Naphthyl-Based PCP Platinum Complexes. Nucleophilic Activation of Coordinated CO and Synthesis of a Pt(II) Formyl Complex

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Received January 23, 2007

A series of naphthyl-based Pt(II) complexes was synthesized and characterized. A single-crystal X-ray study of (PCP)PtCl (2) reveals stacking of the aromatic units between each pair of molecules of 2. Chloride abstraction from 2 under nitrogen atmosphere leads to formation of the unsaturated cationic complex [(PCP)Pt]BF4-(3), with the metal center being stabilized by the counteranion (3a) or by the solvent (3b). Abstraction of the chloride ligand from 2 under CO atmosphere leads to formation of the cationic carbonyl complex [(PCP)Pt(CO)]BF4-(4), containing an electrophilic carbonyl ligand. The latter is attacked by nucleophiles (MeO- and H-) to give the platinum carbonyl complex 5 and a rare platinum formyl complex, 6. Stabilized by the bulky bis-chelating tridentate pincer-type system, the formyl complex 6 was isolated and characterized. Complex 6 is more stable than the previously reported platinum formyls, at room temperature complex 6 is slowly converted (during days) into a hydrido complex, 7.

Introduction

Nucleophilic attack on coordinated carbon monoxide is well known for various nucleophiles.1 Of particular interest is hydride nucleophilic addition, since it can lead to transition-metal formyl complexes,2 which have been proposed as intermediates in the Fischer–Tropsch reaction.2 While stable formyl complexes have been described for several group VIII and group IX late transition metals,3 only two reports of platinum formyl complexes are available:2a in organometallic reactions and mechanisms, in catalysis, and in the design of new materials.3 The high thermal stability of such complexes, particularly those based on an aromatic backbone, permits their use as catalysts at elevated temperatures in various catalytic applications.3 Bulky bis-chelating pincer-type ligands are effective in the stabilization of highly unsaturated cationic complexes and in the stabilization of reactive species.5a,6a

We have recently prepared naphthyl-based PCP-type rhodium complexes and have shown that they can exhibit unusual reactivity modes.5a With an interest in the generation of stable and isolable neutral Pt(II) formyl complexes, we have decided

(II) formyl complex [Na][(η2-Tp)(Me)[(C(O)H)](TpH = hydridotriss(3,5-dimethylpyrazolyl)borate) was formed by hydride attack on the carbonyl ligand of Tp(Me)(CO)). Deprotonation of the metal center at 193 K resulted in the Pt(IV) formyl hydride complex Tp(IV)(Me)[(C(O)H)]H.4a The second example describes a dinuclear platinum formyl complex, formed upon treatment of a dinuclear platinum carbonyl complex with a borohydride reagent.4b

Pincer-type complexes constitute a family of compounds that have attracted much recent interest. They play important roles in organometallic reactions and mechanisms, in catalysis, and in the design of new materials.5 The high thermal stability of such complexes, particularly those based on an aromatic backbone, permits their use as catalysts at elevated temperatures in various catalytic applications.5 Bulky bis-chelating pincer-type ligands are effective in the stabilization of highly unsaturated cationic complexes and in the stabilization of reactive species.5a,6a

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‡ Concentrating and H2O (see Eqs 1 and 2).
§ Aromatic ring.


A platinum carbomethoxy complex is also described. The synthesis and reactivity of the PCP Pt(II) formyl complex. The nucleophilic formyl might kinetically stabilize the susceptibility toward nucleophilic addition. In addition, the steric bulk of the phosphine ligand might kinetically stabilize the formyl.

In this paper we report the synthesis and characterization of novel naphthyl-based PCP Pt(II) complexes. The nucleophilic reactivity of a cationic carbonyl complex is described, including the synthesis and reactivity of the PCP Pt(II) formyl complex. A platinum carbomethoxy complex is also described.

**Results and Discussion**

**Synthesis and Characterization of a Naphthyl-Based PCP Pt(II) Complex.** The PCP naphthyl-based ligand 1 was synthesized by treatment of 1,3-di(bromomethyl)naphthalene with di-tert-butylphosphine in the presence of triethylamine.7 The new complex 2 was obtained as a result of C–H activation and methane elimination in the reaction of ligand 1 with 1 equiv of (COD)Pt(Me)Cl at room temperature for 1 h (Scheme 1).

