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REACTIONS OF TRICARBONYLPHOSPHINE COMPLEXES (bipy)P(4-$XC_6H_4)_3Mo(CO)_3$ (X = Cl, F, Me, MeO) WITH TIN(IV) DERIVATIVES R_nSnCl_{4-n} (n = 0, 1, 3)

M. PANIZO and M. CANO*

Facultad de Ciencias Químicas, Departamento de Química Inorgánica, Universidad Complutense, Madrid-3 (Spain)

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Summary

The reactions of the tricarbonylphosphine complexes (bipy)(P)$Mo(CO)_3$ (bipy = 2,2′-bipyridine and (P) = P(4-$ClC_6H_4)_3$, P(4-$FC_6H_4)_3$, P(4-$CH_3C_6H_4)_3$ and P(4-$CH_3OC_6H_4)_3$) with $SnCl_4$ yield new seven-coordinate compounds of the type (bipy)P(4-$XC_6H_4)_3(CO)_2Mo(Cl)(SnCl_3)$ (X = F, Cl, CH_3). When X = MeO the complex (bipy)$(CO)_3Mo(Cl)(SnCl_3)$ is obtained. The halides CH_3SnCl_3 and $C_6H_5SnCl_3$ enter into similar reactions to give new dicarbonyl complexes (bipy)-P(4-$XC_6H_4)_3(CO)_2Mo(Cl)(SnRCl_2)$ and tricarbonyl complexes (bipy)-$(CO)_3Mo(Cl)(SnRCl_2)$ (R = CH_3, C_6H_5).

Introduction

In previous papers [1–5] we have shown that the mixed carbonyl complexes (NN)(L)$M(CO)_3$ (NN = 2,2′-bipyridine, 1,10-phenanthroline, (phen); L = CO, PPh_3, py), undergo oxidative addition reactions when treated with mercuric derivatives. In all cases the (NN)$M(CO)_3$ moiety is conserved and the ease of displacement of L ligand depends on the nature of the acceptor.

Tin and germanium tetrahalides and organohalides have been found to react readily with several substituted carbonyls of Mo and W to yield neutral or ionic seven-coordinate complexes. For example the (NN)$M(CO)_4$ (NN = bipy, phen, 2,7-dmnapy = 2,7-dimethyl-1,8-naphthyridine; M = Mo, W) compounds yield new complexes (NN)$M(CO)_3(X)(SnX_3)$ and (NN)$M(CO)_3(X)SnX_{3-n}R_n$ on reaction with SnX_4, $RSnX_3$, R_2SnX_2 (X = halogen, R = alkyl, aryl) [6,7], and the complex (diphos)$M(CO)_4$ (diphos = $[(C_6H_5)_2PCH_2]_2$) reacts with two moles of $SnCl_4$ to yield the ionic product $[(diphos)Mo(CO)_4SnCl_3]^+ [SnCl_5OH_2]^-$ [8].

Several crystallographic studies of some derivatives of this type have been reported; an interesting feature of the neutral complexes (bipy)$(CO)_3ClMoSnCH_3Cl_2$ [9] and (DTH)$(CO)_3ClWSnCH_3Cl_2$ [10] is that the Cl atom attached to Mo or W is

