Organometallics **2005,** *24,* 6295−6297

Regioselective Dimetallapolycarbyl Hydrometalation

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Received September 15, 2005

Summary: The reactions of $[L(CO)_2W{\equiv}CC{\equiv}CC{\equiv}CC{\equiv}W(CO)_2L]$ *(L = HB(pz)*$_3$*, HB(pz′)*$_3$*; pz = pyrazol-1-yl, pz′ = 3,5-dimethylpyrazol-1-yl) with* $[RuHCl(CO)(PPh_3)_3]$ *result in regioselective hydroruthenation of one C≡C bond of the* WC_6W *spine to provide the coordinatively unsaturated hex-2-en-4-yn-3-yl-1,6-diylidyne-bridged complex* $[Ru\{C(C{\equiv}CC{\equiv}W(CO)_2L){=}CHC{\equiv}W(CO)_2L\}Cl(CO)(PPh_3)_2]$*.*

In recent times, much effort has been directed toward the synthesis of bimetallic complexes spanned by linear carbon-only ligands, $L_nM{-}C_x{-}ML_n$. This has been driven, in part, by the anticipation that they might show unusual electrooptical properties and the prospect of intermetallic communication.[1] Accordingly, the central emphasis has been on developing synthetic routes to new examples and less so on investigations into the actual reactivity of the carbon spine. The primary exceptions have been various additions of metal−ligand fragments ($[Co_2(CO)_8]$, $[Mo_2(CO)_4(\eta\text{-}C_5H_5)_2]$, etc.) or $C_2(CN)_4$ to individual C≡C bonds within the MC_xM spines, each of which might be considered to compromise conjugation and electronic communication. We have a longstanding interest in hydroruthenation reactions of the complex $[RuHCl(CO)(PPh_3)_3]$ with alkynes,[2] diynes,[3] and phosphaalkynes,[4] which led us to question whether the dimetallapolycarbyls $L_nMC_xML_n$ might also be prone to hydrometalation. Because alkyne hydrometalation generates (often regioselectively) σ-vinyl complexes, the addition of metal hydrides across C≡C bonds in such systems would not completely disrupt conjugation but rather install metal functionalities along the MC_xM chain. We have recently reported the synthesis of the first dimetallaoctatetraynes, $[L(CO)_2W{\equiv}CC{\equiv}CC{\equiv}CC{\equiv}W(CO)_2L]$ (L = HB(pz)$_3$ (**1a**), HB(pz′)$_3$ (**1b**); pz = pyrazol-1-yl; pz′ = 3,5-dimethylpyrazol-1-yl),[5] and herein report that these can indeed be hydrometalated in a regioselective manner.

Treating a solution of **1a** with $[RuHCl(CO)(PPh_3)_3]$ in THF under reflux results in the formation of a complex formulated as $[Ru\{C(C{\equiv}CC{\equiv}W(CO)_2L){=}CHC{\equiv}W(CO)_2L\}Cl(CO)(PPh_3)_2]$ (L = HB(pz)$_3$ (**2a**), Scheme 1) on the basis of spectroscopic data.[6] Both infrared and NMR data indicate two chemically distinct tungsten termini, while the former also includes an absorption at 1931 cm^{-1}, not far removed from that reported for $[Ru\{C(C{\equiv}CPh){=}CHPh\}Cl(CO)(PPh_3)_2]$ (ν_{CO} 1923 cm^{-1}).[3a] In a similar manner the reaction of **1b** with $[RuHCl(CO)(PPh_3)_3]$ provides the corresponding HB(pz′)$_3$ derivative **2b**, despite the added steric encumbrance of pyrazolyl 3,5-disubstitution. The complexes **2** are somewhat difficult to crystallize; however, a range of options exists for their conversion to more tractable derivatives. On the basis of analogy with the corresponding vinyl, enynyl, and phosphaalkenyl complexes[2−4] and by virtue of the coordinative unsaturation at ruthenium and the lability of the chloride ligand, various

