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Preliminary communication

New Fe-Mo and Fe-W fulvalene-bridged heterobimetallic complexes containing the ferrocenyl unit. Crystal structure of $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4Ind)Mo(CO)_2(\eta\text{-}C_3H_5)]$ (Ind = 1-indenyl)

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Abstract

Convenient syntheses of the ferrocene-based fulvalene-bridged heterobimetallic complexes $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4C_nR_m)M(CO)_2(\eta\text{-}C_3H_5)]$ (M = Mo or W; C_nR_m = 1-indenyl, C_5Me_4 or $C_5H_2Me_2$) are described.

Keywords: Iron; Molybdenum; Tungsten; Heterobimetallic complexes; Crystal structure

1. Introduction

Investigations of homobimetallic molecular species of the general type $[L_nM(\mu\text{-bridge})ML_n]$ have been very fruitful for the understanding of electron transfer and charge delocalisation phenomena [1–3]. Many inorganic and organometallic examples of such species have been described since the complexes $[(NH_3)_5Ru^{II}(\mu\text{-pyrazine})Ru^{III}(NH_3)_5]^{5+}$ [4] and $[(\eta\text{-}C_5H_5)Fe^{II}(\mu\text{-}\eta:\eta\text{-}C_{10}H_8)Fe^{II}(\eta\text{-}C_5H_5)]$ [5] first appeared. Considerable progress has been made in extending the range of Class II and III [6] mixed-valence homobimetallic organometallic complexes, and most effort has been focused on complexes which use one or two fulvalenyl ($C_{10}H_8$) or biphenyl ($C_{12}H_{10}$) ligands (or derivatives thereof) to bridge the two metal centres [1e–h,2].

Comparatively fewer studies of electronic cooperativity in heterobimetallic complexes have been reported. In 1981 Taube and coworkers showed, for the first time, that weakly-coupled heterobimetallic, 'mixed valence' ions could be prepared by using the ferrocenyl $[\{(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4)\}$, "Fc"] fragment as one of the metal centres [7]. Subsequently, a range of Fc-based heterobimetallic complexes has been described [8]. However, in contrast to the situation for homobimetallic systems, in the case of heterobimetallic complexes non-fulvalenyl linkages have almost exclusively been employed to connect the Fc unit to the second metal centre. Furthermore, the small number of heterobimetallic complexes that do contain a Fc moiety linked to another metal centre through a fulvalene-type bridge are, with one exception, of the bis(metallocene) type [9]. The paucity of compounds of the type $[(\eta\text{-}C_5H_5)Fe(\mu-\eta:\eta\text{-}C_5H_4C_nR_m)ML_n]$ is acknowledged to reflect the lack of suitable synthetic routes to these compounds [10]. Such heterobimetallic complexes are nevertheless attractive targets, since the ferrocenyl unit has well-behaved redox properties and is chemically robust, while the redox potential and donor/acceptor properties of the second metal centre $\{ML_n\}$ may be varied by changing L and/or M. We report here a convenient synthesis of a series of ferrocene-based fulvalene-bridged compounds of the type $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4C_nR_n)ML_n]$; where $ML_n = Mo(CO)_2(\eta\text{-}C_3H_5)$ or $W(CO)_2(\eta\text{-}C_3H_5)$.

2. Results and discussion

Plenio has recently described the synthesis of the ferrocene-substituted dimethylcyclopentadiene and 1-indene complexes, $[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4C_5H_3Me_2)]$ (**1**) and $[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4IndH)]$ (**2**; Ind = 1-indenyl) [10a]. By analogy we have prepared the 2-fer-

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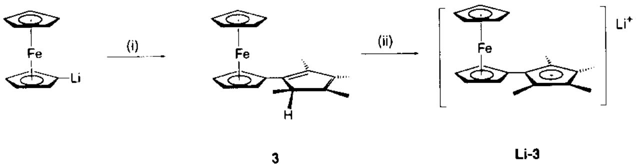
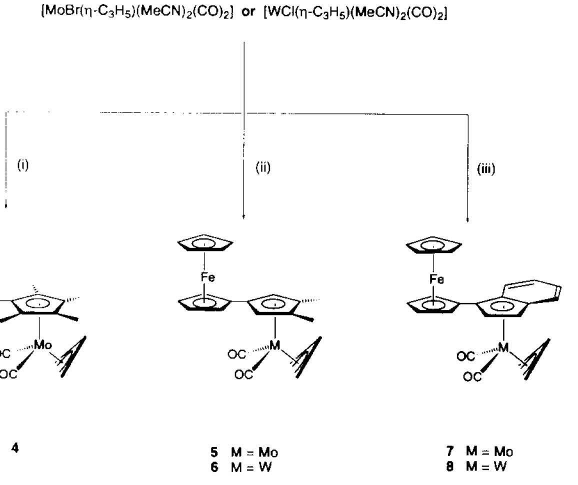


