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A novel synthetic route to cyclobutadiene complexes of molybdenum and tungsten. Crystal and molecular structure of Mo(.eta.-C5H5)(.eta.-C4Ph3Me)(CO)(Cl)

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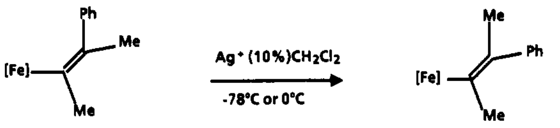
idants catalyze a *Z–E* isomerization reaction. Similar conditions cause the double-bond isomerization reaction shown in eq 7. If these reactions are carried out in the



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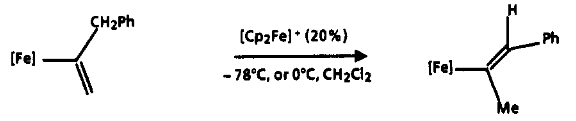

(7)

presence of CO, one does not get CO insertion of the starting materials, but the products expected when the isomerized iron complex is subjected to these conditions. Thus, for example, carrying out reaction 6 in the presence of CO yields the (*E*)-acyl product shown in eq 1. Although these are interesting isomerization reactions, they do limit the types of alkenylacyl complexes and ultimately cleaved esters that one can prepare from this methodology.

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Registry No. [Fe][η^1-C(CH_2Ph)=CH_2], 92366-24-0; [Fe][η^1-(*E*)-COC(Me)=C(Me)Ph], 92366-21-7; [Fe][Nη^1-(*E*)-COC(Me)=C(Me)CH(CO_2Et)$_2$], 92366-22-8; [Fe][η^1-(*E*)-COC(CO_2Me)=C(H)Me], 92366-23-9; [Fe][η^1-(*E*)-COC(Me)=C(H)Ph], 92396-62-8; [Fe][η^1-(*E*)C(Me)=C(Me)Ph], 83096-09-7; [Fe][η^1-(*E*)-C(Me)=(H)Ph], 86563-32-8; [Fe][η^1-(*E*)-C(Me)=C(Me)CH(CO_2Et)$_2$], 87556-41-0; [Fe][η^1-(*E*)-C(CO_2Me)=C(H)Me], 86563-31-7; [Fe][η^1-(*Z*)-C(Me)=C(Ph)Me], 863149-30-8; [Cp_2Fe]BF_4, 1282-37-7; $AgBF_4$, 14104-20-2.

A Novel Synthetic Route to Cyclobutadiene Complexes of Molybdenum and Tungsten. Crystal and Molecular Structure of Mo(η-C_5H_5)(η-C_4Ph_3Me)(CO)(Cl)

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Received June 4, 1984

Summary: Treatment of oxocyclobutenyl complexes of molybdenum and tungsten, **1a–c**, with titanacyclobutane **2**, followed by tetrafluoroboric acid etherate, affords cationic cyclobutadiene complexes **4**, via the intermediacy of methylenecyclobutenyl compounds **3**. Reaction of cationic **4a** with chloride yields the neutral derivative **5**, which has been characterized crystallographically.

While transition-metal complexes containing cyclobutadiene ligands have aroused considerable synthetic and theoretical interest over the past two decades,[3] group 6B complexes of this ligand are rare.[4,5] Cationic group 6B derivatives are unknown,[3,4] though some routes to cationic cyclobutadiene complexes of other transition metals have been reported.[6] Here we describe a novel and versatile synthesis of cationic cyclobutadiene complexes of molybdenum and tungsten, from readily available precursors, and the molecular structure of a neutral molybdenum derivative.

We have reported previously the facile syntheses of oxocyclobutenyl complexes **1** from readily available cyclopropenyl cations.[7] While the ketone functionalities of complexes **1a**,**b** are unreactive toward excess methylenetriphenylphosphorane (75 °C; Me_2SO),[8] treatment with 1 equiv of the titanacycle **2**[9] (50 °C; toluene) effects clean conversion to the methylenecyclobutenyl compounds **3a**,**b**.[10,11] Subsequent protonation with tetrafluoroboric acid in ether affords the cationic cyclobutadiene complexes **4a**,**b**[12] as yellow, air-stable, crystalline solids. In a similar one-pot reaction sequence, treatment of **1c** with **2**, followed by $HBF_4 \cdot Et_2O$, affords the permethylated cationic com-

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(1) (a) Dartmouth College. (b) University of Delaware.

