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Synthesis and spectroscopic properties of the binuclear pentacarbonylcyclopentadienylcycloheptatrienyl complexes $[M(CO)_3(\eta\text{-}C_5R_5)M'(CO)_2(\eta\text{-}C_7H_7)]$ (M,M' = Mo or W; R = H or Me)

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Abstract

A series of metal–metal bonded complexes $[M(CO)_3(\eta\text{-}C_5R_5)\ M'(CO)_2(\eta\text{-}C_7H_7)]$ (**1**: M = M' = Mo, R = H; **2**: M = M' = Mo, R = Me; **3**: M = M' = W, R = H; **4**: M = W, M' = Mo, R = H) have been synthesised from the dicarbonylacetonitrile cations $[M'(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ (M' = Mo or W) and the appropriate anion $Li[M(CO)_3(\eta\text{-}C_5R_5)]$ (M = Mo or W, R = H or Me). Infrared, electronic absorption, and NMR spectra of complexes **1** to **4** are discussed; variable temperature ^{13}C NMR studies on **2** advocate an intermetallic carbonyl scrambling process.

Although there is an extensive chemistry of metal–metal bonded dimolybdenum and ditungsten complexes which contain the fragment $M(CO)_n(\eta$-cyclopentadienyl) (M = Mo or W, $n = 2$ or 3) [1], the corresponding chemistry of the $M'(CO)_2(\eta$-cycloheptatrienyl) moiety (M' = Mo or W) has been restricted to reports of heterobinuclear $[Mo(CO)_2(\eta\text{-}C_7H_7)Mn(CO)_5]$ [2] and $[Mo(CO)_2(\eta\text{-}C_7H_7)Fe(CO)_2(\eta\text{-}C_5H_5)]$ [3]. (To our knowledge no characterisation data have been reported for the latter example.) One constraint on the synthesis of such binuclear dicarbonylcycloheptatrienyl complexes of molybdenum and tungsten is that reaction of anionic reagents with $[M'(CO)_3(\eta\text{-}C_7H_7)]^+$ (M' = Mo or W) or, in some cases, even with $[MoBr(CO)_2(\eta\text{-}C_7H_7)]$, leads to formation of the ligand-bridged dimers $[M'_2(CO)_6(\eta^6,\eta^6\text{-}C_{14}H_{14})]$ ($C_{14}H_{14}$ = ditropyl) by reductive coupling of two cycloheptatrienyl rings [4,5]. Thus whilst $[M(CO)_3(\eta\text{-}C_5H_5)]^-$ (M = Mo or W) reacts with $[Co(CO)_3(\eta^4$-tetramethylcyclobutadiene)$][PF_6]$ to give metal–metal bonded heterobinuclear products by displacement of CO [6], the corresponding reaction of $[M(CO)_3(\eta\text{-}C_5H_5)]^-$ with $[M'(CO)_3(\eta\text{-}C_7H_7)]^+$ (M' = Mo or W) gives only $[\{M(CO)_3(\eta\text{-}C_5H_5)\}_2]$ and $[M'_2(CO)_6(\eta^6,\eta^6\text{-}C_{14}H_{14})]$ [4]. We have recently re-

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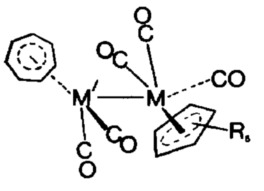
ported the synthesis of the dicarbonylacetonitrile cations $[M'(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ ($M' = Mo$ or W) [7,8], which because of facile displacement of acetonitrile from the metal centre are much more versatile synthetic reagents than the corresponding tricarbonyl cations $[M'(CO)_3(\eta\text{-}C_7H_7)]^+$. In this paper we describe the application of $[M'(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ in the synthesis of metal–metal bonded, asymmetric homobinuclear complexes; a brief account has appeared previously [7].