Interestingly, the cyclometalation to form the naphthyl-based complex 2 occurs at substantially less vigorous conditions than those used in the preparation of its phenyl-based analogue [(C6H3(CH2tBu2)2)Pt(Cl)].8 Both these complexes were prepared in our hands by the reaction of the corresponding PCP ligand with 1 equiv of (COD)Pt(Me)Cl but the phenyl-based analogue was obtained only upon heating at 100 °C for 12 h. Since the methane reductive elimination is expected to be facile and the C–H activation is expected to be similar in both cases, it may be that the barrier for COD displacement is lower for the naphthyl-based ligand, although the reasons for that are not clear.9

The purified complex 2 was characterized by 31P, 1H, and 13C NMR spectroscopy. The 31P{1H} NMR spectrum of 2 exhibits an AB system with a “roof” effect, due to the asymmetry of the phosphines in the complex. The AB system doublet of doublets is centered at 64.7 ppm (J_Pt–P = 374 Hz) with Pt satellites (J_Pt–P = 2858 Hz). In the 13C{1H} NMR spectrum the ipso carbon appears as a virtual triplet at 148.46 ppm (J_Pt–C = 7 Hz). X-ray quality colorless crystals of complex 2 were obtained by recrystallization from pentane at room temperature. The single-crystal X-ray study reveals a slightly distorted square-planar structure (Figure 1). The P–Pt–Cl angles are about 96°, while the P–Pt–C angles are approximately 84°. The aryl C–C bond lengths and angles are quite similar to those of naphthalene. Selected bond angles and bond lengths are given in Table 1. A comparison of the naphthyl-based bisphosphine ligand structure in 2 and the structure of naphthalene10a shows that both C–C bonds near the ipso carbon are slightly elongated (1.389(7) and 1.436(7) Å) relative to the corresponding bonds in naphthalene (1.378 Å), and 1.415 Å); this reflects the strain imposed by the metal center on the ipso carbon.

Interestingly, the crystal packing of complex 2 consists of alternating two-molecule stacks (Figure 2), which are held together by face-to-face π–π interactions between each pair of molecules. The distance between two aryl planes in the stack is 3.54 Å, which is consistent with aromatic stacking.10b

**Cationic PCP Pt(II) Complexes.** Upon reaction of compound 2 with AgBF4 in THF at room temperature for 2 days (protected from light), the 31P{1H} NMR spectrum revealed formation of complexes 3a and 3b in a 1:2 ratio, respectively (Scheme 2).

The 31P{1H} NMR spectrum at room temperature exhibits two doublets of doublets AB systems, which are assigned to two complexes: a minor complex (3a) at 75.4 ppm (J_Pt–P = 332 Hz, J_Pt–P = 2945 Hz) and a major complex (3b) at 71.7 ppm (J_Pt–P = 337 Hz, J_Pt–P = 2850 Hz). In the 13C{1H} NMR

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(9) Rate-determining associative substitution of alkenes in a cyclometalation process of a PCC-type phosphine ligand and a square-planar alkene complex (of Rh and Ir) was reported: Rybickinska, B.; Vignelok, A.; Ben-David, Y.; Milstein, *D. J. Am. Chem. Soc.* 1996, 118, 12406.

spectrum, the ipso carbon of 3a appears as a doublet of doublets at 147.21 ppm ($\delta_{C} = 6$ Hz) and the ipso carbon of 3b appears as doublet of doublets at 144.55 ppm ($\delta_{C} = 6$ Hz). The $^{19}$F NMR spectrum in THF exhibits two signals, a broad peak at $\delta = 169.15$ ppm, attributed to a coordinated $\text{BF}_4$ anion, and a sharp singlet at $\delta = 151.00$ ppm of free $\text{BF}_4^-$. The free $\text{BF}_4^-$ ion appears at $\delta = 151.00$ ppm in the $^{19}$F NMR spectrum in THF. $^{19}$F Coordinated $\text{BF}_4^-$ was reported to appear in the range $-162$ to $-164$ ppm. Reports of coordinated $\text{BF}_4^-$ include cationic rhodium complexes $6$ and a PCN-type cationic platinum complex. $^{16}$ On the basis of the reported data, we can conclude that this anion is coordinated to the metal center in 3a, while it is not coordinated in 3b.

The cationic carbonyl complex 4 was prepared directly from complex 2 by abstraction of the chloride ligand with silver tetrafluoroborate in THF at room temperature under carbon monoxide (Scheme 3). $^{12}$ This reaction goes to completion faster and in higher yield than chloride abstraction under an inert atmosphere followed by reaction with CO.

