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Triphenylarsine derivatives of cyclopentadienyl-molybdenum carbonyls

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Abstract

$C_5H_5Mo(CO)_2AsPh_3Cl$ (I) reacts with benzonitrile in the presence of $AlCl_3$ to form $[C_5H_5Mo(CO)_2(AsPh_3)PhCN]^+ AlCl_4^-$, which has been isolated as the PF_6^- salt II. Reaction of this ionic complex with n-butyllithium did not yield a methyleneamine complex by nucleophilic attack at the coordinated nitrile, but gave a mixture of $[C_5H_5Mo(CO)_2(AsPH_3)]_2$ (III) and $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$ (IV). When dichloromethane was used to extract the crude mixture, $C_5H_5Mo(CO)_2(AsPh_3)Cl$ was an additional product. The outcome of the reaction is rationalised in terms of initial formation of III, which was shown separately to be formed with $C_5H_5Mo(CO)_3Cl$ from IV. When a solution of III was heated or III was reduced with sodium amalgam or lithium methoxide IV was formed together with carbonyl-free decomposition products. IV was formed to react with dichloromethane to form I and $C_5H_5Mo(CO)_3Cl$.

Introduction

Various methods have been used to synthesise methyleneamine (RR′C=N) complexes of the transition metals, and they invariably involve reactions of the preformed ligand [1]. An alternative route would involve nucleophilic attack at a coordinated nitrile molecule, according to:

$$L_nM(N\equiv CR) + X^- \rightarrow L_nM\{N\equiv C(X)R\}^-$$

Intermolecular reactions involving transfer of an alkyl group to an adjacent coordinated nitrile are well documented for main-group compounds [2], but for the transition metals, nucleophilic attack at nitriles, although reported, is not common. Ammonia, for example, reacts with *cis*-$Pt(CH_3CN)_2Cl_2$ to give the amidine complex *trans*-$[Pt(NH_3)_2\{NHC(CH_3)NH_2\}_2]Cl_2$ [3], and *cis*-$[Co(NCCH_2NH_2)en_2Cl]^{2+}$ undergoes an intermolecular nucleophilic attack to produce the ligand isomer $[Co(en)\{NH_2CH_2C(NH_2)=NCH_2CH_2NH_2\}Cl]^{2+}$ [4]. *N*-Lithiobenzamidine {Li-

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NHC(Ph)NH} reacts with $Pt(PhCN)_2Cl_2$ to form a diazaacetylacetato complex $Pt\{HNC(Ph)NC(Ph)NH\}_2$ [5]. Nucleophilic attack is generally enhanced by cationic character in the metal complex containing the electrophilic ligand. For example, amines and alcohols attack at a coordinated carbonyl group of $[C_5H_5Mo(CO)_4]^+$ to form carbamoyl (CONRR′) and carboalkoxy (COOR) [6] groups, respectively, and amines react with $[C_5H_5Mo(CO)_3(C_2H_4)]^+$ to form $[C_5H_5Mo(CO)_3CH_2CH_2NHRR']^+$ and $[\{C_5H_5Mo)CO)_3\}_2NRR']^+$ [7]. Our attempts to treat coordinated nitriles were consequently centred on the related cationic complexes $[C_5H_5Mo(CO)_3(PhCN)]^+$ and $[C_5H_5Mo(CO)_2L(PhCN)]^+$, where we chose L = $AsPh_3$. Nucleophilic attack at the coordinated nitrile to form methyleneamino complexes was not achieved, but new triphenylarsine complexes were isolated.