Results and discussion

Treatment of a thf solution of $[Mo(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ with $Li[Mo(CO)_3(\eta\text{-}C_5H_5)$, or alternatively $Na[Mo(CO)_3(\eta\text{-}C_5H_5)] \cdot 2dme$ (dme = dimethoxyethane), gives a dark green mixture which, after work-up to remove small quantities of $[\{Mo(CO)_3(\eta\text{-}C_5H_5)\}_2]$, affords moderate yields of $[Mo(CO)_3(\eta\text{-}C_5H_5)Mo(CO)_2(\eta\text{-}C_7H_7)]$ (**1**). Complex **1** is also formed by reaction of $[MoCl(CO)_2(\eta\text{-}C_7H_7)]$ with $Na[Mo(CO)_3(\eta\text{-}C_5H_5)] \cdot 2dme$ in thf but this route proved less satisfactory because of difficulty of separating **1** from the larger amounts of $[\{Mo(CO)_3(\eta\text{-}C_5H_5)\}_2]$ also produced. Details of elemental analysis, infrared, UV/visible, and mass spectra of **1** are shown in Table 1, and the ^1H and ^{13}C NMR data in Table 2. The detailed molecular structure of **1** (Fig. 1) has been established by an X-ray crystallographic study [9].

The generality of our route to **1** is demonstrated by the synthesis of a series of analogous binuclear complexes. Thus treatment of $[Mo(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ with $Li[Mo(CO)_3(\eta\text{-}C_5Me_5)]$ affords the pentamethylcyclopentadienyl derivative $[Mo(CO)_3(\eta\text{-}C_5Me_5)Mo(CO)_2(\eta\text{-}C_7H_7)]$ (**2**), and the synthesis of the ditungsten derivative $[W(CO)_3(\eta\text{-}C_5H_5)W(CO)_2(\eta\text{-}C_7H_7)]$ (**3**) proceeds from $[W(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ and $Li[W(CO)_3(\eta\text{-}C_5H_5)]$. We have also obtained one example of a heterobinuclear complex $[W(CO)_3(\eta\text{-}C_5H_5)Mo(CO)_2(\eta\text{-}C_7H_7)]$ (**4**) starting from $[Mo(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ and $Li[W(CO)_3(\eta\text{-}C_5H_5)]$. Full spectroscopic characterisation for each of complexes **2**, **3** and **4** are given in Tables 1 and 2.

Several points of interest arise from a comparison of spectroscopic data for complexes **1** to **4** with those for the symmetric homobinuclear species $[\{M(CO)_3(\eta\text{-}C_5H_5)\}_2]$ (M = Mo or W). The infrared spectra obtained for hexane solutions of **1**,



(1) M = M′ = Mo, R = H

(2) M = M′ = Mo, R = Me

(3) M = M′ = W , R = H

(4) M = W, M′ = Mo, R = H

Table 1

Yield, microanalytical, infrared, UV/visible, and mass spectral data

Complex	Yield (%)	Analysis [a] (%)		ν(CO) [b] (cm^{-1})	UV/visible [c] (nm)	Mass spectrum [d]
		C	H			
1	41	41.3 (41.8)	2.6 (2.5)	1980(w), 1942(s) 1932(m), 1874(w), 1856(m)	306 (22500) 343 (sh) 435 (4700) 665 (2300)	432 M^+ − 2CO 348 M^+ − 5CO
2	32	46.9 (47.3)	4.1 (3.9)	1973(w), 1922(s) 1845(m)	307 (24000) 373 (sh) 458 (4400) 682 (2800)	502 M^+ − 2CO
3	31	31.0 (30.7)	2.0 (1.8)	1973(w), 1941(s), 1914(m), 1873(w) 1854(m)	287 (21100) 328 (12900) 398 (5900) 669 (2100)	608 M^+ − 2CO 524 M^+ − 5CO
4	23	35.6 (35.4)	2.1 (2.1)	1978(w), 1940(s), 1928(m), 1870(w), 1851(m)	299 (24000) 340 (sh) 417 (4600) 665 (2100)	520 M^+ − 2CO 436 M^+ − 5CO

[a] Calculated values are given in parentheses, **1** Mo Found 38.7%, calcd. 39.3%, **2** Mo Found 34.7%, calcd. 34.4%. [b] In hexane; s, strong; m, medium; w, weak intensity. [c] In thf at 293 K, extinction coefficients in parentheses; sh, shoulder. [d] By electron impact mass spectroscopy.

