Chemistry of Polynuclear Metal Complexes with Bridging Carbene or Carbyne Ligands. Part 83.[1] Molybdenum and Tungsten Complexes containing the Alkylidyne Group C{η^6-C_6H_4(OMe-2)Cr$(CO)_3$}

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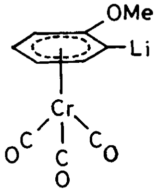
The alkylidyne-metal complexes [CrM{μ-σ:η^6-CC_6H_4(OMe-2)}$(O_2CCF_3)(CO)_5L_2$] [M = Mo or W, L_2 = tmen ($Me_2NCH_2CH_2NMe_2$) or bipy (2,2′-bipyridine)] and [CrM{μ-σ:η^6-CC_6H_4(OMe-2)}-$(CO)_5L$] {M = Mo, L = η-C_5H_5 or HB$(pz)_3$ [hydrotris(pyrazol-1-yl)borate]; M = W, L = η-C_5H_5} have been prepared. In these species the alkylidyne ligand attached to Mo or W is η^6-co-ordinated to a Cr$(CO)_3$ group. Several reactions of the complexes [CrM{μ-σ:η^6-CC_6H_4(OMe-2)}$(CO)_5$-(η-C_5H_5)] with low-valent metal compounds have been studied. These have led to the isolation of various heteronuclear metal cluster compounds with bonds between molybdenum or tungsten and platinum, nickel, cobalt, and rhodium. The n.m.r. data for the new products are reported and discussed in relation to the structures proposed.

In our early work[2] on polynuclear metal compounds with bridging alkylidyne ligands we employed as a precursor the compound [W($\equiv CC_6H_4Me$-4)$(CO)_2$(η-C_5H_5)]. This species served as a useful model since the C≡W bond readily combines with a variety of low-valent metal–ligand fragments involving nearly all of the transition elements. In our more recent studies we have greatly extended the scope of these syntheses. The paradigm molecule [W($\equiv CC_6H_4Me$-4)$(CO)_2$(η-C_5H_5)][3] may be modified in one or other of various ways, provided an isolobal mapping[4] with the new precursor is maintained. Modifications include substituting Cr or Mo for the W atom, various alkyl or aryl groups for the C_6H_4Me-4 substituent[5] and η-C_5Me_5,[6a,b] HB$(pz)_3$ [hydrotris(pyrazol-1-yl)borate],[6c,7] $H_2B(pz)_2$ [dihydrobis(pyrazol-1-yl)borate],[8] or η^5-$C_2B_9H_9R_2$ (R = H or Me)[9] groups for the η-C_5H_5 ligand. Hence, many mononuclear metal–alkylidyne reagents are now available as reagents for the synthesis of complexes containing heteronuclear metal–metal bonds following the general strategy developed earlier. Moreover, as a consequence of the different metals present and the variety of ligands involved, the products obtained display a rich chemistry.

In this paper we introduce a further class of mononuclear metal–alkylidyne complex for use in the preparation of compounds having metal–metal bonds. In the new reagents, the molybdenum or tungsten atoms are ligated by the alkylidyne group CC_6H_4OMe-2, the arene ring of which is η^6-co-ordinated to a Cr$(CO)_3$ moiety. The resulting products thus contain an additional metal–ligand fragment.



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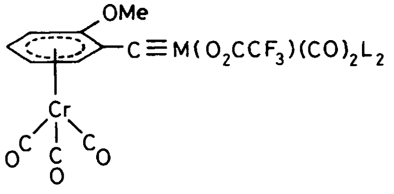
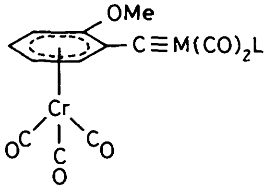


	M	L_2
(2a)	Mo	tmen
(2b)	Mo	bipy
(2c)	W	tmen
(2d)	W	bipy



	M	L
(3a)	Mo	η-C_5H_5
(3b)	W	η-C_5H_5
(3c)	Mo	HB$(pz)_3$

Results and Discussion

The new compounds [CrM{μ-σ:η^6-CC_6H_4(OMe-2)}$(O_2CCF_3)(CO)_5L_2$] [M = Mo or W, L_2 = tmen ($Me_2NCH_2CH_2NMe_2$) or bipy (2,2′-bipyridine)] were prepared by a procedure developed by Mayr and co-workers.[10] The lithium reagent Li[Cr$(CO)_3$(η^6-C_6H_4OMe)] (1),[11] prepared by treating [Cr$(CO)_3$(η^6-C_6H_5OMe)] with n-butyl-lithium, was added to a suspension of the hexacarbonyls [M$(CO)_6$] (M = Mo or W) in thf (tetrahydrofuran) at *ca.* −30 °C. The resulting solution was then treated with an equivalent amount of $(CF_3CO)_2O$, followed by an equivalent amount of the bidentate ligand tmen or bipy. The products (2) (70—80% yield) were isolated by column chromatography on silica gel, and were characterised by the data given in Tables 1 and 2. The ^{13}C-{^1H} n.m.r. spectra of the compounds showed the characteristic resonances for the ligated carbon atom of the alkylidyne group at δ 268.7 (**2a**), 274.4 (**2b**), 257.1 (**2c**), and 263.3 p.p.m. (**2d**).

Treatment of (**2b**) or (**2d**), suspended in Et_2O, with the reagent NaC_5H_5·dme (dme = $MeOCH_2CH_2OMe$)[12] afforded the complexes [CrM{μ-σ:η^6-CC_6H_4(OMe-2)}$(CO)_5$(η-C_5H_5)] [M = Mo (**3a**) or W (**3b**)], isolated by column chromatography on alumina. The reaction between (**2a**) and K[HB$(pz)_3$] in CH_2Cl_2 gave the related compound [CrMo{μ-σ:η^6-CC_6H_4-(OMe-2)}$(CO)_5${HB$(pz)_3$}] (**3c**). Data for the complexes (**3**) are listed in Tables 1 and 2, and as expected the ^{13}C-{^1H} n.m.r. spectra show the diagnostic peaks for the terminally bound alkylidyne carbon nuclei at δ 295.6 (**3a**), 282.9 (**3b**), and 278.1 p.p.m. (**3c**). It is interesting to compare the chemical shift in the spectrum of (**3a**) with that in [Mo($\equiv CC_6H_4OMe$-2)$(CO)_2$-

