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REACTIONS OF SUBSTITUTED CARBONYL COMPLEXES OF MOLYBDENUM(0), $Mo(CO)_4(DAB)$ AND $Mo(CO)_2(PPh_3)_2(DAB)$ WITH Cl_2, Br_2 AND I_2 (DAB = 1,4-DIAZABUTADIENE)

M. IGLESIAS, A. LLORENTE, C. DEL PINO and A. SANTOS
Instituto de Química Inorgánica "Elhuyar", C.S.I.C. Serrano 113-BIS, Madrid-6 (Spain)

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Summary

The reactions of $Mo(CO)_4(DAB)$ compounds (DAB = diacetyldihydrazone (DDH), glyoxalcyclohexylamine (GCy)) with Cl_2, Br_2 and I_2 under mild conditions can be described as oxidative elimination reactions. CO is eliminated, and seven-coordinate molybdenum(II) species containing halogen are generally formed, but frequently ionic or neutral binuclear species through bridging CO or halogen were isolated: $\{[MoX(CO)_2(DDH)_2]_2(\mu\text{-}CO_2)\}X_2$ (X = Cl, Br), $\{[Mo(CO)_2(DDH)_2]_2(\mu\text{-}Br)_2\}Br_2$, $[MoCl(CO)_2(GCy)]_2(\mu\text{-}Cl)_2$. In these compounds DDH acts as monodentate ligand. One binuclear molybdenum(I) complex, $[Mo(CO)_3(DDH)]_2(I_3)_2$, and one molybdenum(III) complex, $[MoBr_2(CO)_3(GCy)]Br$ were also isolated. Only one typical oxidative elimination reaction product, $MoI_2(CO)_3(GCy)$, was isolated. However the reactions of $Mo(CO)_2(PPh_3)_2(DAB)$ compounds (DAB = glyoxaldihydrazone (GDH) and (GCy)) with halogens in the same mild conditions take place with PPh_3 elimination and formation of typical oxidative elimination products $MoX_2(CO)_2(PPh_3)(DAB)$.

Introduction

Two general methods have been used for the preparation of substituted derivatives of Group VI metal carbonyl halides and a number of neutral, cationic and anionic complexes have been synthesized, most of them seven-coordinate metal(II) compounds [1]. The first method involves halogen oxidation of the appropriate substituted metal(0) carbonyl complex while the second one involves the reaction of ligands (N, P, As donors) with the appropriate metal(II) carbonyl halide [2]. In the first method Br_2 or I_2 have usually been used. Reactions with Cl_2 were regarded as impracticable, as formation of higher oxidation state compounds with loss of all the carbonyl groups invariably occurs [2].

Sometimes halocarbonyl complexes of molybdenum(I) or molybdenum(III) have been obtained by halogen oxidation, e.g. $[Mo(CO)_2(diphos)_2]I_3$ [3] and

$[Mo(CO)_2(diars)I_3]$ [4]. Complexes of the last type have been also isolated by reaction with SnX_4 (X = Br, I) [5]. It is noteworthy that very few molybdenum(III) halocarbonyl complexes have been reported. Halogen oxidation of $Mo(CO)_4(LL)$ complexes has been studied for LL = 2,2'-bipyridine ($X_2 = Br_2$, I_2) [5], LL = diphos ($X_2 = Br_2$, I_2) [3] and LL = diars ($X_2 = Br_2$, I_2) [4]. The halogen oxidation of $Mo(CO)_2(LL)_2$ complexes (LL = diphos, diars) has been also reported [3,4,7].

In this work we study the complexes derived from halogen oxidation of $Mo(CO)_4(DAB)$ and $Mo(CO)_2(PPh_3)_2(DAB)$ complexes (DAB = substituted 1,4-diazabutadienes) under mild and controlled conditions, including the reactions with Cl_2.

Results and discussion

Table 1 lists the analytical data and physical properties of the complexes obtained by halogen oxidation of $Mo(CO)_4$-(DDH) (DDH = diacetyldihydrazone), $Mo(CO)_4$-(GCy) (GCy = glyoxalbis(cyclohexyldiimine)), $Mo(CO)_2(PPh_3)_2(GDH)$ (GDH = glyoxaldihydrazone) and $Mo(CO)_2(PPh_3)_2(GCy)$. Table 2 gives the most significant IR frequencies.