The $^{31}$P($^1$H) NMR spectrum of 4 shows a doublet of doublets AB system centered at 84.9 ppm ($\delta_{P-P} = 247$ Hz) with Pt satellites ($\delta_{P-P} = 2334$ Hz). In the $^{13}$C($^1$H) NMR spectrum the ipso carbon exhibits a virtual triplet at 183.87 ppm as a singlet and with Pt satellites ($\delta_{C-P} = 296$ Hz), and a peak of the carbonyl ligand appears at 2080 ppm. The IR spectrum, the carbonyl stretch was observed at 1992 cm$^{-1}$, indicating little back-bonding and a relatively low electron density at the metal center. The noncoordinated $\text{BF}_4^-$ anion appears in the $^{19}$F NMR spectrum as a singlet at $\delta = 150.87$ ppm. Complex 4 was crystallized by slow evaporation of a CH$_2$Cl$_2$/THF solution at room temperature, to give orange crystals. A single-crystal X-ray analysis of 4 exhibits a square-planar structure, with the carbonyl ligand being coordinated trans to the aromatic ring (Figure 3) and $\text{BF}_4^-$ located out of the coordination sphere. $^{13}$ Selected bond lengths and bond angles of complex 4 are given in Table 2. The C–O bond length in complex 4 (1.131(15) Å) is comparable to the bond length of a free CO molecule (1.128 Å). $^{14}$ In view of these data, the carbonyl ligand is expected to be electrophilic.

![Figure 3. View of a molecule of complex 4 at the 30% probability level. Hydrogen atoms and $\text{BF}_4^-$ anion are omitted for clarity.](image)

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<td>$\text{C}(1)-\text{O}(1)$</td>
<td>1.131(15)</td>
<td>$\text{O}(1)-\text{C}(1)-\text{Pt}(1)$</td>
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**Table 2. Selected Bond Lengths and Angles of Complex 4**

**Ligand Phosphine Part**

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<td>$\text{P}(3)-\text{C}(33)$</td>
<td>1.84(3)</td>
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**Nucleophilic Activation of Coordinated Carbon Monoxide.**

**Formation of a Carbomethoxy Complex by Methoxide Attack.** The chemistry of alkoxycarbonyl complexes of transition metals is of significant interest due to their recognition as intermediates in several important catalytic processes, such as carbonylation of alcohols, hydrocarboxylation of olefins to saturated and unsaturated esters, carbonylation of alkyl halides, and synthesis of alkyl carbones and oxalates from alcohols. $^{15}$ Alkoxycarbonyl complexes of platinum, their reactivity, and their role in carbonylation reactions are well known. $^{16}$

The facility of nucleophilic attack on coordinated CO ligands has been shown to be directly related to $\nu_{CO}$ of the CO ligand. $^{17}$ As expected from the relatively high frequency, $\nu_{CO}$ (2080 cm$^{-1}$), of 4, it reacts with NaOMe in THF at room temperature to form the carbomethoxy complex 5 (Scheme 4).

Complex 5 exhibits in the $^{31}$P($^1$H) NMR spectrum a doublet of doublets AB system centered at 70.8 ppm ($\delta_{P-P} = 338$ Hz).

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(13) There is a difference of 0.03 Å in the Pt–P bond lengths for the slightly distorted crystal structure of compound 4, due to $\text{BF}_4^-$ counteranion presence. A difference of only 0.01 Å was obtained for the Pt–P bond lengths in the highly ordered crystal structure of compound 2.


with Pt satellites ($J_{Pt-C} = 2856 \text{ Hz}$). In the $^{13}$C($^1$H) NMR spectrum the ipso carbon exhibits a virtual triplet at 168.79 ppm ($J_{Pt-C} = 754 \text{ Hz}$) with Pt satellites ($J_{Pt-C} = 70 \text{ Hz}$) and a peak of methoxide appears as a singlet at 16.66 ppm with Pt satellites ($J_{Pt-C} = 747 \text{ Hz}$). In the $^{13}$C($^1$H) NMR spectrum measured in THF was $1984, 1203$. (b) Barratt, D. S.; Cole-Hamilton, D. J. J. Chem. Soc 1984, 1203. (c) Smith, G.; Cole-Hamilton, D. J. J. Chem. Soc 1984, 1203.
complex 2 with carbon monoxide in the presence of silver tetrafluoroborate resulted in a stable cationic carbonyl complex 4, which underwent nucleophilic attack by a methoxide anion to form the carboxymethyl complex 5. The same methodology, nucleophilic addition to coordinated CO, has been employed with Na[Et₃BH], as a hydride transfer agent, to prepare the rare naphthyl-based PCP platinum complex 6.