3 and **4** each exhibit five carbonyl stretching frequencies, consistent with predictions based upon the C_s symmetry of the molecules. By contrast the corresponding infrared spectrum of **2** shows only three carbonyl stretching frequencies, yet the identity of **2** as a pentacarbonyl complex with a molecular structure comparable to that of **1** has been established by elemental analysis for molybdenum, and by the close correspondence of the low temperature ^{13}C NMR data for **1** and **2** (see later). We have no definitive explanation for the apparently anomalous infrared spectrum of **2**, but we note that the solid state structure of **1** has four carbonyls located *cis* to the Mo–Mo bond, as found for [{Mo(CO)$_3$(η-C$_5$H$_5$)}$_2$], and that the presence of

Table 2

Proton and ^{13}C NMR spectral data [a]

Complex	^{1}H (δ) [b]	^{13}C (ppm) [c]
1	5.35(s, 7H, C$_7$H$_7$), 5.16(s, 5H, C$_5$H$_5$)	237.8, 228.9, 218.4 (CO), 93.3 (C$_7$H$_7$), 92.1 (C$_5$H$_5$) [d]
2	5.30(s, 7H, C$_7$H$_7$), 1.87(s, 15H, C$_5$Me$_5$)	240.3, 233.6, 220.6 (CO), 102.9 (C_5Me$_5$), 93.0 (C$_7$H$_7$), 10.6 (C$_5\mathit{Me}_5$) [e,f]
3	5.33(s, 5H, C$_5$H$_5$), 5.25(s, 7H, C$_7$H$_7$)	225.9, 216.7, 207.8 (CO), 91.7 (C$_5$H$_5$), 89.4 C($_7$H$_7$)
4	5.38(s, 7H, C$_7$H$_7$), 5.26(s, 5H, C$_5$H$_5$)	226.4, 219.2, 218.0 (CO), 93.8 (C$_7$H$_7$), 91.1 (C$_5$H$_5$)

[a] Chemical shifts downfield from SiMe$_4$, ambient temperature spectra unless stated otherwise, s = singlet. [b] In CDCl$_3$. [c] In CD$_2$Cl$_2$. [d] Recorded at −40 °C. [e] Recorded at −80 °C. [f] Spectrum also recorded at +40 °C in CDCl$_3$, 230.5 (CO), 103.9 (C_5Me$_5$), 93.3 (C$_7$H$_7$), 11.1 (C$_5\mathit{Me}_5$).

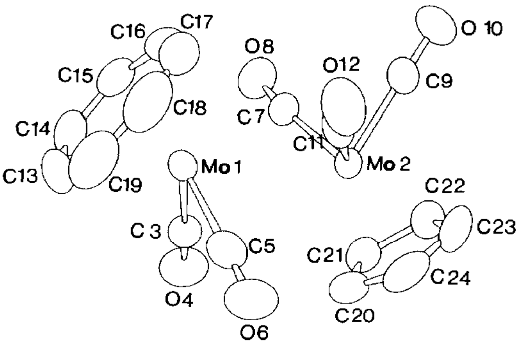


Fig. 1. The molecular structure of [Mo(CO)$_3$(η-C$_5$H$_5$)Mo(CO)$_2$(η-C$_7$H$_7$)] (**1**).

rotamers of **1**, **3** and **4** could account for our observations. In a study analogous to that carried out on [{Mo(CO)$_3$(η-C$_5$H$_5$)}$_2$] [10], the infrared active carbonyl stretching frequencies of **1** and **2** were examined in solvents of differing dielectric constant; no significant changes were observed, although the interpretation of results in polar solvents such as CH$_2$Cl$_2$ or acetone was hindered by loss of resolution.