Table 1. Analytical[a] and physical data for the complexes

Compound	Colour	Yield (%)	$\nu_{max.}(CO)$[b]/cm^{-1}	Analysis (%)	
				C	H
(2a) $[CrMo\{\mu\text{-}\sigma:\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(O_2CCF_3)(CO)_5(tmen)]$	Orange	71	[c]2 011m, 1 961s, 1 927m, 1 890m	[d]39.0 (39.6)	3.6 (3.6)
(2b) $[CrMo\{\mu\text{-}\sigma:\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(O_2CCF_3)(CO)_5(bipy)]$	Orange	79	[c]2 009m, 1 960s, 1 928m, 1 894m	[e]43.9 (44.0)	2.5 (2.2)
(2c) $[CrW\{\mu\text{-}\sigma:\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(O_2CCF_3)(CO)_5(tmen)]$	Orange	76	[c]1 995m, 1 957s, 1 907s, 1 888s	[f]34.5 (34.8)	3.1 (3.2)
(2d) $[CrW\{\mu\text{-}\sigma:\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(O_2CCF_3)(CO)_5(bipy)]$	Dark orange	81	[c]1 999m, 1 956s, 1 906m, 1 886m	[g]38.8 (39.3)	2.0 (2.0)
(3a) $[CrMo\{\mu\text{-}\sigma:\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_5(\eta\text{-}C_5H_5)]$	Red	46	2 009m, 1 967s, 1 932m, 1 903m	46.0 (45.8)	2.7 (2.6)
(3b) $[CrW\{\mu\text{-}\sigma:\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_5(\eta\text{-}C_5H_5)]$	Red	51	1 996m, 1 964s, 1 912m, 1 900m	38.5 (38.6)	2.1 (2.2)
(3c) $[CrMo\{\mu\text{-}\sigma:\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_5\{HB(pz)_3\}]$	Orange	93	2 011m, 1 966s, 1 929m, 1 900m	[h]42.4 (42.6)	2.9 (2.8)
(4a) $[Cr_2Mo_2Pt\{\mu\text{-}\sigma,\sigma':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}_2(CO)_{10}(\eta\text{-}C_5H_5)_2]$	Magenta	63	1 961s, 1 894m, 1 827w (br)	37.6 (37.9)	2.3 (2.1)
(4b) $[Cr_2Mo_2Ni\{\mu\text{-}\sigma,\sigma':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}_2(CO)_{10}(\eta\text{-}C_5H_5)_2]$	Brown	46	[i]1 956s, 1 890m, 1 829w (br)	42.8 (43.1)	2.6 (2.4)
(4c) $[Cr_2W_2Ni\{\mu\text{-}\sigma,\sigma':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}_2(CO)_{10}(\eta\text{-}C_5H_5)_2]$	Brown	42	[i]1 957s, 1 884m, 1 832w (br)	36.1 (36.7)	2.4 (2.1)
(5a) $[CrMoCo_2\{\mu_3\text{-}\sigma,\sigma',\sigma'':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_{11}(\eta\text{-}C_5H_5)]$	Brown	75	[i]2 083m, 2 071w, 2 044m, 2 027s, 2 004m, 1 957m, 1 884m	37.7 (38.0)	1.7 (1.6)
(5b) $[CrWCo_2\{\mu_3\text{-}\sigma,\sigma',\sigma'':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_{11}(\eta\text{-}C_5H_5)]$	Brown	78	[i]2 081m, 2 073w, 2 042m, 2 025s, 2 002m, 1 956m, 1 882m	33.9 (34.1)	1.4 (1.4)
(6) $[CrMoCo_2\{\mu_3\text{-}\sigma,\sigma',\sigma'':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(\mu\text{-}dppm)(CO)_9(\eta\text{-}C_5H_5)]$	Dark brown	97	[i]2 018m, 1 989m, 1 974s, 1 948s, 1 870m	51.6 (52.0)	3.5 (3.2)
(7a) $[CrMoRh\{\mu\text{-}\sigma,\sigma':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_6(\eta\text{-}C_5H_5)(\eta^5\text{-}C_9H_7)]$	Brown	67	2 018w, 1 999m, 1 983s, 1 942m, 1 892m, 1 864w	46.6 (46.8)	2.8 (2.7)
(7b) $[CrWRh\{\mu\text{-}\sigma,\sigma':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_6(\eta\text{-}C_5H_5)(\eta^5\text{-}C_9H_7)]$	Brown	73	2 011w, 1 995m, 1 981s, 1 934m, 1 889m, 1 854w	41.1 (41.7)	2.4 (2.4)

[a] Calculated values are given in parentheses. [b] Measured in Et_2O unless otherwise stated. [c] In thf. [d] N, 4.3 (4.4%). [e] N, 4.5 (4.1%). [f] N, 3.7 (3.9%). [g] N, 3.9 (3.7%). [h] N, 12.9 (13.6%). [i] In CH_2Cl_2.

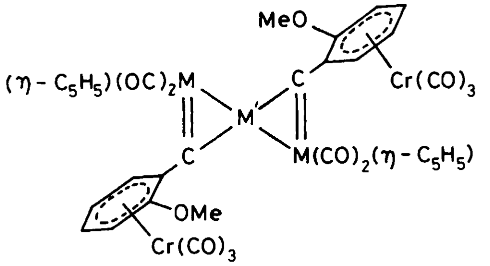
$(\eta\text{-}C_5H_5)]$ (δ 305.9 p.p.m.).[5] Evidently co-ordination of the $Cr(CO)_3$ group to the arene ring in the former species results in a somewhat less deshielded alkylidyne carbon resonance.

Complexes of the type $[M(\equiv CR)(CO)_2(\eta\text{-}C_5H_5)]$ (M = Mo or W, R = alkyl or aryl) undergo well established reactions with $[Pt(C_2H_4)_3]$, $[Ni(cod)_2]$ (cod = cyclo-octa-1,5-diene), or $[Co_2(CO)_8]$. The zerovalent Pt and Ni reagents afford species $[M_2M'(\mu\text{-}CR)_2(CO)_4(\eta\text{-}C_5H_5)_2]$ (M′ = Pt or Ni) in which the platinum or nickel atoms are ligated by two C≡M groups in such a manner that the three metal atoms adopt an essentially linear arrangement. However, the disposition of the edge-bridging alkylidyne groups is such that the two dimetallacyclopropene rings which share a common vertex (Pt or Ni) are at 90° to one another.[6a,7d,13] Octacarbonyldicobalt and the alkylidyne–molybdenum or –tungsten complexes afford a family of species $[MCo_2(\mu_3\text{-}CR)(CO)_8(\eta\text{-}C_5H_5)]$ containing a trimetallatetrahedrane, $\mu_3\text{-}CMCo_2$, core.[5,6b,c,14] In view of these results it was of interest to establish whether the compounds (**3a**) and (**3b**) would undergo similar reactions with $[Pt(C_2H_4)_3]$, $[Ni(cod)_2]$, or $[Co_2(CO)_8]$.

