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REACTIONS OF TRICARBONYL PHOSPHINE COMPLEXES OF MOLYBDENUM(0) WITH MERCURY CHLORIDE

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 tricarbonylphosphine complexes (bipy)(P)Mo(CO)$_3$, (bipy) = 2,2'-bipyridine and (P) = P(4-ClC$_6$H$_4$)$_3$, P(4-FC$_6$H$_4$)$_3$, P(4-CH$_3$C$_6$H$_4$)$_3$ and P(4-CH$_3$OC$_6$H$_4$)$_3$, with HgCl$_2$ give compounds of the type (bipy)$_2$Mo$_2$(CO)$_6$ · HgCl$_2$ and (bipy)Mo(CO)$_3$(HgCl)(Cl), depending on the mol ratio of reactants employed. The reaction proceeds with elimination of the phosphine ligand and the coordination of HgCl$_2$ to molybdenum.

A new tricarbonyl complex (bipy)(dppe)$_2$Mo$_2$(CO)$_6$ (dppe = 1,2-ethanediylbis(diphenylphosphine), $\widehat{\text{P–P}}$) with the bidentate phosphine ligand, is prepared from the reaction of the (bipy)Mo(CO)$_4$ complex and dppe. The tricarbonyl-dppe derivative also reacts with HgCl$_2$ in a 1 : 1 mol ratio, to give (bipy)(dppe)$_2$Mo$_2$(CO)$_6$ · 2HgCl$_2$ and (dppe)$_3$Mo$_2$(CO)$_6$ · HgCl$_2$. An excess of mercuric chloride yields the compound (bipy)(dppe)$_2$Mo$_2$(CO)$_6$ · 4HgCl$_2$.

In addition, the (bipy)Mo(CO)$_3$(HgCl)(Cl) complex is isolated from the solution.

Introduction

Some previous papers [1,2] have been concerned with the preparation of tricarbonyl complexes of Group VI metals containing metal–mercury bonds. Among the compounds investigated are the derivatives of the type [($\widehat{\text{NN}}$)Mo(CO)$_3$]$_2$ · Hg(SCN)$_2$ ($\widehat{\text{NN}}$ = 2,2'-bipyridine (bipy), 1,10-phenanthroline) or [(π-C$_5$H$_5$)Mo(CO)$_3$]$_2$Hg, which contain two Mo(CO)$_3$ units for each mercury atom; a linear bond Mo–Hg–Mo has been revealed in the crystal structure of the [(π-C$_5$H$_5$)Mo(CO)$_3$]$_2$ · Hg complex.

Heteronuclear complexes of the type (arene)Mo(CO)$_3$ · nHgCl$_2$ [3], (bipy)Mo(CO)$_3$(HgCl)(Cl) [4] and (bipy)(PPh$_3$)Mo(CO)$_3$ · HgX$_2$ [2] have also been isolated and the crystal structure of the (bipy)Mo(CO)$_3$(HgCl)(Cl) [5] complex shows the presence of a Mo–Hg bond. All the heteronuclear tricarbonyl complexes with a

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Mo–Hg bond show a notable increase in $\nu(C\equiv O)$ with respect to those of the starting carbonyl compounds.

In the carbonyl complexes, the $C\equiv O$ stretching vibration frequencies are extremely sensitive to changes in the formal oxidation state of the metal. The interaction of the acceptor molecule with the central metal atom leads to an increase of the stretching vibration frequencies of the CO group. These complexes can be formulated as metal/basic-metal acceptor compounds.

In this paper we have investigated new tricarbonylmolybdenum derivatives containing phosphine ligands ($P(4\text{-}ClC_6H_4)_3$, $P(4\text{-}FC_6H_4)_3$, $P(4\text{-}CH_3C_6H_4)_3$, $P(4\text{-}CH_3OC_6H_4)_3$ and $(C_6H_5)_2P\text{–}(CH_2)_2\text{–}P(C_6H_5)_2$ which have been prepared from $(bipy)Mo(CO)_4$. The replacement of a CO group by the phosphine ligand increases the metal basicity, and the higher basicity of the metal atom in the complexes would give rise to an increase in the interaction with the acid metal derivative.