The wavenumbers observed for the carbonyl stretching frequencies of complexes **1** to **4** in solution are broadly consistent with the presence of all terminal carbonyls, in accord with the solid state structure of **1**. It is notable however that carbonyls C(7)–O(8) and C(11)–O(12) (Fig. 1), with an average bridge asymmetry $(D_2-D_1)/D_1$ (Fig. 2) [11] of 0.52, are positioned more symmetrically with respect to the metal–metal bond than the corresponding carbonyls in [{Mo(CO)$_3$(η-C$_5$H$_5$)}$_2$] for which $(D_2-D_1)/D_1 = 0.61$ [11]. The average bridge asymmetry of carbonyls C(3)–O(4) and C(5)–O(6), which are bonded to the cycloheptatrienylmolybdenum fragment, is calculated to be 0.66.

The electronic absorption spectra of complexes **1** to **4** were recorded between 250 and 800 nm in thf. The spectrum at 250 to 500 nm compares closely with those reported for [{M(CO)$_3$(η-C$_5$H$_5$)}$_2$] (M = Mo or W) [12], and the strong, lowest wavelength absorption may be tentatively assigned to a $\sigma \rightarrow \sigma^*$ transition of the metal–metal bond [13]. Features of note are the shift to lower wavelength of the presumed $\sigma \rightarrow \sigma^*$ transition in **1** and **3** compared with that for [{Mo(CO)$_3$(η-C$_5$H$_5$)}$_2$] and [{W(CO)$_3$(η-C$_5$H$_5$)}$_2$], respectively, and the weak absorption which appears as a shoulder to high wavelength of the $\sigma \rightarrow \sigma^*$ transition for all the complexes **1** to **4**.

The molecular ions of complexes **1** to **4** were not observed in electron impact mass spectrometry, but in each case groups of peaks corresponding to $M^+ - 2$CO and $M^+ - 5$CO were present; Table 1 shows the individual peaks of highest intensity. Results for complex **2** were slightly anomalous in that the highest intensity peak in the group attributed to $M^+ - 5$CO had $m/e = 416$.

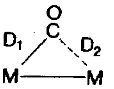


Fig. 2. Diagram for definition of bridge asymmetry.

