

trum of **1** recorded at several temperatures is shown in Figure 3. At 25 °C the Au-P groups are equivalent (broad single resonance δ 49.7) due to a rapid skeletal rearrangement, but at -100 °C the Au-P resonance has split into three peaks in a 1:2:2 ratio. This pattern is consistent with the edge-shared bi-tetrahedral structure. The dynamic rearrangement is not due to rapid phosphine or AuPPh₃ dissociation because P-P and P-H coupling is observed in the 25 °C spectrum. The ¹H NMR spectrum (CD₂Cl₂, 25 °C, 300 MHz) of **1** shows an eight-line multiplet in the hydride region at δ -2.07. Selective phosphorus decoupling gave a sextet [$J(\text{H}-\text{P}_{\text{Au}}) = 9.1$ Hz] with Re-P decoupled and a triplet [$J(\text{H}-\text{P}_{\text{Re}}) = 18.6$ Hz] with Au-P decoupled. Careful integration of this hydride resonance vs. the aromatic region and vs. the para-CH₃ peak in the *p*-tolylphosphine PAR₃ derivative [ReH₄(PAR₃)₂(AuPPh₃)₂]²⁺ consistently gave 4.0 ± 0.1 hydrides.²⁰ The positions of the hydride ligands could not be deduced from the available data. It is likely that hydride bridging between Re and Au occurs due to the crowded nature of the Re core; however, the nonrigid nature of the complex in solution makes this difficult to prove.

The ³¹P{¹H} NMR spectrum (reference trimethyl phosphate, CH₂Cl₂, 25 °C, 121.5 MHz) of **2** showed a doublet of quartets resonance at δ 58.6 [$J(\text{P}-\text{Rh}) = 125$ Hz, $J(\text{P}-\text{P}) = 41$ Hz, integration = 2] assigned to RhP and a triplet of doublets resonance at δ 41.9 [$J(\text{P}-\text{P}) = 41$ Hz, $J(\text{P}-\text{Rh}) = 14$ Hz, integration 3] assigned to AuP. The compound is nonrigid since in the solid state P3 is nonequivalent to P1 and P2. The ¹H NMR spectrum (CD₂Cl₂, 25 °C, 300 MHz) shows a multiplet at δ -4.91 with integration of 1.0 ± 0.1 H (integration vs. phenyl hydrogens). The multiplet spectrum was successfully computer-simulated with use of the above coupling constants and $J(\text{Rh}-\text{H}) = 8.4$, $J(\text{H}-\text{P}_{\text{Rh}}) = 6.5$, and $J(\text{H}-\text{P}_{\text{Au}}) = 13.0$ Hz.²¹ The placement of the hydride trans to the CO group is likely on the basis of the solid-state structure. This position is exactly correct for bridging to Au3. The hydride could of course be bent away from Au3, but the observation that the Rh-Au3 distance is significantly longer than the other Rh-Au bonds supports a bridging formulation. The infrared spectra (solution and KBr disk) of **1** and **2** show no evidence for terminal metal-hydride stretches in the 1800-2200-cm⁻¹ region. The bridging region is complex due to other absorptions, and thus for M(μ -H)Au bands have not been identified.

The formation of **1** and **2** in high yield supports the synthetic approach discussed above for mixed-metal gold clusters. Both of the starting transition-metal hydride compounds are known to easily become coordinatively unsaturated. RhH(CO)(PPh₃)₃ shows extensive phosphine dissociation at room temperature in benzene solution,¹⁵ and ReH₇(PPh₃)₂ undergoes thermal as well as photochemical elimination of H₂ to produce the 16-electron species ReH₅(PPh₃)₂.²² These complexes should also be capable of reducing gold(I). Gold is reduced in the formation of **1** and **2** from AuNO₃PPh₃. This general synthetic approach is currently being successfully used in the synthesis of other mixed-metal gold clusters.

Acknowledgment. This work has been supported by the National Science Foundation (NSF Grant CHE-81-08490) and by the University of Minnesota Graduate School.