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TABLE 1

ANALYTICAL DATA AND SOME PHYSICAL PROPERTIES FOR THE NEW COMPLEXES I–X

Complex [a]	Colour	Analyses (Found (calcd.) (%))			M.p. [b] (°C)	Conductivity [c] (Λ_M) (ohm^{-1} cm^2 mol^{-1})
		C	H	N		
(bipy)P(4-ClC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCl$_3$) (I)	orange-red	38.68 (38.54)	2.20 (2.14)	3.01 (2.99)	180	11
(bipy)P(4-FC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCl$_3$) (II)	orange	40.11 (40.69)	2.36 (2.26)	3.55 (3.16)	160	8.9
(bipy)P(4-CH$_3$C$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCl$_3$) (III)	orange	45.16 (45.38)	3.20 (3.32)	3.19 (3.21)	187	10
(bipy)(CO)$_3$Mo(Cl)(SnCl$_3$) (IV)	yellow	26.07 (26.14)	1.55 (1.44)	4.54 (4.69)	150	0.75 [d]
(bipy)P(4-ClC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCH$_3$Cl$_2$) (V)	orange	39.90 (40.71)	2.47 (2.51)	3.01 (3.06)	210	9
(bipy)P(4-FC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCH$_3$Cl$_2$) (VI)	orange	42.10 (43.05)	2.60 (2.66)	3.55 (3.24)	230	10
(bipy)P(4-CH$_3$OC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCH$_3$Cl$_2$) (VII)	orange	47.01 (47.99)	3.45 (3.55)	3.20 (3.11)	225	8.6
(bipy)(CO)$_3$Mo(Cl)(SnCH$_3$Cl$_2$) (VIII)	yellow	29.41 (29.16)	2.01 (1.91)	4.35 (4.86)	190	2.6 [d]
(bipy)P(4-ClC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnC$_6$H$_5$Cl$_2$) (IX)	orange	44.15 (44.28)	2.50 (2.56)	2.65 (2.87)	195	8.7
(bipy)(CO)$_3$Mo(Cl)(SnC$_6$H$_5$Cl$_2$) (X)	yellow	35.66 (35.73)	2.10 (2.03)	4.48 (4.39)	168	0.5 [d]

[a] All the complexes are diamagnetic. [b] Decomposition temperature. [c] In DMFA solution. [d] In acetone solution.

weakly bound to the Sn atom, giving an unusual five-coordination at Sn. In addition a Mo–Sn bond and a seven-coordination at the transition metal are present.

The reaction

$$(L_2)M(CO)_4 + SnR_{4-n}X_n \rightarrow (L_2)(CO)_3Mo(X)(SnR_{4-n}X_{n-1}) + CO$$

can be classified as oxidative elimination, and provides a method of making compounds with bonds between transition metals and tin or germanium.

A study of new carbonyl complexes containing Mo–Sn bonds and possible chlorine bridges, and of the factors controlling replacement of the ligand L in $(NN)(L)Mo(CO)_3$ complexes, led us to investigate the reactions of $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ with chlorotin derivatives R_nSnCl_{4-n} ($R = CH_3$, C_6H_5; $n = 0, 1, 3$) in which different electronic and steric effects are present.

Results and discussion

In the reactions of the complexes $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ ($X = Cl, F, CH_3, CH_3O$) with tin(IV) derivatives, $SnCl_4$, Ph_3SnCl, $PhSnCl_3$, Me_3SnCl_3 and $MeSnCl_3$ ($Me = CH_3$, $Ph = C_6H_5$), $SnCl_4$ reacts more rapidly than the organometallic halides, and of the latter only the CH_3SnCl_3 and $C_6H_5SnCl_3$ yield products. Some reactions were observed with Ph_3SnCl when more vigorous conditions were used, but at the high temperatures involved the products are decomposed. No reaction was observed with Me_3SnCl.

There are difficulties in isolating the products, especially when the ligands are $P(4\text{-}CH_3C_6H_4)_3$ or $P(4\text{-}CH_3OC_6H_4)_3$; in such cases the products decompose in the reaction medium or mixed products are obtained as oils.

In general the reactions of complexes $(bipy)(P(4\text{-}XC_6H_4)_3Mo(CO)_3$ with tin(IV) chloride and organotin chlorides are of the two types: (a) reactions with retention of the phosphine ligand and CO elimination to give the dicarbonyl complexes $(bipy)P(4\text{-}XC_6H_4)_3(CO)_2Mo(Cl)SnRCl_2)$, and (b) reactions involving elimination of phosphine ligand to yield $(bipy)(CO)_3Mo(Cl)(SnCl_3)$ or $(bipy)(CO)_3Mo(Cl)(SnRCl_2)$.

