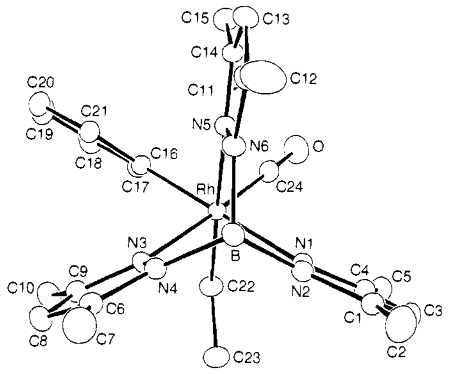
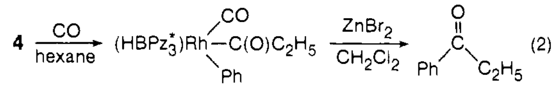


Figure 1. Molecular structure of $(HBPz^*_3)Rh(CO)(C_2H_5)(Ph)$ (4), viewed approximately along the pseudo-3-fold axis. Selected interatomic distances from Rh to C22, 2.106 (4); C16, 2.053 (4); C24, 1.824 (4); N1, 2.170 (3); N3, 2.104 (3); N5, 2.219 (3) Å. Selected angles: C22–Rh–C24, 86.8 (2); C22–Rh–C16, 90.4 (2); C16–Rh–C24, 86.4 (2); average N–Rh–N, 86.1 (1)°.

periments under the same conditions established that **3** does not react with excess C_2H_4 to produce **4** and that **4** is not converted to **3**. Thus one product is not the precursor of the other, and separate pathways (perhaps involving a common intermediate) are indicated. The X-ray crystal structure of **4**[7] (Figure 1) confirms the formulation and demonstrates the high steric demand of the $HBPz^*_3$ ligand; the rhodium atom actually lies 0.127 Å above (i.e., on the boron side) the plane formed by the 3-methyl carbons C5, C10, and C15.

When a hexane solution of **4** (3.7 mM) was pressurized with CO at 950 psig and heated at 100 °C for 2 weeks, complete conversion occurred to two carbonylation products **5** and **6**. **5** has been fully characterized[8] as the phenylpropionyl complex $(HBPz^*_3)Rh(CO)(COC_2H_5)(Ph)$. While **6** is still under investigation,[9] it converts slowly but completely to **5** in the absence of CO. Thus the complete conversion of **4** to **5** is possible as shown in eq 2.



$$\mathbf{4} \xrightarrow[\text{hexane}]{CO} (HBPz^*_3)Rh(CO)(Ph){-}C(O)C_2H_5 \xrightarrow[CH_2Cl_2]{ZnBr_2} PhC(=O)C_2H_5 \qquad (2)$$

5

Under conditions so far utilized (maximum 1000 psi at 100 °C) CO pressure does not induce reductive elimination of ketone from **5** or 1,2-diketone from **6**.[10] However, noting the observation[11] that $ZnBr_2$ promoted reductive elimination of RH from Cp*Ir(PMe$_3$)(R)(H), we treated **5** with excess $ZnBr_2$ (CH_2Cl_2, 25 °C, 4 h) and found that propiophenone was formed in 82% yield. The fate of the rest of complex **5** is not yet known, and carrying out the $ZnBr_2$ reaction under a CO atmosphere did not result in formation of **1**.

The reactions reported here are stoichiometric rather than catalytic, and the key rhodium complex has not been regenerated. Nevertheless, ethylene, benzene, and carbon monoxide have been combined under mild conditions with photochemical assistance to produce propiophenone. We think these reactions provide an encouraging glimpse of the role that C–H activation by transition-metal complexes may play in the manipulation and functionalization of hydrocarbons.

Acknowledgment. We gratefully acknowledge the support of this work by the Natural Sciences and Engineering Research Council.

Supplementary Material Available: Experimental details for syntheses and reactions with spectroscopic and analytical data for **2**, **3**, **4**, and **5**, tables of positional and thermal parameters, interatomic distances and angles, and details of X-ray crystallographic data collection for **4** (26 pages). Ordering information is given on any current masthead page.