We have also investigated the reactions of the new tricarbonylphosphine compounds with $HgCl_2$ as the acceptor metal derivative.

Results and discussion

The $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ complexes

The reactions of $(bipy)Mo(CO)_4$ with $P(4\text{-}XC_6H_4)_3$ ($X = Cl$, F, CH_3, CH_3O) proceed with replacement of CO groups by triarylphosphine ligands. In all cases, products of the type $(bipy)P(4\text{-}XC_6H_4)_3Mo(CO)_3$ have been obtained.

The new complexes and some of their physical properties are listed in Table 1. They are microcrystalline solids with bright colours, which are insoluble in polar organic solvents and non-electrolytes in DMFA solution. Their IR spectra in the solid state show the characteristic bands of the CO and phosphine coordinated ligands. Table 2 gives the most significant IR frequencies and their assignments for the new complexes. It is to be noted that the stretching vibrations of the CO group are modified slightly by the presence of the different phosphine ligands.

The pattern of bands observed in the carbonyl stretching region is similar in all the complexes; showing three bands centered at ca. 1900–1770 cm^{-1}. The *fac*-configuration is expected for these compounds; the same *fac*-configuration is reported for $M(CO)_3(\overset{\frown}{NN})Y$ trisubstituted derivatives in which $\overset{\frown}{NN}$ and Y differ appreciably in their π-accepting abilities, so C_s molecular symmetry is applicable and three infrared-active $C\equiv O$ stretching modes (A, A′, A″) are expected [6].

The UV-VIS spectra of the new tricarbonyl complexes were recorded in DMFA solution in the 200–650 nm region and the absorption maxima (λ_{max}), intensities ($\log \epsilon$) and assignments are listed in Table 3. The bands have been assigned by comparing the spectra with one another and with those of $Mo(CO)_6$ [7] and $(bipy)Mo(CO)_4$ [8].

The ML_{bipy} CT position does not depend on the substituents in the triarylphosphine ligands and only the bands assigned to the LF (ligand field) transition, probably accompanied with $M \rightarrow \pi^*(CO)$ transition, are shifted significantly for the different phosphine ligands.

The $(bipy)(dppe)_2Mo_2(CO)_6$ complex

The formation of tricarbonyl complexes with $\overset{\frown}{P\text{–}P}$ donor ligands has been investigated by Zingales and Canziani [9] using the reaction of $(arene)M(CO)_3$

TABLE 1

ANALYTICAL DATA AND SOME PHYSICAL PROPERTIES OF THE NEW COMPOUNDS

Compound [a]		Colour	Analysis (Found (Calcd.) (%))			M.p. [b]	Conductivity [c]
			C	H	N	(°C)	Ω_M (ohm^{-1} cm^2 mol^{-1})
I	(bipy)P(4-$ClC_6H_4)_3Mo(CO)_3$	violet	53.07 (52.28)	2.85 (2.82)	3.99 (4.13)	212	15.15
II	(bipy)P(4-$FC_6H_4)_3Mo(CO)_3$	violet	57.15 (57.08)	3.15 (3.01)	4.40 (4.29)	203	17.61
III	(bipy)P(4-$CH_3C_6H_4)_3Mo(CO)_3$	violet	62.73 (63.78)	4.48 (4.52)	4.33 (4.37)	201	1.31
IV	(bipy)P(4-$CH_3OC_6H_4)_3Mo(CO)_3$	violet	59.33 (59.69)	4.21 (4.49)	4.03 (3.51)	202	3.69
V	(bipy)(dppe)$_2Mo_2(CO)_6$	violet	61.80 (62.22)	2.20 (2.13)	4.30 (4.26)	204	3.29
VI	(bipy)$_2Mo_2(CO)_6 \cdot HgCl_2$	orange	32.93 (33.09)	1.69 (1.98)	5.49 (5.93)	172	21.64
VII	(bipy)Mo(CO)$_3$(HgCl)(Cl)	red	25.80 (25.70)	1.30 (1.30)	4.40 (4.60)	162	13.9
VIII	(bipy)(dppe)$_2Mo_2(CO)_6 \cdot 2HgCl_2$	yellow-orange	44.59 (44.01)	3.07 (3.01)	1.67 (1.50)	250	– [d]
IX	(dppe)$_3Mo_2(CO)_6 \cdot HgCl_2$	green	55.81 (55.24)	4.01 (3.94)	(0.00) (0.00)	240	– [d]
X	(bipy)(dppe)$_2Mo_2(CO)_6 \cdot 4HgCl_2$	violet	33.77 (34.05)	2.40 (2.33)	1.29 (1.16)	245	– [d]