Results and discussion

The reactions of cyclopentadienyl carbonyl halides e.g. $C_5H_5M(CO)_3Cl$ (M=Mo, W), with halogen acceptors, such as $AlCl_3$, in the presence of a donor molecule, are known to give ionic carbonyl species of the type $[C_5H_5M(CO)_4]^+$, $[C_5H_5M(CO)_2$-$L_2]^+$ $[AlCl_4]^-$ and $[C_5H_5M(CO)_2$-$L]^+$ $[AlCl_4]^-$ (M = Mo, W) [8]. We attempted to synthesise related complexes with L = nitrile (CH_3CN or C_6H_5CN) but had only limited success. The dicarbonyl complex was unstable towards decomposition and attempts to synthesise the second complex also failed, as they did for Barnett et al. [9]. By substituting another two-electron donor, i.e. a phosphine or arsine, for a carbonyl group in the starting complex, it was thought that the stability towards decomposition would be improved, and the lability of the molybdenum–chloride bond enhanced. Triphenylarsine was chosen for this substitution because the arsine forms only a mono-substituted derivative, *cis*-$C_5H_5Mo(CO)_2(AsPh_3)Cl$, whereas triphenylphosphine forms a mixture of mono- and di-substituted products, $C_5H_5Mo(CO)_2(PPh_3)Cl$ and $C_5H_5Mo(CO)_3(PPh_3)_2Cl$ [10]. The latter complexes are formed under both thermal and photochemical conditions. The orange $C_5H_5Mo(CO)_2(AsPh_3)Cl$, produced here by the reaction of $C_5H_5Mo(CO)_3Cl$ with an excess of triphenylarsine in toluene at 50 ° C for 8 h is identical to that described by Barnett [9], but in our work it was produced in half the reaction time and in a much greater yield (93%. vs. 32%).

The complex $[C_5H_5Mo(CO)_2(AsPh_3)(PhCN)]^+$ $[AlCl_4]^-$ was smoothly formed in dichloromethane at ambient temperature when $C_5H_5Mo(CO)_2(AsPh_3)Cl$ was stirred with a two molar excess of each of $AlCl_3$ and benzonitrile. The cation was precipitated as bright yellow crystals of $[C_5H_5Mo(CO)_2(AsPh_3)(C_6H_5CN)]^+$ $[PF_6]^-$ by the addition of NH_4PF_6. The complex is relatively stable to air in the solid state, but quickly decomposes in solution when exposed to air. In solution the infrared spectrum shows two strong absorptions in the carbonyl stretching region, typical of a dicarbonyl complex. However, in the solid state two additional absorptions appear, which may arise either because of solid state effects, or because of the presence of two isomers in the solid. In this context, the two isomeric forms possible for this type of complex, have been separated and studied by several workers [11,12], separation being achieved by fractional crystallisation. Here, a common species appears to be produced in solution, so if isomeric forms do exist in the solid, interchange between these forms must be a low energy process in solution.

Table 1

Spectroscopic data for $(C_5H_5)_2Mo_2(CO)_5L$ and $(C_5H_5)_2Mo_2(CO)_4L_2$ (L=PPh_3, $AsPh_3$)

Complex	Colour	ν(CO) [a] (cm^{-1})					^1H NMR [b] (δ)
$Cp_2Mo_2(CO)_5(AsPh_3)$	red	1967vs	1902vs	1888vs	1873vs	1818s	4.83(5) 5.10(5) 7.46 (15)
$Cp_2Mo_2(CO)_5(PPh_3)$ [12]	red	1968vs	1905vs	1886vs	1873vs	1816s	4.73(5) 5.11(5) 7.44 (15)
$Cp_2Mo_2(CO)_4(AsPh_3)_2$	crimson		1843s	1828vs			[c]
$Cp_2Mo_2(CO)_4(PPh_3)_2$ [12]	red		1853sh	1830s			[c]

[a] Nujol mull. [b] $CDCl_3$ solution. [c] Insufficiently soluble.