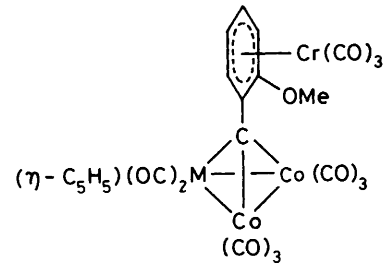
Treatment of (**3a**) with $[Pt(C_2H_4)_3]$ or $[Ni(cod)_2]$, respectively, afforded the pentanuclear metal complexes $[Cr_2Mo_2M'\{\mu\text{-}\sigma,\sigma':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}_2(CO)_{10}(\eta\text{-}C_5H_5)_2]$ [M′ = Pt (**4a**) or Ni (**4b**)], data for which are given in the Tables. Similarly, (**3b**) and $[Ni(cod)_2]$ gave $[Cr_2W_2Ni\{\mu\text{-}\sigma,\sigma':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}_2(CO)_{10}(\eta\text{-}C_5H_5)_2]$ (**4c**). The ^1H and $^{13}C\text{-}\{^1H\}$ n.m.r. properties of the complexes (**4**) are in accord with the structures proposed, and thus the compounds are closely related to those referred to above having C≡M (M = Mo or W) groups ligating Pt or Ni atoms.

In Et_2O at room temperature, compounds (**3a**) and (**3b**), respectively, react with $[Co_2(CO)_8]$ to yield the tetranuclear metal complexes $[CrMCo_2\{\mu_3\text{-}\sigma,\sigma',\sigma'':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_{11}(\eta\text{-}C_5H_5)]$ [M = Mo (**5a**) or W (**5b**)]. These products are evidently similar to those obtained earlier having structures based on a $\mu_3\text{-}CMCo_2$ core. The $^{13}C\text{-}\{^1H\}$ n.m.r. spectra of (**5a**) and (**5b**) reveal resonances for the μ_3-C nuclei at δ 266.1 and 257.0 p.p.m., respectively. These data may be compared with signals for the triply bridging alkylidyne carbons at 270.7 p.p.m. in the spectrum of $[MoCo_2(\mu_3\text{-}CC_6H_4Me\text{-}4)(CO)_8(\eta\text{-}C_5H_5)]$[15] and at 257.0 p.p.m. in the spectrum of $[WCo_2(\mu_3\text{-}CC_6H_4Me\text{-}4)(CO)_8(\eta\text{-}C_5H_5)]$.[14]

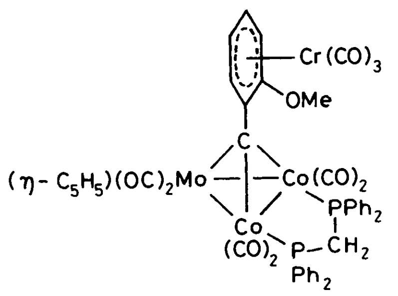
Compound (**5a**) was treated with dppm $(Ph_2PCH_2PPh_2)$ in CH_2Cl_2 in an attempt to link, by displacement of CO, two metal centres involving different transition elements. The product of this reaction, however, was the complex $[CrMoCo_2\{\mu_3\text{-}\sigma,\sigma',\sigma'':\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(\mu\text{-}dppm)(CO)_9(\eta\text{-}C_5H_5)]$ (**6**) in which the dppm ligand bridges the Co–Co bond. Complex (**6**) is thus related to the previously reported compound $[WCo_2(\mu_3\text{-}CC_6H_4Me\text{-}4)(\mu\text{-}dppm)(CO)_6(\eta\text{-}C_5H_5)]$.[14] Both the latter and (**6**) show a single resonance in their $^{31}P\text{-}\{^1H\}$ n.m.r. spectra. For (**6**) there is a broad peak at δ 33.2 p.p.m., in the spectrum measured at room temperature, while that for $[WCo_2(\mu_3\text{-}CC_6H_4Me\text{-}4)(\mu\text{-}dppm)(CO)_6(\eta\text{-}C_5H_5)]$ (at −50 °C) occurs as



	M	M′
(4a)	Mo	Pt
(4b)	Mo	Ni
(4c)	W	Ni



	M
(5a)	Mo
(5b)	W



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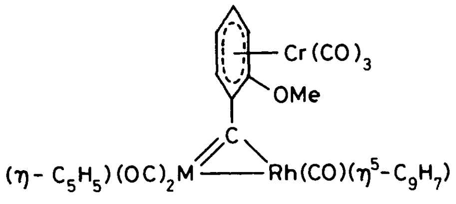

	M
(7a)	Mo
(7b)	W

a sharp band at 27.3 p.p.m. [relative to 85% H_3PO_4 (external)]. Similarly, in their $^{13}C-\{^1H\}$ n.m.r. spectra both compounds show characteristic resonances for the μ_3-C groups: (**6**) at δ 253.1, and $[WCo_2(\mu_3\text{-}CC_6H_4Me\text{-}4)(\mu\text{-dppm})(CO)_6(\eta\text{-}C_5H_5)]$ at 265.0 p.p.m.[14]

Addition of the compounds (**3a**) and (**3b**), respectively, to light petroleum solutions of $[Rh(CO)_2(\eta^5\text{-}C_9H_7)]$ (C_9H_7 = indenyl) afforded the trimetal compounds $[CrMRh\{\mu\text{-}\sigma,\sigma'\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_6(\eta\text{-}C_5H_5)(\eta^5\text{-}C_9H_7)]$ [M = Mo (**7a**) or W (**7b**)], characterised by the data given in Tables 1 and 2. In the $^{13}C-\{^1H\}$ n.m.r. spectra of both compounds there are doublet signals diagnostic for alkylidyne groups bridging the M–Rh bonds: (**7a**), δ 312.9 [J(RhC) 29]; (**7b**), δ 288.7 p.p.m. [J(RhC) 27 Hz]. Moreover, the $^{13}C-\{^1H\}$ data also reveal the presence of two CO ligands attached to molybdenum [(**7a**)] or tungsten [(**7b**)], and one CO group bonded to rhodium. Signals for the latter are doublets in each spectrum, and occur at δ 188.7 [J(RhC) 88] for (**7a**) and at 189.5 p.p.m. [J(RhC) 88 Hz] for (**7b**). Peaks at δ 233.5 (**7a**) and 233.8 p.p.m. (**7b**) are due to the $Cr(CO)_3$ fragments, and resonances for these moieties are observed with similar chemical shifts in the spectra of all the compounds (**2**)—(**6**).