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[6] The reaction also took place in sunlight or incandescent light but more slowly and with some variation in the ratio **3**:**4**.

[7] The X-ray crystallographic study was carried out by Dr. R. G. Ball in the Structure Determination Laboratory of this department. **4** crystallized from hexane in the monoclinic space group $P2_1/n$, $a = 10.752$ (3) Å, $b = 17.591$ (3) Å, $c = 13.751$ (2) Å, $\beta = 104.43$ (2)°, $V = 2519$ Å^3, $Z = 4$; 3601 unique reflections having $I > 3\sigma(I)$ were used in the solution and refinement: $R = 0.035$, $R_w = 0.044$.

[8] Data for **5**: colorless crystals mp 184–185 °C; IR (*n*-hexane) ν_{CO} 2069, 1670 cm^{-1}; ^1H NMR (CD_2Cl_2, ambient, 200 MHz) δ 8.24 (d, 1 H), 7.18 (t, 1 H), 6.96 (t, 1 H), 6.74 (t, 1 H), 6.37 (d, 1 H), 5.94 (s, 1 H), 5.84 (s, 1 H), 5.72 (s, 1 H), 2.50 (s, 3 H), 2.46 (s, 3 H), 2.42 (s, 3 H), 2.35 (m, 2 H, CH_2), 2.25 (s, 3 H), 1.59 (s, 3 H), 1.29 (s, 3 H), 0.82 (t, 3 H, $J = 7$ Hz); the 400 MHz spectrum established that the δ 2.35 multiplet was an AB pattern with $\Delta\delta = 0.093$, $J_{AB} = 19$ Hz; MS (16 eV, 150 °C), 533 (100%) $[M - C_2H_5)]^+$, 505 (25%) $[M - C_2H_5 - CO]^+$, 477 (73%) $[M - C_2H_5 - 2CO]^+$, 400 (8%) $[M - C_2H_5 - C_6H_5 - 2CO]^+$. Anal. ($C_{25}H_{32}BN_6O_2Rh$) C, H; N: calcd 14.95; found 14.30.

[9] Although **6** has not been obtained free of **5**, ^1H and ^{13}C NMR studies by Dr. Glenn J. Sunley have established its probable identity as the benzoyl propionyl complex $(HBPz^*_3)Rh(CO)(COC_6H_5)(COC_2H_5)$.

[10] This is in marked contrast with the reactivity of $(\eta^5\text{-}C_5Me_5)Rh(CO)(Me)(Ph)$ with CO, which leads under milder conditions (6 h, 100 °C, 7.5 atm) to acetophenone in 99% yield along with $[(\eta^5\text{-}C_5Me_5)Rh(CO)]_2$: Sunley, G. J.; Fanizzi, F. P.; Saez, I. M.; Maitlis, P. M. *J. Organomet. Chem.* **1987**, *330*, C27–C30.

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Synthesis and Electrophilic Reactivity of Dicarbonyl(alkene)(arene)manganese Cations[†]

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Received July 15, 1988

A wide variety of nucleophiles add to the arene in (arene)Mn(CO)$_3^+$ (**1**) to give exo functionalized (cyclohexadienyl)Mn(CO)$_3$ complexes (**2**).[1-4] Further elaboration of **2** to generate coordinated cis difunctionalized cyclohexadienes has been reported.[5] Herein we report the synthesis and reactivity of (arene)Mn(CO)$_2$(alkene)$^+$ (**3**); it is anticipated that this new class of electrophilic complexes will provide a general route to *endo*-cyclohexadienyl complexes and *trans*-cyclohexadienes.

