two rings **1** and **2** toward platinum metals. Although the coordination chemistry of several monophosphinine ring systems has been widely studied [10,12], only a few σ-complexes of **2** have been structurally characterised [15,18]. We now describe the syntheses and single-crystal X-ray structural characterisation of the 2,4,6-tritertiarybutylphosphabenzene complexes *trans*-[$PtCl_2(PEt_3)(\eta^1$-$PC_5H_2Bu^t_3)$], *cis*-[$PdCl(C_{10}H_6CHMe(NMe_2)(\eta^1$-$PC_5H_2Bu^t_3)$] and [$AuCl(\eta^1$-$PC_5H_2Bu^t_3)$]. We also have carried out ^{31}P NMR spectroscopic experiments which show that some *trans*-[$PtCl_2(PR_3)(\eta^1$-$P_3C_3Bu^t_3)$] complexes undergo partial isomerisation to the *cis*-isomer at ambient temperature.



1 **2**

2. Results and discussion

The attempted reaction of **1** with di-μ-chloro-bis[(R)-dimethyl(1-ethyl-α-naphthyl)-aminato-C^2,N] palladium(II), **3** [19] (Fig. 1), or [AuCl(tht)] only resulted in the recovery of unreacted starting materials, as shown by ^{31}P NMR spectroscopic monitoring, the only observed resonance being that characteristic of **1** ($\delta_P = 232$ ppm). Changing the solvent and/or varying the reaction temperatures produced no observable effect. Complex **3** was chosen in an attempt to obtain the first chiral triphosphabenzene complex, while [AuCl(tht)] (tht = tetrahydrothiophen) normally readily exchanges its very labile (tht) on treatment with tertiary phosphines.

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This surprising lack of reactivity of **1** (which we had also noted previously [5] towards a variety of metal halides in different oxidation states e.g. $TiCl_4$, $SnCl_4$, $RhCl_3$, $PdCl_2$ and $PtCl_2$), no doubt reflects the significant *s*-character of the phosphorus lone-pair electrons in **1**.

On the other hand, treatment of **2** with $[Pt_2Cl_4(PR_3)_2]$ (R = PEt_3) in CH_2Cl_2 readily resulted in the formation of *trans*-$[PtCl_2(PEt_3)(\eta^1\text{-}PC_5H_2Bu^t_3)]$ **4** whose ^{31}P NMR spectrum shows the characteristically large *trans* P–P coupling constant; (ring phosphorus δ_P = 161.4 ppm; ($^2J(P_2P_1)$ 526.8 Hz; $^1J(PtP_1)$ 2520 Hz; PEt_3 δ_P = 10.9 ppm ($^2J(P_2P_1)$ 526.9 Hz, $^1J(PtP_2)$ 2935 Hz). Comparable coupling constant data found in the related triphosphabenzene complex *trans*-$[PtCl_2(PEt_3)(\eta^1\text{-}P_3C_3Bu^t_3)]$ **5** are $^2J(P_2P_1)$ 508.5 Hz; $^1J(PtP_1)$ 2378 Hz; and $^1J(PtP_2)$ 2884 Hz) [4]. The slightly higher $^1J(PtP_1)$ ring couplings for **4** probably reflect the slightly greater *s*-character in the monophosphabenzene system **1** than in **2**, reflecting the greater overall electron withdrawing properties of phosphorus compared with carbon, which we previously established by both PE measurements and DFT calculations [20].

Structural characterisation of **4** was established by a single-crystal X-ray diffraction study and its molecular structure is shown in Fig. 2, together with selected bond lengths and angles.

In contrast to the unreactivity of **1**. treatment of **2** with 0.5 equivalents of the palladium complex **3** readily results in the formation of $[PdCl(C_{10}H_6CHMeNMe_2)(\eta^1\text{-}PC_5H_2Bu^t_3)]$ **6**. The ^{31}P NMR spectrum of **6** exhibits a single resonance (δ_P = 167.6 ppm) corresponding to the η^1-Pt-bonded phosphorus atom. Interestingly, the ^{31}P resonance of **2** was often observed in solution (δ_P = 180 ppm), indicating that an equilibrium exists between complex **6** and the free ring. In support of these observations, recrystallisation of the reaction mixture often yielded crystals of both **6** and **3** even if **2** were present in excess. A single-crystal X-ray diffraction study revealed the molecular structure of **6**, which is presented in Fig. 3, together with selected bond lengths and angles.