The cationic nitrile complex was treated with n-butyllithium in an attempt to generate the methyleneamino group (N=CRR′) at the metal centre. Such reactions occur between alkyllithiums and nitriles in the absence of transition-metal complexes, and provide convenient methods of preparation of lithiomethyleneamine reagents [13]. Although the nitrile was part of a cationic complex, and the lithio nucleophile is regarded as a strong nucleophile, the reaction did not produce any of the expected complexes. Instead, two new triphenylarsine complexes were isolated, and identified as $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$ and $[C_5H_5Mo(CO)_2(AsPh_3)]_2$. Both complexes have known triphenylphosphine analogues [12] and show a close similarity with them. This is illustrated in Table 1, in which the carbonyl absorptions for the two series of complexes are listed.

In the mass spectrometer, neither of the arsine complexes gave molecular ions, both appearing to lose triphenylarsine very easily. The ions $[(C_5H_5)_2Mo_2(CO)_5]^+$ and $[(C_5H_5)_2Mo_2(CO)_4]^+$, however, were clearly visible for the pentacarbonyl and tetracarbonyl complexes respectively, together with the daughter ions arising from stepwise loss of the carbonyls. For both complexes, the most intense peaks in the spectrum correspond to $AsPh_3{}^+$, $AsPh_2{}^+$ and $AsPh^+$.

When dichloromethane was used to extract the product from the crude mixture, an additional product, $C_5H_5Mo(CO)_2(AsPh_3)Cl$, was obtained in significant yield (17%). Further studies showed that $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$ could be formed from $[(C_5H_5Mo(CO)_2(AsPh_3)]_2$ by gentle heating in toluene, carbonyl-free decomposition products also being formed. It was also found that $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$ reacts with CH_2Cl_2 at the reflux temperature to give $C_5H_5Mo(CO)_3Cl$ and $C_5H_5Mo(CO)_2(AsPh_3)Cl$. Thus, all the products from the reaction of butyllithium with $[C_5H_5Mo(CO)_2(AsPh_3)(PhCN)]^+$ can be accounted for in terms of the initial formation of $[C_5H_5Mo(CO)_2(AsPh_3)]_2$ through reduction and loss of the nitrile.

$$[C_5H_5Mo(CO)_2(AsPh_3)(PhCN)]^+ + BuLi \rightarrow Li^+ + [C_5H_5Mo(CO)_2(AsPh_3)]_2 + Bu_2 + PhCN$$

Alternatively the reduction could proceed by nucleophilic attack by Bu^- at the metal centre, accompanied by displacement of the nitrile, and free nitrile was indeed present in the reaction solution. β-Elimination of butene from a butylmetal complex would lead to formation of the metal hydride $C_5H_5Mo(CO)_2(AsPh_3)H$, which would decompose with loss of hydrogen to form the dimer $[(C_5H_5Mo(CO)_2(AsPh_3)]_2$. The intermediate formation of a hydride provides an alternative explanation for the appearance of chloride products when dichloromethane is used as the solvent. Metal hydrides are well known to react with halo solvents to form halide derivatives e.g. $C_5H_5Mo(CO)_3H$ with CCl_4 [13].

In an attempt to establish whether reduction is primarily responsible for the formation of the dinuclear arsine complexes, $[C_5H_5Mo(CO)_2(AsPh_3)(PhCN)]^+$ PF_6^- was treated with sodium amalgam. Benzonitrile was eliminated, and the sole product was $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$. It is likely that this complex originated from the dimer, $[C_5H_5Mo(CO)_2(AsPh_3)]_2$ through disproportionation, some of the product decomposing to non-carbonyl material, though no direct evidence for formation of this dimer was obtained. The use of lithium methoxide in the place of butyllithium and sodium amalgam also produced the same dinuclear complex, again with no evidence for the prior formation of $[C_5H_5Mo(CO)_2(AsPh_3)]_2$.

Experimental

$C_5H_5Mo(CO)_3Cl$ was prepared by a standard method [14]. Triphenylarsine, acetonitrile, benzonitrile, ammonium hexafluorophosphate, aluminium chloride, lithium, sodium, and organic solvents were supplied by B.D.H. Chemicals Ltd., and n-butyllithium by Alfa Products. The nitriles, and methanol were dried over molecular sieves and purified by distillation. Hydrocarbons and diethyl ether were dried over extruded sodium, and chloroform, dichloromethane and acetone over molecular sieves. Tetrahydrofuran was dried by refluxing for 48 h over potassium, then distilled under nitrogen and stored over freshly extruded sodium wire.