[a] Every compound is diamagnetic. [b] Decomposition temperature. [c] In DMFA solution. [d] The compounds are unstable in DMFA solution.

TABLE 2

INFRARED SPECTRAL DATA [a,b]

Compound	ν(CO)	ν(HgX)
I (bipy)P(4-ClC$_6$H$_4$)$_3$Mo(CO)$_3$	1915sh, 1900vs, 1800vs, 1780vs	
II (bipy)P(4-FC$_6$H$_4$)$_3$Mo(CO)$_3$	1900vs, 1820vs, 1765vs	
III (bipy)P(4-CH$_3$C$_6$H$_4$)$_3$Mo(CO)$_3$	1895vs, 1790vs, 1770vs	
IV (bipy)P(4-CH$_3$OC$_6$H$_4$)$_3$Mo(CO)$_3$	1895vs, 1791vs, 1770vs	
V (bipy)(dppe)$_2$Mo$_2$(CO)$_6$	1895vs, 1805s, 1770s	
VI (bipy)$_2$Mo$_2$(CO)$_6\cdot$HgCl$_2$	1950vs, 1938vs, 1870sh, 1845sh, 1840vs	260s, 248s
VII (bipy)Mo(CO)$_3$(HgCl)(Cl)	1990vs, 1928s, 1900vs, 1880vs	270s, 240s
VIII (bipy)(dppe)$_2$Mo$_2$(CO)$_6\cdot$2HgCl$_2$	1965vs, 1950vs, 1935vs, 1850vs, 1838vs	290m, 280m
IX (dppe)$_3$Mo$_2$(CO)$_6\cdot$HgCl$_2$	1965vs, 1860vs, 1838vs	258s
X (bipy)(dppe)$_2$Mo$_2$(CO)$_6\cdot$4HgCl$_2$	1965vs, 1860vs, 1838vs	258s

[a] sh, shoulder; vs, very strong; s, strong; m, medium. [b] ν in cm^{-1}.

(M = Cr, Mo, W) complexes with dppe. The compound isolated, (dppe)$_3$Mo$_2$(CO)$_6$, was formulated as a dimer containing one or three dppe bridging groups.

We have now investigated the reaction of the (bipy)Mo(CO)$_4$ compound with

TABLE 3

ELECTRONIC SPECTRAL DATA [a]