Supplementary Material Available: Experimental details of the X-ray structure determination, tables of crystal structure data collection and refinement parameters (Table 1), final atom positional and thermal parameters (Tables 2 and 3), and weighted least-squares planes (Tables 4 and 5), figures of ORTEP drawings of the molecular structures (Figures

- (20) Integrations were done on several different samples and with use of a variety of instrument conditions. The results always gave 4 hydride ligands. Our experience with many different polyhydrido clusters gives us confidence in this NMR result. Conductivity measurements on **1** as the *p*-tolyl derivative also confirmed the dicationic formulation so the possible alternate diamagnetic hydride possibilities are 2 or 6. It is unlikely that the NMR integrations are wrong by this amount.
- (21) NMR simulation was done with the Nicolet program NMCSIM and is described in the Nicolet Magnetics Corp. 1280 program manual. The spectrum is included as supplementary material.
- (22) DeWit, D.; Folting, K.; Streib, W. E.; Caulton, K. G. *Organometallics* **1985**, *4*, 1149 and references cited therein.

S1 and S2) and ¹H and ³¹P NMR spectra for **1** and **2** (Figures S3-S5) (28 pages). Ordering information is given on any current masthead page.

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Unusual Route to an Acyl Complex in Cluster Chemistry: Synthesis, Structure, and Reactivity of Ru₃(μ - η^2 -C(O)(C₆H₅))(μ ₃- η^2 -P(C₆H₅)(C₅H₄N))(CO)₉

Sir:

Recent discoveries^{1,2} of catalytic systems for substitution of CO by phosphine ligands in Ru₃(CO)₁₂ have opened the field to studies of monosubstituted cluster complexes that could not be accessed through thermal routes.³

Typically, thermal reaction of diphenylpyridylphosphine (PPh₂py) with Ru₃(CO)₁₂ was previously shown to give Ru₃(CO)₉(PPh₂py)₃, as well as monomeric species.⁴ A reinvestigation of this reaction using either sodium benzophenone ketyl¹ or [PPN]CN² led us to prepare Ru₃(CO)₁₁(PPh₂py) (**1**) selectively and quantitatively.⁵ Notably, the catalysts did not promote subsequent coordination of the nitrogen atom, since the expected complex Ru₃(CO)₁₀(μ - η^2 -PPh₂py) was not observed. By contrast, **1** is observed to be metastable at ambient temperature, yielding the new cluster complex Ru₃(μ - η^2 -C(O)(C₆H₅))(μ ₃- η^2 -P(C₆H₅)(C₅H₄N))(CO)₉ (**2**),⁶ which is fully characterized by IR, ³¹P and ¹³C NMR,⁶ and X-ray diffraction⁷ methods.

As shown in Figure 1, the structure of this 50-e cluster involves an open trinuclear ruthenium framework. Oxidative cleavage of a phosphorus-carbon bond in diphenylpyridylphosphine has provided the organic fragment P(C₆H₅)(C₅H₄N), which is capping one face of the metal triangle through the bridging phosphido group, which spans the open Ru(2)···Ru(3) edge and is also coordinated to Ru(1) through the nitrogen atom.

A further feature of interest in the structure consists of a bridging acyl group -O=C(C₆H₅) across Ru(2)···Ru(3).