IR spectra were recorded on a Perkin–Elmer 456 spectrometer, and proton NMR spectra at 60 MHz on a Varian E.M. 360L spectrometer. NMR samples were dissolved in deuteriochloroform containing $SiMe_4$ as internal reference.

Carbon, hydrogen, and nitrogen contents were determined using a Perkin–Elmer 240 elemental analyser, and molybdenum using a Perkin–Elmer 403 atomic absorption spectrometer. Arsenic was determined, following combustion of the complex in oxygen, by atomic absorption.

Attempted reaction of $C_5H_5Mo(CO)_3Cl$ with C_6H_5CN in the presence of $AlCl_3$

$C_5H_5Mo(CO)_3Cl$ (0.40 g, 1.43 mmol) was dissolved in dichloromethane (10 ml) and $AlCl_3$ (0.19, 1.43 mmol) was added carefully under nitrogen. C_6H_5CN (0.147 g, 1.43 mmol) was then added to the mixture which was refluxed for 16 h. A solution of NH_4PF_6 (0.233 g, 1.43 mmol) in water (10 ml) was then added to the organic mixture. The flask was vigorously shaken, the aqueous layer removed, and the organic layer washed with water (2 × 10 ml). The solvent was removed by evaporation in vacuo, and the residue dried. Extraction with toluene (20 ml) gave a clear red solution containing only $C_5H_5Mo(CO)_3Cl$, identified by infrared spectroscopy.

The same reaction was attempted in refluxing toluene (40 ml) with a reaction time of 2.5 h. A black oil in a black solution was obtained, and contained no metal carbonyl, only decomposition products.

Attempted reaction of $C_5H_5Mo(CO)_3Cl$ with CH_3CN in the presence of $AlCl_3$

This reaction was performed as described above, but only starting material was recovered.

Reaction of $C_5H_5Mo(CO)_3Cl$ with $AsPh_3$

$C_5H_5Mo(CO)_3Cl$ (3.05 g, 10.9 mmol) was dissolved in toluene (150 ml) and $AsPh_3$ (5.00 g, 16.3 mmol) added. The resulting orange solution was kept at 60 °C for 8 h, filtered hot and then set aside to cool. The resulting orange crystals of $C_5H_5Mo(CO)_2(AsPh_3)Cl$ were washed with hexane and dried in vacuo (yield 93%). The solid product was stable in air for short periods, but solutions quickly decomposed if traces of air were admitted.

When toluene was used as the solvent, decomposition occurred to give a black oil. Analyses. Found: C, 53,8; H, 4.08. $C_{25}H_{20}AsClMoO_2$ calcd.: C, 53.7; H, 3.58%. M.p. 160 °C (dec). Infrared spectrum: ν(CO) nujol: 1946vs, 1855vs cm^{-1}; ν(CO) CH_2Cl_2: 1970vs, 1880vs cm^{-1}. ^{1}H NMR spectrum: δ ($CDCl_3$): 7.65s(15), 5.65(5) ppm.