A good way to prepare **3** is outlined in Scheme I. Treatment of **1** in THF with a slight excess of Me_3NO (25 °C, 30 min, N_2, dark) leads to ready displacement of CO[6] to give **4** as a purple solid in 80–90% yield after filtration and solvent removal at ≤30 °C. The heat and light sensitive complex **4** can be stored at −10 °C for weeks; its purity was established as typically 90% by dissolution in CD_3CN, which rapidly generates free THF (determined by NMR) and (arene)Mn(CO)$_2$(NCCD$_3$)$^+$PF$_6^-$. (The complex $[(C_6Me_6)Mn(CO)_2MeCN]PF_6$ was prepared independently and fully characterized by X-ray diffraction.[7]) The THF in **4** is replaced by alkenes to give good yields (≥80%) of **3** as thermally stable yellow-orange salts. The usual procedure with liquid alkenes was to reflux a CH_2Cl_2 solution of **4** and excess

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[†] Dedicated to the memory of Fred A. Snavely, an inspiring teacher, scholar, and friend.

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Scheme I

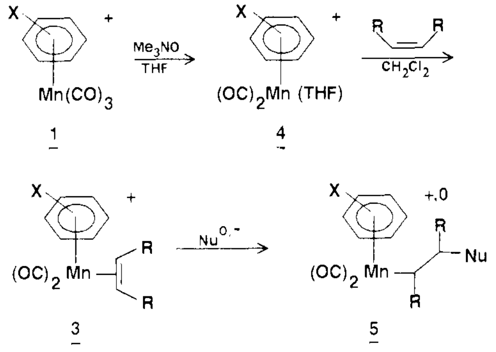

Table I. IR Spectral Data for $[(\mathrm{Arene})\mathrm{Mn(CO)_2L}]^{0,+}$ Complexes[a]

arene	L	ν_{CO}[b] (cm^{-1})	arene	L	ν_{CO}[b] (cm^{-1})
C_6Me_6	THF	1992, 1942	C_6Me_6[e]	propene	2007, 1963
$C_6H_3Me_3$	THF	2008, 1957	C_6HMe_5[g]	propene	2009, 1965
C_6H_6	THF	2022, 1977	C_6Me_6[e]	cyclopentene	2001, 1958
C_6Me_6	H^-	1936, 1876[c]	C_6Me_6	norbornadiene	2003, 1960
C_6Me_6	CN^-	1981, 1931[d]	C_6HMe_5[e,h]	norbornadiene	2005, 1961
C_6Me_6[e,f]	ethene	2012, 1971	C_6H_6	norbornadiene	2025, 1986
C_6HMe_5	ethene	2015, 1972	C_6Me_6[e]	2,3-dihydrofuran[i]	2000, 1957
$C_6H_3Me_3$	ethene	2023, 1983	C_6Me_6	ethyne	2019, 1975

[a] If charged (L neutral), the anion is PF_6^-. [b] In CH_2Cl_2. [c] 1954, 1903 cm^{-1} in pentane. [d] In pentane (containing 1% MeOH), ν_{CO} = 1989, 1946 cm^{-1}. [e] Satisfactory elemental analysis obtained. [f] FAB-MS, m/z 301 (M^+), 273 ($M^+ - C_2H_4$), 245 ($M^+ - C_2H_4 - CO$), 217 ($M^+ - 2CO - C_2H_4$). [g] FAB-MS, m/z 301 (M^+), 259 ($M^+ - C_3H_6$), 231 ($M^+ - C_3H_6 - CO$), 203 ($M^+ - 2CO - C_3H_6$). [h] FAB-MS, m/z 351 (M^+), 259 ($M^+ - C_7H_8$), 231 ($M^+ - C_7H_8 - CO$), 203 ($M^+ - 2CO - C_7H_8$). [i] π-bonded.

alkene for 1 h, followed by filtration, concentration, and addition of diethyl ether to precipitate the product. Purification was effected by recrystallization from CH_2Cl_2/pentane or Soxhlet extraction with diethyl ether. With gaseous alkenes, a CH_2Cl_2 solution of **4** was charged with several atmospheres of alkene and stirred for several hours at ca. 25 °C. Tables I and II give spectral data for selected complexes.