Scheme 1. (i) $C_5OMe_4H_2$, Et_2O, 18 h then HCl(aq), 25%; (ii) ^nBuLi (1.6 M), Et_2O, 1 h, 86%.

rocenyl-1,3,4,5-tetramethylcyclopentadiene (major isomer) derivative $[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4C_5HMe_4)]$ (**3**) [11] from lithioferrocene [12] and 2,3,4,5-tetramethylcyclopentene-1-one [13] in ca. 25% yield (see Scheme 1). The lithio-salts of **1–3**, namely $Li[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4C_5H_2Me_2)]$ (**Li-1**), $Li[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4Ind)]$ (**Li-2**) [10a] and $Li[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4C_5Me_4)]$ (**Li-3**) [11] are readily obtained in gram quantities as weighable solids by treating hexane or Et_2O solutions of the corresponding diene with n-butyllithium. The compounds **Li-1–Li-3** are useful precursors to ferrocene-based, fulvalene-bridged heterobimetallic complexes.

Reaction of **Li-3** with $[MoBr(\eta\text{-}C_3H_5)(MeCN)_2(CO)_2]$ [14a] in THF gave, after chromotography on alumina, the 2,3,4,5-tetramethylfulvalene-bridged Fe–Mo heterobimetallic complex $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4C_5Me_4)Mo(CO)_2(\eta\text{-}C_3H_5)]$ (**4**) in 15% yield (see Scheme 2). Similarly, reaction of **Li-1** and **Li-2** with $[MoBr(\eta\text{-}C_3H_5)(MeCN)_2(CO)_2]$ or $[WCl(\eta\text{-}C_3H_5)(MeCN)_2(CO)_2]$ [14b,c] gave the corresponding Fe–Mo or Fe–W analogues $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4C_5H_2Me_2)M(CO)_2(\eta\text{-}C_3H_5)]$ (M = Mo, **5**; M = W, **6**) and $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4Ind)M(CO)_2(\eta\text{-}C_3H_5)]$ (M = Mo, **7**; M = W, **8**). The spectroscopic data for **4–8** are consistent with their being the exo-allyl isomers depicted in Scheme 2. Monomeric analogues of **4–8**, namely $[(\eta\text{-}C_5R_5)M(CO)_2(\eta\text{-}C_3R'_5)]$ (M = Mo, W) have been reported previously [15].



Scheme 2. (i) $Li[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4C_5Me_4)]$ (**Li-3**), THF, 18 h, 15%; (ii) $Li[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4C_5H_2Me_2)]$ (**Li-1**), hexane, 0.5 h, 70%; (iii) $Li[(\eta\text{-}C_5H_5)Fe(\eta\text{-}C_5H_4Ind]$ (**Li-2**), hexane, 0.5h, 25%.

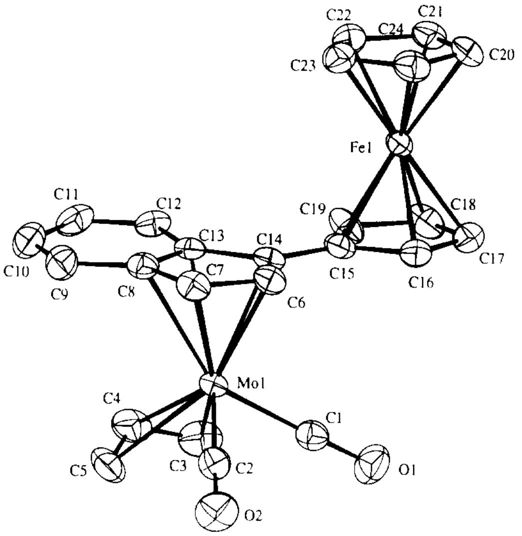


Fig. 1. CAMERON thermal ellipsoid plot (30% probability) of $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4Ind)M(CO)_2(\eta\text{-}C_3H_5)]$ (**7**). Hydrogen atoms omitted for clarity. Selected distances (Å): Fe(1)–$Cp_{cent(1)}$ 1.64, Fe(1)–$Cp_{cent(2)}$ 1.64, Mo(1)–$Cp_{cent(3)}$ 2.04, Mo(1)–C(1) 1.940(4), Mo(1)–C(2) 1.929(3), Mo(1)–C(3) 2.337(4), Mo(1)–C(4) 2.196(4) and Mo(1)–C(5) 2.329(4) where $Cp_{cent(1)}$, $Cp_{cent(2)}$ and $Cp_{cent(3)}$ refer to the computed centroid for the $Fe(\eta\text{-}C_5H_5)$, $Fe(\eta\text{-}C_5H_4)$ and Mo(C_5 ring of Ind) ring carbons respectively.

