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Use of the anions $[W_2(CO)_{10}]^{2-}$ and $[Mo_2(CO)_8(\mu\text{-}H)(\mu\text{-}L)]^-$ (L = bidentate phosphines) in the synthesis of mercury-transition metal clusters

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Abstract

The reaction of the PPN^+ (bis(triphenylphosphine)nitrogen(1 +)) salt of $[W_2(CO)_{10}]^{2-}$ with ClHg–m complexes (m = $Mo(CO)_3Cp$, $W(CO)_3Cp$, $Fe(CO)_2Cp$, $Mn(CO)_5$, and $Co(CO)_4$) gives high yields of the new trimetallic clusters $(PPN)[(OC)_5WHgm]$ along with $(PPN)[W(CO)_5Cl]$, the crystal structure of which has been determined. The W atom in the anion is octahedrally surrounded by five carbonyl groups and one chloride ligand, giving an approximately C_{4v} symmetry. Attempts to use the anion $[Mo_2(CO)_8(\mu\text{-}H)(\mu\text{-}L)]^-$ (L = dppm, dppe, dppp) to give spiked triangular clusters of the type $[Mo_2(CO)_8(\mu\text{-}H)(\mu\text{-}L)(\mu\text{-}Hgm)]$ was unsuccessful; although tetrametallic chain-type complexes of formula $[(OC)_4HMo(\mu\text{-}L)Mo(CO)_4\{HgMo(CO)_3Cp\}]$ are thought to be formed in THF solutions, processes involving ligand metal redistribution and mercury extrusion precluded their isolation.

Introduction

Recent studies in our programme have dealt with the use of $[Fe_2(CO)_8]^{2-}$, $[Fe_2(CO)_6(\mu\text{-}CO)(\mu\text{-}PPh_2)]^-$ and $[Fe_2(CO)_6(\mu\text{-}CO)(\mu\text{-}CR{=}CR_2)]^-$ anions as building blocks for mixed metal clusters. Thus, compounds containing the metal cores Fe_2M (M = Cu, Ag, Au) [1,2], Fe_2Au_2 [3], and Fe_2HgM (M = Mo, W, Fe, Mn, Co) [4] have been described, and several structurally characterized. With the goal of extending this class of derivatives to group 6 metals in this paper, we attempted the synthesis of anionic triangle clusters of mercury of the type $[M_2(CO)_{10}(\mu\text{-}Hgm)]^-$ (M = Cr, Mo, W; m = $Mo(CO)_3Cp$, $W(CO)_3Cp$, $Fe(CO)_2Cp$, $Mn(CO)_5$, $Co(CO)_4$) by the addition of the bimetal mHg^+ unit to the basic $[M_2(CO)_{10}]^{2-}$ species. However, in spite of the apparent structural similarity between $[Fe_2(CO)_8]^{2-}$ and $[M_2(CO)_{10}]^{2-}$ anions, the latter have proved to be more unstable owing to the ease of rupture of their metal–metal bonds, which precludes the isolation of the tetrametal derivatives. Consequently, the resulting species, formulated as $[MHgm]^-$ on

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the basis of analytical and spectroscopic data, contained fewer metal atoms than those initially expected. These observations prompted us to use the anions $[Mo_2(CO)_8(\mu\text{-}H)(\mu\text{-}L)]^-$ (L = dppm, diphenylphosphinomethane; dppe, diphenylphosphinoethane; dppp, diphenylphosphinopropane), considered to be suitable for giving more stable spiked triangular metal clusters. However, although tetrametallic chain-type complexes are thought to be formed in solution, as indicated by ^{31}P NMR spectroscopy, their unstability precluded their isolation.