Reaction of tin(IV) chloride with $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ *(*$X = Cl, F, CH_3, CH_3O$*)*

The tricarbonyl complexes $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ react with $SnCl_4$ in 1 : 1 mole ratio at room temperature under nitrogen; CO evolution is observed. Elimination of $P(4\text{-}CH_3C_6H_4)_3$ and $P(4\text{-}CH_3OC_6H_4)_3$ phosphine ligands occurs to yield a yellow compound whose analytical data, IR and melting point correspond with that of the heptacoordinate complex $(bipy)(CO)_3Mo(Cl)(SnCl_3)$ [6]. However, when the phosphine ligand in the substrate is $P(4\text{-}XC_6H_4)_3$ ($X = Cl, F$), orange products are precipitated, these compounds can be formulated as $(bipy)P(4\text{-}XC_6H_4)_3(CO)_2Mo(Cl)(SnCl_3)$ (I, II) on the basis of their analytical data (Table 1). This type of compound with $X = Me$ (III) is also isolated from the filtrate after the yellow tricarbonyl complex has been removed.

The new complexes are microcrystallines solids, air-stables, and slightly soluble in polar solvents. In DMFA, CH_2Cl_2 and nitrobenzene solutions, changes in colour are observed. The conductances in DMFA are indicative of their neutral character. Table 1 shows some properties of the products isolated.

The infrared spectral data, of the new complexes obtained, in the solid state are listed in Table 2. The pattern of the bands in the carbonyl stretching region is similar for all three complexes I–III; two strong bands are centered at ca. 1920–1840 cm^{-1}, typical of *cis*-dicarbonyl compounds [11,12]. However, complex IV shows three carbonyl bands in the 2000–1800 cm^{-1} region (Table 2), and the same bands are present in CH_2Cl_2 solution.

The new compounds show the characteristic bipyridine and phosphine bands in the 1600–400 cm^{-1} region (13–15). In the lower frequency region (350–200 cm^{-1}) as expected the complexes show bands corresponding to Sn–Cl stretching vibrations [16]. This region is characteristic of a bridging Mo–Cl [16]. In Fig. 1 are reproduced the spectra of four complexes in the 400–200 cm^{-1} region. The pattern of the bands is similar in all cases and their positions are not significantly different. The $SnCl_3$ group bonded to Mo is not appreciably modified by the substitution of CO for the phosphine ligand around the Mo atom. The free $SnCl_3^-$ ion, shows two bands at 289 (A_1) and 252 cm^{-1} (E) which are shifted to higher frequencies upon coordination to metal or other ligand [16].

For complexes I–IV four bands at 340, 300, 270, 260 cm^{-1} are observed, and this is consistent with an increase in the coordination around the tin atom. An X-ray diffraction study has shown that $(bipy)(CO)_3Mo(Cl)(SnMeCl_2)$ contains a chlorine atom bridging the Mo–Sn bond, to give five-coordination around the tin [9]. A similar situation is suggested for the complexes I–IV.

In the ^1H NMR spectrum of $(bipy)P(4\text{-}CH_3C_6H_4)_3(CO)_2Mo(Cl)(SnCl_3)$ a single 2,4-CH_3 resonance is observed. The position of this is different from the parent compound (2.24 ppm) and confirms the higher deshielding of CH_3 protons resulting from the coordination of the $(bipy)P(4\text{-}CH_3C_6H_4)_3(CO)_2Mo$ moiety to the tin atom.

When the reactions are carried out with excess of $SnCl_4$ products in which the phosphine ligand is absent are always observed. Because of partial decomposition it is not possible to establish the formulae of the products.

Reaction of $SnCH_3Cl_3$ with $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ ($X = Cl, F, CH_3, CH_3O$)

The reactions of tricarbonylphosphine complexes $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ with $MeSnCl_3$ in 1 : 1 mol ratio lead to precipitation of the orange microcrystalline

TABLE 2

INFRARED SPECTRAL DATA (ν in cm^{-1})

Complex [a]	ν(CO)	ν(SnCl)
I	1910vs, 1840vs	345s–335sh,310s,275s,260m
II	1920vs, 1845vs	345s–330sh,305s,280s,265m
III	1925vs, 1855vs	335s–325sh,315s,275s,260m
IV	2005s, 1925s, 1880s	345s,310s,270s,260m
V	1900vs, 1818vs	320s,280s
VI	1895vs, 1818vs	315s,280s
VII	1900vs, 1825vs	305s,295s,270m,255m
VIII	2005s, 1910s, 1880s	315s,280s,270m
IX	1900vs, 1820vs	315s,300m,275m
X	2020s, 1930s, 1900s	335s,305s,275m

[a] For the formulae of the complexes I–X see Table 1.