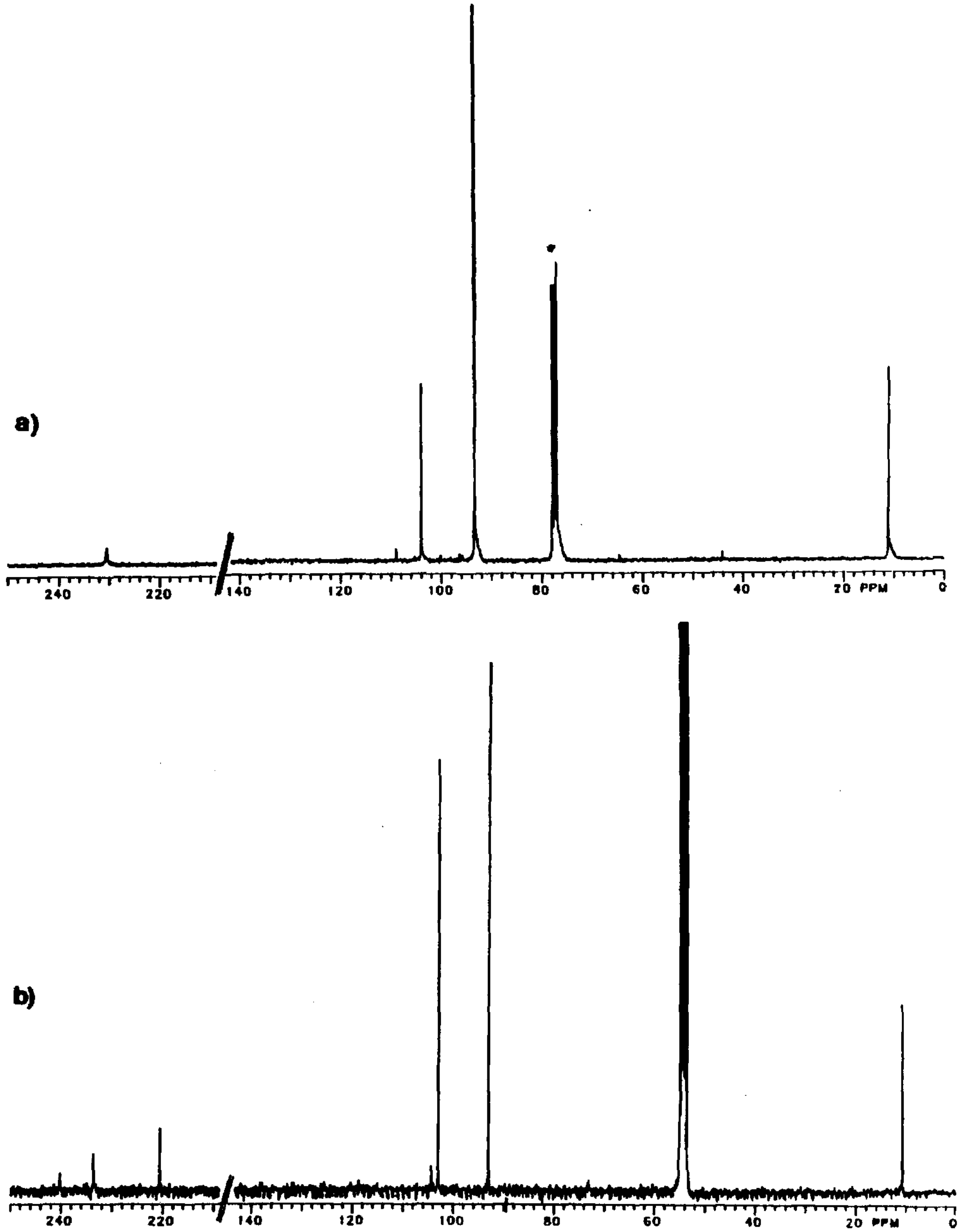


Fig. 3. The variable temperature 75 MHz ^{13}C NMR spectrum of $[Mo(CO)_3(\eta\text{-}C_5Me_5)Mo(CO)_2(\eta\text{-}C_7H_7)]$ (2) (a) +40 °C in $CDCl_3$, (b) −80 °C in CD_2Cl_2, * = solvent.

The ^1H and ^{13}C NMR spectra of complexes **1** to **4** in each case establish the presence of discrete cyclopentadienyl and cycloheptatrienyl rings. Furthermore the carbonyl carbon regions of the ^{13}C NMR spectra of **1** and **2** exhibit variable temperature behaviour; spectra observed for **2** are shown in Fig. 3. At −80 °C in CD_2Cl_2 three sharp singlets are observed for the carbonyl resonances of **2**, and comparisons of data obtained for $[M(CO)_3(\eta\text{-}C_5R_5)M'(CO)_2(\eta\text{-}C_7H_7)]$ and

$[\{M(CO)_3(\eta\text{-}C_5H_5)\}_2]$ (M = Mo [14], M = W *), suggests assignment of the two low field carbonyl resonances in **1** to **3** to the tricarbonylcyclopentadienylmetal fragment (the carbonyl *trans* to the metal–metal bond is identified with the smaller, lower field signal), whilst the third, high field carbonyl resonance arises from the dicarbonylcycloheptatrienylmetal fragment. When the solution in CD_2Cl_2 is warmed to +20 °C, the carbonyl region of **2** coalesces to a singlet signal with a chemical shift of 230.6 ppm (in good agreement with the averaged chemical shift of the resonances observed at −80 °C). Elevated temperature studies in $CDCl_3$ revealed sharpening of this singlet carbonyl resonance, but at temperatures above +40 °C there was extensive decomposition. We suggest that the spectra observed are consistent with an intermetallic carbonyl scrambling process in **2** similar to that described for the binuclear isocyanide complex $[Mo(CO)_3(\eta\text{-}C_5H_5)Mo(CO)_2(CNMe)(\eta\text{-}C_5H_5)]$ [15]. Two processes are involved: (i) local scrambling of carbonyls situated *cis* and *trans* to the metal–metal bond in the tricarbonylpentamethylcyclopentadienylmolybdenum unit, and (ii) internuclear exchange of the four carbonyls located *cis* to the metal–metal bond.