Chlorocarbonyl complexes could be obtained from all these starting compounds by bubbling of a slow stream of Cl_2 diluted with N_2 through a suspension or solution of the corresponding complex in CH_2Cl_2.

$Mo(CO)_4(DDH)$ is oxidized by Cl_2 or Br_2 to give yellow compounds of empirical formula $Mo(CO)_3(DDH)_2X_2$ with evolution of CO. The observation of two terminal and two bridging carbonyl bands in the IR spectra and the conductance values suggests the presence of ionic binuclear species $[X(CO)_2(DDH)_2Mo(\mu\text{-}CO)_2Mo(DDH)_2(CO)_2X]X_2$ (X = Cl, Br) containing monodentate DDH ligands (I, II).

A second complex of empirical formula $Mo(CO)_2(DDH)_2Br_2$, containing terminal carbonyl groups only, was isolated from the reaction with Br_2 in a very small molar ratio. The presence of three carbonyl bands in the IR spectrum rends probable a binuclear seven-coordinate species of the type $[(CO)_2(DDH)_2Mo(\mu\text{-}Br)_2Mo(DDH)_2(CO)_2]Br_2$ containing monodentate DDH ligands (III).

The reaction with I_2 occurs also rapidly with vigorous evolution of CO and with formation of a complex of empirical formula $Mo(CO)_3(DDH)I_3$. This compound, which contains terminal CO groups only, behaves as a 1/1 electrolyte in nitromethane and may be formulated as a seven-coordinate species $[Mo^{III}I_2(CO)_3(DDH)]I$. The alternative formula $[Mo^{I}(CO)_3(DDH)]I_3$ is highly improbable. However the low μ_{eff} value seems to suggest the presence of dimeric units with Mo–Mo bonds of the type $[(DDH)(CO)_3Mo\text{–}Mo(CO)_3(DDH)](I_3)_2$. Several molybdenum(I) complexes containing uncoordinated I_3^- ions have been reported [3] (IV).

All halogen oxidations of $Mo(CO)_4(DDH)$ occur quickly with formation of a precipitate. The compounds are insoluble in the ordinary organic solvents, and moderately soluble in nitromethane and DMF.

The Cl_2 oxidation of $Mo(CO)_4(GCy)$ gives a complex of empirical formula $Mo(CO)_2(GCy)Cl_2$, which behaves as non-electrolyte in nitromethane. The presence of three infrared bands at least in the terminal carbonyl stretching region suggests the dimeric formula $[Cl(CO)_2(GCy)Mo(\mu\text{-}Cl)_2Mo(GCy)(CO)_2Cl]$ for this complex, which contains seven-coordinate molybdenum(II) species (V).

The reaction of $Mo(CO)_4(GCy)$ with Br_2 gives a molybdenum(III) complex of composition $Mo(CO)_3(GCy)Br_3$, which can be formulated as a non-electrolyte, seven-coordinate species containing monodentate GCy or as a 1/1 electrolyte of the type $[MoBr_2(CO)_3(GCy)]Br$ with bidentate GCy (VI). The conductance value for this complex in nitromethane is only slightly lower than that for a 1/1 electrolyte. The magnetic moment of this complex is consistent with the presence of one unpaired electron, as expected for a d^3 seven-coordinate complex.

Oxidation with I_2 gives a $MoI_2(CO)_3(GCy)$ complex (VII), similar to the seven-coordinate species commonly formed in the reactions of halocarbonyls with bidentate ligands [2]. The complex is a non-electrolyte in DMF and nitromethane.

The complexes derived from $Mo(CO)_4(GCy)$ are soluble in CH_2Cl_2 and must be precipitated with diethyl ether or petroleum ether.

From the reaction between Cl_2 and $Mo(CO)_2(PPh_3)_2(GDH)$ were isolated either a brown product of empirical formula $Mo(CO)_2(PPh_3)_2(GDH)_{0.5}Cl_2$ (VIII), or a blue compound characterized as $MoCl_2(CO)_2(PPh_3)_2$. There is evidently some tendence to displacement of GDH in the oxidation with Cl_2. However the Br_2 oxidation gives a brown product of composition $MoBr_2(CO)_2(PPh_3)(GDH)$, in which a PPh_3 molecule has been displaced. A similar compound, which contains lattice CH_2Cl_2, was isolated from the reaction with I_2.