- (1) (a) Bruce, M. I.; Matison, J. G.; Nicholson, B. K.; Rieger, P. H.; Williams, M. L. *J. Chem. Soc., Chem. Commun.* **1982**, 442. (b) Bruce, M. I.; Matison, J. G.; Nicholson, B. K. *J. Organomet. Chem.* **1983**, *247*, 321.
- (2) Lavigne, G.; Kesz, H. D. *J. Am. Chem. Soc.* **1984**, *106*, 4647.
- (3) (a) Candlin, J. P.; Shortland, A. C. *J. Organomet. Chem.* **1969**, *16*, 289. (b) Poë, A. J.; Twigg, M. V. *J. Chem. Soc., Dalton Trans.* **1974**, 1860. (c) Keeton, D. P.; Malik, S. K.; Poë, A. J. *J. Chem. Soc., Dalton Trans.* **1977**, 233. (d) Malik, S. K.; Poë, A. *Inorg. Chem.* **1978**, *17*, 1484.
- (4) Maisonnat, A.; Farr, J. P.; Olmstead, M. M.; Hunt, C. T.; Balch, A. L. *Inorg. Chem.* **1982**, *21*, 3961.
- (5) Both methods afforded the red complex Ru₃(CO)₁₁P(C₆H₅)₂(C₅H₄N) (**1**) in 100% spectroscopic yield within 2 min. IR (ν CO, cm⁻¹; cyclohexane): 2093 (w), 2041 (m), 2025 (s), 1992 (w). This spectrum could be unambiguously assigned to the monosubstituted complex by comparison with related spectra of a variety of substituted complexes (see for example ref 1a). The complex could not be chromatographed, since its conversion into **2** (see ref 6) was accelerated on the column at 25 °C.
- (6) The slow conversion of **1** into **2** is observable in solution at 25 °C. The reaction is complete within 2 h in THF at 40 °C. Chromatographic workup on silica gel (CH₂Cl₂/petroleum ether as eluent) affords the yellow complex Ru₃(μ - η^2 -C(O)(C₆H₅))(μ ₃- η^2 -P(C₆H₅)(C₅H₄N))(CO)₉ (**2**) as the only detectable product (yield 85%, recrystallized). Anal. Calcd for C₂₇H₁₄NO₁₀PRu₃: C, 38.31; H, 1.65; N, 1.65. Found: C, 37.09; H, 1.65; N, 1.58. IR (ν CO, cm⁻¹; hexane): 2060 (w), 2045 (vs), 2020 (s), 1992 (m), 1973 (m), 1955 (w). ¹³C NMR (-60 °C, CD₂Cl₂): δ 302.37 (d, acyl carbon). ³¹P{¹H} NMR (CDCl₃): δ 48.93.
- (7) (a) Crystal data for compound **2**: orthorhombic, space group P2₁2₁2₁, $a = 10.431$ (2) Å, $b = 15.888$ (5) Å, $c = 17.821$ (5) Å, $V = 2953.44$ Å³, $Z = 4$. Data in the range $2 < 2\theta$ (Mo K α) $< 55^\circ$ were collected on an Enraf-Nonius CAD4 diffractometer. Data collection, reduction, and refinement procedures were similar to those detailed in a previous publication.^{7b} Full-matrix least-squares refinements of 379 variables for 2757 reflections with $F_o^2 > 3\sigma(F_o^2)$ led to $R = 0.043$ and $R_w = 0.049$. (b) Lavigne, G.; Lukan, N.; Bonnet, J.-J. *Organometallics* **1982**, *1*, 1040.

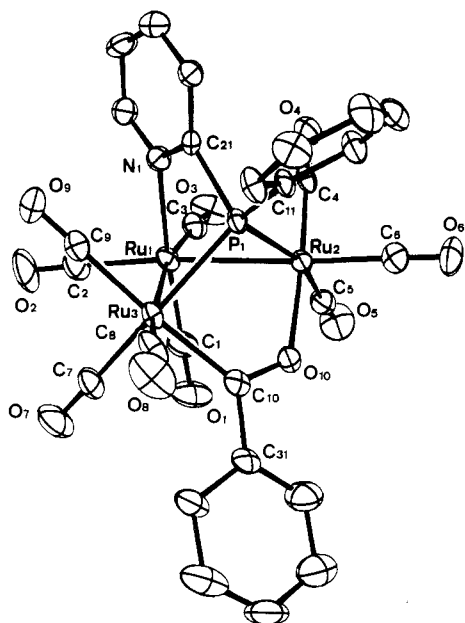


Figure 1. Geometry and atomic labeling scheme for $\text{Ru}_3(\mu\text{-}\eta^2\text{-C(O)(C}_6\text{H}_5))(\mu_3\text{-}\eta^2\text{-P(C}_6\text{H}_5)(\text{C}_5\text{H}_4\text{N))}(\text{CO})_9$ (**2**). Selected parameters. Interatomic distances (Å): Ru(1)–Ru(2), 2.821 (1); Ru(1)–Ru(3), 2.877 (1); Ru(2)–Ru(3), 3.639 (1); Ru(1)–N(1), 2.165 (8); Ru(2)–P(1), 2.356 (2); Ru(3)–P(1), 2.385 (2); Ru(3)–C(10), 2.102 (9); Ru(2)–O(10), 2.100 (6); C(10)–O(10), 1.27 (1). Bond angles (deg): Ru(2)–Ru(1)–Ru(3), 79.39 (3).

