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# Reactivity of platinum and palladium α-ketoacyl complexes toward olefins and acetylenes

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## Abstract

The α-ketoacyl complexes *trans*-$M(PPh_3)_2(Cl)(COCO_2R)$ (M = Pd, R = $(CH_2)_nCHCH_2$, $n = 2, 3, 4$, R = $(CH_2)_2CCMe$; M = Pt, R = $(CH_2)_nCHCH_2$, $n = 2$, 3, 4, R = $(CH_2)_2CCMe$) were prepared by treating $M(PPh_3)_4$ with ROCOCOCl. Attempts to insert the pendant olefin or acetylene into the α-ketoacyl-to-metal bond through heating failed, since CO deinsertion to form corresponding acyl complexes was predominant. Reaction of [*trans*-$Pt(PPh_3)_2(NCMe)(COCOR)]BF_4$ (R = Me, Ph) with norbornylene resulted in the deinsertion of CO from the α-ketoacyl ligand and the formation of [*cis*-$Pt(PPh_3)_2(C_7H_{10}COR)]BF_4$, an insertion of norbornylene into the corresponding acyl ligand.

## Introduction

Insertion of carbon–carbon multiple bonds into transition metal-to-acyl bonds is a well established and well studied reaction, particularly for platinum and palladium acyls [1,2,5]. In the course of our work on the chemistry of platinum and palladium α-ketoacyl complexes [3] we sought to observe an analogous insertion into an α-ketoacyl-to-metal bond. To this end we prepared a series of compounds containing alkyloxalyl ligands bearing pendant unsaturated groups. These compounds are closely related both to complexes reported by Angelici [4] (e.g., *trans*-$MCl(COCO_2Me)(PPh_3)_2$, M = Pd, Pt) and others reported by Norton [2] (e.g., *trans*-$PdCl(CO_2(CH_2)_2CCMe)(PPh_3)_2$ and $PdCl(CO_2(CH_2)_2CHCH_2)(PPh_3)_2$). We also examined normal α-ketoacyl complexes for reactivity toward intermolecular olefin insertion. We failed however, to observe any reactions of this type.

## Results and discussion

Alkyloxalyl chlorides were prepared through reaction 1. **1a–1d** are colorless liquids of low volatility that are stable indefinitely at room temperature when

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protected from moisture.

$$\mathrm{ROH + ClCOCOCl \xrightarrow[Et_2O]{} ROCOCOCl + HCl} \qquad (1)$$

($\mathrm{R = CH_2CH(CH_2)}_n$, $n = 2$, **1a**, $n = 3$, **1b**, $n = 4$, **1c**; $\mathrm{R = MeCC(CH_2)_2}$, **1d**)

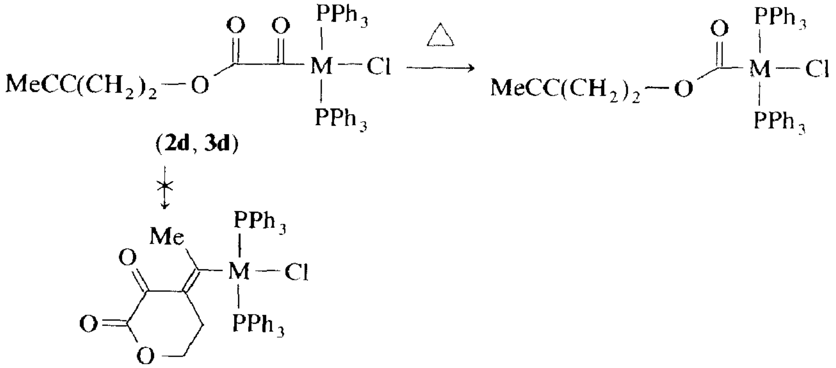
These compounds were allowed to react with $\mathrm{M(PPh_3)_4}$ in benzene solution to give bis(triphenylphosphine)chloro(alkyloxalyl) complexes of platinum and palladium (eq. 2). All products are bright yellow solids, soluble in aromatic hydrocarbons and chlorinated solvents but insoluble in ethers and alkanes. The platinum compounds are indefinitely stable in air both as solids and in solution while the palladium compounds turned dark in the solid phase over a period of months. The palladium compounds are stable in benzene solution although they decompose in chlorinated solvents to produce $\mathrm{PdCl_2(PPh_3)_2}$. A dependence of stability related to hydrocarbon chain length in $\mathrm{CDCl_3}$ solutions was observed by $^{31}$P NMR. **2a** decomposed over a period of hours, while **2c** showed only slight decomposition over several days. Compound **3a** was found to contain 0.25 molecules of $\mathrm{CH_2Cl_2}$ of crystallization when prepared with this solvent as indicated by $^1$H NMR spectra and elemental analysis.