Halogen oxidation of $Mo(CO)_2(PPh_3)_2(GCy)$ gives in all cases violet complexes of composition $MoX_2(CO)_2(PPh_3)(GCy)$, similar to those earlier described.

The complexes derived from reactions of $Mo(CO)_2(PPh_3)_2(DAB)$ compounds with Cl_2 or Br_2 are non-electrolytes in nitromethane, but some behaves as 1/1 electrolytes in DMF. However, the product from I_2, which contains lattice CH_2Cl_2, behaves as a 1/1 electrolyte in nitromethane and an ionic formula of the type $[MoI(CO)_2(PPh_3)(GCy)]I \cdot CH_2Cl_2$ may be proposed for this compound. The magnetic moment of $[MoI(CO)_2(PPh_3)(GCy)]I \cdot CH_2Cl_2$ (2.4 BM) is rather high compared with μ_{eff} values usually found for molybdenum(II) species (1.4–2.0 BM) [8].

The IR spectra of the halogen oxidation products derived from $Mo(CO)_2(PPh_3)_2(DAB)$ compounds show two bands in the $\nu(CO)$ stretching region, which are significantly, displaced towards the higher frequencies as a consequence of metal oxidation. The IR spectra of the products from Cl_2 show in the low-frequency region two bands at about 290–330 cm^{-1} of medium or strong intensity, which have been assigned to terminal $\nu(Mo–Cl)$ stretching frequencies. Bands corresponding to $\nu(Mo–N)$ stretching frequencies have been tentatively assigned in the range 213–248 cm^{-1}. Bands assignable to $\nu(Mo–Br)$ stretching frequencies could be not clearly observed owing to the presence of $\nu(Mo–N)$ bands.

We conclude that the halogen oxidation (Cl_2, Br_2, I_2) under mild and controlled conditions of $Mo(CO)_4(DAB)$ and $Mo(CO)_2(PPh_3)_2(DAB)$ complexes leads to Mo^{II}, Mo^{I} or Mo^{III} complexes, most of them seven-coordinate, and that binuclear species are frequently formed. In general the products from $Mo(CO)_4(DAB)$ complexes differ considerably from those obtained by reaction of halocarbonyl complexes $MoX_2(CO)_4$ with N–N, P–P or As–As bidentate ligands [2].

Experimental

All experiments were performed under dry nitrogen or in vacuo. Solvents and liquid reagents were purified and dried by standard methods. $Mo(CO)_4(DAB)$ and

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TABLE 1

PHYSICAL AND ANALYTICAL DATA OF THE COMPOUNDS OBTAINED FROM THE REACTION OF $Mo(CO)_4(DAB)$ AND $Mo(CO)_2(PPh_3)_2(DAB)$ WITH Cl_2, Br_2, I_2