Similar fluxionality probably operates for the unsubstituted cyclopentadienyl ring complex **1** but with the activation energies higher than for **2** (ring-substitution dependent activation energies for carbonyl scrambling have been reported previously [16]). Thus the ambient temperature ^{13}C NMR spectrum of **1** exhibits three very broad carbonyl resonances, which at −40 °C appear as sharp signals corresponding to those obtained for **2** at −80 °C. Elevated temperature studies were precluded by extensive decomposition. By contrast the ambient temperature ^{13}C NMR spectra of **3** and **4** exhibit three sharp carbonyl resonances similar to those in the low temperature spectra of **1** and **2**. The apparent absence of local and internuclear carbonyl exchange in **3** and **4** accords with our observations on the ambient temperature ^{13}C NMR spectrum of $[\{W(CO)_3(\eta\text{-}C_5H_5)\}_2]$. We also note that local carbonyl scrambling in $[M_2(CO)_6(\eta^5,\eta^5\text{-guaiazulene})]$ (M = Mo or W) is a very low energy process and is virtually indpendent of the identity of M [17]; the complexes $[M(CO)_3(\eta\text{-}C_5R_5)M'(CO)_2(\eta\text{-}C_7H_7)]$ appear to be very different in this respect.

Experimental

The preparation, purification, and reactions of the complexes described were carried out under dry nitrogen. All solvents were dried by standard methods, and distilled and deoxygenated before use. The compounds $[M'(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ (M′ = Mo or W) [7,8], $Li[M(CO)_3(\eta\text{-}C_5H_5)]$ (M = Mo or W) [18], $Na[Mo(CO)_3(\eta\text{-}C_5H_5)]\cdot 2dme$ [19], $Li[C_5Me_5]$ [20] and $[W(CO)_3(NCPr^n)_3]$ [21] were prepared by published procedures or slight modifications thereof. Aldrich Chemical Co. supplied $M(CO)_6$ (M = Mo or W), $LiBEt_3H$, Li Bu^n and C_5Me_5H; Florisil (60–100 mesh) for column chromatography was purchased from FSA Laboratory Supplies, Loughborough.

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* ^{13}C NMR data for $[\{W(CO)_3(\eta\text{-}C_5H_5)\}_2]$ recorded in CD_2Cl_2 at +20 °C. Chemical shifts in ppm referenced to $SiMe_4$. 222.4, 214.9 (CO), 91.5 (C_5H_5).

The ^{1}H NMR spectra were recorded on a Varian Associates SC 300 instrument and the ^{13}C NMR spectra at 75 MHz on a Varian Associates XL 300 spectrometer. Infrared spectra were obtained with a Perkin–Elmer FT 1710 spectrometer, UV/visible spectra with a Shimadzu UV-260 spectrometer, and electron impact mass spectra with an AEI MS 30 instrument. Microanalyses were by the staff of the Microanalytical Service of the Department of Chemistry, University of Manchester.