**Experimental Section**

**General Procedures.** All experiments with metal complexes and phosphine ligands were carried out in oven-dried glassware under an atmosphere of purified nitrogen in a Vacuum Atmospheres glovebox equipped with an MO 40-2 inert gas purifier. All nondeuterated solvents were reagent grade or better. All solvents were degassed with argon and kept in the glovebox equipped with an MO 40-2 inert gas purifier. All solvents were degassed with argon and kept in the glovebox. Deuterated solvents were used as received. All nondeuterated solvents were degassed with argon and kept in the glovebox. To a THF solution of (COD)Pt(Cl)CH₃ (COD = 1,5-cyclooctadiene; 21.0 mg, 0.059 mmol) was added 26.0 mg (0.059 mmol) of ligand 1 (0.059 mmol) was added 26.0 mg (0.059 mmol) of ligand 1 (0.059 mmol) was added 26.0 mg (0.059 mmol) of ligand 1 (0.059 mmol) was added 26.0 mg (0.059 mmol) of ligand 1 (0.059 mmol) was added 26.0 mg (0.059 mmol) of ligand 1 (0.059 mmol) was added 26.0 mg (0.059 mmol) of ligand 1 (0.059 mmol) was added 26.0 mg (0.059 mmol) of ligand 1 (0.059 mmol) was added 26.0 mg (0.059 mmol) of ligand 1. The mixture was stirred at room temperature for 1 h, resulting in a yellow solid solution. The 31P[H] NMR spectrum revealed formation of 2. The solvent was evaporated, and the complex was washed with pentane and dissolved in benzene. Benzene evaporation yielded 33.9 mg (0.050 mmol, 35% clean product yield) of the yellow complex 3 was obtained.

**Preparation of the Naphthyl-Based PCP Ligand C₅₀H₅₅(CH₃PBU₂).** The PCP naphthyl-based ligand 1 was synthesized by treatment of 1,3-dichloromethyl)naphthalene with di-tert-butylphosphine in the presence of triethylamine.

**Reaction of the PCP Ligand (1) with (COD)PtCl(Me).** Formation of [(C₅₀H₅₅(CH₃PBU₂)]PtCl(1)] (2). To a THF solution (5 mL) of (COD)PtClCH₃ (COD = 1,5-cyclooctadiene; 21.0 mg, 0.059 mmol) was added 26.0 mg (0.059 mmol) of ligand 1 in THF (5 mL). The mixture was stirred at room temperature for 1 h, resulting in a yellow solid solution. The 31P[H] NMR spectrum revealed formation of 2. The solvent was evaporated, and the complex was washed with pentane and dissolved in benzene. Benzene evaporation yielded 33.9 mg (0.050 mmol, 35% clean substance yield 85%) of 2. X-ray quality, colorless crystals were obtained from pentane washes at room temperature.

**Analysis.** The NMR spectra were recorded at 250 MHz (1H), 101 MHz (31P), and 235 MHz (19F) using a Bruker DXP 250 spectrometer, at 400 MHz (1H), 100 MHz (13C), 162 MHz (31P), and 376 MHz (19F) using a Bruker AMX 400 NMR spectrometer, and at 500 MHz (1H), 126 MHz (13C), and 202 MHz (31P) using a Bruker DXP 500 spectrometer. All spectra, except 13C spectra of 2 were recorded at 200 MHz using a Bruker DPX 250 spectrometer. All spectra, except 13C spectra of 2 were recorded at 23°C. All NMR chemical shifts were referenced to an external 85% solution of phosphoric acid in D₂O. 19F NMR chemical shifts were referenced to C₆F₆ (7.16 ppm), and referenced to toluene-d₈ (7.16 ppm). Abbreviations used in the description of NMR data are as follows: a, broad; s, singlet; d, doublet; t, triplet; m, multiplet; v, virtual. IR spectra were measured with a Nicolet-460 FT-IR spectrometer. Elemental analyses were performed by the Unit of Chemical Research Support, Weizmann Institute of Science.