An alternative general route to **3** involves treating **1** with Me_3NO in 2,3-dihydrofuran as the solvent. After 10–20 min at room temperature, IR spectra showed ≥90% conversion to (arene)$Mn(CO)_2(C_4H_6O)^+$ (**6**), which contains π-bonded dihydrofuran (isolated as a stable orange salt in 80% yield). IR spectra showed that ethylene (100 psi, 2 h) and even cyclopentene (100-fold excess, 30 min) and acetylene (25 psi, 2 h) quantitatively replace the dihydrofuran from **6** in a CH_2Cl_2 solution at room temperature to give the corresponding complex **3**.

X-ray structures of **3** have been determined for the sets (arene, alkene) being (C_6Me_6, propene), (C_6Me_6, cyclopentene), (C_6HMe_5, norbornadiene), and (C_6HMe_5, 2,3-dihydrofuran).[8] Figure 1 shows the structure of $[C_6HMe_5)Mn(CO)_2$(norbornadiene)]PF_6 (**7**).[9] As expected, the coordinated double bond C3–C4 in **7** is considerably longer (1.365 (4) Å) than the free one (C6–C7 = 1.311 (5) Å). The structural and stereochemical details are quite similar to those for $CpMn(CO)_2$(norbornadiene),[10] except that the latter has a longer coordinated double bond (1.40 (4) Å) due to a relatively greater amount of π-backbonding in this neutral complex.

Complexes **3** are electrophilic and are attacked by phosphine, phosphite, hydride, and cyanide nucleophiles at the alkene to give **5** (IR ν_{CO} bands at 1950 ± 15 and 1880 ± 15 cm^{-1}). In most cases the nucleophilic addition is a clean and rapid reaction in THF, CH_2Cl_2, or pentane, although with some of the more sterically congested systems alkene displacement is a competing reaction. The full scope of these nucleophilic addition reactions, particularly with carbon donors, is being explored.[11] The coordinated acetylene in $(C_6Me_6)Mn(CO)_2(C_2H_2)^+$ is extremely electrophilic and even reacts with the weak base NMe_3 to give the expected σ-vinyl complex (ν_{CO} = 1955, 1903 cm^{-1} in CH_2Cl_2), which has been characterized by X-ray diffraction.[7] Kinetic studies in CH_2Cl_2 show that the reaction **3** → **5** is cleanly second order and that the relative nucleophilic reactivities of a series of phosphorus donors are independent of the nature of the alkene (or arene) and are equal to those found for additions to coordinated cyclic π-hydrocarbons and free carbocations.[12,13]

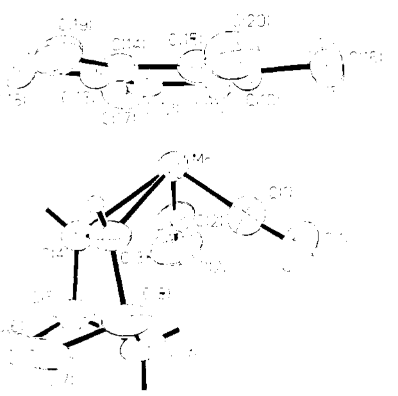

Figure 1. An ORTEP drawing of $(C_6HMe_5)Mn(CO)_2$(norbornadiene)$^+$ with the thermal ellipsoids at the 50% probability level.

Table II. ^{1}H NMR Data for Selected Complexes **3** and **5**[a]