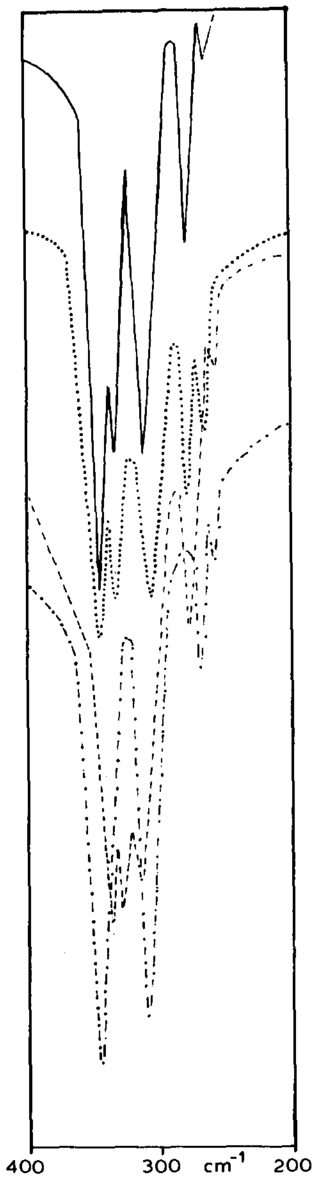

Fig. 1. ν(Sn–Cl) stretching bands in Nujol mulls ——— (bipy)P(4-ClC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCl$_3$), ······ (bipy)P(4-FC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCl$_3$), — — — (bipy)P(4-MeC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCl$_3$), ·—·— (bipy)(CO)$_3$Mo(Cl)(SnCl$_3$).

compounds of the type (bipy)P(4-XC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnCH$_3$Cl$_2$) (X = Cl, F, MeO). The analytical data and some properties of the new compounds are listed in Table 1. When X = Me a yellow cristalline compound is obtained, and this has been shown to be (bipy)(CO)$_3$Mo(Cl)(SnCH$_3$Cl$_2$) [6]. The new compounds are air-stable and have a lower solubility in polar solvents, in which decomposition occurs quite quickly. They are non-electrolytes in DMFA.

The infrared spectra in the solid state show two strong bands in the carbonyl

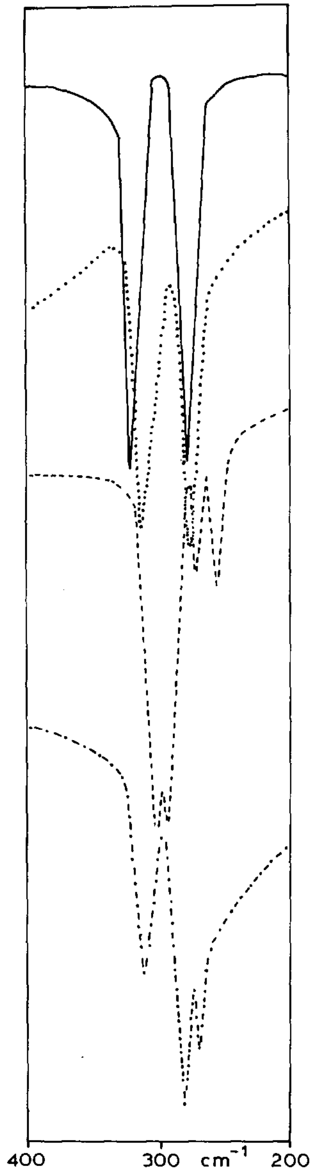

Fig. 2. ν(Sn–Cl) stretching bands in Nujol mulls. ——— (bipy)P(4-ClC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnMeCl$_2$), ······ (bipy)P(4-FC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnMeCl$_2$), — — — (bipy)P(4-MeOC$_6$H$_4$)$_3$-(CO)$_2$Mo(Cl)(SnMeCl$_2$), ·—·—· (bipy)(CO)$_3$Mo(Cl)(SnMeCl$_2$).

stretching region at ca. 1900 and 1820 cm^{-1} (Table 2). These bands are shifted toward lower frequencies with respect to those of the corresponding complexes obtained from SnCl$_4$ (I–III); this is associated with an increase in the electron density at the metal atom and in the d_πM–p_{π^*}(CO) dative bonds because of the CH$_3$ group bonded to the tin.