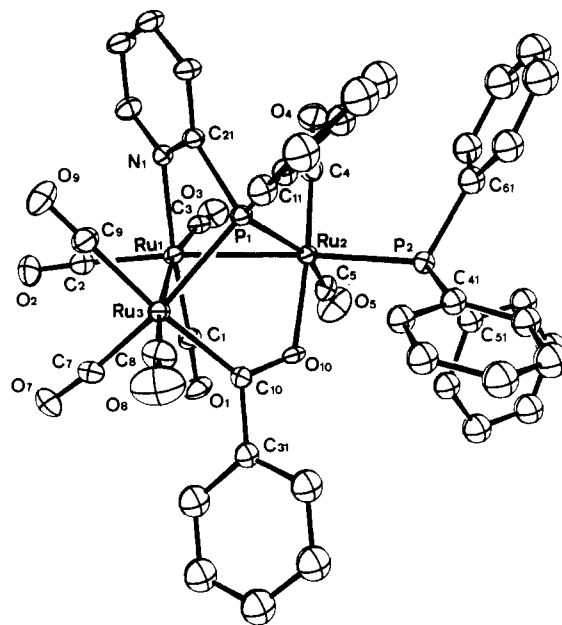
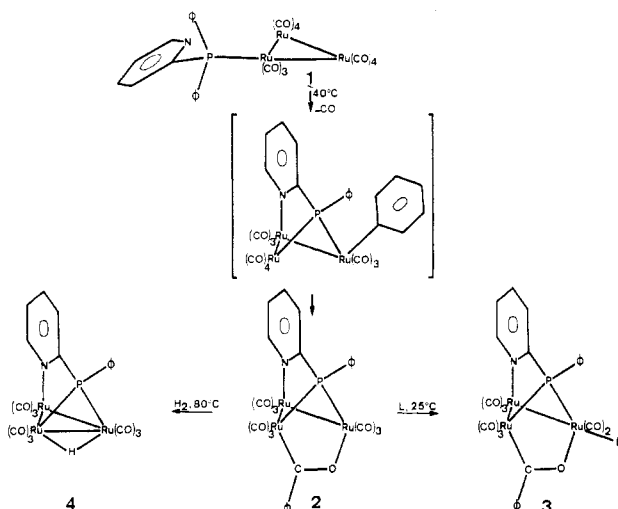


Figure 2. Geometry and atomic labeling scheme for $\text{Ru}_3(\mu\text{-}\eta^2\text{-C(O)(C}_6\text{H}_5))(\mu_3\text{-}\eta^2\text{-P(C}_5\text{H}_4\text{N))}(\text{CO})_8(\text{PPh}_3)$ (**3**). Selected parameters. Interatomic distances (Å): Ru(1)–Ru(2), 2.842 (1); Ru(1)–Ru(3), 2.883 (1); Ru(2)–Ru(3), 3.671 (1); Ru(1)–N(1), 2.158 (3); Ru(2)–P(1), 2.369 (1); Ru(3)–P(1), 2.402 (1); Ru(2)–P(2), 2.405 (1); Ru(3)–C(10), 2.098 (4); Ru(2)–O(10), 2.127 (2); C(10)–O(10), 1.271 (4). Bond angles (deg): Ru(2)–Ru(1)–Ru(3), 79.78 (3); P(1)–Ru(2)–P(2), 106.36 (4); Ru(1)–Ru(2)–P(2), 172.91 (3).

Scheme I



Formation of this acyl group is believed to involve an intermediate with a σ -bonded aryl group, as shown in Scheme I. Though quite likely, this is rarely observed.⁸ Indeed, subsequent reductive elimination of benzene is usually favored, as soon as a hydride ligand is available.⁹ However, in the absence of such a hydride ligand here, a migratory CO insertion takes place to yield the acyl group $\text{C(O)(C}_6\text{H}_5)$, as already observed in one case.¹⁰

Complex **2** can be related to the family of reactive edge-double-bridged species $\text{Ru}_3(\mu\text{-X}, \mu\text{-}\eta^2\text{-C(O)R)}(\text{CO})_{10}$ ¹¹ ($\text{X} = \text{H}$ or halogens), with the additional advantage that the bridgehead P atom in **2** belongs to a face-capping group, which contributes to the stability of the metal framework.