Compound	10^5 Concentration (mol l^{-1})	λ (nm)	log ϵ	Assignment
I (bipy)P(4-ClC$_6$H$_4$)$_3$Mo(CO)$_3$	1.83	520	3.57	CT M $\rightarrow$ Lbipy
		345	3.98	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		298sh	4.32	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		292	4.35	CT M $\rightarrow \pi^*$(CO)
II (bipy)P(4-FC$_6$H$_4$)$_3$Mo(CO)$_3$	7.28	513	3.52	CT M $\rightarrow$ Lbipy
		369sh	3.74	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		335sh	3.86	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		292	4.32	CT M $\rightarrow \pi^*$(CO)
III (bipy)P(4-CH$_3$C$_6$H$_4$)$_3$Mo(CO)$_3$	3.54	520	3.57	CT M $\rightarrow$ Lbipy
		370sh	3.35	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		350sh	3.43	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		275	4.29	CT M $\rightarrow \pi^*$(CO)
IV (bipy)P(4-CH$_3$OC$_6$H$_4$)$_3$Mo(CO)$_3$	4.30	520	3.57	CT M $\rightarrow$ Lbipy
		360sh	3.74	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		330sh	3.87	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		290	4.37	CT M $\rightarrow \pi^*$(CO)
V (bipy)(dppe)$_2$Mo$_2$(CO)$_6$	2.39	520	3.70	CT M $\rightarrow$ Lbipy
		350sh	4.00	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		320sh	4.11	$d \rightarrow d + \mathrm{M} \rightarrow \pi^*(\mathrm{CO})$
		301	4.53	CT M $\rightarrow \pi^*$(CO)
VI (bipy)$_2$Mo$_2$(CO)$_6\cdot$HgCl$_2$	20.7	415sh	3.78	$d \rightarrow d + \mathrm{M} \rightarrow$ Lbipy
		280	4.30	CT M $\rightarrow \pi^*$(CO)
VII (bipy)Mo(CO)$_3$(HgCl)(Cl)	26.6	435	3.27	$d \rightarrow d + \mathrm{M} \rightarrow$ Lbipy
		278	4.68	CT M $\rightarrow \pi^*$(CO)

[a] sh, shoulder.

dppe in a dppe/basic metal mol ratio of 1/1. In this case, we have observed that some of the CO and bipy ligands of the parent complex are replaced by the dppe ligand; to give the final product which can be formulated as (bipy)(dppe)$_2$Mo$_2$(CO)$_6$ (see Table 1).

The new product, which precipitates from a solution in acetone of the reacting compounds is a violet solid, which is air stable and insoluble in most solvents. The complex is diamagnetic and a non-electrolyte in DMFA solution (Table 1). The insolubility of the complex does not permit a molecular-weight determination. The IR spectrum shows the bands of the CO, bipy and dppe ligands as expected for the complex (bipy)(dppe)$_2$Mo$_2$(CO)$_6$. In Table 2 are listed the most significant frequencies and their assignments. In the carbonylic stretching region three bands are present whose positions are not very different from those of the tricarbonyl complexes mentioned above.

Reaction of mercuric chloride with (bipy)P(4-XC$_6$H$_4$)$_3$Mo(CO)$_3$(X = Cl, F, CH$_3$, CH$_3$O)

An acetone solution of (bipy)P(4-XC$_6$H$_4$)$_3$Mo(CO)$_3$ (X = Cl, F, CH$_3$, CH$_3$O) reacts with mercuric chloride in a 1 : 1 mol ratio in the same solvent, to give, in all cases, an orange microcrystalline product (bipy)$_2$Mo$_2$(CO)$_6$ · HgCl$_2$ (Table 1). A different product is obtained if two mol of mercuric chloride are added. In this case P(4-XC$_6$H$_4$)$_3$ · HgCl$_2$ (X = Cl, F, CH$_3$, CH$_3$O) is formed and (bipy)Mo(CO)$_3$(HgCl)(Cl) [1] is isolated from the solution.

The tricarbonyl complex (bipy)$_2$Mo$_2$(CO)$_6$ · HgCl$_2$ is air-stable, insoluble in organic solvents, diamagnetic and a non-electrolyte in DMFA solution. A molecular-weight determination is not possible due to the low solubility of the compound. The IR spectrum in the solid state shows three bands in the carbonyl stretching region (Table 2). The lower band is broad, and has two shoulders. These bands appear to be centered at ca. 45, 45 and 75 cm^{-1} above the frequencies obtained for the tricarbonyl starting materials (Fig. 1). This shift is consistent with an increase in the formal oxidation state of the metal.