(2) Alfred P. Sloan Research Fellow, 1980–1984.

(3) Efraty, A. *Chem. Rev.* **1977**, *77*, 691–744. This provides a general review of cyclobutadiene complexes.

(4) For a recent review of group 6B organometallic compounds, see: "Comprehensive Organometallic Chemistry"; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon Press: Oxford, 1982; Vol. 3.

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(7) Hughes, R. P.; Lambert, J. M. J.; Reisch, J. W.; Smith, W. L. *Organometallics* **1982**, *1*, 1403–1405. Syntheses of **1a** and **1c** are reported here; **1b** is prepared in a method analogous to that used for **1a**.

(8) Greenwald, R.; Chaykovsky, M.; Corey, E. J. *J. Org. Chem.*, **1963**, *28*, 1128–1129.

(9) Titanacyclobutane **2** and its analogues have been shown to be very effective reagents for effecting the conversion of carbonyl functions into olefins. See: Buchwald, S. L.; Grubbs, R. H. *J. Am. Chem. Soc.* **1983**, *105*, 5490–5491, and references cited therein.

(10) Examples of methylenecyclobutenyl complexes of Pd(II) have been reported recently: Ridgewell, P. J.; Bailey, P. M.; Wetherell, S. N.; Kelley, E. A.; Maitlis, P. M. *J. Chem. Soc., Dalton Trans.* **1982**, 999–1004.

(11) Satisfactory microanalysis results were obtained for all compounds reported. In a typical reaction **1a** (0.69 g, 1.35 mmol) was treated, under anaerobic conditions, with a solution of **2** (0.34 g, 1.23 mmol) in toluene (10 mL), and the mixture was heated at 65 °C for 4 h. The solution was cooled, and the solvent was evaporated in vacuo. The solid was extracted with hexane (2 × 30 mL) and filtered through filter aid on a Schlenk filter. Evaporation and recrystallization from hexane yielded **3a** as yellow crystals (0.56 g, 90%): IR (hexanes) ν_{CO} 1987, 1929 cm^{-1}; ^1H NMR (CD_3CN, shifts in ppm downfield from Me_4Si) δ 7.17–7.40 (m, 15 H, Ph), 5.20 (s, 5 H, Cp), 3.32 (s, 2 H, CH_2). Similarly prepared was **3b**: IR (hexanes) ν_{CO} 1983, 1921 cm^{-1}, ^1H NMR ($CDCl_3$) δ 6.98–7.53 (m, 15 H, Ph), 5.22 (s, 5 H, Cp), 3.28 (s, 5 H, CH_2).

(12) Typically, a solution of **3a** (0.50 g, 0.98 mmol) in ether (60 mL) was cooled to 0 °C and treated with tetrafluoroboric acid etherate (0.200 mL) to give a heavy yellow precipitate. The supernatant liquid was removed by using a cannula, and the solid was washed with ether (3 × 15 mL) and recrystallized from methylene chloride/ether to give **4a** as yellow, air-stable crystals (0.53 g, 91%): IR (CH_2Cl_2) ν_{CO} 2054, 2010 cm^{-1}, ^1H NMR ($CDCl_3$) δ 7.18–7.43 (m, 15 H, Ph), 5.65 (s, 5 H, Cp), 2.81 (s, 3 H, Me); ^{13}C NMR ($CDCl_3$) δ 214.8 (CO), 131.6, 130.1, 129.3, 129.0, 127.8 (Ph), 97.3 (Cp), 95.4 (CPh), 89.4 (CPh), 80.8 (CMe), 13.5 (Me). Similarly prepared was **4b**: IR (CH_2Cl_2) ν_{CO} 2042, 1996 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.20–7.65 (m, 15 H, Ph), 5.84 (s, 5 H, Cp), 3.06 (s, 3 H, Me); ^{13}C NMR ($CDCl_3$) δ 200.5 (CO), 130.9, 129.6, 129.3, 128.9, 128.7, 128.6 (Ph), 94.5 (Cp), 79.1 (CPh), 75.7 (CPh), 73.2 (CMe), 11.4 (Me). **4c**: IR (CH_2Cl_2) ν_{CO} 2037, 1985 cm^{-1}, ^1H NMR ($CDCl_3$) δ 2.17 (s, 15 H, C_5Me_5), 2.04 (s, 12 H, C_4Me_4); ^{13}C NMR ($CDCl_3$) δ 220.2 (CO), 106.0 (C_5Me_5), 84.1 (C_4Me_4), 10.2 (C_5Me_5), 8.2 (C_4Me_4).