Complex **2** exhibits high reactivity under mild conditions, as illustrated by its reaction with phosphines at room temperature to yield $\text{Ru}_3(\mu\text{-}\eta^2\text{-C(O)(C}_6\text{H}_5))(\mu_3\text{-}\eta^2\text{-P(C}_6\text{H}_5)(\text{C}_5\text{H}_4\text{N))}(\text{CO})_8(\text{PR}_3)$ (**3**) (Scheme I). Spectroscopic¹² and X-ray data¹³ for **3** show that the PR_3 group occupies an equatorial coordination site localized on the Ru(2), in a cis position relative to the oxygen atom of the acyl group (Figure 2). This extends earlier observations of a stereospecific labilizing effect assigned to the oxygen of acyl^{11,14} or carboxylato groups¹⁵ in cluster complexes.

In attempts to remove the μ -acyl group, molecular hydrogen was bubbled through a solution of **2**, leading to the monohydrido species $\text{Ru}_3(\mu\text{-H})(\mu_3\text{-}\eta^2\text{-P(C}_6\text{H}_5)(\text{C}_5\text{H}_4\text{N))}(\text{CO})_9$ (**4**).¹⁶ However,

- (8) (a) Delavaux, B.; Chaudret, B.; Dahan, F.; Poilblanc, R. *Organometallics* **1985**, *4*, 935. (b) Harding, M. M.; Nicholls, B. S.; Smith, A. K. *J. Chem. Soc., Dalton Trans.* **1983**, 1479.
- (9) (a) It has been recently reported that hydrogen atoms required for the reductive elimination of benzene are easily obtained by ortho metalation of additional phenyl groups.^{9b,c} In the present case, coordination of nitrogen is indeed favored over an ortho metalation process. Consequently, no hydride ligand is available. (b) Lugan, N.; Bonnet, J.-J.; Ibers, J. A. *J. Am. Chem. Soc.* **1985**, *107*, 4484. (c) Bergounhou, C.; Bonnet, J.-J.; Fompeyrine, P.; Lavigne, G.; Lugan, N.; Mansilla, F. *Organometallics*, in press (and references therein).
- (10) (a) Blickensderfer, J. R.; Kaesz, H. D. *J. Am. Chem. Soc.* **1975**, *97*, 2681. (b) Blickensderfer, J. R.; Knobler, C. B.; Kaesz, H. D. *J. Am. Chem. Soc.* **1975**, *97*, 2686.

- (11) Kampe, C. E.; Kaesz, H. D. *Inorg. Chem.* **1984**, *23*, 4646 and references therein.
- (12) Reaction of **2** with PR_3 (PPh_3 , PPh_2Me , PPh_2H) is complete within 90 min at 30 °C. Chromatographic workup (CH_2Cl_2 /petroleum ether as eluent) affords **3** as the only product ($\text{Ru}_3(\mu\text{-}\eta^2\text{-C(O)(C}_6\text{H}_5))(\mu_3\text{-}\eta^2\text{-P(C}_6\text{H}_5)(\text{C}_5\text{H}_4\text{N))}(\text{CO})_8(\text{PPh}_3)$ (**3a**)). IR (νCO , cm^{-1} ; CHCl_3): 2050 (s), 2020 (vs), 2085 (m, br), 2060 (m, br), 2040 (sh). ³¹P{¹H} NMR (CDCl_3): δ 49.53 (d, bridging phosphorus, $J_{\text{P-P}} = 17.64$ Hz), 26.26 (d, terminal phosphorus). ¹³C NMR (-60 °C; CD_2Cl_2): δ 302.6 (s, acyl carbon).
- (13) Crystal data for **3**: monoclinic, $P2_1/n$, $a = 10.650$ (3) Å, $b = 31.023$ (4) Å, $c = 13.016$ (2) Å, $\beta = 97.57$ (2)°, $V = 4262$ Å³, $Z = 4$. Full-matrix least-squares refinement of 383 variables for 5770 observations with $F_o^2 > 3\sigma(F_o^2)$ led to $R = 0.049$ and $R_w = 0.087$.
- (14) Mayr, A.; Lin, Y. C.; Boag, N. M.; Kampe, C. E.; Knobler, C. E.; Kaesz, H. D. *Inorg. Chem.* **1984**, *23*, 4640.
- (15) Darenbourg, D. J.; Pala, M.; Waller, J. *Organometallics* **1983**, *2*, 1285.
- (16) Reaction of **2** with H_2 proceeds cleanly in refluxing cyclohexane within 1 h, giving $\text{Ru}_3(\mu\text{-H})(\mu_3\text{-}\eta^2\text{-P(C}_6\text{H}_5)(\text{C}_5\text{H}_4\text{N))}(\text{CO})_9$ (**4**) (yield 95%). Anal. Calcd for $\text{C}_{20}\text{H}_{10}\text{NO}_9\text{PRu}_3$: C, 32.35; H, 1.35; N, 1.89; P, 4.17. Found: C, 32.47; H, 1.47; N, 2.19; P, 4.26. IR (νCO , cm^{-1} ; cyclohexane): 2075 (s), 2045 (vs), 2024 (vs), 2004 (s), 1982 (s), 1960 (w). ³¹P{¹H} NMR (CDCl_3): δ 118.05. ¹H NMR (CDCl_3): δ -15.30 (hydride ligand, $J_{\text{H-P}} = 25$ Hz). The structure of **4** in Scheme I has been assigned on the basis of these spectroscopic data and by comparison of the IR spectrum (νCO region) with that of $\text{Ru}_3(\mu\text{-H})(\mu_3\text{-}\eta^2\text{-C}_6\text{H}_5\text{-PCH}_2\text{P(C}_6\text{H}_5)_2)(\text{CO})_9$,⁸ which displays the same distribution and symmetry of CO ligands.