Compound	Colour	M.p. (°C) (dec.)	Λ_M (ohm^{-1} cm^2 mol^{-1})		Analysis (Found (calcd.)(%))				Magnetic data μ_{eff}(BM)
			(DMF)	(NO_2CH_3)	C	H	N	X	
$\{[MoCl(CO)_2(DDH)_2]_2(\mu\text{-}CO)_2\}Cl_2$ (I)	yellow	220	125	100	28.1 (27.6)	4.8 (4.2)	23.8 (23.4)	14.1 (14.8)	
$\{[MoBr(CO)_2(DDH)_2]_2(\mu\text{-}CO)_2\}Br_2$ (II)	dull orange	130	227	98	23.0 (23.2)	4.3 (3.5)	20.2 (19.7)	28.0 (28.2)	
$\{[Mo(CO)_2(DDH)_2]_2(\mu\text{-}Br)_2\}Br_2$ (III)	light orange				22.2 (22.2)	3.8 (3.7)	20.3 (20.7)		
$[(DDH)(CO)_3Mo\text{–}Mo(CO)_3(DDH)](I_3)_2$ (IV)	light violet	150	94	92	12.4 (12.4)	1.3 (1.4)	8.0 (8.2)	55.7 (56.4)	0.6
$[MoCl(CO)_2(CGy)]_2(\mu\text{-}Cl)_2$ (V)	violet	140	69	60	42.3 (43.0)	5.3 (5.4)	5.6 (6.3)	16.8 (16.0)	
$[MoBr_2(CO)_3(GCy)]Br$ (VI)	brown violet	120	104	70	32.9 (31.9)	4.4 (3.9)	4.6 (4.4)	36.6 (37.4)	1.8
$MoI_2(CO)_3(GCy)$ (VII)	brown violet	160	6	10	30.6 (31.2)	4.1 (3.7)	4.3 (4.3)	38.9 (38.8)	0.7
$Mo(CO)_2(PPh_3)_2(GDH)_{0.5}Cl_2$ (VIII)	brown		46						
$MoBr_2(CO)_2(PPh_3)(GDH)$ (IX)	brown	132 135	125	24	40.2 (40.0)	3.6 (3.2)	7.6 (8.4)	23.6 (24.2)	
$MoI_2(CO)_2(PPh_3)(GDH)\cdot CH_2Cl_2$ (X)	brown	95	96	63	31.9 (32.9)	3.7 (2.7)	7.6 (6.7)	29.0 (30.2)	0.9
$MoCl_2(CO)_2(PPh_3)(GCy)$ (XI)	violet	86 90	56	31	56.4 (57.8)	5.8 (5.5)	3.6 (3.9)	10.8 (10.1)	
$MoBr_2(CO)_2(PPh_3)(GCy)$ (XII)	violet	85 90	113	31	50.8 (51.4)	5.3 (4.9)	3.8 (3.5)	21 20.2	1.8
$[MoI(CO)_2(PPh_3)(GCy)]I\cdot CH_2Cl_2$ (XIII)	violet	95		85	40.7 (41.9)	4.2 (4.2)	2.9 (2.9)	27.6 (26.1)	2.4

TABLE 2

THE MOST RELEVANT IR FREQUENCIES (cm^{-1}) OF THE COMPOUNDS $Mo(CO)_4(DAB)$, $Mo(CO)_2(PPh_3)_2(DAB)$ AND THOSE OF THEIR REACTION PRODUCTS WITH Cl_2, Br_2 and I_2

Compound	ν(CO)	δ(M–CO)	ν(M–C)	ν(M–Cl)	ν(M–N)
$Mo(CO)_4(DDH)$	2020s, 1910s, 1870s, 1835s	640m, 600m, 585m, 545m	500w, 470m, 460m, 370s		238m, 220w
$\{[MoCl(CO)_2(DDH)_2]_2(\mu\text{-}CO)_2\}Cl_2$ (I)	1980s, 1900s, 1780s, 1720s	630m, 595m, 575m, 540m	510w, 480m, 460m, 410m	328s, 290m	248m, 230s
$\{[MoBr(CO)_2(DDH)_2]_2(\mu\text{-}CO)_2\}Br_2$ (II)	1980s, 1910s, 1780s, 1220s	630m, 605m, 570m, 540m	510w, 480m, 460m, 410w		248sh, 230sh
$\{[Mo(CO)_2(DDH)_2]_2(\mu\text{-}Br)_2\}Br_2$ (III)	1930s, 1920sh, 1910s	610m, 580m, 540m	510w, 480m, 460m, 410w		248w, 228m
$[(DDH)(CO)_3Mo\text{–}Mo(CO)_3(DDH)](I_3)_2$ (IV)	2020s, 1975s, 1935s	610m, 580m, 550m	520w, 500m, 470m		240w, 220w
$Mo(CO)_4(GCy)$	2010s, 1940s, 1880s, 1810s	640m, 600m, 570m, 530m	495w, 480m, 459m, 370s		230m, 214sh
$[MoCl(CO)_2(GCy)]_2(\mu\text{-}Cl)_2$ (V)	2020w, 1980s, (1920-1910)s	590m, 555m		330s, 308m, 230w	227w, 213sh
$[MoBr_2(CO)_3(GCy)]Br$ (VI)	2040s, 1980s, 1915s	580m, 560m, 530m		220s	212sh
$MoI_2(CO)_3(GCy)$ (VII)	2020s, 1980s, 1930s	580m, 540m, 510m			
$Mo(CO)_2(PPh_3)_2(GDH)$	1850s, 1750s				238s, 218m
$MoCl_2(CO)_2(PPh_3)_2(GDH)_{0\,5}$ (VIII)	2015s, 1950s, 1900s, 1885s	535m, 510s, 500sh, 490sh	480sh, 445w	320s, 298m	228m
$MoBr_2(CO)_2(PPh_3)(GDH)$ (IX)	1960s, 1890s	530m, 505s	450w		250w, 230w
$MoI_2(CO)_2(PPh_3)(GDH)\cdot CH_2Cl_2$ (X)	1960s, 1885s	540m, 525m	450w, 420w		
$Mo(CO)_2(PPh_3)_2(GCy)$	1850s, 1770s				235sh, 228m
$MoCl_2(CO)_2(PPh_3)(GCy)$ (XI)	1945s, 1925sh, 1860s, 1840sh	535m, 505m		310m-s, 280m	
$MoBr_2(CO)_2(PPh_3)(GCy)$ (XII)	2020m, 1950s, 1890sh, 1865m	535s, 515m			250w, 230w
$[MoI(CO)_2(PPh_2)(GCy)]I\cdot CH_2Cl_2$ (XIII)	1970sh, 1950sh, 1920m, 1875m				250w, 220w