Alkylidyne groups bridging Mo–Rh or W–Rh bonds, as in the compounds (**7**), occur in the complexes $[MRh(\mu\text{-}CC_6H_4Me\text{-}4)(CO)_2(L)(\eta\text{-}C_5H_5)(\eta^5\text{-}C_9H_7)]$ [M = Mo or W, L = CO or PMe_3].[15–17] The chemical shifts for the μ-C nuclei in the $^{13}C-\{^1H\}$ n.m.r. spectra for the species with L = CO are δ 337.3 (M = Mo) and 312.4 p.p.m. (M = W).[15] Both signals are doublets [J(RhC) 30 Hz] as seen in the spectra of (**7**).

An interesting feature of the synthesis described in this paper is the spectator role played by the $Cr(CO)_3$ group in all the reactions. The carbonyl groups in this fragment are evidently strongly bound to the chromium centre, and thus loss of CO and condensation reactions to form clusters of higher nuclearity were not observed. Indeed, several of the new compounds described herein were thermally unstable with respect to loss of their $Cr(CO)_3$ fragments, indicating that the chromium–arene bond is weaker than the Cr–CO bonds. However, with suitable reagents and appropriate conditions it may be possible to use the compounds reported herein as precursors to heteronuclear metal compounds with bonds between chromium and other transition elements.

Experimental

The experimental procedures used and the instrumentation employed for the spectroscopic measurements have been described earlier.[18] Light petroleum refers to that fraction of b.p. 40—60 °C. The compound $[Cr(CO)_3(\eta^6\text{-}C_6H_5OMe)]$ required for the synthesis of (**1**) was prepared by the method previously reported.[19] Analytical and other data for the new compounds are listed in Table 1.

Synthesis of the Complexes $[CrM\{\mu\text{-}\sigma\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(O_2CCF_3)(CO)_5L_2]$ (M = Mo *or* W, L_2 = tmen *or* bipy).—The method used to prepare the complexes (**2**) follows from a procedure developed by Mayr and co-workers.[10] An Et_2O (50 cm^3) solution of $[Cr(CO)_3(\eta^6\text{-}C_6H_5OMe)]$ (2.44 g, 10.0 mmol) at −30 °C was treated with a hexane solution (6.5 cm^3) of n-butyl-lithium (10 mmol). After 30 min, the resulting mixture was added to a suspension of $[Mo(CO)_6]$ (2.64 g, 10.0 mmol) in thf (50 cm^3) previously cooled to −30 °C. The temperature of the Schlenk tube was allowed to warm to *ca.* −20 °C, and the mixture was stirred for *ca.* 6 h, after which period monitoring by i.r. revealed that all the $[Mo(CO)_6]$ had been consumed. The resulting red solution was cooled to −78 °C and treated with $(CF_3CO)_2O$ (1.4 cm^3, 10 mmol). The mixture initially turned green but after 10 min stirring became bright red. The ligand tmen (1.8 cm^3, 12 mmol) was added, and the mixture was warmed slowly to room temperature. Volatile material was removed *in vacuo*, the residue was suspended in light petroleum–CH_2Cl_2 (45 cm^3, 2:3), and the solution chromatographed on a