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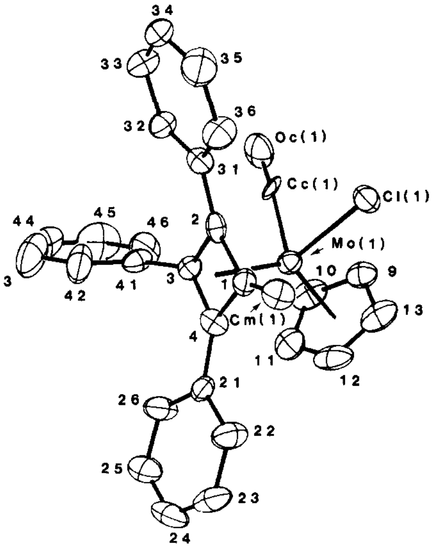
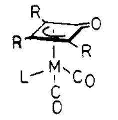
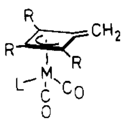


Figure 1. ORTEP diagram and labeling scheme for one of two independent molecules of [MoCl(CO)(η^5-C_5H_5)(η^4-C_4Ph_3Me)], **5**. Bond distances (Å) (for the molecule shown): Mo(1)–CNT4 = 2.021 (10), Mo(1)–CTN5 = 1.998 (10), Mo(1)–C(1) = 2.273 (10), Mo(1)–C(2) = 2.231 (10), Mo(1)–C(3) = 2.219 (10), Mo(1)–C(4) = 2.338 (9), Mo(1)–Cl(1) = 2.526 (3), Mo(1)–Cc(1) = 1.975 (11), C(1)–C(2) = 1.44 (1), C(2)–C(3) = 1.46 (1), C(3)–C(4) = 1.46 (1), C(1)–C(4) = 1.45 (1). Bond angles (deg): Cc(1)–Mo(1)–Cl(1) = 87.3 (2), Cc(1)–Mo(1)–CNT4 = 104.0 (2), Cc(1)–Mo(1)–CNT5 = 110.4 (2), Cl(1)–Mo(1)–CNT4 = 103.6 (1), Cl(1)–Mo(1)–CNT5 = 109.2 (1), CNT4–Mo(1)–CNT5 = 132.8 (2). CNT4 = centroid of η^4-C_4 ring; CNT5 = centroid of η^5-C_5 ring.

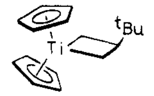
pound **4c**[12]. Reaction of the molybdenum complex **4a** with PPN^+Cl^-[13] in refluxing THF proceeds smoothly with CO displacement to give the neutral complex **5**,[14] which has been crystallographically characterized. The tungsten analogue **4b** is resistant to CO substitution under these conditions.



1. a. M=Mo;L=Cp;R=Ph.
b. M=W;L=Cp;R=Ph.
c. M=Mo;L=Cp*;R=Me.



3. a. M=Mo;L=Cp;R=Ph.
b. M=W;L=Cp;R=Ph.



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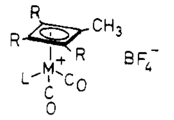
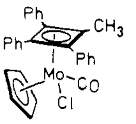


4. a. M=Mo;L=Cp;R=Ph.
b. M=W;L=Cp;R=Ph.
c. M=Mo;L=Cp*;R=Me.



5.

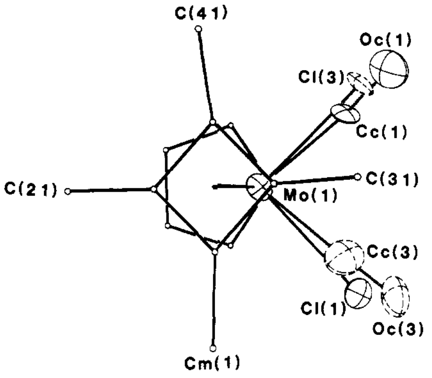


Figure 2. A depiction of the CO and Cl disorder in [MoCl(CO)(η^5-C_5H_5)(η^4-C_4Ph_3Me)]. The broken-line atoms were refined to an occupancy of 25%.

Compound **5** crystallizes as discrete, well separated molecules in a distorted tetrahedral complex (Figure 1)[15] and bears a formal resemblance to seven-coordinate, "four-legged, piano-stool" complexes if the cyclobutadiene ligand is treated as a two-lobed, four-electron donor.