The facile reaction of **2** with [AuCl(tht)] in CH_2Cl_2 affords **7** whose ^{31}P NMR spectrum shows a single resonance (δ_P = 154.6 ppm), which is appreciably shifted to low frequency compared to the resonance observed for the free ring. A single-crystal X-ray diffraction study confirms the molecular structure of **7** shown in Fig. 4, together with selected bond length and angle data.

As expected, the geometry around the metal centre is linear and the Au–P (2.2194(15) Å) distance is not unusual for this type of complex. Interestingly the AuCl fragment projects out from the sp^2-hybridised ring P and above the plane of the aromatic ring with a ring plane–Au–P angle of approximately 12.3°. A detailed comparison of bond lengths and other structural features is not possible since the molecular structure of the free ring **2** is yet to be determined.

In summary, we have show that the σ-donor properties of the 2,4,6-tritertiarybutyl-1,3,5-triphosphabenzene ring **1** towards transition metals are significantly less than those of the corresponding 2,4,6-tritertiarybutylphosphabenzene ring **2** despite the P lone-pair electrons in both aromatic rings being flanked by two bulky C^{t}Bu substituents. Furthermore, although the structurally confirmed t*rans*-stereochemistry of **4** is in full agreement with that originally proposed by us [4] for the analogous 2,4,6-tritertiarybutyl-1,3,5-triphosphabenzene systems, we propose that both are the *kinetic* rather than *thermodynamic* products in view of ^{31}P NMR spectroscopic studies (*vide infra*).

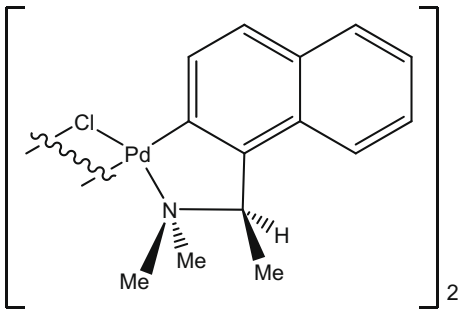

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Fig. 1. Di-μ-chloro-bis[(R)-dimethyl(1-ethyl-α-naphthyl)-aminato-C^2,N] palladium(II).

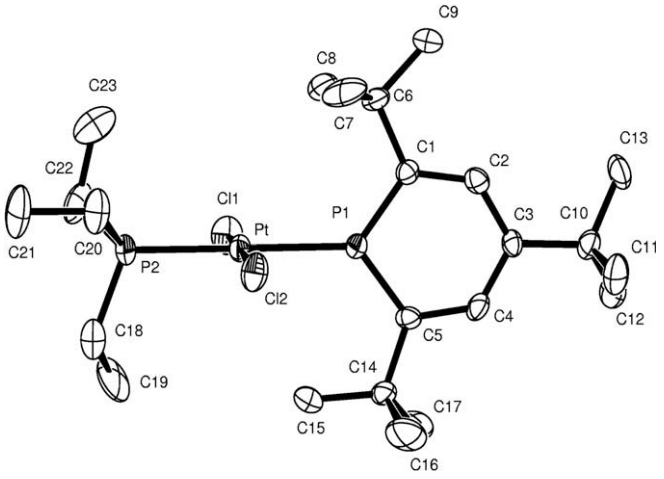

Fig. 2. Molecular structure of *trans*-$[PtCl_2(PEt_3)(\eta^1\text{-}PC_5H_2Bu^t_3)]$ **4**. Selected bond lengths (Å) and angles (°): Pt–P(2) 2.285(2), Pt–Cl(2) 2.309(2), Pt–Cl(1) 2.320(2), Pt–P(1) 2.335(2), P(1)–C(5) 1.726(8), P(1)–C(1) 1.741(8), C(4)–C(5) 1.397(11), C(2)–C(3) 1.391(11), C(3)–C(4) 1.394(11), C(1)–C(2) 1.391(11). C(5)–P(1)–C(1) 107.3(4), C(5)–P(1)–Pt 126.5(3), C(1)–P(1)–Pt 125.9(3), C(2)–C(1)–C(6) 121.2(7), C(2)–C(1)–P(1) 118.8(6), C(6)–C(1)–P(1) 120.0(6), C(1)–C(2)–C(3) 127.4(7), C(2)–C(3)–C(4) 120.1(7), C(4)–C(5)–P(1) 118.3(6), C(3)–C(4)–C(5) 128.0(7).