Reaction of $C_5H_5Mo(CO)_2(AsPh_3)Cl$ with PhCN in the presence of $AlCl_3$

$C_5H_5Mo(CO)_2(AsPh_3)Cl$ (4.60 g, 8.23 mmol) was dissolved in dichloromethane (25 ml) then PhCN (1.69 g, 16.4 mmol) and $AlCl_3$ (2.19 g, 16.5 mmol) added. The solution was then stirred for 16 h. A solution of NH_4PF_6 (1.50 g, 9.20 mmol) in water (15 ml) was carefully added, and the mixture vigorously shaken. The greenish aqueous layer was then removed, and the dark red organic layer was washed with water (5 × 10 ml). Slow removal of the solvent in vacuo, produced yellow crystals of $[C_5H_5Mo(CO)_2(AsPh_3)(PhCN)]^+ [PF_6]^-$ which were recrystallised from CH_2Cl_2/hexane mixtures (yield 82%). Analyses. Found: C, 49.9; H, 3,58; N, 1.68. $C_{31}H_{25}AsF_6MoNP$ calcd.: C, 49.8; H, 3.24; N, 1.82%. Infrared spectra. ν(CO) nujol: 1989vs, 1970m,sh, 1920vs, 1894m cm^{-1}; ν(CO) CH_2Cl_2: 1997vs, 1918vs cm^{-1}; ν(CN) nujol: 2248w; ν(PF_6^-) nujol 844vs cm^{-1}.

Reaction of $[C_5H_5Mo(CO)_2(AsPh_3)(PhCN)]^+ [PF_6]^-$ with n-butyllithium

A suspension of the complex (4.00 g, 5.19 mmol) in THF was frozen at 77 K and n-butyllithium (5.19 mmol, in hexane solution) added. The mixture was warmed to room temperature and stirred for 2 h producing a red solution and a pink slurry. The solution was filtered, and the residue was washed with toluene then dried in vacuo to yield an off-white powder, identified by IR spectroscopy as $LiPF_6$. The filtrate was reduced to dryness, and the residue extracted with toluene (30 ml). After filtration from a crimson solid, the solution was reduced to small bulk (5 ml), ether was added, and the mixture cooled to − 10 °C. Red crystals of $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$ were collected and dried in vacuo (yield 0.16 g).

The crimson precipitate was found to be insoluble in a whole range of organic solvents and could not be recrystallised. It was washed with acetone (3 × 5 ml), water (3 × 5 ml), and acetone (3 × 5 ml) to remove impurities. After drying in vacuo, it was identified as $[C_5H_5Mo(CO)_2(AsPh_3)]_2$ (yield 0.56 g, 17%).

Chromatographic separation of the components of the crude product mixture in dichloromethane on a silica gel column (CH_2Cl_2 eluant), gave first a red fraction containing $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$, followed by an orange fraction containing $C_5H_5Mo(CO)_2(AsPh_3)Cl$. $[C_5H_5Mo(CO)_2(AsPh_3)]_2$ remained on the column and could not be eluted.

The red crystals. Analyses: Found: C, 52.5; H, 3.28; Mo, 26.7. $C_{33}H_{25}AsMo_2O_5$ calcd.: C, 51.6; H, 3.26; Mo, 25.0%. Infrared spectra: ν(CO) CH_2Cl_2: 1968vs, 1896vs,br, 1885vs 1812vs cm^{-1}; ν(CO) nujol: 1967vs, 1902vs, 1888vs, 1873vs, 1818s cm^{-1}; ^{1}H NMR spectrum: δ ($CDCl_3$): 4.83s(5), 5.10s(5), 7.46s(15) ppm. Mass spectrum: The parent ion, $[(C_5H_5)_2Mo_2\text{-}(CO)_5(AsPh_3)]^+$, was not observed. The peak at highest mass occurred at m/e 462, corresponding to $[(C_5H_5)_2Mo_2(CO)_5]^+$. Daughter ions correspond to the stepwise loss of the five carbonyl groups at m/e 434, 406, 378, 350, and 322. $[AsPh_3]^+$, $[AsPh_2]^+$ and $[AsPh]^+$ ions gave the most intense peaks in the spectrum at m/e 306, 229, and 152 respectively.

