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CARBONYL COMPLEXES OF MOLYBDENUM AND TUNGSTEN WITH SULFUR DONORS

V *. REACTIONS OF CARBONYL COMPLEXES OF MOLYBDENUM(0) WITH UNINEGATIVE (X, Y)-DONOR LIGANDS (X, Y = S, N, O)

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Summary

The reactions of the zerovalent carbonyl complexes $Mo(CO)_6$ and $Mo(CO)_4(bipy)$ with a series of uninegative bidentate (X,Y)-donor ligands (X,Y = xanthates, dithiocarbamates, *o*-aminophenoxide, *o*-aminothiophenoxide, 2-picolinate and thioacetate) lead to new anionic tetracarbonyl complex anions $[Mo^0(X,Y)(CO)_4]^-$. These anions, which can be isolated as their tetraphenylphosphonium salts, contain the (X,Y)-ligand as a bidentate group. In the case of (X,Y) = monothioacetate the decarbonylated species $[PPh_4][Mo^{II}(TA)_3]$ is formed. The reacions of the new complexes with allyl bromide and methyl iodide are described.

Introduction

The reactions of the Group VI metal(0) carbonyl complexes with chelating neutral ligands have been widely studied, but few examples of reactions with anionic chelating ligands have been reported. The methods most frequently used to prepare carbonyl complexes containing anionic potentially bidentate ligands involve displacement of CO or CO and halogen from the starting compounds $M(CO)_6$ and $[M(CO)_5X]^-$ (M = Mo, W), respectively. Products of the type $[M(CO)_5L]^-$ (L = Mexant$^-$, RCOO$^-$) (xant = xanthate) and $[M(CO)_4L]^-$ (L-*o*-aminothiophenoxide, monothiothenoyltrifluoracetone, *o*-methylmercaptophenol, 2-(diphenylphosphino)-

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[*] For Part IV see ref. 10.

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ethanothiol, diketonates, pyrazolyl borates,) have been isolated from these reactions, in which the anionic ligand acts as monodentate or as chelating bidentate group, respectively [1–4].

Complexes of the type [$Mo(CO)_4L$] with (S,S)-donor ligands (S,S = ethylxanthate, *N,N*-diethyldithiocarbamate) have been identified in DMSO solution as products of the reaction of $Mo(CO)_6$ with the corresponding alkali salt [5]. To date all attempts to isolate these species have been unsuccessfull.

All these results previously described prompted us to investigate the reactions of $Mo(CO)_6$, $Mo(CO)_4$(bipy) and sometimes $Mo(CO)_3$(bipy)(py) or $Mo(CO)_5$(py) with a series of anionic (X,Y)-donor ligands (X,Y = S, N, O) containing donor atoms of various sizes and basicities. The ligands used were: xanthates (Rxant; R = Me, Et, Cy (cyclohexyl)), *N,N*-dialkyldithiocarbamates (R_2dtc; R = Et, pyr (1-pyrrolidyl)) and *N*-alkyldithiocarbamates (RHdtc; R = Me) ((S,S)-donor ligands); *o*-aminothiophenoxide (o-ATP) ((S,N)-donor ligand); *o*-aminophenoxide (o-AP) and 2-picolinate (2-pic) ((O,N)-donor ligands) and thioacetate (TA) ((S,O)-donor ligand).

Results and discussion

Reactions of $Mo(CO)_6$, $Mo(CO)_4$(bipy), $Mo(CO)_5$(py) and $Mo(CO)_3$(bipy)(py) with anionic (X,Y)-donor ligands