$$\mathrm{M(PPh_3)_4 + ROCOCOCl \xrightarrow[benzene]{}} \textit{trans}\text{-}\mathrm{M(PPh_3)_2(Cl)(COCO_2R)} \qquad (2)$$

($\mathrm{M = Pd}$, $\mathrm{R = (CH_2)_nCHCH_2}$, $n = 2$, **2a**, $n = 3$, **2b**, $n = 4$, **2c**; $\mathrm{R = (CH_2)_2CCMe}$, **2d**; $\mathrm{M = Pt}$, $\mathrm{R = (CH_2)_nCHCH_2}$, $n = 2$, **3a**; $n = 3$, **3b**, $n = 4$, **3c**; $\mathrm{R = (CH_2)_2CCMe}$, **3d**)

**2a**–**2d** and **3a**–**3d** show singlets in their $^{31}$P NMR spectra (ca. 18.20 ppm) consistent with square planar structures whose triphenylphosphine ligands are mutually *trans*. In the cases of **3a**–**3d** these singlets are accompanied by $^{195}$Pt satellites. In the series **3a**–**3c** an increase in $^{195}\mathrm{Pt}$–$^{31}\mathrm{P}$ coupling constant (3214.1 to 3241.3 Hz) with hydrocarbon chain length was observed. Two $\nu$(CO) absorption bands were seen in the IR spectra of these compounds. Small frequency increases with increasing chain length exist in the absorptions for the carbonyl $\alpha$ to the metal in both the series **2a**–**2c** (1642 to 1656 $\mathrm{cm^{-1}}$) and **3a**–**3c** (1665 to 1677 $\mathrm{cm^{-1}}$). These trends in spectroscopic data and those of stability are perhaps related to the ease with which the pendant unsaturated group may interact with the metal. This explanation requires that the interaction becomes more difficult with increasing chain length.

When any of the complexes **2** and **3** were refluxed in toluene for several hours CO deinsertion occurred which gave alkyl chloroformate complexes (e.g., eq. 3).



**(2d, 3d)**

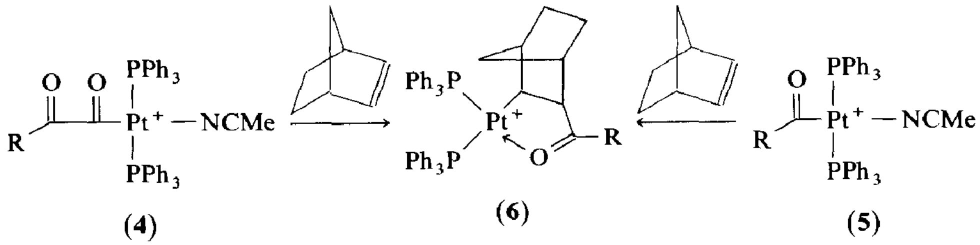
(3)

This reaction was monitored by $^{31}P$ NMR spectroscopy. No products arising from an insertion into the α-ketoacyl-to-metal bond occurred. Thus in this case CO deinsertion from an α-ketoacyl-to-metal bond is much more favorable than olefin or acetylene insertion into an α-ketoacyl-to-metal bond.