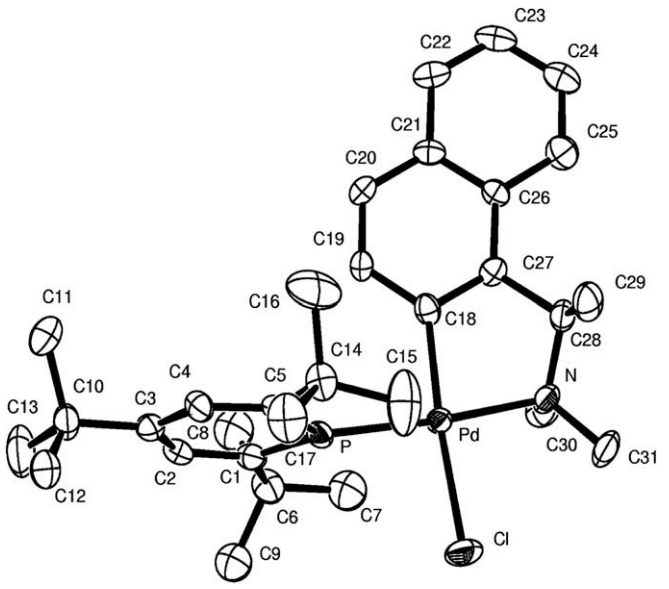

Fig. 3. Molecular structure of *cis*-$[PdCl(C_{10}H_6CHMeNMe_2)(\eta^1\text{-}PC_5H_2Bu^t_3)]$ **6**. Selected bond lengths (Å) and angles (°): Pd–C(18) 2.008(4), Pd–N 2.112(3), Pd–P 2.2358(11), Pd–Cl 2.4057(11), P–C(1) 1.716(4), P–C(5) 1.729(4), C(1)–C(2) 1.386(6), C(4)–C(5) 1.392(6), C(3)–C(4) 1.400(6), C(2)–C(3) 1.401(6). C(18)–Pd–N 81.27(14), C(18)–Pd–P 93.23(12), N–Pd–P 172.91(9), C(18)–Pd–Cl 175.31(12), N–Pd–Cl 94.88(9), P–Pd–Cl 90.80(4), C(1)–P–C(5) 106.6(2), C(1)–P–Pd 126.11(14), C(5)–P–Pd 126.35(15), C(2)–C(1)–P 120.2(3), C(4)–C(5)–P 119.3(3), C(5)–C(4)–C(3) 127.0(4), C(1)–C(2)–C(3) 126.6(4), C(4)–C(3)–C(2) 120.2(4).

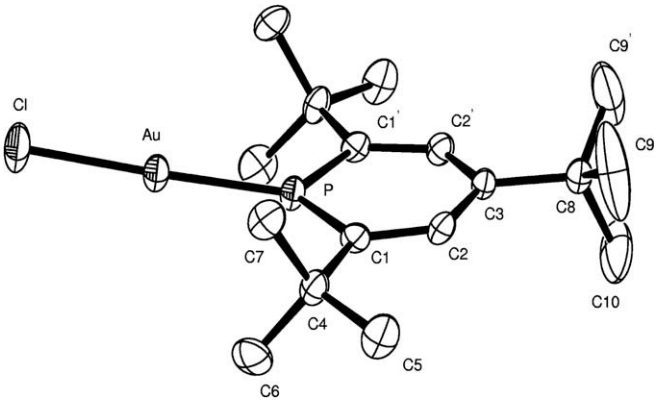

Fig. 4. Molecular structure of [AuCl(η-$PC_5H_2Bu^t_3$)]. **7**. Selected bond lengths (Å) and angles (°): Au–P 2.2194(15), Au–Cl 2.2654(17), P–C(1) 1.726(4), C(1)–C(2) 1.387(5), C(3)–C(2) 1.398(5). P–Au–Cl 177.76(6), C(1)'–P–C(1) 108.0(3), C(1)–P–Au 125.29(13), C(2)–C(3)–C(2)' 120.8(5), C(1)–C(2)–C(3) 127.0(4), C(2)–C(1)–P 118.5(3).

