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Preferential displacement of fluorophosphine ligands from fluorophosphinetungstencarbonyl complexes by reaction with trimethylamine oxide

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Abstract

The photolysis of [W(CO)$_4$dppe] in toluene in the presence of a large excess of PF$_3$, using a medium pressure mercury lamp, gives [W(PF$_3$)$_4$dppe] (**I**). The thermal reaction of solutions of *cis*-[W(CO)$_4$(Ph$_3$P)(CH$_3$CN)] with fluorophosphine ligands L gives *cis*-[W(CO)$_4$(Ph$_3$P)L] (L = PF$_3$ (**II**), Me$_2$NPF$_2$ (**III**), MeN(PF$_2$)$_2$ (**IV**)). Compounds **I**–**IV** were characterized by IR, mass and NMR spectroscopy. They react with Me$_3$NO in acetonitrile, with loss of the fluorophosphine ligand. The CO ligands in **II**–**IV** are not attacked by the amine oxide.

Introduction

Phosphorus trifluoride and other fluorophosphines resemble carbon monoxide in their ability to stabilize low oxidation states in transition metal complexes. The syntheses and structures of these systems, and their potential applications in catalysis, have been reviewed by several authors [1–4]. The thermal stability of binary metal–PF$_3$ complexes and of some fluorophosphine–metal hydrides is often greater than that of the carbonyl analogues. Trifluorophosphine metal halide complexes, on the other hand, generally show diminished stability, severely limiting the derivative chemistry of such systems. Stability is improved, however, if σ-donating tertiary phosphine ligands are also present in the halide complexes. The σ-donor and π-acceptor properties of phosphine ligands can be varied, depending on the nature of the attached groups [5, 6]. Thus the relative stabilities of different oxidation states can be influenced. Phosphorus trifluoride and its amino derivatives R$_2$NPF$_2$ and RN(PF$_2$)$_2$ have provided a rich source of low oxidation state metal complexes [1–4, 7, 8]. In the work described below, we set out to prepare complexes of the type [W(CO)$_x$(Ph$_3$P)L$_{5-x}$] and [W(CO)$_x$(dppe)L$_{4-x}$] (L = PF$_3$, Me$_2$NPF$_2$, MeN(PF$_2$)$_2$; dppe = bis(diphenylphosphino)ethane), in the expectation that the arylphosphine ligand would permit the subsequent syntheses of stable derivatives containing the L ligands. The syntheses and characterization of the new compounds [W(PF$_3$)$_4$dppe] (**I**) and *cis*-[W(CO)$_4$(Ph$_3$P)L] (L = PF$_3$ (**II**), Me$_2$NPF$_2$ (**III**), MeN(PF$_2$)$_2$ (**IV**), and their reactions with Me$_3$NO are described.

Experimental

All reactions were carried out under pre-purified nitrogen in Schlenk tubes or in Carius tubes fitted with Kontes greaseless stopcocks. Solvents were dried and distilled under nitrogen before use. Mass spectra were obtained by the electron impact method using a Hewlett Packard model 5985 mass spectrometer. IR spectra were measured using a Perkin-Elmer model 983G instrument, in CaF$_2$ solution cells or as KBr discs. NMR spectra were obtained on Bruker WB-100 or WB-400 instruments. Melting points were measured on Fisher Johns melting point equipment.

Common reagents were purchased from commercial suppliers. PF$_3$ was purified by fractionation in a high vacuum system through traps cooled to −96, −120 and −196 °C. Product condensing at −196 °C was used in reactions. Me$_3$NO was dried by pumping under high vacuum, and sublimed.