Table 2. Hydrogen-1 and carbon-13 n.m.r. data[a] for the complexes

Compound	^{1}H(δ)	^{13}C(δ)[b]
(2a)	2.59, 2.65, 3.04, 3.09 (s × 4, 12 H, NMe), 2.79 (m, 4 H, NCH_2), 3.78 (s, 3 H, OMe), 4.80 [t, 1 H, C_6H_4, J(HH) 6], 4.98 [d, 1 H, C_6H_4, J(HH) 6], 5.58 [t, 1 H, C_6H_4, J(HH) 6], 5.77 [d, 1 H, C_6H_4, J(HH) 6]	268.7 (C≡Mo), 232.4 (CrCO), 223.3, 223.2 (MoCO), 161.1 [C(O)O], 143.5 [$C^1(C_6H_4)$], 117.6 [q, CF_3, J(FC) 293], 99.1, 97.7, 93.7, 83.9, 72.8 (C_6H_4), 60.0 (NCH_2), 55.9, 55.7, 55.6, 50.2, 50.0 (NMe, OMe)
(2b)	3.73 (s, 3 H, OMe), 4.64 [t, 1 H, C_6H_4, J(HH) 7], 4.82 [d, 1 H, C_6H_4, J(HH) 7], 5.41 [t, 1 H, C_6H_4, J(HH) 7], 5.55 [d, 1 H, C_6H_4, J(HH) 7], 7.56 [t, 2 H, bipy, J(HH) 6], 8.09 [t, 2 H, bipy, J(HH) 6], 8.17 [d, 2 H, bipy, J(HH) 6], 9.21 [d, 2 H, bipy, J(HH) 6]	274.4 (C≡Mo), 232.2 (CrCO), 224.5, 224.3 (MoCO), 161.1 [C(O)O], 154.9, 154.8, 154.6, 154.5 (bipy), 144.9 [$C^1(C_6H_4)$], 139.7, 139.6, 125.9, 125.8, 121.9, 121.6 (bipy), 117.1 [q, CF_3, J(CF) 291], 98.9, 97.0, 93.2, 83.1, 72.5 (C_6H_4), 53.5 (OMe)
(2c)	2.69, 2.75, 3.21, 3.25 (s × 4, 12 H, NMe), 2.90 (m, 4 H, NCH_2), 3.83 (s, 3 H, OMe), 4.82 [t, 1 H, C_6H_4, J(HH) 6], 4.97 [d, 1 H, C_6H_4, J(HH) 6], 5.55 [t, 1 H, C_6H_4, J(HH) 6], 5.71 [d, 1 H, C_6H_4, J(HH) 6]	257.1 (C≡W), 231.6 (CrCO), 219.6, 219.5 (WCO), 159.5 [C(O)O], 142.6 [$C^1(C_6H_4)$], 117.4 [q, CF_3, J(FC) 292], 104.0, 97.7, 93.0, 84.0, 72.7 (C_6H_4), 60.5 (NCH_2), 56.6, 56.3, 55.3, 50.6, 50.3 (NMe, OMe)
(2d)	3.62 (s, 3 H, OMe), 4.66 [t, 1 H, C_6H_4, J(HH) 7], 4.86 [d, 1 H, C_6H_4, J(HH) 7], 5.43 [t, 1 H, C_6H_4, J(HH) 7], 5.56 [d, 1 H, C_6H_4, J(HH) 6], 7.55 [t, 2 H, bipy, J(HH) 6], 8.13 [t, 2 H, bipy, J(HH) 6], 8.24 [d, 2 H, bipy, J(HH) 6], 9.35 [d, 2 H, bipy, J(HH) 6]	263.3 (C≡W), 232.7 (CrCO), 223.6, 223.2 (WCO), 161.3 [C(O)O], 155.7, 155.4, 155.1, 154.9 (bipy), 144.5 [$C^1(C_6H_4)$], 139.9, 139.8, 126.6, 126.5, 122.3, 122.2 (bipy), 117.0 (CF_3), 104.2, 97.4, 93.0, 83.7, 73.1 (C_6H_4), 55.6 (OMe)
(3a)	3.77 (s, 3 H, OMe), 4.71 [t, 1 H, C_6H_4, J(HH) 6], 4.89 [d, 1 H, C_6H_4, J(HH) 6], 5.51 [t, 1 H, C_6H_4, J(HH) 6], 5.62 (s, 5 H, C_5H_5), 5.83 [d, 1 H, C_6H_4, J(HH) 6]	295.6 (C≡Mo), 232.0 (CrCO), 228.7, 228.3 (MoCO), 143.7 [$C^1(C_6H_4)$], 99.8, 97.6, 93.3 (C_6H_4), 93.0 (C_5H_5), 83.3, 72.2 (C_6H_4), 55.9 (OMe)
(3b)	3.77 (s, 3 H, OMe), 4.77 [t, 1 H, C_6H_4, J(HH) 6], 4.90 [d, 1 H, C_6H_4, J(HH) 7], 5.51 [t, 1 H, C_6H_4, J(HH) 6], 5.69 (s, 5 H, C_5H_5), 5.80 [d, 1 H, C_6H_4, J(HH) 6]	282.9 (C≡W), 231.1 (CrCO), 218.9, 218.4 (WCO), 141.9 [$C^1(C_6H_4)$], 104.5, 96.4, 92.6 (C_6H_4), 91.2 (C_5H_5), 83.0, 72.2 (C_6H_4), 55.6 (OMe)
(3c)	3.82 (s, 3 H, OMe), 4.81 (m br, 1 H, C_6H_4), 4.93 [d, 1 H, C_6H_4, J(HH) 7], 5.51 [t, 1 H, C_6H_4, J(HH) 6], 5.83 (m br, 1 H, C_6H_4), 6.21 (m, 3 H, $C_3H_3N_2$), 7.59—7.65 (m, 4 H, $C_3H_3N_2$), 7.92 (m, 2 H, $C_3H_3N_2$)	278.1 (C≡Mo), 232.5 (CrCO), 225.7, 225.5 (MoCO), 144.4 ($C_3H_3N_2$), 142.9 [$C^1(C_6H_4)$], 135.5, 135.4, 105.3 ($C_3H_3N_2$), 99.5, 97.0, 93.0, 83.1, 72.3 (C_6H_4), 55.8 (OMe)
(4a)	3.81 (s, 6 H, OMe), 4.76 [t, 2 H, C_6H_4, J(HH) 6], 5.06 [d, 2 H, C_6H_4, J(HH) 6], 5.65 (s, 10 H, C_5H_5), 5.68 [t, 2 H, C_6H_4, J(HH) 6], 6.02 [d, 2 H, C_6H_4, J(HH) 6]	309.1 [μ-C, J(PtC) 790], 232.8 (CrCO), 206.9 (MoCO), 141.2 [$C^1(C_6H_4)$], 107.3, 96.8, 94.7 (C_6H_4), 94.4 (C_5H_5), 83.7, 73.4 (C_6H_4), 55.9 (OMe)
(4b)	3.77 (s, 6 H, OMe), 4.73—4.85, 4.95—5.05 (m × 2, 4 H, C_6H_4), 5.45 (s, 10 H, C_5H_5), 5.55—5.72, 6.03—6.19 (m × 2, 4 H, C_6H_4)	312.8 (μ-C), 233.4 (CrCO), 232.4, 228.4 (MoCO), 139.5 [$C^1(C_6H_4)$], 111.8, 95.4 (C_6H_4), 94.2 (C_5H_5), 93.8, 82.9, 72.9 (C_6H_4), 54.4 (OMe)
(4c)	3.74 (s, 6 H, OMe), 4.73—4.81, 4.92—5.04 (m × 2, 4 H, C_6H_4), 5.45 (s, 10 H, C_5H_5), 5.51—5.76, 5.90—6.03 (m × 2, 4 H, C_6H_4)	296.5 (μ-C), 233.7 (CrCO), 229.5, 222.8 (WCO), 140.1 [$C^1(C_6H_4)$], 106.7, 95.1, 94.3 (C_6H_4), 92.8 (C_5H_5), 83.6, 73.2 (C_6H_4), 55.6 (OMe)
(5a)	3.76 (s, 3 H, OMe), 4.90 [t, 1 H, C_6H_4, J(HH) 6], 5.13 [d, 1 H, C_6H_4, J(HH) 6], 5.28 (s, 5 H, C_5H_5), 5.65 [t, 1 H, C_6H_4, J(HH) 6], 6.08 [d, 1 H, C_6H_4, J(HH) 6]	[c] 266.1 (μ-C), 232.9 (CrCO), 202.5—200.4 (br, MoCO, CoCO), 136.8 [$C^1(C_6H_4)$], 122.9, 98.9, 94.2 (C_6H_4), 92.7 (C_5H_5), 82.7, 71.4 (C_6H_4), 54.4 (OMe)
(5b)	3.76 (s, 3 H, OMe), 4.86 [t, 1 H, C_6H_4, J(HH) 6], 5.09 [d, 1 H, C_6H_4, J(HH) 6], 5.30 (s, 5 H, C_5H_5), 5.60 [t, 1 H, C_6H_4, J(HH) 6], 6.06 [d, 1 H, C_6H_4, J(HH) 6]	[c] 257.0 (μ-C), 232.9 (CrCO), 201.7—199.7 (br, WCO, CoCO), 136.8 [$C^1(C_6H_4)$], 124.8, 99.4, 94.1 (C_6H_4), 92.7 (C_5H_5), 82.9, 71.8 (C_6H_4), 54.4 (OMe)
(6)	3.07—3.11 (m, 2 H, CH_2P), 3.38 (s, 3 H, OMe), 4.60—4.82 (m, 1 H, C_6H_4), 5.29—5.33 (m, 1 H, C_6H_4), 5.45 (s, 5 H, C_5H_5), 5.50—5.66 (m, 1 H, C_6H_4), 5.93—6.05 (m, 1 H, C_6H_4), 7.02—7.68 (m, 20 H, Ph)	253.1 (μ-C), 234.6 (CrCO), 211.6—209.5 (br, MoCO, CoCO), 138.5—127.7 [$C^1(C_6H_4)$, Ph], 125.3, 100.1, 94.8 (C_6H_4), 92.9 (C_5H_5), 83.4, 74.5 (C_6H_4), 53.3 (OMe), 29.6 (m, CH_2)
(7a)	3.81 (s, 3 H, OMe), 4.82 [t, 1 H, C_6H_4, J(HH) 6], 5.07 [d, 1 H, C_6H_4, J(HH) 7], 5.55 (s, 5 H, C_5H_5), 5.57—5.61 (m, 1 H, C_6H_4), 5.72—5.85 (m, 4 H, C_6H_4, C_9H_7), 6.96—7.10 (m, 4 H, C_9H_7)	312.9 [d, μ-C, J(RhC) 29], 233.5 (CrCO), 231.3, 226.2 (MoCO), 188.7 [d, RhCO, J(RhC) 88], 141.1 [$C^1(C_6H_4)$], 126.5—94.9 (C_6H_4, C_9H_7), 94.3 (C_5H_5), 84.8—72.6 (C_6H_4, C_9H_7), 55.6 (OMe)
(7b)	3.81 (s, 3 H, OMe), 4.69—4.75, 4.85—5.07 (m × 2, 2 H, C_6H_4), 5.55—5.85 (m, 5 H, C_9H_7, C_6H_4), 5.59 (s, 5 H, C_5H_5), 6.94—7.07 (m, 4 H, C_9H_7)	288.7 [d, μ-C, J(RhC) 27], 233.8 (CrCO), 230.9, 226.7 (WCO), 189.5 [d, RhCO, J(RhC) 88], 141.2 [$C^1(C_6H_4)$], 126.6—94.0 (C_6H_4, C_9H_7), 93.1 (C_5H_5), 84.1—72.6 (C_6H_4, C_9H_7), 55.7 (OMe)