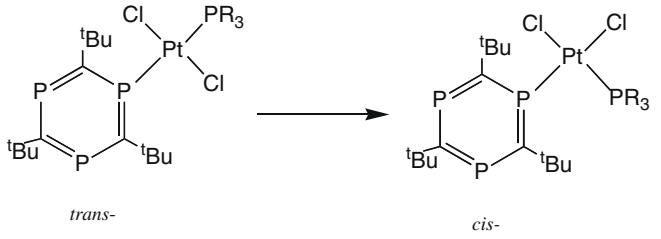

Fig. 5. *Trans*- to *cis*-isomerisation of [$PtCl_2(PEt_3)(P_3C_3Bu^t_3)$].

Thus, as previously reported,[4], the reaction of the chloro-bridged dimers [$Pt_2Cl_4(PR_3)_2$] with triphosphabenzene **1** immediately *quantitatively* afforded the *trans*-η^1-complexes [$PtCl_2(PR_3)(P_3C_3Bu^t_3)$] ($PR_3$ = PEt_3 **5**, PMe_3 **8**, PMe_2Ph **9** and $PMePh_2$ **10**) which were fully spectroscopically characterised by ^{31}P and ^{195}Pt NMR spectroscopy as the sole products.

In more detailed solution NMR studies we have found that a slow isomerisation process takes place in *some* (but not all) of the above systems. In the case of the *trans*-complexes **8** and **9** a spontaneous partial isomerisation to the *cis*-isomers **8a** and **9a** is observed at room temperature (Fig. 5), the relatively slow rate of isomerisation being easily followed by comparison of the relative intensities of the peaks in the ^{31}P NMR spectrum corresponding to each isomer (data are listed in Section 3). The ratio of isomers observed at different times were found to be (i) **8**: *cis*:*trans* = 0:100 (40 min); 2:98 (5 h); 20:80 (20 h); 26:74 (22 h). (ii) **9**: *cis*:-*trans* = 3:97 (30 min); 47:53 (17 h); 57:43 (41 h) whence it can be seen that both the rate of formation and degree of conversion from *trans*- to *cis*-isomers are higher for **9** than for **8**. Thus, although the *trans*-isomer is formed initially as the kinetic product of the bridge splitting reactions, the thermodynamically favoured product is the *cis*-isomer.

3. Experimental

Standard Schlenk tube procedures were employed and all solvents were rigorously dried and redistilled before use. Starting materials and metal complexes were made by published literature procedures.

3.1. Preparation of trans-[$PtCl_2(PR_3)(\eta^1$-$PC_5H_2Bu^t_3)$] **4**

$PC_5H_2Bu^t_3$ (0.1 g, 3.8×10^{-4} mol) and [$Pt_2Cl_4(PEt_3)_2$] (0.145 g, 1.98×10^{-4} mol) were combined and dissolved in CH_2Cl_2 (20 ml). After stirring for 12 h, the solvent was removed to give the product as a yellow solid. *n*-Pentane (5 ml) was added to a sample of the product ~80 mg and the resulting suspension was filtered hot into a round-bottomed flask. After 1 month at room temperature yellow crystals of the product were obtained. Yield = 180 mg, 73%. ^{1}H NMR (400 MHz, CD_2Cl_2): 8.10, 8.05 (m, ring H's), 1.96 (m, 6H, P{CH_2CH_3}$_3$), 1.79 (s, 18H, Bu^t), 1.39 (s, 9H, Bu^t), 1.27, 1.23 (2 overlapping t, 9H, P{CH_2CH_3}$_3$, 7.61 Hz). ^{31}P NMR (CD_2Cl_2, 161.9 MHz): (δ_P = 161.4 (d, ring P, $^2J(P_AP_X)$ 526.8, $^1J(PtP_A)$ 2520 Hz), (δ_P = 10.9 (d, PEt_3, $^2J(P_XP_A)$ 526.9, ^1J(PtPx) 2935 Hz).