Results and discussion

PPN^+ salt (PPN^+ = bis(triphenylphosphine)nitrogen(1 +)) of $[W_2(CO)_{10}]^{2-}$ reacts in THF solutions at −30 °C with ClHgm (m = various metal fragments) to give the trimetal anionic compounds $[(OC)_5WHgm]^-$ and $[W(CO)_5Cl]^-$ according to eq. 1:

$$[W_2(CO)_{10}]^{2-} + ClHgm \rightarrow [(OC)_5WHgm]^- + [W(CO)_5Cl]^- \qquad (1)$$

(m = $Mo(CO)_3Cp$ (**1**); $W(CO)_3Cp$ (**2**); $Mn(CO)_5$ (**3**); $Co(CO)_4$ (**4**); $Fe(CO)_2Cp$ (**5**)) This reaction can be formally interpreted in terms of the rupture of the anion $[W_2(CO)_{10}]^{2-}$ into $[W(CO)_5]^{2-}$ and $W(CO)_5$ units, followed by the capture of the Hgm^+ and Cl^- ions, respectively. This explanation is supported by the weakness of the metal–metal bond in $[W_2(CO)_{10}]^{2-}$ as suggested by the long interatomic distance (3.123(7) Å) [5] found in the closed $[Mo_2(CO)_{10}]^{2-}$. The process reported seems to parallel that described for the synthesis of bimetallic Cr–In complexes, $[(OC)_5CrInBr]\cdot THF$, from $Na_2[Cr_2(CO)_{10}]$ and $InBr_3$ [6]. Interestingly, it was possible to isolate complexes **1**–**5**, since no metal–ligand redistribution processes occurred. This confirms our initial observations that redistribution reactions are disfavoured for anionic unsymmetrical mercury clusters [4].

The reaction represented in equation 1 could not be repeated with the analogous $[M_2(CO)_{10}]^{2-}$ (M = Cr, Mo) anions because the resulting derivatives were found to be very thermal-unstable, and decomposition products predominated. Only the complex $(PPN)[(OC)_5CrHg\{Mo(CO)_3Cp\}]$, **6**, was isolated and analytically characterized. In this and in other metal clusters the presence of the $HgMo(CO)_3Cp$ unit appears to increase the stability of the final product, although the reasons for this remain unclear. The new complexes reported are structurally interesting in that although an extensive group of neutral complexes with symmetrical M–Hg–M skeletons (M = transition metals) [7] has been described, anionic species containing asymmetric M–Hg–M′ arrays are relatively few. To our knowledge, the only examples of complexes the type $(PPN)[(OC)_4FeHgm]$ (m = metal fragment), are those recently prepared in our laboratories [8] by a route involving a metal ligand redistribution between the dianion $[\{Fe(CO)_4\}_2Hg]^{2-}$ and m_2Hg.

Compounds **1**–**6** were isolated as their PPN^+ salts in high yields; all of them are yellow-orange, crystalline, and air-unstable solids. Complexes **1** and **2** can be manipulated in air for short periods. The analytical, and IR and ^1H spectral data are listed in Table 1. In spite of numerous attempts to obtain single crystals of **1**–**6** for an X-ray study, we were able to crystallize only the salt $(PPN)[W(CO)_5Cl]$, which was structurally characterized. The crystal contains well-separated $(PPN)^+$ and $[W(CO)_5Cl]^-$ ions, without short interionic contacts. The anion possesses ap-

Table 1

Analytical and IR spectra of the complexes

Compounds	IR (KBr) $\nu(CO)$ (cm^{-1})	Analyses (Found (calc.)) (%)			^{1}H NMR (ppm)
		C	H	N	
1	2020m, 1925s, 1900s, 1885s, 1850s, 1835s, 1805s	45.1 (44.98)	2.70 (2.68)	0.99 (1.07)	5.21
2	2025m, 1920s, 1905s, 1890s, 1850s, 1835s, 1805s	42.5 (42.14)	2.57 (2.51)	0.99 (1.0)	5.27
3	2045w, 2020m, 1965sh, 1935s, 1910s, 1890s, 1880s, 1835m	45.0 (43.9)	2.50 (2.39)	1.24 (1.11)	-
4	2040s, 2000m, 1965vs, 1950sh, 1900s, 1890sh, 1850m	46.1 (43.78)	2.62 (2.43)	1.20 (1.13)	-
5	2010m, 1910s, br, 1880s, br, 1840m	47.1 (46.48)	2.95 (2.82)	1.24 (1.13)	4.53
6	2010m, 1920s, 1895s, 1880s, 1850s, 1840s, 1810s	50.11 (50.02)	2.98 (2.98)	1.24 (1.19)	5.23

proximately C_{4v} symmetry with the principal interatomic distances $d(W-Cl) = 2.560(3)$, $d(W-C_{trans}) = 1.961(10)$, and $d(W-C_{cis}) = 2.016(17)$ Å. No significant differences from the structure reported for the NMe_4^+ salt are observed [9]. The PLUTO drawing of the molecule with the atom numbering scheme is shown in Fig. 1; relevant bond angles and distances are given in Table 2 and atomic coordinates in Table 3.