Preparation of $[Mo(CO)_3(\eta\text{-}C_5H_5)Mo(CO)_2(\eta\text{-}C_7H_7)]$ *(1)*

A green solution of $[Mo(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ (1.864 g, 5.02 mmol) in thf (70 cm^3) was treated with $Na[Mo(CO)_3(\eta\text{-}C_5H_5)] \cdot 2dme$ (2.333 g, 5.21 mmol). After 45 min stirring at room temperature the mixture was evaporated to dryness and the green residue extracted with toluene. Filtration of the toluene solution, evaporation to dryness, and recrystallisation of the residue from diethyl ether-n-hexane gave the product as a green solid; yield 1.006 g (41% based on $[Mo(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$). Complex **1** was also obtained in 27% yield from $[Mo(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ (2.600 g, 7.00 mmol) and $Li[Mo(CO)_3(\eta\text{-}C_5H_5)]$ (prepared from cyclopentadiene (0.481 g, 7.40 mmol), $LiBEt_3H$ (7.0 cm^3 of a 1.0 *M* solution in thf) and $Mo(CO)_6$ (1.85 g, 7.01 mmol)) stirred in thf for 90 min. The purification procedure was as above. Although the recrystallisation procedure described was reasonably efficient in separating **1** from small quantities of the biproduct $[\{Mo(CO)_3(\eta\text{-}C_5H_5)\}_2]$, trace quantities of $[\{Mo(CO)_3(\eta\text{-}C_5H_5)\}_2]$ were still evident in the solution infrared spectrum. Attempted separation by column chromatography on alumina, silica, or Florisil resulted in extensive decomposition of **1**.

Preparation of $[Mo(CO)_3(\eta\text{-}C_5Me_5)Mo(CO)_2(\eta\text{-}C_7H_7)]$ *(2)*

A solution of $Li[C_5Me_5]$ in thf (25 cm^3) (prepared from C_5Me_5H (0.367 g, 2.70 mmol) and $LiBu^n$ (1.68 cm^3 of a 1.6 *M* solution in hexane)) was refluxed with $Mo(CO)_6$ (0.722 g, 2.73 mmol) for 24 h. The resulting solution of $Li[Mo(CO)_3(\eta\text{-}C_5Me_5)]$ was treated with $[Mo(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ (0.997 g, 2.69 mmol) and the mixture stirred at room temperature for 20 min then evaporated to dryness. Work-up as described for **1** afforded **2** as a dark green solid; yield 0.476 g (32%).

Preparation of $[W(CO)_3(\eta\text{-}C_5H_5)W(CO)_2(\eta\text{-}C_7H_7)]$ *(3)*

A solution of $Li[C_5H_5]$ in thf (30 cm^3) (prepared from C_5H_6 (0.128 g, 1.97 mmol) and $LiBEt_3H$ (1.4 cm^3 of a 1.0 *M* solution in thf)) was refluxed with $[W(CO)_3(NCPr^n)_3]$ (0.579 g, 1.22 mmol) for 1 h. The resulting yellow solution was treated with $[W(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ (0.588 g, 1.22 mmol) and the mixture stirred at room temperature for 15 min then evaporated to dryness. The residue, which contained **3** and trace quantities of $[\{W(CO)_3(\eta\text{-}C_5H_5)\}_2]$, was dissolved in toluene and transferred to a n-hexane-Florisil chromatography column. Elution with n-hexane-toluene (2/1) gave a green band, which afforded pure, green **3**; yield 0.249 g (31%).

The green heterobinuclear derivative $[W(CO)_3(\eta\text{-}C_5H_5)Mo(CO)_2(\eta\text{-}C_7H_7)]$ (**4**) was similarly prepared in 23% yield by treatment of $[Mo(CO)_2(NCMe)(\eta\text{-}C_7H_7)][BF_4]$ (0.392 g, 1.06 mmol) with a thf solution of $Li[W(CO)_3(\eta\text{-}C_5H_5)]$ (prepared from C_5H_6 (0.080 g, 1.23 mmol), $LiBEt_3H$ (1.2 cm^3 of a 1.0 *M* solution in thf) and $[W(CO)_3(NCPr^n)_3]$ (0.503 g, 1.06 mmol) in thf (30 cm^3)). Purification was as described for **1**.

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