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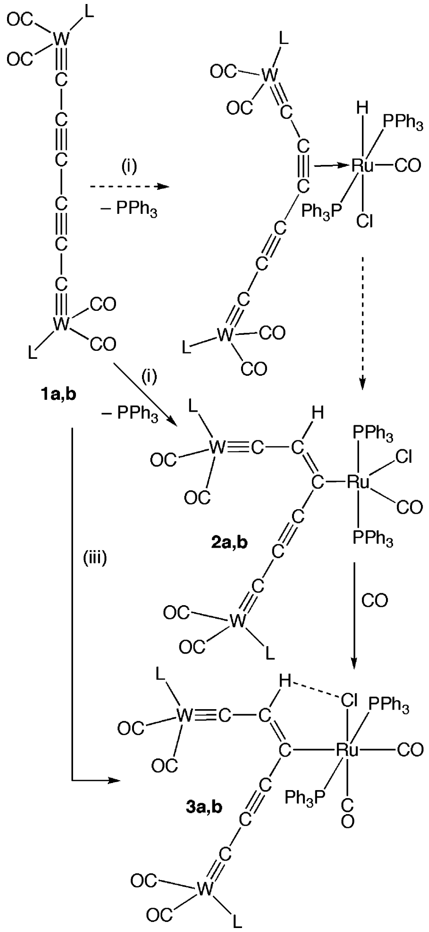
(6) **2a**: a solution of **1a** (50 mg, 0.051 mmol) and $[RuHCl(CO)(PPh_3)_3]$ (46 mg, 0.049 mmol) in THF (10 mL) was heated under reflux for 1 h and then cooled. Addition of hexane and slow evaporation provided a red solid, which was filtered, washed with hexane, and dried in vacuo. Yield: 46 mg (56%). IR (Nujol): 2040 ($\nu_{C\equiv C}$), 1978, 1955, 1872 sh (ν_{WCO}), 1929 (ν_{RuCO}) cm^{-1}. IR (CH$_2$Cl$_2$): 1982, 1958, 1896, 1874 (ν_{WCO}), 1931 (ν_{RuCO}) cm^{-1}. NMR (these and subsequent measurements at 25 °C): ^{1}H (C$_6$D$_6$) δ_H 8.06−6.94 (m, 42 H, C$_6$H$_5$ + H^{3,5}(pz)), 5.93 (2 H), 5.68 (2 H), 5.58 (1 H), 5.54 (1 H) (s × 4, 6 H, H^4(pz)), 5.76 (s, 1 H, Ru−C=CH) ppm; ^{13}C{^{1}H} (C$_6$D$_6$) δ_C 297.9 (W≡*C*CH=), 251.7 (W≡*C*C≡), 227.7, 226.9 (WCO), 195.7 (RuCO), 161.5 (RuC=), 147.2 (2 C), 146.8 (1 C), 145.2 (1 C), 146.0 (2 C) (C^5(pz)), 134.9−127.3 (m, C$_6$H$_5$ + C^3(pz) + methine C(H)=, obscured by benzene solvent signal), 106.6−105.5 (m, C^4(pz)) ppm; ^{31}P{^{1}H} (C$_6$D$_6$) δ_P 35.3 ppm. MS (ESI): *m/z* 1634 [M − Cl]$^+$. Anal. Found: C, 46.84; H, 3.39; N, 9.87. Calcd for $C_{65}H_{51}B_2ClN_{12}O_5P_2RuW_2$: C, 46.81; H, 3.08; N, 10.08. **2b**: a solution of **1b** (50 mg, 0.044 mmol) and $[RuHCl(CO)(PPh_3)_3]$ (42 mg, 0.044 mmol) in THF (10 mL) was heated at reflux for 1 h and cooled. The mixture was filtered, and the solvent was removed under reduced pressure. The dark red residue was crystallized from CH$_2$Cl$_2$ and ethanol, filtered, washed with petroleum ether, and dried in vacuo, affording a dark red solid (ethanol monosolvate). Yield: 62 mg (75%). IR (Nujol): 1976, 1952 (ν_{WCO}), 1927 (ν_{RuCO}), 1888, 1866 (ν_{WCO}) cm^{-1}. IR (CH$_2$Cl$_2$): 1979, 1954 (ν_{WCO}), 1925 (ν_{RuCO}), 1893, 1866 (ν_{WCO}) cm^{-1}. NMR: ^{1}H (C$_6$D$_6$) δ_H 7.86−7.01 (m, 30 H, C$_6$H$_5$), 5.77 (2 H), 5.53 (2 H), 5.39 (1 H), 5.35 (1 H) (s × 4, 6 H, H^4(pz)), 2.54 (3 H), 2.51 (6 H), 2.41 (6 H), 2.33 (3 H), 2.21 (6 H), 2.12 (6 H), 2.10 (3 H), 2.03 (3 H) (s × 8, 36 H, pzCH$_3$-3,5) ppm; ^{13}C{^{1}H} (C$_6$D$_6$) δ_C 287.9 (W≡*C*CH=), 241.6 (W≡*C*C≡), 227.9, 227.0 (WCO), 207.6 (unresolved t, RuCO), 153.0 (2 C), 152.2 (1 C), 152.0 (1 C), 151.9 (2 C) (C^5(pz)), 144.9 (1 C), 144.2 (1 C), 143.9 (2 C), 143.6 (2 C) (C^3(pz)), 135.0 (vt, J_{PC} = 5.5 Hz, C^{2,6}(C$_6$H$_5$)), 132.2 (vt, J_{PC} = 22.0 Hz, C^1(C$_6$H$_5$)), 130.4 (C^4(C$_6$H$_5$)), 128.5 (vt, J_{PC} = 4.8 Hz, C^{3,5}(C$_6$H$_5$)), 106.9 (3 C), 106.8 (1 C), 106.3 (2 C) (C^4(pz)), 17.4 (2 C), 17.0 (2 C), 15.4 (1 C), 15.1 (1 C) (pzMe-3), 12.7 (3 C), 12.4 (3 C) (pzMe-5) ppm (unambiguous assignments could not be made for the remaining ^{13}C nuclei of the C$_6$H ligand due to poor signal to noise); ^{31}P{^{1}H} (C$_6$D$_6$) δ_P 33.2 ppm. MS (ESI): *m/z* 1841 [M − Cl + MeCN]$^+$, 1800 [M − Cl]$^+$, 1619 [M − PPh$_3$ + 2(MeCN)]$^+$, 1538 [M − PPh$_3$]$^+$. Anal. Found: C, 50.80; H, 4.32; N, 8.63. Calcd for $C_{77}H_{75}B_2ClN_{12}O_5P_2RuW_2{\cdot}CH_3CH_2OH$: C, 50.41; H, 4.34; N, 8.93 (ethanol confirmed by ^{1}H NMR integration).