$Mo(CO)_2(PPh_3)_2(DAB)$ were prepared by published methods [9,10]. Elemental analysis for C, H, and N were performed by Elemental Microanalysis Ltd., Devon, England. Infrared spectra in the 4000–200 cm^{-1} region were recorded as KBr discs or Nujol mulls between CsI plates on a Perkin–Elmer 325 spectrometer. Magnetic susceptibilities were determined by the Gouy method, using a 4411 Sartorius electronic microbalance, a B-M-4 Bruker magnet and a VN K 3300 Leybold Heraeus Cryostat. Molar conductivity measurements were made on a Philips 6M 4144/01 conductivity bridge using a PR 9512/00 measurement cell.

Reaction of $Mo(CO)_4(DDH)$ *with* Cl_2

A slow stream of dry chlorine diluted with nitrogen was bubbled into a stirred suspension of $Mo(CO)_4(DDH)$ (0.2 g, 0.6 mmol) in CH_2Cl_2 (20 ml). After a few minutes this orange compound in suspension yielded an abundant yellow precipitate (I), which was filtered off and washed several times with diethyl ether and dried in vacuo. The evolution of CO in the reaction was not noticeable because of the Cl_2/N_2 flow.

Reaction of $Mo(CO)_4(DDH)$ *with* Br_2

A solution of Br_2 in CH_2Cl_2 (0.96 g, 0.6 mmol) was added dropwise with stirring to $Mo(CO)_4(DDH)$ (0.2 g, 0.6 mmol) suspended in the same solvent. Reaction started immediately with vigorous evolution of CO; the colour changed from orange to light yellow and a bulky orange precipitate (II) appeared. The precipitate was filtered off, washed with diethyl ether and dried in vacuo. From the filtrate we were able to isolate in a very low yield, an orange-yellow precipitate which had a composition corresponding to compound III.

Reaction of $Mo(CO)_4(DDH)$ *with* I_2

Solid iodine (0.3 g, 1.2 mmol) was added with stirring to $Mo(CO)_4(DDH)$ (0.2 g, 0.6 mmol) suspended in CH_2Cl_2 (20 ml); a vigorous evolution of CO started and the solution turned red after 5 min. The violet precipitate IV was filtered off, washed several times with diethyl ether, and dried in vacuo.

Reaction of $Mo(CO)_4(GCY)$ *with* Cl_2

A slow stream of dry chlorine diluted with nitrogen was bubbled into a stirred solution of $Mo(CO)_4(GCy)$ (0.2 g, 0.5 mmol) in CH_2Cl_2 (20 ml). After 10 minutes the colour changed to red, and 5 minutes later the Cl_2 flow was interrupted. The solution was concentrated and V was precipitated with diethyl ether or petroleum ether, filtered off, washed and dried in vacuo. It is light violet.

The evolution of CO is not noticeable because of the gas stream.