While no other examples of structurally characterized molybdenum analogues could be found for comparison, **5** closely resembles the structure of the isoelectronic compound $V(C_5H_5)(C_4Ph_4)(CO)_2$.[16] In **5** the CNT5–Mo(1)–CNT4 angle is 132.8 (2)° while the corresponding angle in the V complex is 137.7°[17] larger perhaps to accommodate the fourth phenyl ring. In **5** the orientation of the C_4 ring is staggered in its relationship to the Cc(1)–Mo(1)–Cl(1) angle; the methyl group is unsymmetrically positioned toward the majority chlorine position (see Figure 2). In view of the observed Cl/CO disorder, it is somewhat surprising that the methyl group and the C(41) phenyl ring are not similarly disordered. As is also true for the V complex, the cyclobutadiene ring is planar and constructed of equal bond lengths [average 1.45 (2) Å], and right angles [average 89.9 (2)°].

Assuming that the cationic precursors **4** adopt the same ground-state conformation as that in **5**, free rotation about the metal–C_4-ring axis in solution is demonstrated by NMR observation of a single methyl resonance for the tetramethylcyclobutadiene ligand of **4c**;[12] hindered rotation of tetrakis(trifluoromethyl)cyclobutadiene ligands in analogues of complexes **4** and **5** have been reported.[5c]

This synthetic methodology is mild and promises to be versatile; extensions to oxocyclobutenyl complexes of other

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(13) PPN^+ = bis(triphenylphosphine)nitrogen(1+).

(14) A solution of **4a** (0.11 g, 0.18 mmol) and PPN^+Cl^- (0.13 g, 0.23 mmol) in THF (25 mL) was refluxed overnight to give a clear orange solution. Evaporation and chromatography of the residue on Florisil afforded an orange band on elution with methylene chloride/ether (95/5). Evaporation and recrystallization of the residue from methylene chloride/ether yielded orange crystals of **5** (0.09 g, 94%): IR (CH_2Cl_2) ν_{CO} 1960 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.05–7.44 (m, 15 H, Ph), 5.20 (s, 5 H, Cp), 2.53 (s, 3 H, Me); ^{13}C NMR (acetone-d_6) δ 228.7 (CO), 134.2, 131.3, 129.1, 128.4, 128.0, 127.3 (Ph), 98.6 (Cp), 84.3 (CPh), 81.5 (CPh), 80.6 (CMe), 12.5 (Me).

(15) Crystal data (23 °C): triclinic; space group $P\bar{1}$, a = 11.041 (3) Å, b = 14.556 (4) Å, c = 16.969 (5) Å, α = 103.29 (2)°, β = 92.73 (2)°, γ = 107.64 (2)°, V = 2509.2 (2) Å^3, Z = 4, U = 17.4 cm^{-1}. Of 6871 reflections collected on a Nicolet R3 diffractometer, 6401 were unique and 4767 were considered observed at the $F > 3\sigma(F)$ level. The structure was solved by heavy-atom methods and subsequent difference Fourier syntheses. The asymmetric unit consists of two independent (but not significantly different) complexes each accompanied by a highly disordered molecule of acetone (the recrystallization solvent). Within each Mo complex, further disorder was found regarding interchange of the CO and Cl positions. Occupancy refinement revealed that in each of the two independent molecules the majority structure was as shown in Figure 1 (molecule A, 75%; molecule B, 64%). All non-hydrogen atoms, except as noted below, were refined with anisotropic thermal parameters and with hydrogen atoms in fixed, idealized, updated locations. At convergence R_F = 0.0523, R_{wF} = 0.0524, and GOF = 1.408; a disordered solvent carbon atom, C(A1), and one of the minority CO carbon atoms, C(C3), became nonpositive definite and were restricted to isotropic refinement. Due to insufficient resolution in the intensity data, the following constraints were imposed on the disordered CO and Cl ligands: Mo(1)–C(C1) and Mo(2)–C(C2) distances restricted to 1.975 (2)Å; C(C1)–O(C1) and C(C2)–O(C2) distances restricted to 1.100 (2)Å; fixed positional parameters for the minority atoms Cl(3), Cl(4), C(C3), C(C4), O(C3), and O(C4).

(16) Gusev, A. I.; Alexandrov, G. G.; Struchkov, Yu. T. *J. Struct. Chem. (Engl. Transl.)* **1969**, *10*, 556.

