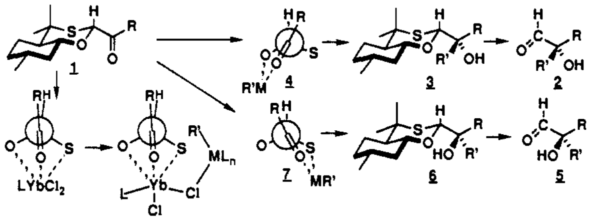
Table I. Reaction of 2-Acyl-1,3-oxathiane **1** with 1-Pentynylmetals (R′M)[a]

	1 (R = Me)		**1** (R = Et)		**1** (R = Ph)	
R′M (equiv)	M = Li **3** + **6**[b] [**3**/**6**][c]	M = MgBr **3** + **6** [**3**/**6**]	M = Li **3** + **6** [**3**/**6**]	M = MgBr **3** + **6** [**3**/**6**]	M = Li **3** + **6** [**3**/**6**]	M = MgBr **3** + **6** [**3**/**6**]
R′M (2)	84 [74/26]	99 [66/34]	94 [75/25]	78 [84/16]	98 [91/9]	98 [97/3]
R′M (2) + $CeCl_3$ (3)	66 [11/89]	57 [10/90]	99 [7/93]	46 [38/62]	99 [44/56]	41 [38/62]
R′M (2) + $YbCl_3$ (3)	99 [3/97]	35 [2/98]	99 [2/98]	39 [1/99][d]	52 [6/94]	0
R′M (2) + YCl_3 (3)	63 [4/96]	18 [4/96]	99 [6/94]	99 [6/94]	56 [6/94]	0

[a] Experimental procedure is described in ref 9. [b] Yield (%). [c] Ratio was determined by ^{1}H NMR. [d] Chemical yield 99% (**3**/**6** = 0/100) when 5-min-aged R′MgBr + $YbCl_3$ reagent was used.

Scheme I



6 exclusively; however, the yield of product is low. Reactions with YCl_3- and $CeCl_3$-mediated species show high diastereoselectivity giving **6**. Results are included in Table I.[9]

Considerable attention has been focused on the reaction with $YbCl_3$-mediated Grignard reagents. A THF solution of R′MgBr was treated at 0 °C with a suspension of $YbCl_3$ in THF, and the mixture was immediately used in the reaction with **1** at −78 °C for 2 h, or the mixture was stirred at 0 °C for an aging period before reaction with **1**. Combinations of aging time, yield, and ratio **3**/**6**; 0 min, >99%, 0.2/99.8; 5 min, >99%, 0/100; 30 min, 78%, 0/100; 2 h, 39%, 0.4/99.6; 18 h, 0%. Reaction of **1** (R = Et) with a 2-h-aged reagent at 0 °C for 3 h gives **6** (95.4% de) quantitatively.

These results suggest the following: (1) Reaction of R′MgBr with $YbCl_3$ gives a reactive species R′MgBr·$YbCl_3$ (**8**) which shows high reactivity and diastereoselectivity. (2) Transmetalation giving $(R'YbCl_2)_2$ (**9**) proceeds slowly at 0 °C and is completed within 18 h; unchanged substrate **1** was recovered in the reaction with **9** at −78 °C. (3) Since the reaction of **1** with **8** proceeds smoothly at −78 °C but sluggishly with **9** at the same temperature, the nucleophilic reactivity of **8** is higher than that of **9**.

It is important to note that ytterbium plays the principal role in the control of diastereoselectivity of the reaction, in spite of the existence of two metal species, ytterbium and magnesium, in the reaction mixture. These observations suggested that the same type of diastereoselective reaction could proceed by the addition of a Grignard reagent to a mixture of **1** and $YbCl_3$. This expectation was realized in the addition of a THF solution of 1-pentynylmagnesium bromide to a 1/3 mixture of **1** and $YbCl_3$ in THF at −78 °C to give **6** (R = Me, 98.6% de) in quantitative yield.

Although the mechanism of reaction of **1** with Yb-mediated reagents has not been clearly understood, a possible mechanism to account for the high diastereoselectivity is depicted in chelate transition structure **11** (reaction with **8**; ML_n = MgBr, L′ = Cl) and **12** (reaction with **9**; ML_n = $YbCl_2$, L′ = R′).[10,11]

Acknowledgment. This work was supported by the Ministry of Education, Science and Culture by a Grant-in-Aid for Scientific Research in Priority Areas (01649005) and by the Japan Society for the Promotion of Science by a Grant-in-Aid for an International Joint Research Project.