In the lower frequency region (350–200 cm^{-1}), characteristic of Sn–Cl vibrations, the pattern of the bands is similar for both complexes (bipy)P(4-XC$_6$H$_4$)$_3$-

$(CO)_2Mo(Cl)(SnCH_3Cl_2)$ (X = Cl, F) (Fig. 2), showing bands at ca. 320 and 280 cm^{-1}. However the complex $(bipy)P(4\text{-}CH_3OC_6H_4)_3(CO)_2Mo(Cl)(SnCH_3Cl_2)$ exhibits a higher number of the bands in this region (Fig. 2). The IR spectrum of the tricarbonyl complex $(bipy)(CO)_3Mo(Cl)(SnCH_3Cl_2)$ [6] shows also three bands in this region. As mentioned above, this complex involves a heptacoordinate molybdenum atom and a five-coordinate tin atom resulting from chlorine-bridging of an Mo–Sn bond [9]. In the compounds investigated such coordination for the Mo atom may always be present, but five-coordination around the tin is more probable in the complex with $P(4\text{-}CH_3OC_6H_4)_3$. The ^1H NMR data could be obtained because of the low solubility of the complexes.

Reaction of $SnC_6H_5Cl_3$ with $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ (X = Cl, F, CH_3, CH_3O)

The reactions of $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ with $PhSnCl_3$ in a 1:1 mol ratio also involves oxidative addition of the tin species with loss of the CO or phosphine ligands. In all cases two complexes, one orange and other yellow, are obtained, but these can be separated only for the reaction of $(bipy)P(4\text{-}ClC_6H_4)_3Mo(CO)_3$. On the basis of the analytical data (see Table 1) the complexes can be formulated as

TABLE 3

ELECTRONIC SPECTRAL DATA

Complex [a]	10^4 Concentration (mol l^{-1})	λ (nm)	log ε	Assignment
I	3.58	470	3.16	CT M $\rightarrow L_{bipy}$
		370sh	3.26	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$
		290	3.95	CT M $\rightarrow \pi^*$(CO)
II	1.40	470	2.97	CT M $\rightarrow L_{bipy}$
		360sh	3.03	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$
		275	4.20	CT M $\rightarrow \pi^*$(CO)
III	3.58	470	3.16	CT M $\rightarrow L_{bipy}$
		370sh	3.26	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$
		290	3.95	CT M $\rightarrow \pi^*$(CO)
IV [b]	2.24	520	2.69	CT M $\rightarrow L_{bipy}$
		410	3.05	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$
V	4.66	450	3.13	CT M $\rightarrow L_{bipy}$
		355	3.31	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$
		300	3.87	CT M $\rightarrow \pi^*$(CO)
VI	6.75	445	2.87	CT M $\rightarrow L_{bipy}$
		360	3.03	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$
		300	3.70	CT M $\rightarrow \pi^*$(CO)
VII	3.46	455	3.17	CT M $\rightarrow L_{bipy}$
		355	3.44	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$
		285	4.00	CT M $\rightarrow \pi^*$(CO)
VIII [b]	2.32	520	2.38	CT M $\rightarrow L_{bipy}$
		410	3.13	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$
IX	0.50	450	2.98	CT M $\rightarrow L_{bipy}$
		365	3.24	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$
		285	4.47	CT M $\rightarrow \pi^*$(CO)
X [b]	2.09	520	2.66	CT M $\rightarrow L_{bipy}$
		405	3.04	M $\rightarrow \pi^*$(CO)+ $d \rightarrow d$

[a] See Table 1 for the formulae of the complexes. [b] In acetone solution.

(bipy)P(4-ClC_6H_4)$_3$(CO)$_2$Mo(Cl)(SnPhCl_2) (IX) and (bipy)(CO)$_3$Mo(Cl)(SnPhCl_2) (X).