The deinsertion products formed from the palladium complexes, **2a** and **2d**, were previously synthesized by Norton through oxidative addition reactions of alkyl chloroformates. Norton reported that when these acyl compounds were refluxed in xylene, olefin and acetylene acyl-to-palladium insertion products were observed.

Abstraction of the Cl ligand by treatment with $AgBF_4$ in $CDCl_3$ gave a large number of unidentifiable products. Upwards of thirty metal species were observed by $^{31}P$ NMR in $CDCl_3$ solutions of **2a–2d** and **3a–3d** treated with an equimolar amount of $AgBF_4$.

Syntheses of the methyl (**4a**) and phenyl (**4b**) α-ketoacyl solvent-coordinated platinum cations formed by treatment of the corresponding α-ketoacyl chloro complex with $AgBF_4$ in acetonitrile have been reported in a previous publication [3]. When norbornylene was added to solutions of **4a** or **4b** a reaction occurs which was complete in nine days at room temperature (eq. 4). The $^{31}P$ NMR spectra of the products of this reaction show two doublets with their $^{195}Pt$ satellites. These absorptions are identical to those of the products formed by the reaction of the acyl acetonitrile cations (**5a–5b**) with norbornylene. They were identified thus as the metallaoxocyclopentanes **6a–6b**. This geometry is consistent with the $^{31}P$ NMR absorptions indicating two inequivalent phosphorus atoms. Abnormally low C–O stretching frequencies were observed in their IR spectra (e.g., 1592 $cm^{-1}$ for **6a** and 1540 $cm^{-1}$ for **6b**). These absorptions indicate a lowered bond order that supports carbonyl oxygen to metal coordination. The large $^{195}Pt-^{31}P$ coupling constants of the more upfield phosphorus absorptions (e.g., 5029.6 Hz for **6a** and 4949.7 Hz for **6b**) identify them as belonging to the phosphorus *trans* to the oxygen and the smaller $^{195}Pt-^{31}P$ coupling constants of the more downfield absorptions (e.g., 1634.7 Hz for **6a** and 1610.8 Hz for **6b**) as belonging to the phosphorus *trans* to the carbon coordinated to the metal. This is due to the higher electron density available to the platinum-to-phosphorus bond *trans* to the weaker platinum-to-oxygen bond relative to the lesser electron density available to the bond *trans* to the coordinated carbon. **6a–6b** are platinum analogues of palladium compounds characterized by spectroscopic methods as well as single crystal X-ray crystallography in other publications from our laboratory [5].



(4)

(R = Me, **a**; Ph, **b**)

It is apparent that CO is lost sometime during the interaction of the olefin with the α-ketoacyl complex involved in this insertion reaction since the α-ketoacyl cations are thermally stable in solution. This CO loss is different in nature from that of **2a**–**2c** and **3a**–**3c** whose CO deinsertion occurs as a distinct reaction from that of their olefin insertion reaction.

All of the reactions herein described strive to facilitate the π-coordination of a multiple bond to a 4-coordinate, square planar metal bearing an α-ketoacyl ligand in order to permit insertion into the α-ketoacyl-to-metal bond. π-coordination of the unsaturated molecule is fostered by 1) removal of a ligand from the metal to create a more reactive 3-coordinate, 14 electron species, 2) replacement of a ligand with a more labile solvent molecule and 3) furnishing the metal complex with a ligand possessing an unsaturated group capable of coordination to the metal intramolecularly, a kinetically favored process. The ultimate result, however, was acyl migration, which has been established as an irreversible reaction [3,6]. For this reason all attempts to observe the sought after insertion reaction failed. In most experiments the multiple bond proceeded to insert into the acyl ligand formed since alkyl migration for an acyl ligand is a comparatively less facile process.