For the complex [(π-C$_5$H$_5$)Mo(CO)$_3$]$_2$Hg Fischer and Noack [10] interpreted the presence of five bands in terms of a single isomer with a "skew"-conformation of the Mo(CO)$_3$(C$_5$H$_5$) groups about a linear Mo–Hg–Mo system. Our IR data indicate a strong splitting of the bands in the carbonyl stretching region, with respect to those of the parent compounds. This is tentatively accounted for on the assumption that it is a dimer compound with a Mo–Hg–Mo linear bond. The compound shows the typical bipyridine ligand bands in the 1600–400 cm^{-1} region [11,12]. However, no trace of the triarylphosphine ligand could be detected. In the lower frequencies region, the bands about 260 and 248 cm^{-1} were assigned to ν(Hg–Cl). These positions are significantly different from those found in linear HgX$_2$ molecules (ν(Hg–Cl) = 375 cm^{-1} [13]; and even in MeHgX (ν(Hg–Cl) = 315 cm^{-1}) [14] in which a strong covalent Hg–Me bond exists. The low ν(Hg–Cl) values suggest a weak Hg–X bond involving a mercury atom bound to a transition metal [15].

On the basis of the IR spectra, the observed increase in ν(C≡O) and decrease in ν(Hg–X), and the elimination of the triarylphosphine ligand, it is possible to classify the reactions we have studied as "oxidative elimination reactions".

The UV-VIS spectra of the new tricarbonyl complexes were recorded in DMFA solution from 200–650 nm, and the absorption maxima (λ_{max}) and intensities (log ϵ) are listed in Table 3. The absorption bands have been assigned similarly to those of

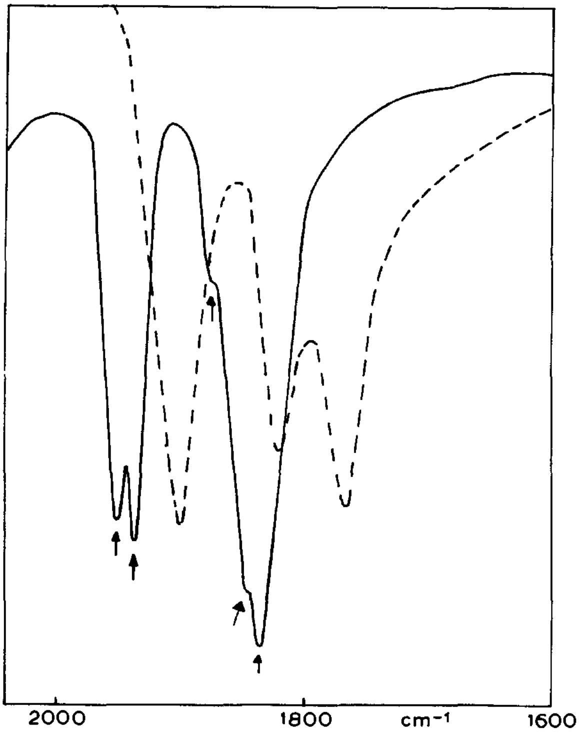

Fig. 1. Carbonyl stretching bands in the solid state ------ (bipy)P(4-$FC_6H_4)_3Mo(CO)_3$, ——— $(bipy)_2Mo_2(CO)_6 \cdot HgCl_2$.

the corresponding parent compounds. The absorption spectrum is similar to that of (bipy)$Mo(CO)_3$(HgCl)(Cl) [1] of crystal structure data which suggest a Mo–Hg bond.

When $HgCl_2$ is added to (bipy)P(4-$XC_6H_4)_3Mo(CO)_3$ (X = Cl, F, CH_3, CH_3O) in a 2 : 1 mol ratio, white precipitates are obtained. The isolated products, P(4-$XC_6H_4)_3 \cdot HgCl_2$, were identificated by analysis and IR spectra.