The failure of the attempts to obtain the spiked triangular metal clusters from $[M_2(CO)_{10}]^{2-}$ species prompted us to use the closely related $[M_2(CO)_8(\mu\text{-}H)(\mu\text{-}L)]^-$ (L = dppm, dppe, dppp) anions, since they were thought likely to give more thermally and air-stable derivatives than those formed from the unsupported anions. The NEt_4^+ salt of the molybdenum complex [10] was chosen as starting material, and its reaction with $ClHgMo(CO)_3Cp$ in THF was monitored by ^{31}P NMR spectroscopy. The following features were observed: (i) When the reagents are allowed to react at −50 °C, the ^{31}P NMR spectrum exhibits, along with the signal belonging to the starting anion, two doublets (for L = dppm: $\delta(P) = -112.6$ and

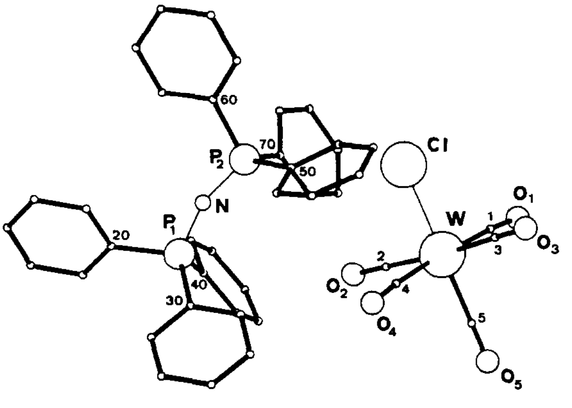


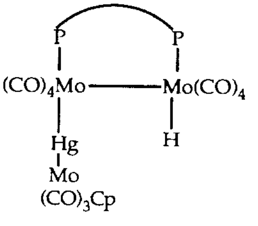
Fig. 1. PLUTO drawing of the molecule with the atom numbering scheme.

Table 2

Selected bond lengths (Å) and angles (°) for $(PPN)[W(CO)_5Cl]$ with esd's in parentheses

Bond distances			
W–Cl	2.560(3)	P1–N1	1.568(5)
W–C1	2.030(10)	P2–N1	1.562(7)
W–C2	2.020(7)	C1–O1	1.142(12)
W–C3	1.992(8)	C2–O2	1.153(10)
W–C4	2.020(12)	C3–O3	1.164(11)
W–C5	1.961(10)	C4–O4	1.143(17)
		C5–O5	1.110(12)
Bond angles			
C4–W–C5	88.8(4)	Cl–W–C5	179.2(3)
C3–W–C5	91.4(4)	Cl–W–C4	91.3(3)
C3–W–C4	90.0(4)	Cl–W–C3	89.3(3)
C2–W–C5	92.2(4)	Cl–W–C2	86.9(3)
C2–W–C4	89.7(4)	Cl–W–C1	92.3(3)
C2–W–C3	176.2(3)	P1–N–P2	151.9(4)
C1–W–C5	87.6(3)	W–C1–O1	176.1(7)
C1–W–C4	176.4(4)	W–C2–O2	177.6(8)
C1–W–C3	89.8(4)	W–C3–O3	179.2(9)
C1–W–C2	90.8(4)	W–C4–O4	175.1(9)
		W–C5–O5	179.9(8)