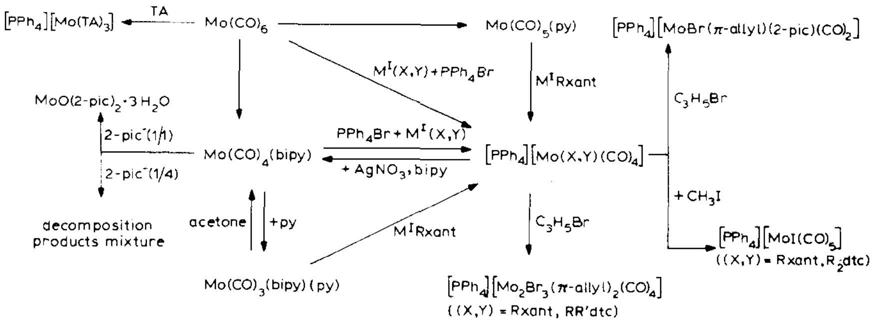
$Mo(CO)_6$ and $Mo(CO)_4$(bipy) were treated with the alkali salts (or the free ligand in a basic medium) in refluxing acetone under an inert atmosphere.

The reactions of $Mo(CO)_6$ with the ligands mentioned above gave the anionic Mo^0 6-coordinated complexes, [PPh_4][Mo(X,Y)$(CO)_4$] ((X,Y) = Rxant, R_2dtc, 2-pic). Sometimes traces of a pentacarbonyl species, [PPh_4][Mo(X,Y)$(CO)_5$], were detected. In the reaction with NaMeHdtc the unstable pentacarbonyl species is formed predominantly, contaminated with tetracarbonyl species. The reaction with monothioacetic acid in a basic medium leads to a complete replacement of CO, with formation of the deep red 6-coordinated Mo^{II} complex [PPh_4][Mo$(TA)_3$]. In all these cases the (X,Y) group acts as a bidentate ligand. The reactions of $Mo(CO)_6$ with *o*-aminophenoxide or *o*-aminothiophenoxide gave mixtures of which were not characterized products.

The reactions of $Mo(CO)_4$(bipy) with xanthates and dialkyldithiocarbamates yield the same products as $Mo(CO)_6$, the 2,2′-bipyridine ligand being displaced. When the ligand is MeHdtc there is no reaction even when an excess of methyldithiocarbamate is used. The reaction with pyridine-2-carboxylate (2-pic) is different, and gives a mixture of products from which a purple oxo complex of composition MoO$(2\text{-pic})_2 \cdot 3H_2O$ was isolated when a molar ratio 1/1 of reactants was used. Use of an excess of ligand (molar ratio 1/4) causes decomposition. *o*-Aminophenoxide and *o*-aminothiophenoxide did not react with $Mo(CO)_4$(bipy) under mild conditions, and under more vigorous conditions only decomposition products were formed. The pyridine complexes $Mo(CO)_5$(py) and $Mo(CO)_3$(bipy)(py) also give products of composition [PPh_4][Mo(Rxant)$(CO)_4$] in reactions with alkali xanthates.

Scheme 1 summarizes the observations described above.

The new anionic complexes isolated behave as 1/1 electrolytes in DMF and are diamagnetic and moderately stable to air and moisture, except in the case of [PPh_4][Mo(MeHdtc)$(CO)_5$] and [PPh_4][Mo(MeHdtc)$(CO)_4$], which decompose



SCHEME 1

gradually even when stored at low temperatures under an inert atmosphere and in the absence of light. All the compounds are soluble in acetone, $CHCl_3$, CH_2Cl_2, DMF, DMSO, and nitromethane.

Table 1 lists the analytical and molar conductance data, and the most characteristic infrared frequencies of the new compounds.

The infrared spectra of all $[Mo(X,Y)(CO)_4]^-$ complexes in the carbonyl stretching region show four bands typical of the *cis*-$[M(CO)_4L_2]$ complexes [6]; the $\nu(CO)$ frequencies for these anionic complexes are generally lower than those of $Mo(CO)_4(bipy)$ (2000, 1912, 1865 and 1810 cm^{-1}), as expected for a higher electron density at molybdenum, which enhances the back-bonding to the carbonyl groups. Sometimes only three bands appear, because of an overlap of the two central bands to produce a broad, very intense band. On the other hand the $\nu(CO)$ frequencies of the dithiocarbamate derivatives are in general lower than those of the analogous xanthate complexes. This may be due to the higher donor ability of a dithiocarbamate ligand with respect to a xanthate group [7].