In most cases attempts at separation involving solutions in polar solvents led to decomposition. The IR data for the isolated products suggest that mixed dicarbonyl- and tricarbonyl-phosphine compounds are obtained; the analytical data show that the products are not pure single species.

The infrared spectral data of complexes IX and X are listed in Table 2.

Electronic spectra

The electronic spectra of the complexes were recorded in DMFA solutions from 200–900 nm, and the absorption maxima (λ_{max}) and intensities (log ε) are listed in Table 3. The absorption bands can be assigned by analogy with those of the corresponding parent compounds [4]. The absorption spectra within each series of compounds are similar, with only small differences depending on the ligand and the tin derivative.

Experimental

All reactions were carried out under nitrogen using standard Schlenk tube techniques and freshly distilled, dried, and degassed solvents.

Analyses

C, H, N analyses were carried out by Elemental Micro-Analyses Ltd., Amberley, Beaworth (Devon), U.K.

Physical measurements

Infrared spectra in the 400–200 cm^{-1} region were recorded on a Perkin–Elmer 325 spectrophotometer, with KBr discs or Nujol mulls.

Conductance measurements were performed in DMFA solution at room temperature with a Philips PW 9506 digital conductivity meter and a cell PR9512/00.

Electronic spectra in the 650–240 nm region were recorded on a Kontron Uvikon 820 spectrophotometer solutions in DMFA being used.

Preparation of the complexes

(Bipy)P(4-XC_6H_4)$_3$(CO)$_2$Mo(Cl)(SnCl_3) (X = Cl, F, CH_3)

A solution of $SnCl_4$ (0.02 ml) in acetone/dichloromethane (15 ml) was added to (bipy)P(4-XC_6H_4)$_3$Mo(CO)$_3$ (0.12 g) (X = Cl, F) and the mixture was stirred under nitrogen for 70 h. The initially violet solution gave an orange precipitate, which was filtered off, washed with acetone and ether, and dried in vacuo (I–II).

When the phosphine ligand present in the parent compound is P(4-$CH_3C_6H_4$)$_3$; the product which separates from the reaction mixture is the yellow tricarbonyl complex (bipy)(CO)$_3$Mo(Cl)(SnCl_3) [6]. Addition of petroleum ether to the filtrate gave III as an orange solid similar to I and II. When X = CH_3O the complex isolated is (bipy)(CO)$_3$Mo(Cl)(SnCl_3) [6].

(Bipy)P(4-XC_6H_4)$_3$(CO)$_2$Mo(Cl)(SnCH_3Cl_2) (X = Cl, F, CH_3O)

A solution of MeSnCl_3 (0.06 g) in acetone (15 ml) was added to (bipy)P(4-

$XC_6H_4)_3Mo(CO)_3$ (X = Cl, F, CH_3O) (0.16 g) and the stirred mixture was refluxed under nitrogen for 12 h. The solution was cooled to room temperature, and an orange precipitate was formed; this was filtered off, washed with warm acetone/ethanol, and dried in vacuo (V–VII). When X = CH_3 the reaction was carried out in CH_2Cl_2; the product which separated in this case was the yellow tricarbonyl complex $(bipy)(CO)_3Mo(Cl)(SnCH_3Cl_2)$ [6].

(Bipy)P(4-ClC$_6$H$_4$)$_3$(CO)$_2$Mo(Cl)(SnC$_6$H$_5$Cl$_2$)

A solution of $PhSnCl_3$ (0.09 g) in dichloromethane (15 ml) was added to $(bipy)P(4\text{-}ClC_6H_4)_3Mo(CO)_3$ (0.22 g) and the stirred mixture was refluxed under nitrogen for 5 h. The initially violet solution gave an orange-red solution, which was evaporated under vacuo to leave an orange oil; this was dissolved in dichloromethane, and addition of petroleum ether then gave crystals. After recrystallization from diethyl ether an orange solid and a yellow solution were obtained. The precipitate was filtered off, washed with acetone, and dried in vacuo (IX). The yellow solution was concentrated under vacuo, and a yellow precipitate obtained by addition of petroleum ether (X).

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