attempts to trace the resulting organic product are yet unsuccessful.¹⁷

In summary, we find it significant that at an originally electronically saturated metal cluster complex, both the oxidative cleavage of a P-C bond and the subsequent migration of the organic group can be made to occur at very mild conditions, in contrast to the stability of diphenylpyridylphosphine toward $\text{Ru}_3(\text{CO})_{12}$ in thermally induced substitution reactions.⁴

Acknowledgment. Financial support from the CNRS is

gratefully acknowledged. We also thank Johnson Matthey for a generous loan of RuCl_3 .

Supplementary Material Available: Listings of crystal and intensity data, positional and thermal parameters, and selected interatomic distances and bond angles for complex 2 (Tables I-V) and complex 3 (Tables VI-X) (18 pages). Ordering information is given on any current masthead page.

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(17) Analysis by GC gave no evidence for any of the following products: benzene, benzaldehyde, benzyl alcohol, diphenylglyoxal.

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Articles

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Synthesis of New Hybrid Phosphine Amide Complexes of Rhodium(I) and Iridium(I). Intramolecular "Chelate-Assisted" Oxidative Addition of an N-H Bond to Iridium(I)

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Received May 2, 1985

The hybrid phosphine amide ligands *o*- $\text{Ph}_2\text{PC}_6\text{H}_4\text{NHC(O)Ph}$ (PNH(CPhO)) and *o*- $\text{Ph}_2\text{PC}_6\text{H}_4\text{C(O)NHPh}$ (P(CO)NHPh) react with $[\text{MCl}(1,5\text{-COD})]_2$ to give the monomeric complexes $\text{MCl}(1,5\text{-COD})\text{PNH}(\text{CPhO})$ and $\text{MCl}(1,5\text{-COD})\text{P}(\text{CO})\text{NHPh}$ (M = Rh, Ir). The complexes are 4-coordinate with a P-bonded monodentate phosphine amide ligand. P,N-Chelate complexes $[\text{M}(1,5\text{-COD})(\text{PN}(\text{CPh}(\text{OH})))]\text{ClO}_4$ and $[\text{M}(1,5\text{-COD})(\text{P}(\text{CO})\text{NHPh})]\text{ClO}_4$ (M = Rh, Ir) are formed by the addition of 1 equiv of the ligand to $[\text{M}(1,5\text{-COD})(\text{THF})_2]\text{ClO}_4$. The complexes are proposed to have the hybrid ligand coordinated through a nitrogen in the iminoyl form. Solutions of the complexes in acetonitrile solvent undergo intramolecular exchange. For $[\text{Rh}(1,5\text{-COD})\text{P}(\text{CO})\text{NHPh}]^+$ this pathway involves replacement of the iminoyl by MeCN, but for $[\text{Rh}(1,5\text{-COD})\text{PNH}(\text{CPhO})]^+$ the process involves addition of MeCN into the fifth coordination position. In the case where iminoyl substitution occurs, the dangling ligand arm has the amide structure. The complexes $\text{RhCl}(1,5\text{-COD})\text{PNH}(\text{CPhO})$, $\text{RhCl}(1,5\text{-COD})\text{P}(\text{CO})\text{NHPh}$, and $\text{IrCl}(1,5\text{-COD})\text{P}(\text{CO})\text{NHPh}$ react with base to give respectively $\text{Rh}(1,5\text{-COD})\text{PN}(\text{CPhO})$, $\text{Rh}(1,5\text{-COD})\text{P}(\text{CO})\text{NPh}$, and $\text{Ir}(1,5\text{-COD})\text{P}(\text{CO})\text{NPh}$.