(17) This angle was generated from the atomic coordinates provided in ref 16.

metals[7,18] and to other ligands are currently in progress.

Acknowledgment. R.P.H. acknowledges generous financial support from the National Science Foundation, the donors of the Petroleum Research Fund, administered by the American Chemical Society, and the Alfred P. Sloan Foundation. Funds to purchase the University of Delaware diffractometer were in part provided by the National Science Foundation.

Registry No. **1a**, 82661-46-9; **1b**, 92144-96-2; **1c**, 92144-97-3; **2**, 92144-98-4; **3a**, 92144-99-5; **3b**, 75687-68-2; **4a**, 92145-01-2; **4b**, 92145-03-4; **4c**, 92145-05-6; **5**, 92145-06-7.

Supplementary Material Available: Tables of atomic coordinates and temperature factors, bond lengths, bond angles, anisotropic temperature factors, hydrogen coordinates, and temperature factors (Tables I–V) and observed and calculated structure factors (Table VI) (32 pages). Ordering information is given on any current masthead page.

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(18) Donaldson, W. A.; Hughes, R. P. *J. Am. Chem. Soc.* **1982**, *104*, 4846–4859.

Reactions of Coordinated Molecules. 41. Addition of a Pt–H Bond across a M≡C Bond: A Direct Route to a Heterodinuclear μ-Alkylidene Complex

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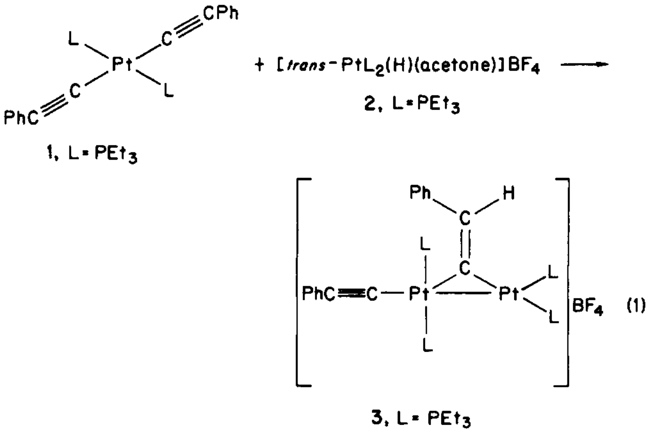
Summary: When the alkylidyne complex $(\eta\text{-}C_5H_5)(OC)_2W{\equiv}C\text{-}C_6H_4CH_3\text{-}p$ is treated with $[trans\text{-}Pt(PEt_3)_2(H)(acetone)]BF_4$, the μ-alkylidene complex $\{(\eta\text{-}C_5H_5)(OC)_2\overline{W[\mu\text{-}\eta^1,\eta^3\text{-}C(C_6H_4CH_3\text{-}p)(H)]Pt}(PEt_3)_2\}BF_4$ is formed. This reaction demonstrates the conversion of a terminal carbyne ligand to a μ-alkylidene complex with concomitant formation of a heterodinuclear cluster.

We reported recently that the Pt–H bond of the cationic metal hydride complex $[trans\text{-}Pt(PEt_3)_2(H)(acetone)]BF_4$, **2**, adds across the C–C triple bond of the neutral phenyl acetylide complex $trans\text{-}Pt(PEt_3)_2(C{\equiv}CPh)_2$, **1**, to give the cationic dinuclear cluster **3** that contains a μ-phenylvinylidene ligand (eq 1).[1]



1, L = PEt$_3$

2, L = PEt$_3$

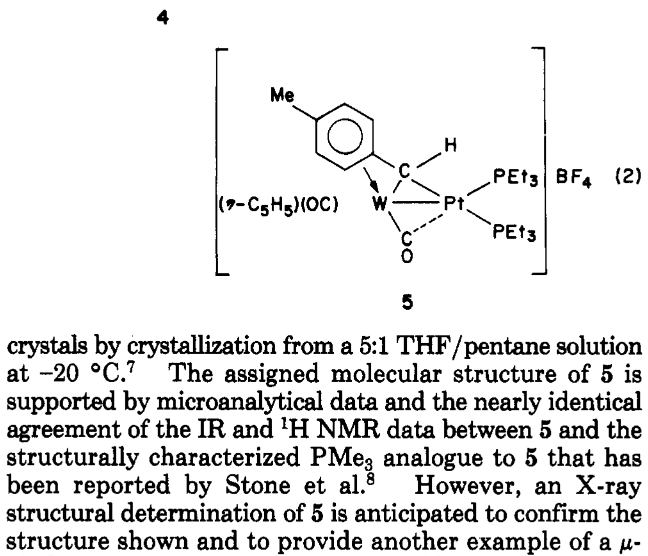
3, L = PEt$_3$

(1)