−163.7 ppm, $J(PP) = 129.2$ Hz; for L = dppe: $\delta(P) = -99.7$ and −110.3 ppm, $J(PP) = 49$ Hz; for L = dppp: $\delta(P) = -102.9$ and −117.0 ppm, $J(PP) = 0$ Hz). Furthermore NEt_4Cl separates at this time. (ii) After several minutes at this temperature a new signal appears at −139.7, −102.4, and −113.2 ppm for L = dppm, dppe, and dppp, respectively. When the solution is warmed the intensity of this singlet greatly increases, while the initial doublets begin to disappear. When the temperature is allowed to rise to 0 °C only the singlet remains, along with several new minor signals. (iii) During the reaction, obvious mercury deposition occurs, and from the resulting solution the complex $[Mo(CO)_3Cp]_2Hg$ can be isolated and characterized by IR and ^1H NMR spectroscopy. These results are tentatively interpreted in terms of a reaction pathway in which the initial step involves chloride replacement of the complex $ClHgMo(CO)_3Cp$ by the anion, giving NEt_4Cl and the tetrametallic complex $[(OC)_4HMo(\mu\text{-}L)Mo(CO)_4\{HgMo(CO)_3Cp\}]$, which requires a metal–metal bond in order for both molybdenum atoms to attain the 18-electron configuration, as is shown in this figure:



This species would via a metal ligand redistribution, give the very unstable pentametallic complex $[(OC)_4HMo(\mu\text{-}L)Mo(CO)_4]_2Hg$, which was not isolated or detected spectroscopically, which, with deposition of mercury, give the unidentified

Table 3

Atomic coordinates ($\times 10^4$) and thermal parameters for compound (PPN)[W(CO)$_5$Cl] with esd's in parentheses

Atom	x	y	z	U_{eq} [a]
W1	2517(0)	4891(0)	352(0)	585(6)
Cl1	2447(2)	4004(2)	2326(2)	943(13)
P1	7605(1)	103(1)	3501(1)	438(6)
P2	7646(1)	1522(1)	5596(1)	427(6)
N1	7815(3)	654(4)	4753(4)	596(19)
C1	1391(5)	5725(5)	222(6)	711(28)
C2	1583(6)	3484(6)	−430(6)	812(32)
C3	3454(6)	6233(7)	1233(7)	863(33)
C4	3646(7)	4106(8)	392(7)	1009(41)
C5	2558(5)	5562(6)	−1181(7)	755(31)
O1	746(4)	6203(5)	81(5)	1033(28)
O2	1060(6)	2653(5)	−880(6)	1286(34)
O3	4010(5)	7018(7)	1746(6)	1360(36)
O4	4302(6)	3716(8)	364(7)	1595(49)
O5	2576(4)	5929(4)	−2032(5)	816(22)
C20	7443(4)	−1434(4)	3634(4)	460(19)
C21	6883(4)	−2155(5)	2687(5)	599(23)
C22	6742(5)	−3330(5)	2849(7)	796(31)
C23	7149(5)	−3777(5)	3939(8)	813(35)
C24	7693(6)	−3081(6)	4859(6)	769(32)
C25	7870(4)	−1900(5)	4719(5)	603(24)
C30	8604(3)	555(4)	2932(4)	456(18)
C31	9036(4)	−218(5)	2512(5)	542(21)
C32	9808(4)	171(6)	2082(6)	733(29)
C33	10137(4)	1327(6)	2051(6)	731(28)
C34	9715(4)	2099(6)	2447(6)	722(27)
C35	8950(4)	1727(5)	2902(5)	617(23)
C40	6508(3)	425(4)	2437(4)	444(18)
C41	6500(4)	1159(5)	1513(5)	579(22)
C42	5626(5)	1464(6)	802(6)	734(28)
C43	4770(5)	9973(6)	1010(6)	750(28)
C44	4767(4)	251(6)	1900(6)	752(28)
C45	5627(4)	−34(5)	2630(6)	634(24)
C50	8607(3)	2738(4)	5953(4)	455(19)
C51	8470(4)	3760(5)	6341(6)	672(25)
C52	9237(5)	4669(5)	6701(8)	897(33)
C53	10135(5)	4541(7)	6642(8)	916(35)
C54	10289(4)	3551(8)	6273(7)	866(34)
C55	9522(4)	2616(6)	5916(5)	652(25)
C60	7675(3)	813(4)	6969(4)	422(17)
C61	8050(4)	1408(5)	8037(5)	647(23)
C62	8115(5)	832(7)	9092(5)	808(30)
C63	7815(6)	−321(7)	9084(6)	767(30)
C64	7447(5)	−929(5)	8037(7)	707(29)
C65	7357(4)	−366(5)	6969(5)	579(23)
C70	6532(3)	2087(4)	5027(4)	429(17)
C71	6386(4)	2726(5)	4006(5)	575(22)
C72	5540(4)	3187(5)	3541(5)	676(25)
C73	4851(4)	3032(6)	4111(7)	809(30)
C74	4990(5)	2428(8)	5123(8)	961(37)
C75	5824(4)	1935(6)	5589(5)	699(26)