10.1021/om050796g CCC: $30.25

Scheme 1. Dimetallaoctatetrayne Hydroruthenation (a Series, L = HB(pz)$_3$; b Series, HB(pz′)$_3$)[a]



[a] Legend: (i) *mer*-[RuHCl(CO)(PPh$_3$)$_3$], THF, reflux; (ii) CO (1 atm); (iii) *cct*-[RuHCl(CO)$_2$(PPh$_3$)$_2$], THF, reflux.

ligand addition or substitution derivatives are available. Thus, treating **2a** with carbon monoxide (1 atm) results in the quantitative formation of the hexacarbonyl complex [Ru{C(C≡CC≡W(CO)$_2$L)=CHC≡W(CO)$_2$L}Cl(CO)$_2$(PPh$_3$)$_2$] (**3a**),[7] as indicated by the presence of two ruthenium-associated carbonyl absorptions in the infrared spectrum (1978, 2044 cm^{-1}) and a single resonance in the ^{31}P{^{1}H} NMR spectrum (δ_P 23.8) (cf. ν_{CO} 1976, 2042 cm^{-1} and δ_P 24.9 for [Ru{C(C≡CPh)=CHPh}Cl(CO)$_2$(PPh$_3$)$_2$]).[3c] In a similar manner, the complex **3b** was obtained from **2b** and CO (1 atm).[7] An alternative approach to the complexes **3** was also developed by starting with a dicarbonyl precursor, [RuHCl(CO)$_2$(PPh$_3$)$_2$]. Although this complex is less reactive than [RuHCl(CO)(PPh$_3$)$_3$], reaction nevertheless ensues in THF under reflux to provide **3** in somewhat improved overall yields.