[W(CO)$_4$dppe] [9], [W(CO)$_5$(CH$_3$CN) [10], Me$_2$NPF$_2$ [11] and MeN(PF$_2$)$_2$ [12, 13] were prepared by published methods. [W(CO)$_5$(Ph$_3$P)] [14] and *cis*-[W(CO)$_4$(Ph$_3$P)(CH$_3$CN)] [15] were prepared according to Koelle's procedure [10]. The new compounds described below were characterized by analysis, mass, IR and NMR (^{1}H, ^{31}P, ^{19}F) spectroscopy. All of the compounds are stable under nitrogen and to brief exposure

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to air, but they slowly decompose in solution, depositing a blue metallic mirror.

Synthesis of [W(PF$_3$)$_4$dppe] (I)

Toluene (50 ml) and [W(CO)$_4$dppe] (410 mg, 0.591 mmol) were placed in a quartz Carius tube fitted with a Kontes stopcock. The solution was degassed in three freeze/thaw cycles, PF$_3$ (20.3 mmol) was transferred to the tube, and the mixture was photolyzed until all of the CO was expelled. At intervals the reaction was stopped, the solution was frozen in liquid nitrogen, and the evolved CO was measured by means of a Sprengle pump. When reaction was complete, the solvent and excess PF$_3$ were removed under vacuum and the product [W(PF$_3$)$_4$ dppe] was recrystallized from dichloromethane/hexane as white needles (yield 20%; m.p. 230 °C).

Synthesis of cis-[W(CO)$_4$(Ph$_3$P)(PF$_3$)] (II)

A solution of *cis*-W(CO)$_4$(Ph$_3$P)(CH$_3$CN)] (395 mg, 0.65 mmol) in dichloromethane (30 ml) in a Carius tube was degassed three times. PF$_3$ (0.71 mmol) was transferred to the tube, and reaction was allowed to occur at 50 °C for 3 h. The greenish-yellow solution was concentrated under vacuum, the product *cis*-[W(CO)$_4$(Ph$_3$P)(PF$_3$)] was precipitated by adding hexane and further purified by recrystallization from hexane (yield 76%). *Anal.* Found: C, 41.0; H, 2.34. Calc.: C, 40.87; H, 2.32%.

Synthesis of cis[W(CO)$_4$(Ph$_3$P)(Me$_2$NPF$_2$)] (III)

A solution of *cis*-[W)CO)$_4$(Ph$_3$P(CH$_3$CN)] (647 mg, 1.08 mmol) in dichloromethane (15 ml) in a Carius tube was degassed twice, and Me$_2$NPF$_2$ (1.25 mmol) was added. After 12 h at room temperature, the solvent and excess Me$_2$NPF$_2$ were removed under vacuum. The residue was purified by column chromatography on silica gel (230–400 mesh; 1:2 CH$_2$Cl$_2$: hexane as eluant) to give *cis*-[W(CO)$_4$(Ph$_3$P)(Me$_2$NPF$_2$)] (yield 30%; m.p. 105 °C). *Anal.* Found: C, 43.1; H, 3.13; N, 1.95. Calc. C, 42.92; H, 3.13; N, 2.09%.

Synthesis of cis-[W(CO)$_4$(Ph$_3$P)(MeN(PF$_2$)$_2$)] (IV)

A solution of *cis*-[W(CO)$_4$(Ph$_3$P)(CH$_3$CN)] (435 mg, 0.73 mmol) in dichloromethane (10 ml) in a Carius tube was degassed twice, and MeN(PF$_2$)$_2$ (1.21 mmol) was added. The tube was heated at 50 °C for 12 h. The product *cis*-[W(CO)$_4$(Ph$_3$P)(MeN(PF$_2$)$_2$)] (**IV**) was precipitated by adding hexane and further purified by crystallization from CH$_2$Cl$_2$/hexane (yield 33%, m.p. 100 °C).*Anal.* Found: C, 38.3; H, 2.52; N, 1.83. Calc.: C, 38.07; H, 2.48; N, 1.93%.