[a] Chemical shifts (δ) in p.p.m., coupling constants in Hz, measurements at room temperature in $CDCl_3$ unless otherwise stated. [b] Hydrogen-1 decoupled, chemical shifts are positive to high frequency of $SiMe_4$. [c] Measured in CD_2Cl_2–CH_2Cl_2 at −50 °C.

water-cooled silica gel column (3 × 50 cm). Elution with light petroleum–CH_2Cl_2 (2:3) removed a yellow fraction containing unreacted $[Cr(CO)_3(\eta^6\text{-}C_6H_5OMe)]$ (0.40 g). Elution with thf removed a red eluate. The latter was concentrated *in vacuo* to *ca.* 20 cm^3, and cooled to −78 °C to afford orange *microcrystals* of $[CrMo\{\mu\text{-}\sigma{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(O_2CCF_3)(CO)_5(tmen)]$ **(2a)** (4.51 g).

A similar procedure was used to prepare the compounds $[CrM\{\mu\text{-}\sigma{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(O_2CCF_3)(CO)_5L_2]$ [L_2 = bipy, M = Mo **(2b)** or W **(2d)**; L_2 = tmen, M = W **(2c)**]. The quantities of product obtained were 5.3 g **(2b)**, 5.5 g **(2c)**, and 6.2 g **(2d)** using 10 mmol of the various reagents, as for **(2a)**.

Synthesis of the Complexes $[CrM\{\mu\text{-}\sigma{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_5(\eta\text{-}C_5H_5)]$ (M = Mo *or* W).—Solid NaC_5H_5·dme (dme = 1,2-dimethoxyethane)[12] (0.85 g, 6.0 mmol) was added to a suspension of **(2b)** (3.38 g, 5.0 mmol) in Et_2O (50 cm^3) at room temperature. The mixture was stirred for 30 min, and then filtered through an alumina pad (3 × 3 cm). Solvent was removed *in vacuo*, and the residue was dissolved in light petroleum–CH_2Cl_2 (1:1, 5 cm^3) and chromatographed on a water-cooled alumina column (3 × 80 cm). Elution with light petroleum (100 cm^3) afforded a colourless eluate containing bipy (0.39 g). Elution with light petroleum–CH_2Cl_2 (1:1) gave an orange-red eluate. Reduction in volume of the latter *in vacuo* to *ca.* 15 cm^3, and cooling to −78 °C, gave red *microcrystals* of $[CrMo\{\mu\text{-}\sigma\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_5(\eta\text{-}C_5H_5)]$ (**3a**) (1.08 g).

The compound $[CrW\{\mu\text{-}\sigma\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_5(\eta\text{-}C_5H_5)]$ (**3b**) (1.42 g) was similarly prepared from (**2d**) (5 mmol) and $NaC_5H_5\cdot dme$ (6 mmol).