## Experimental

### *General procedure*

$Pt(PPh_3)_4$ [7], $Pd(PPh_3)_4$ [7], $Pt(PPh_3)_2(Cl)(COCOMe)$ [3] and $Pt(PPh_3)_2(Cl)(COCOPh)$ [3] were prepared by literature methods. Benzene and diethyl ether were dried by reflux over sodium benzophenone ketyl and degassed. Acetonitrile was predried over 4Å molecular sieves followed by reflux over and fractional distillation from calcium hydride. Chloroform-*d* was dried by reflux over $P_2O_5$ and degassed. Oxalyl chloride and unsaturated alcohols were obtained from Aldrich and degassed prior to use. All manipulations of acid chlorides, zerovalent metal compounds and cationic acetonitrile complexes were carried out under anaerobic conditions using Schlenk techniques or in a Vacuum Atmospheres glove box equipped with a dri-train.

### *Analytical instrumentation*

IR spectra were recorded on a Perkin–Elmer model 281B spectrometer. $^1H$ NMR spectra were recorded on a Bruker WP200 FT-NMR spectrometer, while $^{31}P$ NMR spectra were recorded on a Varian CFT-20 spectrometer. Elemental analyses were performed by Schwartzkopf Analyses, Inc.

### *3-Butenyloxalyl chloride (1a)*

3-Butene-1-ol (24.2 ml, 0.281 mol) was added dropwise over 10 min to a stirred solution of oxalyl chloride (24.5 ml, 0.281 mol) in 300 ml diethyl ether chilled to 0 °C with an ice bath. The reaction mixture was allowed to stir for 15 min at 0 °C, then overnight at room temperature. The solvent was distilled away at ambient pressure while protected from atmospheric moisture by a Drierite-filled drying tube to yield a yellow-brown syrupy liquid. This was fractionally distilled with an 18 cm Vigreux column under reduced pressure to separate 32 ml of colorless **1a** (0.228 mol, 81% yield) (b.p. 67 °C/18 torr) from the dialkyl oxalate (b.p. 114 °C/18 torr). $^1H$ NMR ($CDCl_3$): $\delta$ 2.48 (q, 2H, $J$(H–H) 6.7, 1.3 Hz), 4.37 (t, 2H, $J$(H–H) 6.7 Hz), 5.12 (m, 2H), 5.74 (m, 1H); IR (neat): 1759, 1790 $cm^{-1}$; d 1.16 g $ml^{-1}$.

### *4-Pentenyloxalyl chloride (**1b**)*

4-Penten-1-ol (11.4 ml, 0.111 mol) gave as above **1b** (16 ml, 0.101 mol, 91% yield) (b.p. 95–96 °C/30 torr). $^1$H NMR ($CDCl_3$): $\delta$ 1.88 (quintet, 2H, $J$(H–H) 7.0 Hz), 2.20 (q, 2H, $J$(H–H) 5.3 Hz), 4.38 (t, 2H, $J$(H–H) 6.6 Hz), 5.02 (m, 1H), 5.11 (m, 1H), 5.80 (m, 1H); IR (neat) 1758, 1786 cm$^{-1}$; d 1.12 g ml$^{-1}$.

### *5-Hexenyloxalyl chloride (**1c**)*

5-Hexen-1-ol (29.5 ml, 0.246 mol) gave **1c** (40 ml, 0.231 mol, 94% yield) (b.p. 94 °C/10 torr). $^1$H NMR ($CDCl_3$): $\delta$ 1.47 (quintet, 2H, $J$(H–H) 7.5 Hz), 1.75 (quintet, 2H, $J$(H–H) 7.1 Hz), 2.07 (q, 2H, $J$(H–H) 7.2 Hz), 4.33 (t, 2H, $J$(H–H) 6.6 Hz), 4.95 (m, 1H), 4.97 (m, 1H), 5.74 (m, 1H); IR (neat): 1758, 1789 cm$^{-1}$; d 1.10 g ml$^{-1}$.

### *3-Pentynyloxalyl chloride (**1d**)*

3-Pentyn-1-ol (7.8 ml, 0.085 mol) gave **1d** (10 ml, 0.078 mol, 94% yield) (b.p. 91–92°/12 torr). $^1$H NMR ($CDCl_3$): $\delta$ 1.71 (t, 3H, $J$(H–H) 2.5 Hz), 2.58 (m, 2H), 4.35 (t, 2H, $J$(H–H) 6.9 Hz); IR (neat): 1758, 1790 cm$^{-1}$; d 1.18 g ml$^{-1}$.