Recently we have synthesized some new hybrid phosphine amide ligands in order to induce N-H addition to a low-valent transition-metal center. The compounds can form chelate complexes having either 5- or 6-membered rings, and we anticipated that the insertion of the metal center into the N-H bond would be intramolecularly facilitated by chelation to the phosphorus anchor.¹ Following a preliminary communication,² we have published two articles describing the synthesis of the hybrid ligands, along with the chemistry of the palladium(II) and platinum(II) complexes.^{3,4} In these first two papers we reported the synthesis of P-bonded monodentate Pd(II) and Pt(II) complexes of the phosphine amides, and we described the reactions of these complexes with added bases and acids in order to reversibly convert the compounds to chelated phosphine amido complexes. Thermolysis yielded cyclometalated products, and a platinum(II) complex was characterized having a monodentate P-bonded phosphine amide ligand with an "agostic" N-H bond. With these palladium(II) and platinum(II) complexes no metal hydrides were observed, nor were complexes having a protonated amide ligand bonded to the metal ion. Complexes of rhodium(I) and iridium(I) are more reactive to oxidative ad-

dition than their d^8 congeners of palladium(II) and platinum(II), and furthermore for amide chelation these metal centers are more susceptible to achieving pentacoordination.⁵

This article describes the reaction chemistry of our phosphine amide ligands with chloro-bridged rhodium(I) and iridium(I) alkene complexes. The particular ligands used are *o*- $\text{Ph}_2\text{PC}_6\text{H}_4\text{NHC(O)Ph}$ (PNH(CPhO)) and *o*- $\text{Ph}_2\text{PC}_6\text{H}_4\text{C(O)NHPh}$ (P(CO)NHPh) (Figure 1).³ The former compound can be used to prepare 5-membered ring chelate complexes, and the latter the analogous 6-membered ring complexes. Deprotonation of the coordinated amide nitrogen can be used to synthesize the corresponding amido complexes. In this paper we describe the synthesis and reaction chemistry of new chelate complexes having both the phosphorus atom and the uncharged amide nitrogen complexed to rhodium(I) and iridium(I). In addition to these d^8 complexes we also describe the synthesis of the first amido hydride complexes of iridium(III), which have been formed by the oxidative addition of the amide N-H bond to iridium(I).

Experimental Section

Many of the experimental details have been described in the two earlier papers.^{3,4} The air-sensitive complexes prepared in this paper have been handled by Schlenk techniques or in a Vacuum Atmospheres dry-box. Our Schlenk techniques use a standard double-manifold setup and single-side-arm Schlenk vessels. Liquid transfer is effected by stainless-steel transfer tubes through Suba Seal septa (Strem). NMR spectra of air-sensitive complexes were measured in 10-mm septum-capped tubes (Wilmad Co.). Rhodium trichloride and iridium trichloride were sup-

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- (2) Hedden, D.; Roundhill, D. M.; Fultz, W. C.; Rheingold, A. L. *J. Am. Chem. Soc.* **1984**, *106*, 5014-5016.
- (3) Hedden, D.; Roundhill, D. M. *Inorg. Chem.*, in press.
- (4) Hedden, D.; Roundhill, D. M.; Fultz, W. C.; Rheingold, A. L. *Organometallics*, in press.

- (5) Collman, J. P.; Hegedus, L. S. "Principles and Applications of Organotransition Metal Chemistry"; University Science Books: Mill Valley, CA, 1980.