The crimson complex. Analyses: Found: C, 55.5; H, 3.61; As, 13.8, Mo, 21.3. $C_{50}H_{40}As_2Mo_2O_4$ calcd.: C, 57.3; H, 3.82; As, 14.3, Mo, 18.3%. Infrared spectrum: ν(CO) (nujol) 1843vs, 1828vs cm^{-1}. Mass spectrum: The parent ion $[(C_5H_5)_2Mo_2\text{-}(CO)_4(AsPh_3)_2]^+$, was not observed. The peak of highest mass observed occurred at m/e 434, corresponding to $[(C_5H_5)_2Mo_2(CO)_4]^+$. Daughter ions correspond to loss the stepwise loss of the four carbonyl groups at m/e 406, 378, 350, and 322. Again the $[AsPh_3]^+$, $[AsPh_2]^+$ and $[AsPh]^+$ ions gave the most intense peaks in the spectrum at m/e 306, 229, and 152, respectively.

Action of heat on $[C_5H_5Mo(CO)_2(AsPh_3)]_2$

A suspension of the complex in toluene (15 ml) was stirred for 2 h at 50 °C. The red solution was filtered to leave a brown carbonyl-free residue. Concentration of the solution in vacuo to 2 ml and addition of a drop of hexane produced red crystals of $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$ (0.07 g), identified by infrared spectroscopy.

Reaction of $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$ with CH_2Cl_2

A solution of the complex (0.02 g) in CH_2Cl_2 (10 ml) was refluxed for 2 h. During which time four new carbonyl absorptions developed at 2059m, 1978vs,br, 1970vs and 1880vs cm^{-1}, attributed to $C_5H_5Mo(CO)_3Cl$ and $C_5H_5Mo(CO)_2\text{-}(AsPh_3)Cl$.

Reaction of $[C_5H_5Mo(CO)_2(AsPh_3)(PhCN)]^+ [PF_6]^-$ with $LiOCH_3$

To a frozen suspension of the complex (1.38, 1.68 mmol) in THF (10 ml) at 77 K was added lithium methoxide (1.68 mmol in methanol), and the mixture was allowed to warm to ambient temperature. The orange solution was stirred for a further 10 min, then the solvent was removed in vacuo to leave a brown gum. Washing with acetone (5 ml) gave a fawn carbonyl-free solid, and a dark-red solution, which was reduced in volume in vacuo to 3 ml. Addition of hexane (2 ml) produced dark red crystals of $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$, identified by infrared spectroscopy.

Reaction of $[C_5H_5Mo(CO)_2(AsPh_3)(PhCN)]^+ [PF_6]^-$ with sodium amalgam

Sodium amalgam was prepared from 0.5 g of sodium and 10 ml of mercury. To this was added a solution of the complex (1.30 g, 1.68 mmol) in THF (20 ml), and the mixture was shaken vigorously. The mercury was removed from the red-brown solution after 10 min, the solution was filtered, and then evaporated to dryness in vacuo. Extraction of the residue with toluene gave an off-white solid and a red solution. The solid was shown by infrared spectroscopy to be sodium hexafluorophosphate. The red solution was evaporated to dryness in vacuo, and extracted with CH_2Cl_2. The product from this solution was identified by infrared spectroscopy as $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$.

Reaction of $[C_5H_5Mo(CO)_3]_2$ with triphenylarsine

A solution of the complex (0.564 g, 1.15 mmol) and the arsine (0.450 g, 1.47 mmol) in THF (25 ml) was refluxed for 70 h but no appreciable change occurred. The solution was transferred to a silica flask and irradiated with a 1 kW ultraviolet lamp placed 60 cm from the flask for 8 h. The solvent was then removed completely in vacuo and the residue extracted with chloroform (10 ml). Ether (20 ml) was added to the filtered chloroform solution and the mixture cooled to $-8\,°C$. The crystals obtained were a mixture of $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$ and starting material.

Better separation of the complexes was achieved by chromatographing the filtered chloroform solution on a silica-gel column (30×4 cm) using a 1/1 $CHCl_3$/hexane mixture as eluant. The starting material (0.223 g) was eluted first, followed by the red product $(C_5H_5)_2Mo_2(CO)_5(AsPh_3)$ (0.426 g, 48%).

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