Recrystallisation of $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4Ind)Mo(CO)_2(\eta\text{-}C_3H_5)]$ (**7**) from hexane afforded single crystals suitable for an X-ray diffraction analysis [16]. The molecular structure of **7** shown in Fig. 1 supports the solution state structures proposed for **4–8** and reveals the presence of mutually trans $\{(\eta\text{-}C_5H_5)Fe\}$ and exo-$\{Mo(CO)_2(\eta\text{-}C_3H_5)\}$ fragments linked by a planar $\mu\text{-}\eta:\eta\text{-}C_5H_4Ind$ unit. The computed torsion angle between the normals to the C_5H_4 and indenyl C_5 ring least-squares planes is 2.8°, confirming the coplanarity of the $\eta\text{-}C_5H_4$ and η-Ind rings. All other angles and distances about the Fe and Mo centres are as expected from comparison with unimetallic complexes of the type $[Fe(\eta\text{-}C_5R_5)_2]$ [17] and $[(\eta\text{-}C_5R_5)M(CO)_2(\eta\text{-}C_3H_5)]$ (R = H, M = Mo; R = Me, M = W), respectively [15b,c]. A preliminary crystal structure determination [18] for $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4C_5Me_4)Mo(CO)_2(\eta\text{-}C_3H_5)]$ (**4**) also shows the basic structural motif of mutually trans $\{(\eta\text{-}C_5H_5)Fe\}$ and exo-$\{Mo(CO)_2(\eta\text{-}C_3H_5)\}$ fragments in **4–8**.

It is interesting to compare the solution ν(CO) IR data for the Mo–Fe heterobimetallics $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4C_5Me_4)Mo(CO)_2(\eta\text{-}C_3H_5)]$ (**4**; ν(CO) = 1932, 1846 cm^{-1}) and $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4Ind)Mo(CO)_2(\eta\text{-}C_3H_5)]$ (**7**; 1944, 1861 cm^{-1}) with those for the molybdenum monomers $[(\eta\text{-}C_5Me_5)Mo(CO)_2(\eta\text{-}C_3H_5)]$ (**9**; η(CO) = 1931; 1845 cm^{-1}) [15e] and $[(\eta\text{-}C_9H_7)Mo(CO)_2(\eta\text{-}C_3H_5)]$ (**10**; ν(CO) = 1945, 1862 cm^{-1}) [15a]. The negligible differences in frequency between the pairs of ν(CO) bands for **4** and **9** and between those for **7** and **10** imply that there is little 'leakage' of electron density between Fe and Mo in the ground state of the complexes **4**–**8**. The apparent inductive effect of the $\{(\eta\text{-}C_5H_5)Fe(C_5H_4)\}$ substituent on the Mo-bound ring is qualitatively about that of a single H atom or Me group. The ν(CO) values for the compounds **4** (ν(CO) = 1932, 1846 cm^{-1}), **5** (ν(CO) = 1936, 1851) and **7** (ν(CO) = 1944, 1861 cm^{-1})] suggest that new ligands $(\eta\text{-}C_5H_5)Fe(C_5H_4C_nR_m)$ should, by selection of the appropriate $\{C_nR_n\}$ fragment, allow the electron density (and hence $E_{1/2}$) at the second centre to be tuned in other complexes of the type $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4C_nR_m)ML_n]$. Electrochemical studies of the heterobimetallic complexes **4**–**8** are in progress.

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[9] These are: $[Fe(\mu\text{-}\eta:\eta\text{-}C_{10}H_8)_2M]^{n+}$ (M = Ru, Ni, Co) [19a–d, 10c], $(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_{10}H_8)Ni(\eta\text{-}C_5H_5)$] [19d], $[Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4C_5Me_4)_2ZrCl_2]$ and $[Fe(\mu\text{-}\eta:\eta\text{-}C_5H_4Ind)_2ZrCl_2]$ (ind = 1-indenyl) [10b] and $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}\eta:\eta\text{-}C_{10}H_8)\text{-}W(CO)_3Me]$ [19e].

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[11] Satisfactory elemental analysis have been obtained for all of the new compounds except for the oil **3** (which was characterised by ^1H NMR and EI mass spectrometry) and **Li-3**, which was obtained as a very air- and moisture-sensitive pale orange powder and used without further characterisation.