[a] $U_{eq} = \frac{1}{3}\Sigma[U_{ij}, a_i^* a_j^* a_i \cdot a_j \cdot \cos(a_i, a_j)] \cdot 10^4$.

final products. Several comments can be offered on this proposed mechanism. First, the formation of the tetrametallic chain-type complex $[(OC)_4HMo(\mu\text{-}L)Mo(CO)_4\{HgMo(CO)_3Cp\}]$ instead of the spiked triangular cluster $[Mo_2(CO)_8(\mu\text{-}H)(\mu\text{-}L)(\mu\text{-}Hg\{Mo(CO)_3Cp\})]$ can be understood on the basis of the long Mo–Mo distance (about 3.4 Å) reported for the anions in the literature [10], which disfavours the formation of the initially expected 3c–2e bond found in other systems. Secondly the isolation of $[Mo(CO)_3Cp]_2Hg$ strongly confirms that a redistribution process has occurred, and we have noted above that ligand redistribution reactions are favoured for neutral mercury complexes. Finally, although we have recently reported the isolation and structural characterization of the air-stable complex $(NEt_4)_2[\{(OC)_4FeSnCl_2Fe(CO)_4\}_2Hg]$ [11] containing the heptametallic unsupported skeleton FeSnFeHgFeSnFe, it is well known that long metallic chains containing mercury tend to break down, to give mercury and clusters with fewer metal atoms [12].

Experimental

Solvents were dried by standard methods and all manipulations and reactions were performed in Schlenk-type vessels under nitrogen. Elemental analyses for C, H, and N were carried out at the Institut de Bio-Orgànica de Barcelona. ^1H NMR and ^{31}P NMR spectra were recorded on a Bruker WP 80SY spectrometer; ^1H shifts are relative to $Si(CH_3)_4$ and ^{31}P shifts are relative to $P(OCH_3)_3$. Infrared spectra were recorded on a Perkin–Elmer 1330 spectrophotometer. Compounds $(PPN)_2[W_2(CO)_{10}]$ [13], $(NEt_4)[Mo_2(CO)_8(\mu\text{-}H)(\mu\text{-}L)]$ (L = dppm, dppe, dppp) [10], and ClHg–m (m = $Mo(CO)_3Cp$, $W(CO)_3Cp$, $Fe(CO)_2Cp$, $Mn(CO)_5$, and $Co(CO)_4$) [14] were prepared as previously described.

Synthesis of $(PPN)[(CO)_5W–Hg–m]$ (m = $Mo(CO)_3Cp$, $W(CO)_3Cp$, $Fe(CO)_2Cp$, $Mn(CO)_5$, and $Co(CO)_4$) (1–5) and $(PPN)[W(CO)_5Cl]$

Details of the synthesis of **1** also apply to **2**–**5**. A solution of $ClHgMo(CO)_3Cp$ (0.14 g, 0.29 mmol) in THF (25 ml) was added dropwise to a vigorously stirred suspension of $(PPN)_2[W_2(CO)_{10}]$ (0.5 g, 0.29 mmol) in THF (40 ml) at −20 °C. The solution was allowed to warm to 10 °C, and the solvent was removed under vacuum. The residue was extracted with 50 ml of diethyl ether at 10 °C, and the extract filtered through Celite and then cooled, to give yellow microcrystals of **1**. Yield 0.31 g (83%). A second extraction of the resulting residue with 30 ml of diethyl ether afforded yellow microcrystals of $(PPN)[W(CO)_5Cl]$ (0.117 g, 45%).