Crystal data for ***4****:* $C_{23}H_{44}Cl_2P_2Pt$, M = 648.51, monoclinic, space group $P2_1/n$ (No.14), a = 11.9240(3) Å, b = 15.9942(5) Å, c = 15.1677(3) Å, β = 107.503(2)°, V = 2758.78(12) Å^3, T = 173(2) K, Z = 4, D_c = 1.56 Mg m^3, μ = 5.40 mm^{-1}, λ = 0.71073 Å, crystal size 0.15 × 0.10 × 0.10 mm^3, 20 506 measured reflections, 5394 independent reflections, 4472 reflections with $I > 2\sigma(I)$, Final indices R_1 = 0.043, wR_2 = 0.097 for $I > 2\sigma(I)$, R_1 = 0.057, wR_2 = 0.102 for all data. Data collection: KappaCCD, Program package WINGX, Abs correction MULTISCAN Refinement using SHELXL-97, Drawing using ORTEP-3 for Windows. There are two residual peaks of ca. 2 e Å^{-3} which make no chemical sense and are assumed to be artifacts

3.2. Preparation of [$PdCl(C_{10}H_6CHMe(NMe_2)(\eta^1$-$PC_5H_2Bu^t_3)$] **6**

$PC_5H_2Bu^t_3$ (0.035 g, 1.3×10^{-4} mol) and **5** (0.044 g, 6.5×10^{-5} mol) were combined, dissolved in toluene and stirred vigorously for 24 hours. The solution was reduced to 2 ml in volume and filtered hot into a round-bottomed flask. Storage at −50 °C for 1 week resulted in yellow crystals. Yield = 28 mg, 35%. ^{31}P NMR (CD_2Cl_2, 161.9 MHz): (δ_P = 167.6 (s, ring P).

Crystal data for ***6****:* $C_{31}H_{45}ClNPPd.(C_7H_8)$, M = 696.63, orthorhombic, space group $P2_12_12_1$ (No. 19), a = 12.1168(3) Å, b = 13.9137(2) Å, c = 21.5795(5) Å, V = 3638.08(13) Å^3, T = 173(2) K, Z = 4, D_c = 1.27 Mg m^3, μ = 0.65 mm^{-1}, λ = 0.71073 Å, crystal size 0.25 × 0.25 × 0.10 mm^3, 23 598 measured reflections, 7120 independent reflections, 6164 reflections with $I > 2\sigma(I)$, Final indices R_1 = 0.039, wR_2 = 0.083 for $I > 2\sigma(I)$, R_1 = 0.052, wR_2 = 0.090 for all data. Data collection: KappaCCD, Program package WINGX, Abs correction MULTISCAN Refinement using SHELXL-97, Drawing using ORTEP-3 for Windows. The disordered methyl C atoms for the C(6) ^{t}Bu group were left isotropic.

3.3. Preparation of [$AuCl(\eta^1$-$PC_5H_2Bu^t_3)$] **7**

$PC_5H_2Bu^t_3$ (0.03 g, 1.1×10^{-4} mol) and [AuCl(tht)] (0.036 g, 1.1×10^{-4} mol) were combined and dissolved in toluene (20 ml). After stirring for 20 h, the solvent was removed and the remaining solid dissolved in *n*-pentane (5 ml). The solution was filtered hot into a round-bottomed flask and stored at room temperature for 1 week. Colourless crystals of the product were obtained. Yield = 45 mg, 80%. ^{1}H NMR (400 MHz, C_6D_6): 7.95, 7.89 (s, ring H's), 1.41 (s, 18H, Bu^t), 1.07 (s, 9H, Bu^t). ^{31}P NMR (C_6D_6, 161.9 MHz): (δ_P = 154.6 (s, ring P).

Crystal data for ***7****:* $C_{17}H_{29}AuClP$, M = 496.79, monoclinic, space group $P2_1/m$ (No. 11), a = 6.1103(1) Å, b = 15.5516(5) Å, c = 10.2639(3) Å, β = 104.442(2)°, V = 944.51(4) Å^3, T = 173(2) K, Z = 2, D_c = 1.75 Mg m^3, μ = 8.01 mm^{-1}, λ = 0.71073 Å, crystal size 0.25 × 0.20 × 0.10 mm^3, 14 613 measured reflections, 1912 independent reflections, 1805 reflections with $I > 2\sigma(I)$, Final indices R_1 = 0.028, wR_2 = 0.072 for $I > 2\sigma(I)$, R_1 = 0.030, wR_2 = 0.074 for all data. Data collection: KappaCCD, Program package WINGX, Abs correction MULTISCAN Refinement using SHELXL-97, Drawing using ORTEP-3 for Windows. The molecule lies on a crystallographic mirror plane.