complex	^{1}H NMR (δ)
$[(C_6Me_6)Mn(CO)_2$(ethene)]PF_6	2.59 (s, C_2H_4), 2.35 (Me)
$[(C_6HMe_5)Mn(CO)_2$(ethene)]PF_6	5.91 (s, CH), 2.76 (s, C_2H_4), 2.32–2.37 (Me)
$[(C_6HMe_5)Mn(CO)_2$(propene)]PF_6	5.87 (s, CH), 3.85 (m, CH), 2.80 (d, J = 8, *cis*-CH_2), 2.53 (d, J = 14, *trans*-CH_2), 2.33 (br, Me), 1.58 (d, J = 6, Me)
$[(C_6Me_6)Mn(CO)_2$-(norbornadiene)]PF_6	6.73 (s, free =CH), 4.04 (s, complexed =CH), 3.31 (s, CH), 2.32 (Me), 1.52 (d, J = 10, *anti*-CH_2), 0.00 (d, J = 10, *syn*-CH_2)
$[(C_6HMe_5)Mn(CO)_2$-(2,3-dihydrofuran)]PF_6	7.06 (s, =CH), 5.80 (s, CH), 4.15 (m, CH_2), 3.41 (m, =CH), 2.99, 2.92 (m, CH_2), 2.33 (br, Me)
$(C_6Me_6)Mn(CO)_2(CH_2CH_2CN)$	2.18 (obscured, CH_2), 2.10 (Me), 0.72 (t, J = 9, CH_2)
$(C_6Me_6)Mn(CO)_2(CH_2CH_3)$	2.08 (Me), 1.18 (t, J = 7.4, CH_3), 0.67 (q, J = 7.4, CH_2)

[a] In CD_2Cl_2.

Complexes **3** are analogous to the thoroughly studied alkene complexes $CpFe(CO)_2$(alkene)$^+$ (**8**), which have found significant synthetic applications.[14-19] There are, however, substantial differences between **3** and **8**. Kinetic studies show that **3** (arene = C_6Me_6) is about 150 times less electrophilic than **8**,[20] suggesting

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(8) Halpin, W. A.; Sweigart, D. A. Unpublished results.

(9) Crystal data for **7**: space group $P2_1/n$ with a = 8.818 (3) Å, b = 16.807 (4) Å, c = 14.284 (3) Å, β = 92.50 (2)°, V = 2114.8 Å^3, Z = 4, d_{calcd} = 1.56 g cm^{-3}. Data collected at 20 °C on a Nicolet R3m diffractometer with Mo Kα radiation, μ = 7.38 cm^{-1}, 2θ limits 3.5–50°, 392 variables refined with 3743 unique reflections $I > 1.0\sigma(I)$ to R = 0.051 (R_w = 0.048).

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that unwanted redox pathways are less likely for nucleophilic additions to **3**. Unlike **8**, the complexes **3** offer a pathway for functionalization of the coordinated cyclic π-hydrocarbon. Thus, there is precedent[21] for thermal migration of the σ-hydrocarbon in **5** to the arene to yield endo functionalized cyclohexadienyl complexes (redox-promoted migration probably will prove to be more convenient[22-24]). Through the intermediacy of **3**, such cyclohexadienyl complexes may be available with a wide range of endo substituents, and these may be converted to trans difunctionalized cyclohexadienes by using published procedures.[5]

Finally, we note that the chemistry described above can lead to C–H activation, at least with reactive C–H bonds. Thus, treatment of **1** with Me_3NO in furan was found to effect electrophilic substitution on the furan to give **5** as a σ-vinyl complex (ν_{CO} = 1950, 1897 cm^{-1} in CH_2Cl_2). Similarly, IR evidence strongly suggests C–H bond cleavage to give neutral σ-bonded complexes when oxiranes are added to a CH_2Cl_2 solution containing a mixture of **1** and Me_3NO.

Acknowledgment. We are grateful to Professor G. B. Carpenter for very useful discussions. This work was supported by a grant from the National Science Foundation (No. CHE-8521189).

Supplementary Material Available: Tables of atomic coordinates, thermal parameters, bond lengths, and bond angles for **7** (7 pages); tables of observed and calculated structure factors for **7** (22 pages). Ordering information is given on any current masthead page.