Results and discussion

Starting with [W(CO)$_4$dppe], the replacement of all of the CO groups was achieved by photolyzing toluene solutions containing excess PF$_3$, over a period of several hours. The mass spectrum of **I** showed the parent ion grouping of peaks arising from the several isotopes of tungsten ($m/e=934$ for the 30.64% abundant ^{184}W isotope), together with fragments corresponding to the sequential loss of four PF$_3$ groups. Strong peaks at m/e 554 and 474 corresponded to the loss of C$_2$H$_4$ and Ph$_2$P from the base peak, [Wdppe]$^+$. The fragmentation pattern was analogous to that reported for [Mo(PF$_3$)$_4$dppe] [16]. The IR spectrum corroborated the absence of carbonyl groups and showed bands at 867–807 cm^{-1} due to P–F vibrational modes [12], as well as features due to the dppe ligand.

The ^{1}H NMR spectrum of **I** showed phenyl and methylene multiplets in a 5:1 intensity ratio. The ^{31}P NMR spectrum consisted of two closely spaced, low field second order quartet patterns due to the PF$_3$ groups, and a high field broad doublet due to dppe. The ^{19}F spectrum consisted of two signals; one was a simple doublet of doublets, while the other signal was a second order multiplet (see Table 5). These results confirm the magnetic inequivalence of the two pairs of PF$_3$ groups.

Attempts to prepare members of the series [W(CO)$_x$(dppe)(Me$_2$NPF$_2$)$_{4-x}$] via the photolysis of solutions of [W(CO)$_4$dppe] and Me$_2$NPF$_2$ were unsuccessful. The dppe ligand as well as CO was eliminated, giving products of the type [W(CO)$_x$(Me$_2$NPF$_2$)$_{6-x}$]. An earlier study showed that [Mo(CO)$_4$dppe] also loses dppe in photolytic reactions with Me$_2$NPF$_2$ [16].

The photolytic substitution of the CO groups in [W(CO)$_5$(Ph$_3$P)] by PF$_3$ was studied under various conditions in an effort to obtain a dominant product, but mixtures of [W(CO)$_x$(Ph$_3$P)(PF$_3$)$_{5-x}$] that were difficult to separate were formed. The photolysis of mixtures of [W(CO)$_6$] and PF$_3$ also gives a suite of products, unless a large excess of PF$_3$ is used [2]. King and Gimeno [12] have shown that the photolysis of [W(CO)$_6$] and MeN(PF$_2$)$_2$ gives a complicated mixture of mono- and binuclear species. The displacement of CH$_3$CN from *cis*-[W(CO)$_4$(Ph$_3$P)(CH$_3$CN)] by thermal reaction with fluorophosphine ligands L was found to be satisfactory, giving the new compounds *cis*-[W(CO)$_4$(Ph$_3$P)L] (L=PF$_3$ (**II**), Me$_2$NPF$_2$ (**III**), MeN(PF$_2$)$_2$ (**IV**). Similarly, the thermal reaction of [W(CO)$_5$(CH$_3$CN)] with MeN(PF$_2$)$_2$ gives [W(CO)$_5$(MeN(PF$_2$)$_2$)] and [(W(CO)$_5$)$_2$(MeN(PF$_2$)$_2$)] [17].

The *cis* configuration is proposed for complexes **II**–**IV**, based on the similarities of their IR spectra (carbonyl region) with those of known *cis*-[W(CO)$_4$L$_2$] complexes

TABLE 1. IR spectra[a] (carbonyl region) of *cis*-[W(CO)$_4$L$_2$] complexes

Compound	ν(CO) (cm^{-1})			
[W(CO)$_4$(Ph$_3$P)(PF$_3$)] (**II**)	2051m	1967sh	1939s	
[W(CO)$_4$(Ph$_3$P(Me$_2$NPF$_2$)] (**III**)	2035s	1938sh	1915s	
[W(CO)$_4$(Ph$_3$P)(MeN(PF$_2$)$_2$)] (**IV**)	2043m	1955m	1931s	
[W(CO)$_4$(MeN(PF$_2$)$_2$)$_2$)]	2070m	1991m	1965vs	1977s
[W(CO)$_4$(MeN(PF$_2$)$_2$)]	2062m	1990s	1970vs	1977vs

[a]In dichloromethane.