Crystallographic grade crystals of **3a** were obtained, and the results of a structure determination are summarized in Figure 1. This confirms (a) the pseudooctahedral geometries at ruthenium and tungsten, notwithstanding the geometric constraints of HB(pz)$_3$ chelation (N−W−N angles 79.83−81.83°), (b) the internally referenced trans influences of the alkylidyne ligands on tungsten relative to the carbonyl ligands (29 and 35σ for W1 and W2, respectively),[8] and (c) the connectivity and hydrometalation regioselectivity of the WC$_6$W spine. Thus, the bond lengths W1≡C101, C101−C102, C102≡C103, C103−104, C104=C105, C105−C106, and C106≡W2 (Figure 1) fall respectively within norms for W≡C, C−C, C≡C, C−C, C=C, C−C, and C≡W bonds. The Ru1−C104 separation of 2.155(3) Å is unusually long for octahedral ruthenium vinyls, while the Ru1−C105−C105 angle of 125.8(2)° is somewhat small, this angle typically lying at about 130°.[9,10] While this presumably reflects the steric bulk of the substituents, it should be noted that there is a weak hydrogen-bonding interaction between the vinylic proton and the chloride ligand (H105···Cl1 = 2.52(2) Å; C105−H105···Cl1 = 126.7(2)°), though it is not clear whether this interaction is a cause or effect of the modest swiveling of the vinyl ligand. The trans influence of the vinyl ligand, relative to chloride, is expressed in the Ru1−C6 bond (1.945(3) Å) being elongated (25σ) relative to that of the adjacent carbonyl (Ru1−C5 = 1.870(3) Å).