### *trans-$Pd(PPh_3)_2(Cl)(COCO_2(CH_2)_2CHCH_2)$ (**2a**)*

**1a** (0.5 ml) was added to a solution of 1.04 g (0.89 mmol) $Pd(PPh_3)_4$ in 20 ml benzene. The reaction mixture was stirred for 15 min at 25 °C followed by solvent removal through rotary evaporation. Diethyl ether (100 ml) was added to the residual oil. The precipitate formed was collected on a fritted glass Büchner funnel and then dissolved in 12 ml benzene and filtered. Benzene was removed from the filtrate by rotary evaporation to give 0.45 g **2a** (0.57 mmol, 64% yield). $^1$H NMR ($C_6D_6$); $\delta$ 1.64 (q, 2H, $J$(H–H) 7.0 Hz), 3.02 (t, 2H, $J$(H–H) 7.1 Hz), 4.84 (m, 1H), 4.89 (m, 1H), 5.42 (m, 1H), 6.94–7.09, 7.96, 8.03 (br, 30H); $^{31}$P NMR ($CDCl_3$): $\delta$ 18.49; IR (KBr pellet) 1665, 1718 cm$^{-1}$; Anal. Found: C, 63.56; H, 4.93. $C_{42}H_{37}ClO_3P_2Pd$ calcd.: C, 63.56; H, 4.70%.

### *trans-$Pd(PPh_3)_2(Cl)(COCO_2(CH_2)_3CHCH_2)$ (**2b**)*

**1b** (0.5 ml) was allowed to react as above with 1.01 g $Pd(PPh_3)_4$ (0.88 mmol) to yield 0.59 g **2b** (0.71 mmol, 81% yield). $^1$H NMR ($C_6D_6$): $\delta$ 1.01 (quintet, 2H, $J$(H–H) 7.2Hz), 1.68 (q, 2H, $J$(H–H) 7.3 Hz), 3.00 (t, 2H, $J$(H–H) 6.9 Hz), 4.87 (m, 1H), 4.92 (m, 1H), 5.56 (m, 1H), 6.98–7.06, 7.96–8.03 (br, 30H); $^{31}$P NMR ($CDCl_3$): $\delta$ 18.49; IR (KBr pellet): 1670, 1718 cm$^{-1}$; Anal. Found: C, 65.21; H, 4.98. $C_{43}H_{39}ClO_3P_2Pd$ calcd.: C, 63.95; H, 4.87%.

### *trans-$Pd(PPh_3)_2(Cl)(COCO_2(CH_2)_4CHCH_2)$ (**2c**)*

**1c** (0.5 ml) was allowed to react with 1.01 g $Pd(PPh_3)_4$ (0.88 mmol) to yield 0.59 g **2c** (0.71 mmol, 81% yield). $^1$H NMR ($C_6D_6$) $\delta$ 1.02 (m, 2H), 1.77 (q, 2H, $J$(H–H) 6.8 Hz), 2.99 (t, 2H, $J$(H–H) 6.4 Hz), 3.38 (t, 2H, $J$(H–H) 6.2 Hz), 4.93 (m, 1H), 4.97 (m, 1H), 5.65 (m, 1H), 7.00–7.08, 7.97–8.06 (br, 30H); $^{31}$P NMR: $\delta$ 18.51; IR (KBr pellet) 1677, 1713 cm$^{-1}$; Anal. Found: C, 65.20; H, 5.13. $C_{44}H_{41}ClO_3P_2Pd$ calcd.: C, 64.23; H, 5.03%.