(Table 1) and on the values of the ^2J(P–P) coupling constants for **II** and **III** that are in the range (20–30 Hz) typical for *cis* rather than *trans* geometry in octahedral, bis(phosphine)metal–tetracarbonyl complexes.

The mass spectra (Table 2) of each of the compounds **II–IV** showed the parent ion and fragments corresponding to the parallel loss of carbonyl and fluorophosphine ligands. The signal due to [W(CO)$_4$(Ph$_3$P)]$^+$ (m/e = 558) was relatively strong, compared to [W(CO)$_4$(Ph$_3$P)L]$^+$, for **II** and **III**, but weak for **IV**. It appears that PF$_3$ and Me$_2$NPF$_2$ are lost more readily than CO, whereas MeN(PF$_2$)$_2$ is lost less readily, possibly due to incipient cyclization by the bidentate ligand.

The proton decoupled ^{31}P NMR spectrum (Tables 3 and 4) of compound **II** consisted of a low field doublet of quartets due to coordinated PF$_3$, and a higher field doublet due to Ph$_3$P, together with satellites showing coupling with the 14.4% abundant spin 1/2 ^{183}W nucleus. The ^{19}F NMR spectrum (Table 5) was a doublet also showing coupling to ^{183}W. The ^1J(P–F) coupling in **II** is identical, within experimental error, with that observed in *cis*-[W(CO)$_4$(PF$_3$)$_2$] (1281 Hz [1a]), but is ~130 Hz smaller than in free PF$_3$ (1410 Hz [18]). The phosphorus coordination chemical shifts are 24 and 25 Hz, respectively, based on δ^{31}P(PF$_3$) = 97 ppm [19]. The corresponding ^2J(P–P) and the ^2J(W–F) couplings in *cis*-[W(CO)$_4$(PF$_3$)$_2$] [20] and **II** have similar values.

TABLE 2. Mass spectra[a] of *cis*-[W(CO)$_4$(Ph$_3$P)L] complexes

Fragment	PF$_3$ (**II**)	Me$_2$NPF$_2$ (**III**)	MeN(PF$_2$)$_2$ (**IV**)
P	646	671	725
P − CO	618	643	697
P − 2CO	590	615	669
P − 3CO	562	586	641
P − 4CO	534	558	613
P − L	558	558	558
P − L − CO	530	530	530
P − L − 2CO	502	502	502
P − L − 3CO	474	474	
?		465[b]	465[b]
P − L − 4CO[c]	446	446	446
?		387[d]	387[d]
?	366	366	366

[a]Electron impact excitation. [b]465 = [W(Ph$_3$P)F]$^+$. [c]Base peak. [d]387 = [W(C$_6$H$_5$)(C$_6$H$_4$F)P]$^+$.

The ^{1}H NMR spectrum of **III** consisted of a phenyl multiplet and a doublet of triplets for the methyl protons (^3J(P–H) = 11.0 Hz; ^4J(F–H), 3.0 Hz). The proton decoupled ^{31}P NMR spectrum (Tables 3 and 4) consisted of a low field doublet of triplets due to the PF$_2$ group, and a high field doublet due to the Ph$_3$P ligand, together with satellite peaks due to coupling with ^{183}W. The ^2J(P–P) couplings in **III** and *cis*-[W(CO)$_4$(Me$_2$NPF$_2$)$_2$] [21, 22] are 28 and 21 Hz, respectively.