The formation of **3** by this route represents a new type of bridge-assisted, addition reaction for cluster synthesis.[2] This reaction parallels the well-studied addition of **2** (and related complexes) to the C–C triple bonds of alkynes.[3] The Pt–H bond polarity of **2** is presumably $^{\delta-}$Pt–H$^{\delta+}$,[3a] so the addition of **2** to **1** occurs with the expected regioselectivity of having the electrophilic hydrogen atom attack C_β of the phenyl acetylide ligand.[4] This mode of addition converts the terminal phenyl acetylide ligand into a μ-phenylvinylidene ligand.

The possibility of adding reagents like **2** to other types of unsaturated bonds as a general method for preparing homo- and heterodinuclear clusters is under investigation. We now wish to report that the Pt–H bond of **2** adds across the M–C triple bond of a terminal alkylidyne ligand to form a heterodinuclear cluster which has a μ-alkylidene ligand.

When a solution of 0.175 g (0.43 mmol) of the neutral, terminal alkylidyne complex $(\eta\text{-}C_5H_5)(OC)_2W{\equiv}C\text{-}C_6H_4CH_3\text{-}p$, **4**,[5] in 5 mL of acetone is added to an acetone solution of **2** (0.43 mmol)[6] and the reaction solution is stirred for an additional 1 h at 25 °C, the heterodinuclear cluster **5** is formed (eq 2). Complex **5** is isolated as amber crystals by crystallization from a 5:1 THF/pentane solution at −20 °C.[7] The assigned molecular structure of **5** is supported by microanalytical data and the nearly identical agreement of the IR and ^{1}H NMR data between **5** and the structurally characterized PMe$_3$ analogue to **5** that has been reported by Stone et al.[8] However, an X-ray structural determination of **5** is anticipated to confirm the structure shown and to provide another example of a $\mu\text{-}\eta^1,\eta^3\text{-}C(C_6H_4CH_3\text{-}p)(R)$ ligand type.[8,9] Complex **5** is



4

5

(2)

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(1) Afzal, D.; Lenhert, P. G.; Lukehart, C. M. *J. Am. Chem. Soc.* **1984**, *106*, 3050–3052.

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(4) (a) Bruce, M. I.; Wallis, R. C. *Aust. J. Chem.* **1979**, *32*, 1471–1485. (b) Bruce, M. I.; Swincer, A. G. *Aust. J. Chem.* **1980**, *33*, 1471–1483. (c) Berke, *Z. Naturforsch., B.: Anorg. Chem., Org. Chem.* **1980**, *B35*, 86–90. (d) Bruce, M. I.; Swincer, A. G.; Wallis, R. C. *J. Organomet. Chem.* **1979**, *171*, C5–C8. (d) Davison, A.; Selegue, J. P. *J. Am. Chem. Soc.* **1978**, *100*, 7763–7765.

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(6) Complex **2** is prepared by a procedure analogous to that used in preparing the PF$_6^-$ salt; see: Clark, H. C.; Fiess, P. L.; Wong, C. S. *Can. J. Chem.* **1977**, *55*, 177–178.

(7) Detailed characterization data for complex **5** are as follows: yield 0.18 g, 45%; mp 145–148 °C dec; IR(CH$_2$Cl$_2$) ν(CO) 1960 (vs), 1810 (br, m) cm^{-1}; ^{1}H NMR (CDCl$_3$) δ 1.03 (m, 18, CH$_3$, J_{HH} = 4 Hz, J_{PH} = 8 Hz), 1.94 (m, 12, CH$_2$, J_{HH} = 4 Hz, J_{PH} = 8 Hz), 2.32 (s, 3, aryl CH$_3$), 4.76 (s, 5, C$_5$H$_5$), 5.94 (d, 1, C$_6$H$_4$, J_{HH} = 6 Hz), 6.74 (d, 1, μ-CH, J_{PH} = 7 Hz), 7.00, 7.20, 7.41 (m, 3, C$_6$H$_4$). Anal. Calcd for $C_{27}H_{40}BF_4O_2P_2PtW$: C, 34.97; H, 4.67. Found: C, 34.83; H, 4.70.

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