Selected ^1H NMR data: **3** (250 MHz, $CDCl_3$, RT); 4.40–4.30 [2H, overlapping 2×m, C_5H_4], 4.20–4.15 [2H, overlapping 2×m, C_5H_4], 4.05 (5H, s, C_5H_5), 2.85 [1H, q of q (J = 7.6, 1.3), C_5Me_4H]; 2.06 [3H, d(J = 1.3), 5-C_5Me_4H], 1.84 (3H, s, 3- or 4-C_5Me_4H), 1.81 (3H, s, 4- or 3-C_5Me_4H), 1.08 [3H, d (J = 7.6), 1-C_5Me_4H].

4 (250 MHz, $CDCl_3$, RT); 4.26, 4.24 (2×2H, 2×virtual t, C_5H_4), 4.11 (5H, s, C_5H_5), 2.67 [1H, t of t (J = 10.7, 7.2), $CH(CH_2)_2$], 2.15 [2H, d (J = 7.2), syn-$CH(CH_2)_2$], 2.11 (6H, s, 2,5- or 3,4-C_5Me_4), 2.00 (6H, s, 3,4- or 2,5-C_5Me_4), 0.94 [2H, d (J = 10.7), anti-$CH(CH_2)_2$].

5 (250 MHz, $CDCl_3$, RT), 5.24 (2H, s, $C_5Me_2H_2$), 4.20–4.10 (4H, overlapping 2×virtual t, C_5H_4), 4.03 (5H, s, C_5H_5), 3.09 [1H, t of t (J = 10.8 7.1), $CH(CH_2)_2$)] 2.39 [2H, d (J = 7.1), syn-$CH(CH_2)_2$], 2.07 (6H, s, $C_5Me_2H_2$), 0.93 [2H, d (J = 10.8), anti-$CH(CH_2)_2$].

7 (400 MHz, C_3D_6O, RT), 7.72 [1H, d (J = 8.7), 4- or 7-C_9H_6], 7.33 [1H, d (J = 8.5), 7- or 4-C_9H_6], 7.25 [1H, d of d (J = 8.7, 7.7), 5- or 6-C_9H_6], 7.11 [1H, d of d (J = 8.5, 7.7), 6- or 5-C_9H_6], 6.18 [1H, d (J = 2.8) 2- or 3-C_9H_6], 6.06 [1H, d (J = 2.8) 3- or 2-C_9H_6], 4.79, 4.70, 4.37, 4.32 (4×1H, 4×virtual q, C_5H_4), 4.04 (5H, s, C_5H_5), 2.14, 1.98 [2×1H, 2×d of d (J = 7.2, 1.4), syn-$CH(CH_2)_2$], 0.96, (1H, m, $CH(CH_2)_2$], 0.79, 0.71 [2×1H, 2×d of d, (J = 10.9, 1.4), anti-$CH(CH_2)_2$].

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[16] X-ray data: $C_{23}H_{20}FeMoO_2$, M = 480.19, crystal size = 0.5×0.3 ×0.1 mm^3, monoclinic, space group = $P2_1/n$, a = 12.794(4), b = 11.659(5), c = 14.095(4) Å, β = 112.23(4) °, V = 1946.21 $Å^3$, Z = 4, D_c = 1.639, μ = 13.89 cm^{-1}, $F(000)$ = 968, Mo K_α (λ = 0.71069 Å), $2 < 2\theta < 50°$, scan mode ω-2θ, total unique data 3629, observations $[I > 3\sigma(I)]$ 2815, variables 254, observations/variables 11.1, Chebyshev parameters 2.39, −2.35, 1.77, −0.574, R = 0.029, R_w = 0.029. Tables of atomic coordinates

and bond lengths and angles have been deposited with the Cambridge Crystallographic Data Centre.

Data were collected on a Hilger and Watts Y290 four-circle diffractometer. The data were corrected for Lorentz and polarisation effects and systematically absent reflections were rejected. The Mo and Fe atom positions were located by direct methods and subsequent difference Fourier syntheses revealed the positions of all other non-hydrogen atoms. Hydrogen atoms were placed in estimated positions (C–H = 0.96 Å) with fixed isotropic thermal parameters (1.3× the equivalent isotropic temperature factor of the carbon atom to which they were bonded) and their positions recalculated between successive least-squares cycles. A DIFABS absorption correction [20] was applied (min/max correction 0.95/1.12) to the fully-refined isotropic model. Non-hydrogen atoms were subsequently refined with anisotropic thermal parameters. A Chebyshev weighting scheme [21] was applied and the data were corrected for the effects of anomalous dispersion and isotropic extinction (via an overall isotropic extinction parameter [22]) in the final stages of refinement. All crystallographic calculations were performed using the CRYSTALS suite [23] on an Akhter 486DX computer. Neutral atom scattering factors were taken from the usual sources [24].

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