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(7) **3a**: a solution of **1a** (21 mg, 0.021 mmol) and [RuHCl(CO)$_2$(PPh$_3$)$_2$] (15 mg, 0.021 mmol) in THF (10 mL) was heated at reflux for 3 h. Addition of hexane and slow evaporation gave a dark red solid, which was filtered, washed with hexane, and dried under vacuum. Yield: 25 mg (70%). IR (Nujol): 2040, 1974 (br) (ν_{RuCO}), 1954 (br), 1873 (br) (ν_{WCO}) cm^{-1}. IR (CH$_2$Cl$_2$): 2044 (ν_{RuCO}), 2021 ($\nu_{C\equiv C}$), 1978 (ν_{RuCO}), 1958, 1877 (br) (ν_{WCO}) cm^{-1}. NMR: ^{1}H (C$_6$D$_6$) δ_H 8.10−6.93 (m, 42 H, C$_6$H$_5$ + H^{3,5}(pz)), 5.87 (t, 2 H, $^3J_{HH}$ = 2.1 Hz, H^4(pz)), 5.72 (t, 2 H, $^3J_{HH}$ = 2.1 Hz, H^4(pz)), 5.60 (t, 1 H, $^3J_{HH}$ = 2.3 Hz, H^4(pz)), 5.55 (t, 1 H, $^3J_{HH}$ = 2.1 Hz, H^4(pz)) ppm (N.B.: the signal due to RuC=CH was not unequivocally identified; δ_H 6.46 for [Ru{C(C≡CPh)=CHPh}Cl(CO)$_2$(PPh$_3$)$_2$][3c]); ^{13}C{^{1}H} (C$_6$D$_6$) δ_C 287.9 (W≡*C*CH=), 241.6 (W≡*C*C≡), 227.7, 226.8 (WCO), 195.7 (unresolved t × 2, RuCO), 161.5 (Ru−*C*=CH), 147.2 (2 C), 146.0 (2 C), 144.2 (1 C), 144.0 (1 C) (C^5(pz)), 135.2 (1 C), 134.5 (3 C) (C^3(pz)), 134.8 (vt, J_{PC} = 5.5 Hz, C^{2,6}(C$_6$H$_5$)), 132.4 (vt, J_{PC} = 23.4 Hz, C^1(C$_6$H$_5$)), 130.6 (C^4(C$_6$H$_5$)), 106.1 (1 C), 106.0 (2 C), 105.8 (1 C), 105.6 (2 C) (C^4(pz)) ppm (signals for C^3(pz) and C^{3,5}(C$_6$H$_5$) are obscured by the C^{2,6}(C$_6$H$_5$) and benzene solvent signals, respectively); ^{31}P{^{1}H} (C$_6$D$_6$) δ_P 23.8 ppm. MS (ESI): *m/z* 1697 (1) [M]$^+$, 1666 (2) [M − CO]$^+$, 1632 [M − (Cl + CO)]$^+$. Satisfactory elemental microanalytical data were not obtained. Crystal data for C$_{66}$H$_{51}$B$_2$ClN$_{12}$O$_6$P$_2$RuW$_2$·3C$_6$H$_6$: M_w = 1930.29, monoclinic, $P2_1/n$, a = 13.461(3) Å, b = 18.110(4) Å, c = 33.907(7) Å, β = 95.00(3)°, V = 8234(3) Å^3, Z = 4, ρ_{calcd} = 1.557 g cm^{-3}, μ(Mo Kα) = 3.099 mm^{-1}, T = 200(2) K, red plate, 18 891 independent measured reflections ($2\theta \leq 55°$), R1 = 0.0279, wR2 = 0.0604 for 15 209 independent observed absorption-corrected reflections ($I > 2\sigma(I)$), 994 parameters, CCDC 283725. Procedure a for **3b**: a solution of **2b** (50 mg, 0.027 mmol) in CH$_2$Cl$_2$ (10 mL) was partially evacuated and then back-filled and stoppered under a slight overpressure of CO gas. The mixture was stirred for 1 day, after which addition of hexane and slow evaporation provided a dark red solid, which was filtered, washed with hexane, and dried under vacuum. Procedure b for **3b**: a suspension of **1b** (100 mg, 0.087 mmol) and [RuHCl(CO)$_2$(PPh$_3$)$_2$] (60 mg, 0.083 mmol) in THF (10 mL) was heated at reflux for 6 h. Addition of hexane and slow evaporation provided a red solid, which was filtered, washed with hexane, and dried under vacuum. Yield: 150 mg (97%). IR (Nujol): 2042, 1969 (ν_{RuCO}), 1950 (br), 1870 (br) (ν_{WCO}) cm^{-1}. IR (CH$_2$Cl$_2$): 2048, 1976 (ν_{RuCO}), 1952, 1874 (br) (ν_{WCO}) cm^{-1}. NMR: ^{1}H (C$_6$D$_6$) δ_H 8.02−6.96 (m, 30 H, C$_6$H$_5$), 5.66 (2 H), 5.63 (2 H), 5.34 (1 H), 5.31 (1 H) (s × 4, 6 H, H^4(pz)), 2.55 (6 H), 2.53 (3 H), 2.50 (6 H), 2.38 (3 H), 2.21 (6 H), 2.17 (6 H), 2.09 (3 H), 2.06 (3 H) (s × 8, 36 H, pzCH$_3$-3,5) ppm; ^{13}C{^{1}H} (C$_6$D$_6$) δ_C 283.6 (W≡*C*C=C), 244.9 (W≡*C*C≡C), 228.6, 227.4 (WCO), 195.9, 194.8 (RuCO), 163.1 (Ru−C=), 153.1−152.3 (m, C^5(pz)), 144.2 (1 C), 144.0 (1 C), 143.7 (2 C), 143.5 (2 C) (C^3(pz)), 134.8 (br, C^{2,6}(C$_6$H$_5$)), 130.5 (C^4(C$_6$H$_5$)), 107.1−106.3 (m, C^4(pz)), 53.3 (W≡CC≡*C*), 18.1−15.3 (pzCH$_3$-3), 12.5 (3 C), 12.4 (3 C) (pzCH$_3$-5) ppm (signals for C^1(C$_6$H$_5$) are obscured by the C$_6$D$_6$ solvent peak; resonances corresponding to the remaining nuclei of the C$_6$H ligand could not be unequivocally identified); ^{31}P{^{1}H} (C$_6$D$_6$) δ_P 19.8 ppm. MS (high-resolution ESI): *m/z* 1865.3562 [M + H]$^+$ (calcd 1865.3423), 1842.4229 [M − Cl − CO + MeCN]$^+$ (calcd 1842.3972). Anal. Found: C, 50.12; H, 4.07; N, 8.81. Calcd for C$_{78}$H$_{75}$B$_2$ClN$_{12}$O$_6$P$_2$RuW$_2$: C, 50.25; H, 4.05; N, 9.02.

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(9) Table S1 (Supporting Information) collates structural data[10] for a range of octahedral σ-vinyl complexes of ruthenium to have been characterized since such data were last tabulated in ref 2b.

(10) Conquest, Version 1.7; Cambridge Crystallographic Data Centre, Cambridge, U.K., Aug 2005 release.