Synthesis of $(PPN)[(CO)_5Cr–Hg–Mo(CO)_3Cp]$ (6)

A solution of $ClHgMo(CO)_3Cp$ (0.20 g, 0.41 mmol) in THF (20 ml) was added dropwise to a vigorously stirred suspension of $(PPN)_2[Cr_2(CO)_{10}]$ (0.60 g, 0.41 mmol) in THF (30 ml) at −40 °C. The solution was allowed to warm to 0 °C and then concentrated to dryness. The solid was extracted with 30 ml of diethyl ether at 0 °C and the extract filtered through Celite, and after cooling, orange microcrystals of **6** were formed. Yield 0.29 g (60%).

A second extraction of the resulting residue with 30 ml of diethyl ether afforded yellow microcrystals of $(PPN)[Cr(CO)_5Cl]$. Yield 0.21 g (65%).

^{31}P NMR study of the reactions involving $(NEt_4)[Mo_2(CO)_8(\mu\text{-}H)(\mu\text{-}L)]$ (L = dppm, dppe, dppp) and $ClHgMo(CO)_3Cp$

Details of the experiment carried out with $(NEt_4)[Mo_2(CO)_8(\mu\text{-}H)(\mu\text{-}dppm)]$ also apply to the molybdenum derivatives containing dppe or dppp. A solution of $ClHgMo(CO)_3Cp$ (0.08 g, 0.16 mmol) in THF (10 ml) was added to solution of $(NEt_4)[Mo_2(CO)_8(\mu\text{-}H)(\mu\text{-}dppm)]$ (0.150 g, 0.16 mmol) in THF (20 ml) at −50 °C and the ^{31}P NMR spectrum was recorded. After 10 min the spectrum was again recorded and the mixture then was allowed to warm slowly to 0 °C. Mercury and NEt_4^+ salts were removed by filtration through Celite, and from the filtrate the complex $[Mo(CO)_3Cp]_2Hg$ (0.033 g, 55%) was isolated and characterized by IR and ^{1}H NMR spectroscopy.

Crystallographic section

Yellow crystals of $(PPN)[W(CO)_5Cl]$ were grown from acetone–diethyl ether (1 : 1). A prismatic crystal of approximate dimensions of 0.40 × 0.32 × 0.25 mm was sealed in a capillary and mounted on a Philips PW-1100 four-circle diffractometer. The unit-cell parameters were measured from 41 reflections ($2 < \theta < 21°$) and refined by least-squares method. Intensities were collected with graphite-monochromatized Mo-K_α radiation, using the $\omega/2\theta$ scan technique with scan width 1.40° and scan speed 0.055° s^{-1}. 6435 reflections were measured in the range $2 < \theta < 25°$, 6193 of which with $I > 2\sigma$ were assumed as observed. Data were corrected for Lorentz polarization effects and an empirical absorption correction following the DIFABS procedure [15].

Crystal data. $[C_{41}H_{30}ClO_5NP_2W]$. $M = 897.94$, triclinic, a 14.658(1), b 11.917(1), c 11.894(1) Å; α 87.82(1), β 108.31(1), γ 99.82(1)°. V 1926.97(3) Å^3, space group $P\bar{1}$, $Z = 2$, $D_c = 1.5476$ g cm^{-3}, $F(000) = 888$, Mo-K_α radiation, $\lambda = 0.7107$ Å, $\mu(\text{Mo-}K_\alpha) = 32.55$ cm^{-1}.

The structure was solved by Patterson method and the rest of the non-hydrogen atoms were located by alternate cycles of difference Fourier maps and isotropic least-squares refinement then by anisotropic refinement using full-matrix least squares of non-hydrogen. The H atoms were placed in calculated positions with C–H distance of 1.00 Å, and they were included in the last cycles of refinement as fixed contributors. An empirical weighting scheme was applied so as to give no trends in $\langle w\Delta F^2\rangle$ versus $\langle |F_o|\rangle$ and $\langle(\sin\theta)/\lambda|\rangle$. Final R and R_w were 0.042 and 0.039 respectively. Final $\Delta\rho \pm 0.87$ e Å^{-3}; the highest peaks are closed to the W atoms; no extinction correction. Scattering factors were taken from the International Tables of X-ray Crystallography [16]. All calculations were performed on a VAX 6410 computer using the computing program XRAY76 [17] and several local programs. Tables of all bond distances and angles, final hydrogen parameters, anisotropic thermal parameters, and a list of structure factors can be obtained from the authors.

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