**Formation of [[C₅₀H₅₅(CH₃PBU₂)]PtBF₄] (3).** To a THF solution (25 mL) of (PCP)PtCl₂ (79.0 mg, 0.117mmol) was added 24.0 mg (0.123 mmol) of AgBF₄, resulting in immediate formation of white precipitate of AgCl. The reaction mixture was stirred vigorously at room temperature for 2 days in a vial protected from light, resulting in a suspension of precipitated AgCl. The 31P[H] NMR spectrum at room temperature revealed formation of two new complexes, 3a (minor) and 3b (major), in a 1:2 ratio, respectively. The suspension was filtered through a frit covered with Celite, and the solvent was evaporated. The resulting yellow solid was washed with pentane and dissolved in benzene. After evaporation of benzene, 30 mg (0.041 mmol, 35% clean product yield) of the yellow complex 3 was obtained.

**Synthesis of [[C₅₀H₅₅(CH₃PBU₂)]Pt(CO)(BF₄)] (4).** To a 8 mL screw-cap vial, containing 29 mg (0.148 mmol) of AgBF₄, protected from light, was added a THF solution (5 mL) of (PCP)PtCl₂ (100 mg, 0.148 mmol). The reaction mixture was sealed with a Teflon septum cap, and 10 mL (0.400 mmol) of CO gas was injected. The reaction mixture was shaken vigorously at room temperature for 12 h using a mechanical shaker. The 31P[H] NMR spectrum revealed the formation of (PCP)Pt(CO)(BF₄) complex 4. The brown reaction mixture suspension was filtered through a
frit covered with Celite, and the solvent was evaporated. The resulting solid was washed with pentane and dissolved in toluene. Complex 4 was precipitated from a toluene (2 mL)/pentane (10 mL) two-phase mixture at −30 °C. After removing the liquid and drying the precipitate, 43.2 mg (0.057 mmol, 39% pure product yield) of 4 was obtained as a brown solid. Small needle-like crystals of 4 were obtained from a saturated THF solution at room temperature. X-ray quality orange crystals of 4 were obtained by recrystallization of these crystals from a CH₂Cl₂/THF solution at room temperature.

31P{1H} NMR (CDCl₃): 84.9 (dd (AB), J₀₋₁ = 247 Hz, J₁₋₂ = 2334 Hz). 1H NMR (CDCl₃): 7.98 (d, J₈₋₇ = 8 Hz, 1H, Ar), 7.85 (s, 1H, Ar), 7.67 (d, J₇₋₆ = 8 Hz, 1H, Ar), 7.43 (t, J₆₋₅ = 7 Hz, 1H, Ar), 7.39 (t, J₅₋₄ = 7.5 Hz, 1H, Ar), 4.11 (bd, J₄₋₃ = 7.9 Hz, 2H, Ar-CH₂-P), 3.89 (bd, J₃₋₂ = 7.6 Hz, 2H, Ar-CH₂-P), 1.35 (m, 36H, P-t-Bu). 13C{1H} NMR (CDCl₃): 183.87 (s, J₁₋₀ = 980 Hz, Pt-CO), 160.51 (vt, J₀₋₉ = 7 Hz, J₉₋₈ = 260 Hz, ipso), 150.50 (t, J₈₋₇ = 7 Hz, Ar), 148.82 (t, J₇₋₆ = 7 Hz, Ar), 134.32 (s, Ar), 128.21 (s, Ar), 127.60 (s, Ar), 127.18 (s, Ar), 123(s, Ar), 37.17 (s, P-C(CH₃)₃), 34.87 (d, J₀₋₉ = 30 Hz, Ar-CH₂-P), 33.24 (d, J₀₋₉ = 32 Hz, Ar-CH₂-P), 29.18 (m, P-C(CH₃)₃). (Assignment of 13C{1H} NMR signals was confirmed by 13C{31P} DEPT.) IR (film): 1580 cm⁻¹. 

The cooled mixture was stirred at room temperature for 10 min, resulting in a red solution. The hydride (38.5 mg, 0.960 mmol). The colloidal mixture was stirred overnight at room temperature, resulting in a red solution. The hydride (38.5 mg, 0.960 mmol). The colloidal mixture was stirred at room temperature for 7 days, resulting in a red solution. The hydride (38.5 mg, 0.960 mmol). The colloidal mixture was stirred at room temperature for 7 days, resulting in a red solution. The hydride (38.5 mg, 0.960 mmol). The colloidal mixture was stirred at room temperature for 7 days, resulting in a red solution. The hydride (38.5 mg, 0.960 mmol). The colloidal mixture was stirred at room temperature for 7 days, resulting in a red solution. The hydride (38.5 mg, 0.960 mmol). The colloidal mixture was stirred at room temperature for 7 days, resulting in a red solution. The hydride (38.5 mg, 0.960 mmol). The colloidal mixture was stirred at room temperature for 7 days, resulting in a red solution.