Reaction of mercuric chloride with the (bipy)(dppe)$_2$Mo$_2$(CO)$_6$ complex

The reaction of the (bipy)(dppe)$_2$Mo$_2$(CO)$_6$ complex with mercuric chloride in a 1 : 1 mol ratio proceeds via an oxidative addition reaction without loss of the ligands present, to give the adduct (bipy)(dppe)$_2$Mo$_2$(CO)$_6 \cdot 2HgCl_2$. The compound precipitates out of acetone solution as a yellow-orange microcrystalline solid. In addition, a new complex has been isolated when the mol ratio of reactants is 1 : 1 and the reaction is carried out in a more dilute solution. This compound is a green solid and the formula (dppe)$_3$Mo$_2$(CO)$_6 \cdot HgCl_2$ is attributed to it. When an excess of mercuric chloride is added to a solution of the reactants the yellow-orange adduct gives a violet precipitate which can be formulated as (bipy)(dppe)$_2$Mo$_2$(CO)$_6 \cdot$ 4HgCl$_2$.

The analytical data (Table 1) for the three compounds are consistent with the proposed formulae. The compounds are insoluble in organic solvents similar to other carbonyl diphosphine complexes of the type $Mo_2(CO)_6[C_2H_4P_2(C_6H_5)_2]_3$ [9]. The complexes are non-conductors in DMFA solution.

The IR spectrum of the yellow orange complex contains five bands in the carbonyl stretching region. These bands are considerably displaced with respect to those of the parent carbonyl complex (bipy)(dppe)$_2$Mo$_2$(CO)$_6$ (Fig. 2), which may suggest a Hg-bridged dimer [10]. In addition, bands at 290 and 280 cm^{-1} are tentatively assigned to the ν(Hg–Cl) vibration of the Hg–Cl group.

In the IR spectrum of the (dppe)$_3$Mo$_2$(CO)$_6$ · HgCl$_2$ green complex it is to be noted that the band at ca. 755 cm^{-1} (γ(C–H)), very characteristic of the bipyridine ligand, is absent. On the other hand three bands are present in the carbonyl stretching region (Fig. 2), these can be associated with the formation of the simple adduct of the tricarbonyl complex described by Zingales and Canziani [9]. Similarly the IR spectrum of the adduct (bipy)(dppe)$_2$Mo$_2$(CO)$_6$ · 4HgCl$_2$ also shows three bands in the carbonyl stretching region.

The solution from the reaction of (bipy)(dppe)$_2$Mo$_2$(CO)$_6$ with HgCl$_2$ in a 1:2 mol ratio gives, on concentration, a red precipitate whose analytical data and IR spectrum are consistent with those expected of the (bipy)Mo(CO)$_3$(HgCl)(Cl) compound [16].