The ^{19}F NMR spectrum of **III** (Table 5) was a doublet of septuplets. The ^1J(P–F) coupling and the coordination chemical shift for the Me$_2$NPF$_2$ ligand in **III** have essentially the same values as the corresponding parameters in *cis*-[W(CO)$_4$(Me$_2$NPF$_2$)$_2$] [21]. The ^1J(P–F) coupling is about 90 Hz larger (8%) in free Me$_2$NPF$_2$ (1196 Hz [23]), when compared to the value in the tungsten complexes. The ^1J(W–PF) values in **III** and *cis*-[W(CO)$_4$(Me$_2$NPF$_2$)$_2$] [22] are 401 and 370 Hz [17], respectively.

The ^{1}H NMR spectrum of **IV** showed phenyl and methyl signals in a 5:1 intensity ratio. The latter signal was a doublet of multiplets arising from numerous couplings within this multiple spin system.

The proton decoupled ^{31}P NMR spectrum of **IV** (Tables 3 and 4) consisted of a high field broad signal due to Ph$_3$P, and two poorly resolved, nearly coincident sets of triplets due to the coordinated and the free PF$_2$ groups. The coordination chemical shift of the attached PF$_2$ group is almost zero.

The ^{19}F NMR spectrum of **IV** (Table 5) consisted of two sets of second order signals, one slightly downfield of the other and likely attributable to the coordinated PF$_2$ group.

The potential cyclization of the mono-ligate, bidentate MeN(PF$_2$)$_2$ ligand in compound **IV** was studied. When a solution of **IV** in decalin was heated at 180 °C for 2 h, some thermal decomposition occurred, but most of **IV** remained unchanged. Treatment of **IV** with Me$_3$NO (in an attempt to remove CO) resulted in elimination of the fluorophosphine ligand (see below). A frozen glass of **IV** in toluene at −196 °C was irradiated with

TABLE 3. ^{31}P NMR data[a] for the Ph_3P group in *cis*-$[W(CO)_4(Ph_3P)L]$ complexes

Compound	$\delta^{31}P$	$\Delta(\delta^{31}P)$	$^1J(P–W)$	$^2J(P–P)$
$[W(CO)_5(Ph_3P)]$[b]	21	27		
$[W(CO)_4(Ph_3P(CH_3CN)]$[b]	30	36	121	
$[W(CO)_4(Ph_3P)(PF_3)]$ (**II**)[b]	20	26	242	25
$[W(CO)_4(Ph_3P)(Me_2NPF_2)]$ (**III**)[c]	21	27	239	28
$[W(CO)_4(Ph_3P)(MeN(PF_2)_2)]$ (**IV**)[c]	21	27		

[a]Chemical shifts in ppm relative to external 85% H_3PO_4; coupling constants in Hz. [b]In C_6D_6. [c]In CH_2Cl_2.

TABLE 4. ^{31}P NMR data[a] for the fluorophosphine ligands in *cis*-$[W(CO)_4LL']$ complexes

Compound	$\delta^{31}P$	$\Delta(\delta^{31}P)$	$^1J(P–F)$	$^1J(P–W)$
$[W(CO)_4(Ph_3P)(PF_3)]$ (**II**)[b, c]	121	24	1281	490
$[W(CO)_4(PF_3)_2]$[d]	122[e]	24[f]	1282[e]	
$[W(CO)_4(Ph_3P)(Me_2NPF_2)]$ (**III**)[g]	158	15[h]	1105	401
$[W(CO)_4(Me_2NPF_2)_2]$[e]	156	13	1096	370
$[W(CO)_4(Ph_3P)(MeN(PF_2)_2)]$ (**IV**)[g]	(144)[i]	(3)[i]		
$[W(CO)_4(MeN(PF_2)_2)]$[j]	112	−29	1275	

[a]Chemical shifts in ppm relative to external 85% H_3PO_4; coupling constants in Hz. [b]In C_6D_6. [c] $^2J(W–F) = 28$ Hz. [d] $^2J(W–F) = 30$ Hz [20]. [e]Ref. 21. [f]Based on $\delta^{31}P(PF_3) = 97$ ppm [19]. [g]In CH_2Cl_2. [h]Based on $\delta^{31}P(Me_2NPF_2) = 143$ ppm [21]. [i]Approximate value due to poor resolution and near coincidence of coordinated and free PF_2 groups. [j]Refs. 20 and 24.