3.4. Formation of cis-[$PtCl_2(PR_3)(\eta^1$-$P_3C_3Bu^t_3)$] (R = PMe_3 8a, PMe_2Ph 9a) from the corresponding trans-isomers

$P_3C_3Bu^t_3$ and 0.5 molar equivalents of $[PtCl_2(PR_3)]_2$ (PR_3 = PMe_3, PMe_2Ph) were combined, dissolved in a minimal volume of CH_2Cl_2 or $CHCl_3$ and the solution stirred for 30 min to afford a yellow solution of *trans*-[$PtCl_2(PR_3)(\eta^1$-$P_3C_3Bu^t_3)$] (PR_3 = PMe_3, PMe_2Ph) in *quantitative* yield. Removal of the solvent *in vacuo* yielded a yellow powder. *Trans*- to *cis*-isomerism was monitored by ^{31}P NMR spectroscopy: Compound **8**: *cis*:*trans* = 0:100 (40 min); 2:98 (5 h); 20:80 (20 h); 26:74 (22 h). Compound **9**: *cis*:*trans* = 3:97 (30 min); 47:53 (17 h); 57:43 (41 h).

$^{31}P\{1H\}$ NMR data for *trans*-isomers **8** and **9**: (δ_P in ppm; *J* in Hz) (**8**): $^{31}P\{1H\}$ NMR (121.5 MHz, $CDCl_3$): AB_2XY system, δ_P = 264.8 [d, $^2J_{P(A)P(B)}$ 36.3, P(B)]; δ_P = 203.0 [dt; $^2J_{P(A)P(X)}$ 543.3, $^2J_{P(A)P(B)}$ 36.3, $^1J_{PtP(A)}$ 2418; P(A)]; δ_P = −18.3 [d; $^2J_{P(A)P(X)}$ 543.3, $^1J_{PtP(X)}$ 2886; P(X)]. ^{195}Pt NMR (107.496 MHz, $CDCl_3$): δ = −3705 (dd; $^1J_{PtP(A)}$ 2418, $^1J_{PtP(X)}$ 2886). (**9**): δ_P = 265.4 [d, $^2J_{P(A)P(B)}$ 36.6, P(B)]; δ_P = 202.5 [dt; $^2J_{P(A)P(X)}$ 543.6, $^2J_{P(A)P(B)}$ 36.6, $^1J_{PtP(A)}$ 2487; P(A)]; δ_P = −11.4 [d; $^2J_{P(A)P(X)}$ 543.6, $^1J_{PtP(X)}$ 2920; P(X)]. ^{195}Pt NMR (107.496 MHz, $CDCl_3$): δ = −3710 (dd; $^1J_{PtP(A)}$ 2487, $^1J_{PtP(X)}$ 2884).

$^{31}P\{1H\}$ NMR data for *cis*-isomers **8a** and **9a**: (**8a**): $^{31}P\{1H\}$ NMR (121.5 MHz, $CDCl_3$): AB_2XY system, δ_P = 274.2 [d, $^2J_{P(A)P(B)}$ 42.0, P(B)]; δ_P = 173.3 [td; $^2J_{P(A)P(B)}$ 42.0, $^2J_{P(A)P(X)}$ 25.3, $^1J_{PtP(A)}$ 4196; P(A)]; δ_P = −23.2 [d; $^2J_{P(A)P(X)}$ 25.3, $^1J_{PtP(X)}$ 3261; P(X)]. (**9a**): δ = 275.9 [d, $^2J_{P(A)P(B)}$ 42.2, P(B)]; δ_P = 172.2 [td; $^2J_{P(A)P(B)}$ 42.2, $^2J_{P(A)P(X)}$ 25.0, $^1J_{PtP(A)}$ 4192; P(A)]; δ_P = −19.3 [d; $^2J_{P(A)P(X)}$ 25.0, $^1J_{PtP(X)}$ = 3352; P(X)].

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2009.12.005.

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