Synthesis of the Complex $[CrMo\{\mu\text{-}\sigma\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_5\{HB(pz)_3\}]$.—Solid $K[HB(pz)_3]$ (1.25 g, 5.0 mmol) was added to a CH_2Cl_2 (40 cm^3) solution of (**2a**) (3.38 g, 5 mmol) at room temperature. After stirring the mixture for 24 h, solvent was removed *in vacuo*, and the residue was treated with Et_2O (50 cm^3). The resulting suspension was filtered through an alumina pad (3 × 3 cm). Solvent was removed *in vacuo*, and the residue was dissolved in light petroleum–CH_2Cl_2 (1:1, 8 cm^3) and chromatographed on a water-cooled alumina column (3 × 50 cm). Elution with the same solvent mixture afforded, after removal of solvent *in vacuo*, orange *microcrystals* of $[CrMo\{\mu\text{-}\sigma\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_5\{HB(pz)_3\}]$ (**3c**) (2.88 g).

Synthesis of the Complexes $[Cr_2M_2M'\{\mu\text{-}\sigma,\sigma'\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}_2(CO)_{10}(\eta\text{-}C_5H_5)_2]$.—(*i*) A light petroleum (20 cm^3) solution of $[Pt(C_2H_4)_3]$ at 0 °C was prepared *in situ* from $[Pt(cod)_2]$ (0.42 mmol).[20] This solution was transferred *via* a canula, using ethylene pressure, into a suspension of (**3a**) (0.40 g, 0.85 mmol) in light petroleum (40 cm^3) at 0 °C. The resulting mixture was warmed to room temperature, and after 30 min the solvent was removed *in vacuo*. The residue was dissolved in light petroleum–CH_2Cl_2 (7:3, 15 cm^3) and chromatographed on a water-cooled alumina column (3 × 30 cm). Elution with the same solvent mixture yielded a purple eluate. The latter was concentrated *in vacuo* to *ca.* 20 cm^3, and cooled to −78 °C to give red-purple *microcrystals* of $[Cr_2Mo_2Pt\{\mu\text{-}\sigma,\sigma'\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}_2(CO)_{10}(\eta\text{-}C_5H_5)_2]$ (**4a**) (0.31 g).

(*ii*) Solid $[Ni(cod)_2]$ (0.12 g, 0.42 mmol) was added to a suspension of (**3a**) (0.40 g, 0.84 mmol) in light petroleum (70 cm^3) at 0 °C. The mixture was warmed to room temperature and stirred for 6 h. Solvent was removed *in vacuo*, and the residue was dissolved in light petroleum–CH_2Cl_2 (7:3, 15 cm^3) and chromatographed on a water-cooled alumina column (3 × 30 cm). Elution with the same solvent mixture removed a brown eluate. The latter was concentrated *in vacuo* to *ca.* 15 cm^3 and cooled to −78 °C to afford brown *microcrystals* of $[Cr_2Mo_2Ni\{\mu\text{-}\sigma,\sigma'\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}_2(CO)_{10}(\eta\text{-}C_5H_5)_2]$ (**4b**) (0.19 g).

(*iii*) In a similar manner, addition of $[Ni(cod)_2]$ (0.10 g, 0.35 mmol) to (**3b**) (0.39 g, 0.70 mmol) in light petroleum (50 cm^3) afforded, after chromatography, brown *microcrystals* of $[Cr_2W_2Ni\{\mu\text{-}\sigma,\sigma'\text{:}\eta^6\text{-}C_6H_4(OMe\text{-}2)\}_2(CO)_{10}(\eta\text{-}C_5H_5)_2]$ (**4c**) (0.17 g).

Reactions with Octacarbonyldicobalt.—(*i*) Solid $[Co_2(CO)_8]$ (0.10 g, 0.29 mmol) was added to an Et_2O (30 cm^3) solution of (**3a**) (0.14 g, 0.29 mmol) at room temperature. After stirring for 40 min, solvent was removed *in vacuo* and the residue was dissolved in light petroleum–CH_2Cl_2 (7:3, 8 cm^3) and chromatographed on a water-cooled alumina column (3 × 30 cm). Elution with the same solvent mixture yielded a brown eluate. The latter was reduced in volume *in vacuo* to *ca.* 15 cm^3 and cooled to −78 °C to give brown *microcrystals* of $[CrMoCo_2\{\mu_3\text{-}\sigma,\sigma',\sigma''\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_{11}(\eta\text{-}C_5H_5)]$ (**5a**) (0.16 g).

The dppm derivative (**6**) of compound (**5a**) was obtained in the following manner. Solid dppm (0.11 g, 0.30 mmol) was added to a CH_2Cl_2 (30 cm^3) solution of (**5a**) (0.18 g, 0.25 mmol) at room temperature, and the mixture was stirred for 6 h. Solvent was removed *in vacuo*, and the residue was dissolved in light petroleum–CH_2Cl_2 (2:3, 8 cm^3) and chromatographed on alumina. Elution with the same solvent mixture gave a brown solution, which was reduced in volume *in vacuo* to *ca.* 15 cm^3 and cooled to −78 °C to give dark brown *microcrystals* of $[CrMoCo_2\{\mu_3\text{-}\sigma,\sigma',\sigma''\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(\mu\text{-}dppm)(CO)_9(\eta\text{-}C_5H_5)]$ (**6**) (0.26 g). This product was also prepared by irradiating a mixture of (**5a**) and dppm in thf with u.v. light (500-W medium-pressure Hanovia mercury vapour lamp).

(*ii*) The compound $[CrWCo_2\{\mu_3\text{-}\sigma,\sigma',\sigma''\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_{11}(\eta\text{-}C_5H_5)]$ (**5b**) (0.17 g) was prepared from (**3b**) (0.15 g, 0.26 mmol) and $[Co_2(CO)_8]$ (0.09 g, 0.26 mmol) in a similar manner to that used to obtain (**5a**).

Synthesis of the Complexes $[CrMRh\{\mu\text{-}\sigma,\sigma'\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_6(\eta\text{-}C_5H_5)(\eta^5\text{-}C_9H_7)]$.—(*i*) Carbon monoxide was bubbled (*ca.* 10 min) through a light petroleum (30 cm^3) solution of $[Rh(\eta\text{-}C_2H_4)_2(\eta^5\text{-}C_9H_7)]$ (0.17 g, 0.62 mmol) at 0 °C to generate $[Rh(CO)_2(\eta^5\text{-}C_9H_7)]$ *in situ*.[7a] Solid (**3a**) (0.29 g, 0.62 mmol) was added, and the mixture was warmed to room temperature and stirred for 30 min. Solvent was removed *in vacuo*, and the residue was dissolved in light petroleum–CH_2Cl_2 (7:3, 10 cm^3) and chromatographed on a water-cooled alumina column (3 × 60 cm). Elution with the same solvent mixture afforded a brown eluate. The latter was reduced in volume to *ca.* 20 cm^3 and cooled to −78 °C to give brown *microcrystals* of $[CrMoRh\{\mu\text{-}\sigma,\sigma'\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_6(\eta\text{-}C_5H_5)(\eta^5\text{-}C_9H_7)]$ (**7a**) (0.29 g).