The ^1H NMR spectra of the new complexes are consistent with the proposed formulae. The ethyl groups of *N,N*-diethyldithiocarbamate ligand give a single resonance, and so both are equivalent on the NMR time scale, because of free rotation around the C–N bond. No other anionic $[Mo(X,Y)(CO)_4]^-$ complexes with xanthate, dithiocarbamate or 2-picolinate ligands have been previously studied, and we cannot make comparisons.

The chemical shift for the methyl group in $[Mo(Mexant)(CO)_4]^-$ (δ 3.93 ppm) is similar to that of $[W(Mexant)(CO)_5]^-$ (δ 3.94 ppm) [4], in which the methylxanthate anion acts as a monodentate ligand. In general the δ values for bidentate xanthates or dithiocarbamates are higher than those of monodentate ones, but the nature of metal can account for the similarity observed. As expected, the δ values of the R groups of Rxant and R_2dtc ligands in these anionic metal(0) complexes are lower than those reported for Mo^{II} neutral complexes [8–10]. Table 2 gives the ^1H NMR spectral data for the compounds isolated.

The electronic spectra of the tetracarbonyl complexes in DMF solution were recorded in the 270–900 nm region. Table 2 summarizes the absorption maxima (λ nm) and molar extinction coefficient values (ϵ) observed for these complexes. The absorption maxima in the 370–460 nm region were assigned tentatively to $d \leftarrow d$ transitions, sometimes accompanied by an intraligand transition (IL). The remaining absorption bands were assigned to $\pi^*(CO) \leftarrow M$ CT or $\pi^*(CO) \leftarrow M$ CT + IL

(continued on p. 372)

TABLE 1

ANALYTICAL, PHYSICAL AND INFRARED DATA FOR THE NEW COMPLEXES

Compound	Colour	Analysis (Found (calc.) (%))				Λ_m (ohm^{-1} cm^2 mol^{-1})	IR data (cm^{-1})	
		C	H	N	S		ν(C≡O)	Other bands
$[PPh_4][Mo(Mexant)(CO)_4]$	yellow	54.01 (55.06)	3.44 (3.51)			110 [a]	2002 1883, 1875sh 1849 1802	360 (ν(Mo–CO)
$[PPh_4][Mo(Exant)(CO)_4]$	yellow	55.44 (55.71)	3.95 (3.74)			95 [a]	1993 1864 1801	370 ν(Mo–CO)
$[PPh_4][Mo(Cyxant)(CO)_4]$	yellow	57.72 (58.20)	4.21 (4.29)		9.01 (8.88)	97 [a] 153 [b]	2002 1879 1848 1801	366 ν(Mo–CO)
$[PPh_4][Mo(Et_2dtc)(CO)_4]$	yellow	57.11 (57.08)	4.29 (4.32)	2.01 (2.02)		115 [a]	1993, 1988 1857 1836 1798	368 ν(Mo–CO)
$[PPh_4][Mo(pyrdtc)(CO)_4]$	yellow	56.66 (57.17)	4.04 (4.04)	2.02 (2.02)		60 [a]	1994 1852br 1786	365 ν(Mo–CO) 353

$[PPh_4][Mo(MeHdtc)(CO)_5]$	yellow					2060 1918 1860	
$[PPh_4][Mo(MeHdtc)(CO)_4]$	yellow					1990 1860 1835 1790	
$[PPh_4][Mo(2\text{-pic})(CO)_4]$	earth-yellow	60.84 (61.02)	3.49 (3.59)	1.98 (2.09)	128 [a]	1993 1870 1842 1798	359 ν(Mo–CO)
$[PPh_4][Mo(TA)_3]$	deep red	54.39 (54.58)	4.17 (4.39)		56 [a]		1468 ν(C=O) 947 899 (ν(C=S)
$MoO(2\text{-pic})_2 \cdot 3H_2O$	purple	35.44 (35.15)	3.61 (3.42)	6.79 (6.83)			1678 1648 ν(C=O)(2-pic)
$[PPh_4][MoBr(\pi\text{-allyl})(2\text{-pic})(CO)_2]$	brown-orange	56.33 (57.26)	4.13 (3.95)	1.85 (1.91)		1922 1827	

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[a] In DMF solution (conc. $\simeq 10^{-3}$). [b] In acetone solution (conc. $\simeq 10^{-3}$).