TABLE 5. ^{19}F NMR data[a] for *cis*-$[W(CO)_4(Ph_3P)L]$ complexes

Compound	^{19}F	$^1J(P–F)$	(^{19}F)
$[W(CO)_4(Ph_3P)(PF_3)]$ (**II**)[b]	−9.1	1280[c]	29[d]
$[W(CO)_4(Ph_3P)(Me_2NPF_2)]$ (**III**)[b, e]	−34	1101[f]	32[g]
$[W(CO)_4(Ph_3P)(MeN(PF_2)_2)]$ (**IV**)[h]	−28[i]	(1117)[j]	47[k]
	−71[l]	(1236)[j]	

[a]Chemical shifts in ppm relative to external $CFCl_3$; coupling constants in Hz. [b]In CH_2Cl_2. [c] $^2J(W–F) = 28$ Hz. [d] $\delta^{19}F(PF_3) = -38$ ppm in acetonitrile. [e]Doublet of septuplets, $^4J(F–H) = 3$ Hz. [f] $^2J(W–F) = 30$ Hz [20]. [g] $\delta^{19}F$(free Me_2NPF_2) $= -65.7$ ppm in acetonitrile; $\delta^{19}F$(pure liquid) $= 65.3$ ppm [7]. [h]In toluene. [h]In toluene. [i]Coordinated PF_2 group. [j]Approximate value. [k] $\delta^{19}F$(free $MeN(PF_2)_2$) $= -74.7$ ppm [25]. [l]Free PF_2 group.

UV light for 30 min; the ^{19}F NMR spectrum of the thawed solution showed only the presence of **IV**. Photolysis of toluene solutions at ambient temperature or at 0 °C gave numerous products, as evidenced by ^{19}F NMR spectroscopy. A very small sample of yellow crystals, with a mass spectrum corresponding to the chelate product $[W(CO)_3(Ph_3P)(MeN(PF_2)_2)]$, and showing new IR absorption bands, was obtained from the reaction mixture.

In order to prepare the more highly substituted complexes $[W(CO)_3(Ph_3P)(L)_2]$, a method for the sequential, selective removal of CO groups from **II**–**IV** was sought. The use of tertiary amine oxides is known to effect the partial substitution of CO in carbonyl complexes [10]. It was argued that the π-acid nature of the fluorophosphine ligands in **II**–**IV** would enhance or at least maintain the electrophilic character of the carbonyl groups, and thereby facilitate nucleophilic attack by trimethylamine oxide to produce carbon dioxide. σ-Donating ligands, on the other hand, ultimately inhibit the attack of amine oxide upon carbonyl groups. To test the above hypothesis, compounds **II**–**IV** were allowed to react with Me_3NO in acetonitrile. In each case the fluorophosphine ligand rather than CO was eliminated, and *cis*-$[W(CO)_4(Ph_3P)(CH_3CN)]$ was identified by IR and ^{31}P NMR spectroscopy as the main metal-containing complex. The oxide OPF_3 was detected by ^{19}F NMR spectroscopy as the only volatile fluorine-containing product from the reaction of *cis*-$[W(CO)_4(Ph_3P)(PF_3)]$ with Me_3NO. These results indicate that the fluorophosphine ligands in **II**–**IV** are more sensitive to nucleophilic attack by amine oxide than the carbonyl ligands. Furthermore, in the case of **IV**, attack occurs at the sterically more hindered, coordinated PF_2 site, rather than at the pendant PF_2 site. Compound **I** also reacts readily with Me_3NO, releasing OPF_3, whereas $[W(CO)_4dppe]$ is unreactive.

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