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Intramolecular Arene Hydrogenation by Niobium Aryloxide Compounds: Stereochemistry of Cyclohexadiene Formation

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The high-valent early d-block, lanthanide and actinide metal-hydride bond has been shown over the last few years to be an extremely important functional group, implicated in a wide range of stoichiometric and catalytic reactivity.[2-9] In this communication we wish to report on the observation of a very facile hydrogenation of an arene ring by a high-valent niobium aryloxy, hydride intermediate.[10-13]

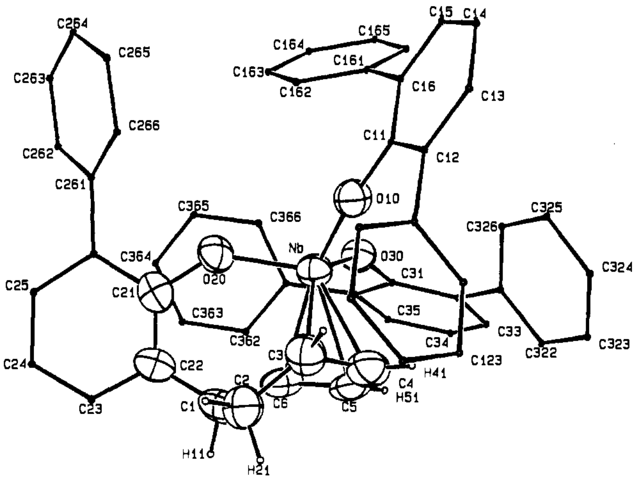
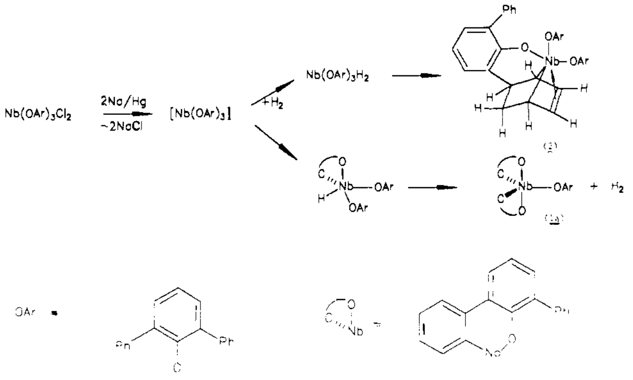


Figure 1. ORTEP view of $Nb(OC_6H_3Ph\text{-}\eta^4\text{-}C_6H_7)(OAr\text{-}2,6Ph_2)_2$ (**2**) emphasizing the central coordination sphere. Selected bond distances (Å) and angles (deg): Nb–O(10) = 1.928 (9); –O(20) = 1.923 (9); –O(30) = 1.932 (9); –C(3) = 2.27 (1); –C(4) = 2.27 (2); –C(5) = 2.37 (1); –C(6) = 2.40 (1); C(1)–C(2) = 1.52 (2); C(2)–C(3) = 1.51 (2); C(3)–C(4) = 1.41 (2); C(4)–C(5) = 1.40 (2); C(5)–C(6) = 1.38 (2); C(6)–C(1) = 1.52 (2); O(10)–Nb–O(20) = 107.8 (4); –O(30) = 107.3 (4); O(20)–Nb–O(30) = 108.9 (4); Nb–O(10)–C(11) = 165.8 (9); Nb–O(20)–C(21) = 138.8 (9); Nb–O(30)–C(31) = 147.8 (8).

Scheme I



The reduction of toluene solutions of the mixed chloro, aryloxide compounds $M(OAr\text{-}2,6Ph_2)_3Cl_2$ (M = Nb, Ta; OAr-2,6Ph$_2$ = 2,6-diphenylphenoxide)[14] with sodium amalgam (2 Na per M) takes place over a period of hours to produce yellow-brown suspensions. Workup allows the isolation of the bright yellow, biscyclometalated compounds $M(OC_6H_3Ph\text{-}C_6H_4)_2(OAr\text{-}2,6Ph_2)$ (M = Nb, **1a**; Ta, **1b**) in high yields (Scheme I). A previous single-crystal X-ray diffraction analysis of **1b** has been reported.[15] In the case of M = Nb, the reduction solution was found to also contain a second, deep-red minor component (**2**). The ^1H NMR

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