TABLE 2

ELECTRONIC AND ^{1}H NMR SPECTRA OF THE $[PPh_4][Mo(X,Y)(CO)_4]$ COMPLEXES

(X, Y)	Electronic spectra [a]			^{1}H NMR spectra	
	λ (nm)	ε	Assignment	δ	Assignment
Mexant	376	2183	$d \leftarrow d$ + IL	3.93 [b]	CH_3
	311	7695	$\pi^*(CO) \leftarrow$ M CT + IL	7.87	H_{arom}
Etxant	375	3034	$d \leftarrow d$ + IL	1.28 [c]	CH_3
	312	14321	$\pi^*(CO) \leftarrow$ M CT + IL	4.32	CH_2–O
				7.83	H_{arom}
Cyxant	376	2473	$d \leftarrow d$ + IL	1.57 [b]	CH_2
	314	9250	$\pi^*(CO) \leftarrow$ M CT + IL	5.19	CH–O
				7.80	H_{arom}
Et_2dtc	455	153	$d \leftarrow d$	1.25 [c]	CH_3
	332	2346	$\pi^*(CO) \leftarrow$ M CT + IL	3.73	CH_2–N
	300	8253	$\pi^*(CO) \leftarrow$ M CT + IL	7.70	H_{arom}
pyrdtc	460	158	$d \leftarrow d$	1.86 [b]	CH_2
	328	2933	$\pi^*(CO) \leftarrow$ M CT + IL	3.53	CH_2–N
	292	12609	$\pi^*(CO) \leftarrow$ M CT + IL	7.80	H_{arom}
2-pic	372	374	$d \leftarrow d$		
	298	1020	$\pi^*(CO) \leftarrow$ M CT + IL		

[a] In DMF solution. [b] In $(CD_3)_2SO$ solution. [c] In $CDCl_3$ solution.

transitions [11–12]. The electronic spectrum of the $[PPh_4][Mo(TA)_3]$ complex in DMF solution consists in three maxima: $\lambda_1 = 352$ ($\epsilon_1 = 4800$), $\lambda_2 = 435$ ($\epsilon_2 = 5500$) and $\lambda_3 = 505$ ($\epsilon_3 = 6600$), which can be assigned to $d \leftarrow d$ transitions accompanied by charge transfer (CT) or intraligand transitions.

Reactions of the $[PPh_4][Mo(X,Y)(CO)_4]$ compounds with allyl bromide and methyl iodide (see Scheme 1)

The $[Mo(X,Y)(CO)_4]^-$ derivatives ((X,Y) = xanthates or dithiocarbamates) react under mild conditions with an excess of allyl bromide or methyl iodide to give the previously reported complexes $[Mo_2Br_3(\pi\text{-allyl})_2(CO)_4]^-$ [13] and $[MoI(CO)_5]^-$ [14] respectively, in which the (S,S)-donor ligands have been displaced. The xanthate ligand of the $[Mo(Rxant)(CO)_4]^-$ complexes can be also displaced by addition of a silver salt to an acetone solution of the complexes. After the silver xanthate has been filtered off, $Mo(CO)_4$(bipy) can be isolated from the solution by addition of an excess of 2,2′-bipyridine.

The complex $[PPh_4][Mo(2\text{-pic})(CO)_4]$ behaves differently towards the halides. Thus the reaction with an excess of allyl bromide at room temperature gives $[PPh_4][MoBr(\pi\text{-allyl})(2\text{-pic})(CO)_2]$, and can be regarded as a typical C_3H_5Br oxidative addition with CO elimination. Molybdenum(II) complexes of the same type containing other uninegative bidentate ligands, as the β-diketonate group, have been described previously [15]. The infrared spectrum of this new complex shows two strong bands, approximately of the same intensity, in the ν(CO) stretching frequencies region (Table 1), which are typical of the *cis*-dicarbonyl complexes [16]. Methyl iodide reacts with $[Mo(2\text{-pic})(CO)_4]^-$ to give a mixture of unidentified products, which do not contain CO ligands.