Reaction of $Mo(CO)_4(GCy)$ *with* Br_2

A solution of Br_2 in CH_2Cl_2 (0.5 mmol) was added dropwise to a stirred solution of $Mo(CO)_4(GCy)$ (0.2 g, 0.5 mmol). Evolution of CO and a change of colour from violet to brown were observed. After 10 minutes the solution was concentrated and VI was precipitated with diethyl ether or petroleum ether. The brown-violet solid was washed and dried in vacuo.

Reaction of $Mo(CO)_4(GCy)$ *with* I_2

Solid iodine (0.5 mmol) was added to a stirred solution of $Mo(CO)_4(GCy)$ (0.2 g, 0.5 mmol) in CH_2Cl_2 (20 ml). Evolution of CO and change of colour were observed. After 10 minutes the solution was concentrated and VII was precipitated with ethyl ether or petroleum ether, washed with diethyl ether, and dried. It is brown violet in colour.

Reaction of $Mo(CO)_2(PPh_3)_2(GDH)$ *with* Cl_2

A stream of dry chlorine diluted with nitrogen was bubbled into a solution of $Mo(CO)_2(PPh_3)_2(GDH)$ (0.3 g, 0.4 mmol) in CH_2Cl_2 (20 ml); the colour quickly changed from violet to brown. The gas flow was interrupted and the suspended solid was filtered off. The filtrate was concentrated and light petroleum ether was added. The resultant brown solid (VIII) was filtered off, washed with petroleum ether, and dried in vacuo.

Reaction of $Mo(CO)_2(PPh_3)_2(GDH)$ *with* Br_2

A solution of Br_2 (0.02 ml, 0.4 mmol) in CH_2Cl_2 (20 ml) was added dropwise to a stirred solution of $Mo(CO)_2(PPh_3)_2(GDH)$ (0.3 g, 0.4 mmol). The colour of the solution changed from violet to brown. The solvent was removed under reduced pressure to leave a brown solid (IX) which was dissolved in CH_2Cl_2, precipitated with petroleum ether and washed with the same solvent then dried in vacuo.

Reaction of $Mo(CO)_2(PPh_3)_2(GDH)$ *with* I_2

Solid iodine (0.13 g, 0.5 mmol) was added to a stirred solution of $Mo(CO)_2(PPh_3)_2(GDH)$ (0.2 g, 0.26 mmol) in CH_2Cl_2 (20 ml). After 1 h stirring at room temperature the precipitate (X) was filtered off, washed with petroleum ether, and dried in vacuo.

Reaction of $Mo(CO)_2(PPh_3)_2(GCy)$ *with* Cl_2

A stream of dry chlorine diluted with nitrogen was bubbled with stirring into $Mo(CO)_2(PPh_3)_2(GCy)$ (0.3 g, 0.33 mmol) in suspension in CH_2Cl_2 (20 ml). The solution turned from blue to violet, and the chlorine flow was interrupted and the solution filtered. The filtrate was concentrated and petroleum ether added. The mauve precipitate (XI) was filtered off, washed with more petroleum ether and dried in vacuo.

Reaction of $Mo(CO)_2(PPh_3)_2(GCy)$ *with* Br_2

A solution of Br_2 (approximately 0.33 mmol) in CH_2Cl_2 (10 ml) was added dropwise to a stirred solution of $Mo(CO)_2(PPh_3)_2(GCy)$ (0.3 g, 0.33 mmol). The colour changed immediately from blue to brown and the solvent was pumped off. The purple solid (XII) was dissolved in CH_2Cl_2 and precipitated with petroleum ether, filtered off, washed with petroleum ether and dried in vacuo.

Reaction of $Mo(CO)_2(PPh_3)_2(GCy)$ *with* I_2

Solid iodine (0.35 g, 0.4 mmol) was added to a stirred solution of $Mo(CO)_2(PPh_3)_2(GCy)$ (0.35 g, 0.4 mmol) in CH_2Cl_2 (30 ml). After 3 h the solution was filtered and the solvent was evaporated at low pressure. The violet solid resulting (XIII) was dissolved in CH_2Cl_2, precipitated with hexane, filtered off, washed with hexane, and dried in vacuo.

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