(*ii*) The compound $[CrWRh\{\mu\text{-}\sigma,\sigma'\text{:}\eta^6\text{-}CC_6H_4(OMe\text{-}2)\}(CO)_6(\eta\text{-}C_5H_5)(\eta^5\text{-}C_9H_7)]$ (**7b**) (0.45 g) was prepared in a similar manner from (**3b**) (0.43 g, 0.77 mmol) and $[Rh(\eta\text{-}C_2H_4)_2(\eta^5\text{-}C_9H_7)]$ (0.21 g, 0.77 mmol) treated with CO.

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References

1 Part 82, A. F. Hill, H. D. Hönig, and F. G. A. Stone, *J. Chem. Soc., Dalton Trans.*, preceding paper.

2 F. G. A. Stone, in 'Inorganic Chemistry: Toward the 21st Century,' ed. M. H. Chisholm, *A.C.S. Symp. Ser.*, 1983, **211**, 383.

3 E. O. Fischer, T. L. Lindner, G. Huttner, P. Friedrich, F. R. Kreissl, and J. O. Besenhard, *Chem. Ber.*, 1977, **110**, 3397.

4 R. Hoffmann, *Angew. Chem., Int. Ed. Engl.*, 1982, **21**, 711; F. G. A. Stone, *ibid.*, 1984, **23**, 89.

5 S. J. Dossett, A. F. Hill, J. C. Jeffery, F. Marken, P Sherwood, and F. G. A. Stone, *J. Chem. Soc., Dalton Trans.*, 1988, 2453.

6 (*a*) G. P. Elliott, J. A. K. Howard, T. Mise, I. Moore, C. M. Nunn, and F. G. A. Stone, *J. Chem. Soc., Dalton Trans.*, 1986, 2091; (*b*) E. Delgado, J. Hein, J. C. Jeffery, A. L. Ratermann, F. G. A. Stone, and L. J. Farrugia, *ibid.*, 1987, 1191; (*c*) M. D. Bermúdez, E. Delgado, G. P. Elliott, N. H. Tran-Huy, F. Major-Real, F. G. A. Stone, and M. J. Winter, *ibid.*, p. 1235; (*d*) J. Hein, J. C. Jeffery, P. Sherwood, and F. G. A. Stone, *ibid.*, p. 2211.

7 (*a*) M. Green, J. A. K. Howard, A. P. James, C. M. Nunn, and F. G. A. Stone, *J. Chem. Soc., Dalton Trans.*, 1986, 187; (*b*) M. Green, J. A. K. Howard, A. P. James, A. N. de M. Jelfs, C. M. Nunn, and F. G. A. Stone, *ibid.*, p. 1697; (*c*) S. V. Hoskins, A. P. James, J. C. Jeffery, and F. G. A. Stone, *ibid.*, p. 1709; (*d*) S. H. F. Becke, M. D. Bermúdez, N. H. Tran-Huy, J. A. K. Howard, O. Johnson, and F. G. A. Stone, *ibid.*, 1987, 1229.

8 M. D. Bermúdez, F. P. E. Brown, and F. G. A. Stone, *J. Chem. Soc., Dalton Trans.*, 1988, 1139.

9 M. J. Attfield, J. A. K. Howard, A. N. de M. Jelfs, C. M. Nunn, and F. G. A. Stone, *J. Chem. Soc., Dalton Trans.*, 1987, 2219; F-E. Baumann, J. A. K. Howard, R. J. Musgrove, P. Sherwood, and F. G. A. Stone, *ibid.*, 1988, 1891 and refs. therein; F-E. Baumann, J. A. K. Howard, R. J. Musgrove, P. Sherwood, M. A. Ruiz, and F. G. A. Stone, *J. Chem. Soc., Chem. Commun.*, 1987, 1881.

10 G. A. McDermott, A. M. Dorries, and A. Mayr, *Organometallics*, 1987, **6**, 925.

11 M. F. Semmelhack, J. Bisaha, and N. Czarny, *J. Am. Chem. Soc.*, 1979, **101**, 768.

12 J. C. Smart and C. J. Curtis, *Inorg. Chem.*, 1977, **16**, 1788.

13 T. V. Ashworth, M. J. Chetcuti, J. A. K. Howard, F. G. A. Stone, S. J. Wisbey, and P. Woodward, *J. Chem. Soc., Dalton Trans.*, 1981, 763; J. C. Jeffery, D. B. Lewis, G. E. Lewis, M. J. Parrott, and F. G. A. Stone, *ibid.*, 1986, 1717.

14 M. J. Chetcuti, P. A. M. Chetcuti, J. C. Jeffery, R. M. Mills, P. Mitrprachachon, S. J. Pickering, F. G. A. Stone, and P. Woodward, *J. Chem. Soc., Dalton Trans.*, 1982, 699.

15 J. A. Abad, E. Delgado, M. E. Garcia, M. J. Grosse-Ophoff, I. J. Hart, J. C. Jeffery, M. S. Simmons, and F. G. A. Stone, *J. Chem. Soc., Dalton Trans.*, 1987, 41.

16 J. A. Abad, L. W. Bateman, J. C. Jeffery, K. A. Mead, H. Razay, F. G. A. Stone, and P. Woodward, *J. Chem. Soc., Dalton Trans.*, 1983, 2075.

17 J. C. Jeffery, C. Sambale, M. F. Schmidt, and F. G. A. Stone, *Organometallics*, 1982, **1**, 1597.

18 J. C. Jeffery, M. J. Parrott, and F. G. A. Stone, *J. Chem. Soc., Dalton Trans.*, 1988, 3017.

19 C. A. L. Mahaffy and P. L. Pauson, *Inorg. Synth.*, 1979, **19**, 155.

20 J. L. Spencer, *Inorg. Synth.*, 1979, **19**, 213.

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