Experimental

All experiments were carried out under oxygen free dry nitrogen. Analytical grade solvents were used.

Reagents

The $Mo(CO)_4(bipy)$, $Mo(CO)_3(bipy)(py)$ and $Mo(CO)_5(py)$ complexes were prepared as previously described [17–20] as were potassium methylxanthate; potassium ethylxanthate, sodium cyclohexylxanthane, sodium *N,N*-diethyldithiocarbamate and sodium *N*-methyldithiocarbamate [21,22].

Molybdenum hexacarbonyl, ammonium 1-pyrrolidyldithiocarbamate, *o*-aminothiophenol, *o*-aminophenol, 2-picolinic and and thioacetic acid were purchased.

Analyses

C, H, N analyses were carried out by Elemental Micro-Analysis Ltd Laboratories, Amberley, Bearworthy (Devon) (England).

Physical measurements

Conductance measurements were performed on DMF solutions at room temperature using a Philips conductivity bridge PW 9506120 and a conductivity cell PW 9510160. Infrared spectra in the 4000–200 cm^{-1} region were recorded on a Perkin–Elmer 325 spectrophotometer, using KBr disks. ^1H NMR spectra were measured at 90 MHz with a Perkin–Elmer R12 spectrometer in deuterodimethylsulphoxide and deuterochloroform solution with TMS as internal standard. Electronic spectra were recorded in DMF solution on a Kontron Uvikon 820 spectrophotometer.

Preparation of complexes

$[PPh_4][Mo(X,Y)(CO)_4]$ complexes ((X,Y) = Mexant, Etxant, Cyxant, Et_2dtc, pyrdtc, MeHdtc). Method A. The alkali salt of the (X,Y) ligand was added to a suspension of $Mo(CO)_6$ in acetone in an 1/1 molar ratio and the stirred mixture was boiled for 15–20 min. After concentration of the solution, diethyl ether was added and the solid formed was filtered off. A suspension of PPh_4Br in 1/1 molar ratio was added to the filtrate with stirring, and the solution was concentrated almost to dryness. The complex was precipitated with light petroleum (b.p. 40–60°C) in the case of xanthates and with diethyl ether in the case of dithiocarbamates.

Method B. A solution of the corresponding alkali xanthate or dithiocarbamate in acetone was added to a suspension of $Mo(CO)_4(bipy)$ in the same solvent in a 1/1 molar ratio, and the mixture was refluxed for 30 min. The solution was then cooled to room temperature and filtered. An equimolar amount of PPh_4Br was added to the filtrate, and the resulting orange-red solution was evaporated to dryness. The solid obtained was redissolved in chloroform and precipitated with light petroleum (b.p. 40–60°C).

Method C. Equimolar amounts of $Mo(CO)_5(py)$ and alkali xanthate were stirred in acetone for a short time and then an equivalent amount of PPh_4Br was added. After filtration and addition of diethyl ether a yellow solid of composition $[PPh_4][Mo(Rxant)(CO)_4]$ appeared, and this was filtered off, washed with diethyl ether and dried under vacuum. The complexes so obtained were identical with those prepared by the previous methods.

Method D. The xanthate complexes $[PPh_4][Mo(Rxant)(CO)_4]$ were also prepared from equimolecular amounts of $Mo(CO)_3(bipy)(py)$ and alkali xanthate, which were stirred in acetone for 15 minutes. After addition of an equivalent amount of PPh_4Br and filtration, diethyl ether was added to the filtrate. A yellow solid appeared, and this was filtered off, washed with diethyl ether, dried under vacuum, and recrystallized from $CH_2Cl_2/EtOH$.