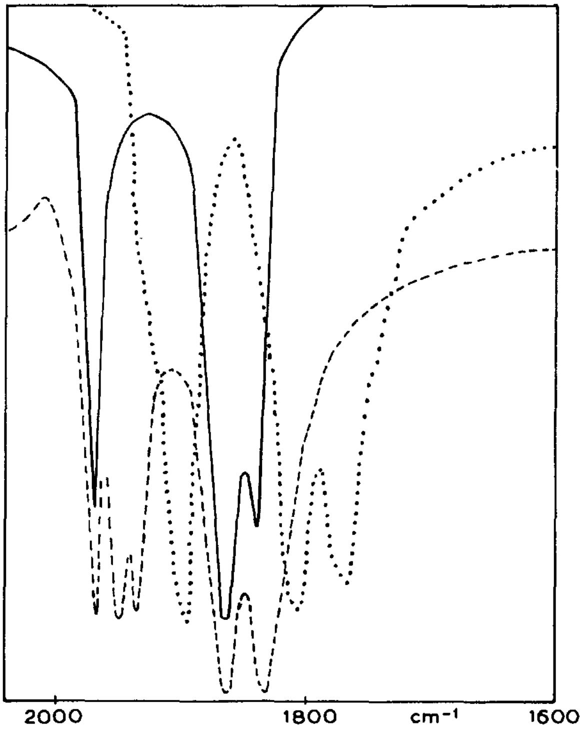

Fig. 2. Carbonyl stretching bands in the solid state ······ (bipy)(dppe)$_2$Mo$_2$(CO)$_6$, - - - - - - (bipy)(dppe)$_2$Mo$_2$(CO)$_6$·2HgCl$_2$, ——— (dppe)$_3$Mo$_2$(CO)$_6$·HgCl$_2$.

Experimental

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

Reagents. Molybdenum hexacarbonyl (Probus) was finely powdered in a blender and kept over P_2O_5 in a desiccator for 12 h before use.

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

Physical measurements. Infrared spectra in the 4000–200 cm^{-1} region were recorded on a Perkin–Elmer 325 spectrophotometer, the samples being prepared as KBr disks or Nujol mulls. Conductance measurements were performed in DMFA solution at room temperature with a Radiometer Copenhagen CDM 2e conductivity bridge and a cell with a constant of a 1 cm^{-1}. Electronic spectra in the 650–240 nm region were scanned in DMFA solution on a Kontron Uvikon 280 spectrophotometer.

Preparation of the complexes

The (bipy)P(4-$XC_6H_4)_3Mo(CO)_3$ (X = Cl, F, CH_3, CH_3O) complexes were prepared according to a literature method [17]. When X = Cl, CH_3 the products precipitate from the reaction medium, while when X = F, CH_3O, the compounds are isolated by partial evaporation of the solution. The products so obtained are dark violet solids.

The (bipy)(dppe)$_2Mo_2(CO)_6$ (V) complex was prepared as described in the literature for the (bipy)(L)Mo(CO_3) compounds [17].

Reaction of mercuric chloride with the (bipy)P(4-$XC_6H_4)_3Mo(CO)_3$ (X = Cl, F, CH_3, CH_3O) and (bipy)(dppe)$_2Mo_2(CO)_6$ complexes

A solution of $HgCl_2$ (0.14 g) in acetone (15 ml) was added to (bipy)P(4-$XC_6H_4)_3Mo(CO)_3$ (0.16 g) and the resultant solution was stirred under nitrogen for 1 h. The initial violet solution gave an orange precipitate which was filtered off, washed with acetone and ether and dried in vacuo to give VI. The same precipitate was obtained with all the triarylphosphine ligands.

For the reaction when the mol ratio is 1 : 2, mercuric chloride (0.33 g) in acetone (15 ml) was added to (bipy)P(4-$XC_6H_4)_3Mo(CO)_3$ (0.27 g) and the mixture was stirred rapidly under nitrogen for 1 h. The initial violet solution gave an orange-red solution which was filtered and concentrated in vacuo to give a bright red crude product, which was filtered off and purified by dissolution in methylene chloride, concentration and cooling, to yield VII [1,16].

For the reaction of the (bipy)(dppe)$_2Mo_2(CO)_6$ complex in a 1 : 1 mol ratio, this compound (0.64 g) was added to mercuric chloride (0.26 g) in acetone (20 ml). The mixture was stirred under nitrogen for 72 h. The yellow-orange precipitate was filtered off, washed with acetone and ether and dried in vacuo to give VIII. The solution was concentrated and a crude bright red compound (VII) was isolated.

When the reaction was carried out in more dilute solution (acetone, 60 ml) a green complex (IX) was isolated.

For a mol ratio in the reaction of 1 : 2, mercuric chloride (0.52 g) in acetone (15 ml) were added to the parent tricarbonyl complex (0.64 g) and the mixture stirred for 72 h. The violet precipitate was filtered off, washed with acetone and ether and dried in vacuo, to leave X.

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