### *trans-$Pd(PPh_3)_2(Cl)(COCO_2(CH_2)_2CCMe)$ (**2d**)*

**1d** (0.5 ml) was allowed to react with 1.06 g $Pd(PPh_3)_4$ (0.91 mmol) to yield 0.73 g **2d** (0.89 mmol, 98% yield). $^1$H NMR ($C_6D_6$): $\delta$ 1.43 (t, 2H, $J$(H–H) 2.5Hz), 2.03

(m, 2H), 3.56 (t, 3H, $J$(H–H) 7.6 Hz), 6.96–7.03, 7.95–8.02 (br, 30H); $^{31}$P NMR: $\delta$ 18.39; IR (KBr pellet): 1668, 1722 cm$^{-1}$; Anal. Found: C, 64.23; H, 4.69. $C_{43}H_{37}ClO_3P_2Pd$ calcd.: C, 64.03; H, 4.62%.

### *trans-Pt(PPh$_3$)$_2$(Cl)(COCO$_2$(CH$_2$)$_2$CHCH$_2$) · 0.25CH$_2$Cl$_2$ (3a)*

**1a** (1.0 ml) was added to a solution of 3.00 g Pt(PPh$_3$)$_4$ (2.41 mmol) in 20 ml benzene. The reaction mixture was stirred for 1 h at 25 °C after which the solvent was removed by rotary evaporation. Diethyl ether (100 ml) was added to the residual oil. The precipitate formed was collected on a fritted glass Büchner funnel and then dissolved in 10 ml of methylene chloride and filtered. Solvent was removed from the filtrate by rotary evaporation to give 2.18 g of a yellow solid, **3a** (2.34 mmol, 97% yield). $^{1}$H NMR (CDCl$_3$): $\delta$ 1.98 (q, 2H, $J$(H–H) 7.2 Hz), 3.43 (t, 2H, $J$(H–H) 7.3 Hz), 4.96 (m, 1H), 5.01 (m, 1H), 5.29 (s, 0.5H), 5.57 (m, 1H), 7.35–7.80 (br, 30H); $^{31}$P NMR (CDCl$_3$): $\delta$ 18.16 ($J$(Pt–P) 3214.1 Hz); IR (KBr pellet): 1642, 1710 cm$^{-1}$; Anal. Found: C, 55.77; H, 4.42. $C_{42}H_{37}ClO_3P_2Pt \cdot 0.25CH_2Cl_2$ calcd.: C, 55.85; H, 4.16%.

### *trans-Pt(PPh$_3$)$_2$(Cl)(COCO$_2$(CH$_2$)$_3$CHCH$_2$) (3b)*

**1b** (1.0 ml) was allowed to react as above with 3.24 g Pt(PPh$_3$)$_4$ (2.60 mmol) to yield 2.10 g **3b** (2.35 mmol, 90% yield). $^{1}$H NMR (CDCl$_3$): $\delta$ 1.36 (quintet, 2H, $J$(H–H) 7.2 Hz), 1.86 (q, 2H, $J$(H–H) 7.2 Hz), 3.41 (t, 2H, $J$(H–H) 6.9 Hz), 4.92 (m, 1H), 4.99 (m, 1H), 5.69 (m, 1H), 7.34–7.80 (br, 30H); $^{31}$P NMR (CDCl$_3$): $\delta$ 18.72 ($J$(Pt–P) 3222.0 Hz); IR (KBr pellet): 1650, 1712 cm$^{-1}$; Anal. Found: C, 57.43; H, 4.35. $C_{43}H_{39}ClO_3P_2Pt$ calcd.: C, 56.55; H, 4.30%.