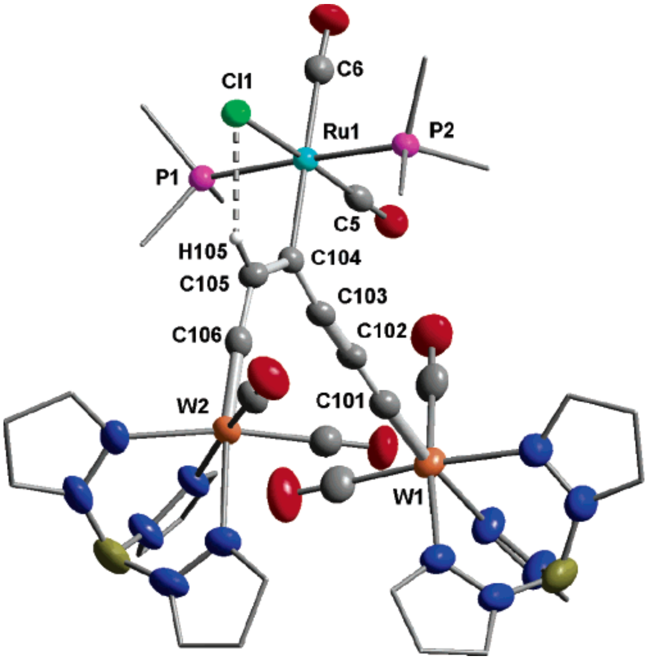

Figure 1. Molecular geometry of the complex **3b** in a crystal of **3b**·C_6H_6 (pyrazolyl and phenyl carbon atoms simplified for clarity). Selected bond distances (Å) and angles (deg): W1≡C101 = 1.842(3), Ru1–C5 = 1.870(3), Ru1–C6 = 1.945(3), Ru1–C104 = 2.155(3), Ru1–P2 = 2.4054(9), Ru1–P1 = 2.4090(9), Ru1–Cl1 = 2.4550(9), W2≡C106 = 1.832(3), C101–C102 = 1.365(4), C102≡C103 = 1.223(4), C103–C104 = 1.397(4), C104=C105 = 1.372(4), C105–C106 = 1.417(4), H105···Cl1 = 2.524(15); C102–C101–W1 = 173.4(3), C103–C102–C101 = 178.1(3), C102–C103–C104 = 173.0(3), C105–C104–C103 = 118.0(3), C105–C104–Ru1 = 125.8(2), C103–C104–Ru1 = 116.15(19), C104–C105–C106 = 123.8(3), C105–C106–W2 = 175.9(2), C105–H105···Cl1 = 126.7(2).

The regioselectivity of the hydrometalation is noteworthy in that the ditungstaoctatetrayne has four potential multiple bonds (two W≡C and two C≡C) across which the Ru–H bond might in principle add. Alkyne hydrometalation is a fundamental and widely encountered organometallic transformation; however, in keeping with the isolobal perspective, it has also been demonstrated that metal–carbon triple bonds may be hydrometalated.[11,12] Given that one of the earliest demonstrations of this process involved hydroplatination of the complexes [W(≡CR)(CO)$_2${HB(pz)$_3$}] (R = Ph, NEt_2, 2-furyl, Me),[11b] we may safely conclude that hydroruthenation of the W≡C linkage of **1a** is at least plausible though probably excluded for **1b** on steric grounds. Thus, we suspect that the observed regiochemistry, in which the C≡C bond is preferentially reduced, reflects a thermodynamic preference. The H_{β}–Ru_{γ} arrangement (cf. Ru_{β}–H_{γ}) presumably also reflects the best accommodation of the steric bulk of the "W(CO)$_2${HB(pz)$_3$}" fragments, as remote as possible from ruthenium.

Metal–hydride bonds are but one example of metal–element bonds that can add across the triple bond of alkynes (or alkylidynes). Accordingly, one might envisage the installation of a metal center cis to a range of heteroatom substituents (B, Si, S, N, O) along a dimetallapolycarbyl chain which still retains a degree of conjugation by virtue of the resulting sp^2 carbon centers—a possibility we are currently investigating.

Supporting Information Available: A CIF file, giving details of the crystal structure determination of **3a**·3C_6H_6 (CCDC 283725), and Table S1, collating relevant structural data for 5- and 6-coordinate ruthenium σ-vinyls. This material is available free of charge via the Internet at http://pubs.acs.org.

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