Reaction of $Mo(CO)_6$ *with 2-picolinic acid in basic medium. Formation of* $[PPh_4][Mo(CO)_4(2\text{-}pic)]$

2-Picolinic acid in methanolic KOH was added in an 1/1 molar ratio to a suspension of $Mo(CO)_6$ in acetone. The mixture was refluxed for 15 min then evaporated under reduced pressure. The oil formed was dissolved in acetone. PPh_4Br was added to the solution, the mixture was filtered, and diethyl ether was added to the filtrate until an oil formed. After decantation of the supernatant solution, the oil was dissolved in acetone and diethyl ether was added as precipitant agent. In some cases it is necessary to repeat this process several times. The yellow solid so formed was filtered off, washed with diethyl ether, and dried under vacuum.

Reactions of $Mo(CO)_4(bipy)$ *with 2-picolinic acid in basic medium*

$Mo(CO)_4(bipy)$ and an equimolecular amount of 2-picolinic acid were refluxed in acetone for 3 h in the presence of Et_3N. The purple solid formed was filtered off, washed with CH_2Cl_2, and dried under vacuum. The analytical data correspond to an empirical formula $MoO(2\text{-pic})_2 \cdot 3H_2O$.

Reaction of $Mo(CO)_6$ *with monothioacetic acid in basic medium. Formation of* $[PPh_4][Mo(TA)_3]$

A solution of monothioacetic acid in methanolic KOH was added to a suspension of $Mo(CO)_6$ in acetone and the mixture was boiled for 30 min, then cooled to room temperature. PPh_4Br in equimolecular amount was added and the resulting mixture was evaporated to dryness under reduced pressure. Addition of acetone and then of diethyl ether gave a deep-red solid precipitate which was filtered off, washed with water, and dried under vacuum. The microcrystalline complex contains acetone, which can be displaced by prolonged treatment under vacuum.

Reactivity of the $[PPh_4][Mo(X,Y)(CO)_4]$ *complexes*

Reaction of $[PPh_4][Mo(S,S)(CO)_4]$ *((S,S) = xanthate and dithiocarbamate) with allyl bromide. Formation of* $[PPh_4][Mo_2Br_3(\pi\text{-allyl})_2(CO)_4]$. Allyl bromide (5 ml) was added to 0.075 mmol of the solid $[Mo(S,S)(CO)_4]^-$ salt. The mixture was stirred for 10 min, and then a few ml of acetone and cold diethyl ether was added. The resulting mixture was filtered and the solution evaporated to dryness. Addition of acetone and n-pentane gave an oil, which crystallized upon addition of light petroleum (b.p. 40–60°C). The solid was filtered off, washed with pentane, and dried. The product was characterized by analysis and IR spectroscopy as $[PPh_4][Mo_2Br_3(\pi\text{-allyl})_2(CO)_4]$.

Reaction of $[PPh_4][Mo(2\text{-}pic)(CO)_4]$ *with allyl bromide. Formation of* $[PPh_4][MoBr(\pi\text{-allyl})(2\text{-}pic)(CO)_2]$. The complex was prepared by stirring of $[PPh_4][Mo(2\text{-pic})(CO)_4]$ in an excess of allyl bromide. After 10 min several ml of acetone and diethyl ether were added and the mixture was filtered. Addition of diethyl ether and n-pentane to the filtrate gave an orange solid, which was filtered off, washed with n-pentane, and dried under reduced pressure.

Reaction of [PPh_4][$Mo(S, S)(CO)_4$] complexes ((S, S) = xanthate or dithiocarbamate) with methyl iodide. Formation of [PPh_4][$MoI(CO)_5$]. The reaction was carried out in an excess of methyl iodide. After 10 min stirring several ml of acetone and cold diethyl ether were added. The white solid formed (PPh_4I) was filtered off, and the yellow filtrate was treated with cold n-pentane. The yellow solid which separated was filtered off, washed with n-pentane, and dried under vacuum. The complex was characterized as [PPh_4][$MoI(CO)_5$].

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