### *trans-Pt(PPh$_3$)$_2$(Cl)(COCO$_2$(CH$_2$)$_4$CHCH$_2$) (3c)*

**1c** (0.5 ml) was allowed to react as above with 0.51 g Pt(PPh$_3$)$_4$ (0.41 mmol) to yield 0.35 g **3c** (0.38 mmol, 94% yield). $^{1}$H NMR (CDCl$_3$): $\delta$ 1.17–1.32 (m, 4H), 1.98 (q, 2H, $J$(H–H) 6.4 Hz), 3.40 (t, 2H, $J$(H–H) 6.6 Hz), 4.96 (t, 1H, $J$(H–H) 1.3 Hz), 5.02 (m, 1H), 5.75 (m, 1H), 7.33–7.80 (br, 30H); $^{31}$P NMR: $\delta$ 18.29 ($J$(Pt–P) 3241.3 Hz); IR (KBr pellet): 1756, 1707 cm$^{-1}$; Anal. Found: C, 57.70; H, 4.58. $C_{44}H_{41}ClO_3P_2Pt$ calcd.: C, 58.05; H, 4.54%.

### *trans-Pt(PPh$_3$)$_2$(Cl)(COCO$_2$(CH$_2$)$_2$CCMe) (3d)*

**1d** (0.5 ml) was allowed to react as above with 0.50 g Pt(PPh$_3$)$_4$ (0.40 mmol) to yield 0.24 g **3d** (0.27 mmol, 67% yield). $^{1}$H NMR (CDCl$_3$): $\delta$ 1.68 (t, 3H, $J$(H–H) 7.7 Hz), 2.00 (m, 2H), 3.47 (t, 2H, $J$(H–H) 2.5 Hz), 7.36–7.79 (b, 30H); $^{31}$P NMR: $\delta$ 18.02 ($J$(Pt–P) 3203.8 Hz); IR (KBr pellet): 1647, 1710 cm$^{-1}$; Anal. Found: C, 57.24; H, 4.01. $C_{43}H_{37}ClO_3P_2Pt$ calcd.: C, 57.75; H, 4.17%.

### *[cis-Pt(PPh$_3$)$_2$(C$_7$H$_{10}$COMe)]BF$_4$ (6a)*

Pt(PPh$_3$)$_2$(Cl)(COCOMe) (32 mg, 39 $\mu$mol) was allowed to react with AgBF$_4$ (8 mg, 39 $\mu$mol) in 3 ml acetonitrile. AgCl was removed from the reaction mixture by centrifugation and the solvent removed from the supernant. The residue was dissolved in 3 ml CDCl$_3$ and placed in a 10 mm NMR tube with 0.5 g norbornylene. Reaction was found to be complete in 9 days by $^{31}$P NMR. Solvent was removed to isolate a white solid. $^{1}$H NMR (CDCl$_3$): $\delta$ 1.10 (m, 2H), 1.26 (m, 2H), 1.42 (m, 2H), 1.85 (m, 2H), 2.25 (m, 3H), 2.51 (m, 1H), 3.14 (m, 1H), 7.12–7.62 (br, 30H); $^{31}$P NMR ($CDCl_3$): $\delta$ 10.98 ($J$(P–P) 14.1 Hz, $J$(Pt–P) 5029.6 Hz), 26.06 ($J$(P–P) 14.0 Hz, $J$(Pt–P) 1634.7 Hz); IR (KBr pellet): 1592, 1025, 1035 cm$^{-1}$.

### $[cis\text{-}Pt(PPh_3)_2(C_7H_{10}COPh)]BF_4$ *(6b)*

A solution of **4b** was prepared by treating 65 mg *trans*-$Pt(PPh_3)_2(Cl)(COCOPh)$ (79 $\mu$mol) with 15 mg $AgBF_4$ (79 $\mu$mol). Norbornylene (0.5 g) was added as above. Reaction was completed in 9 days. $^1$H NMR ($CDCl_3$): $\delta$ 1.14 (m, 2H), 1.45, (m, 2H) 1.64 (m, 2H), 1.98 (m, 2H), 2.55 (m, 1H), 3.58 (m,1H), 7.12–7.63 (br, 35H); $^{31}$P NMR ($CDCl_3$): $\delta$ 10.89 ($J$(P–P) 14.8 Hz, $J$(Pt–P) 4949.7 Hz), 26.04 ($J$(P–P) 15.4 Hz, $J$(Pt–P) 1610.8 Hz); IR (KBr pellet): 1540, 1010, 1060 cm$^{-1}$.

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