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[9] (a) Typical procedure using $CH_3CH_2CH_2C{\equiv}CYbCl_2$: Finely pulverized $YbCl_3{\cdot}6H_2O$ (485 mg, 1.25 mmol) was heated to 150 °C under vacuum for 2 h. Then 4 mL of THF was added to the cooled material all at once with vigorous stirring. The mixture was sonicated. To this suspension, maintained at −78 °C, was added a THF solution of 1-pentynyllithium (R′Li, 0.83 mmol; 1.22 mL of 0.68 M solution), and the reaction mixture was stirred at −78 °C for 2 h. A THF solution of **1** (R = Et, 107 mg, 0.42 mmol in 4 mL of THF) was then added at −78 °C. The reaction mixture was stirred at −78 °C for 3 h and then quenched with saturated NH_4Cl solution. (b) Typical procedure using R′MgBr + $YbCl_3$: A THF solution of R′MgBr was added to a suspension of $YbCl_3$ in THF at 0 °C. The resultant mixture was stirred at 0 °C for 2 h and then used for the reaction with **1** at −78 °C as described above.

[10] Synergistic rate enhancement with both O and S (relative rate: 3-ethyl-4-heptanone/3-(phenylthio)-4-heptanone/3-methoxy-4-heptanone/1-methoxy-1-(phenylthio)-2-pentanone = 1/2.2/27/49, obtained by competitive reactions with $MeYbCl_2$) suggests that Yb coordinates with two O and one S to give **10**. As the S–C bond is longer than O–C, attack of R′Mg or R′Yb species to **10** from the less hindered side produces **6** via **11** or **12**.

[11] The activation energy of the reaction of dimeric RLi with a carbonyl compound is lower than that of the monomeric species: Kaufmann, E.; Schleyer, P. v. R.; Houk, K. N.; Wu, Y.-D. *J. Am. Chem. Soc.* **1985**, *107*, 5560.

Tungsten Nitrene Complexes $Tp'W(CO)_2(NR)^+$ (R = Ph, Bu^t) Generated from Amido Complexes $Tp'W(CO)_2(NHR)$

Li Luan, Peter S. White, M. Brookhart,* and
J. L. Templeton*

Department of Chemistry, University of North Carolina
Chapel Hill, North Carolina 27599-3290

Received June 11, 1990

Metal nitrene (or imido) chemistry has expanded[1] since olefin amination with d^0 osmium(VIII) reagents was achieved.[2] Zirconium nitrene monomers display insertion reactions[3] and activate CH bonds,[4] and a d^6 iridium nitrene, Cp*IrNBu^t, inserts a variety of electrophilic organic substrates.[5] Nonetheless, well-characterized electrophilic nitrene complexes remain elusive. Aziridine products form from nitrene transfer to cyclooctene after acylation of manganese porphyrin nitride with trifluoroacetic anhydride,[6] and tosyl nitrene insertion reactions have been mediated with metal porphyrins.[7,8] Group VI nitrene complexes such as $X(dppe)_2W(NH)^+$,[9] $L_2Cl_2(CO)W(NPh)$,[10] and $(Et_2NCS_2)_2Mo(NPh)_2$[11] do

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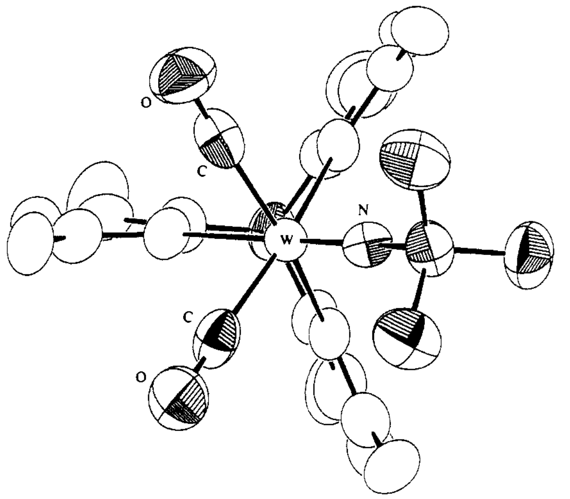


Figure 1. ORTEP drawing of $Tp'(CO)_2W(NBu^t)^+$.

not appear to add nucleophiles at nitrogen, but trapping of the proposed transient, $(OC)_5W(NPh)$, with PPh_3 reflects nitrogen electrophilicity in this low-valent nitrene,[12] and conversion of the high-valent $(RO)_2Cr(NBu^t)_2$ reagent to $[(RO)(Bu^tN)Cr(NPhBu^t)]_n$ with Ph_2Zn[13] also involves net nucleophilic addition to the nitrene ligand. We now report a convenient synthesis of cationic carbonyl nitrene derivatives of tungsten(IV) whose chemistry suggests electrophilic character at nitrogen.

Oxidation of $[Et_4N][Tp'W(CO)_3]$ [Tp′ = hydrotris(3,5-dimethylpyrazolyl)borate] with elemental iodine yields $Tp'(CO)_3WI$, which reacts with either aniline or *tert*-butylamine to form a dicarbonyl(amido)tungsten(II) d^4 complex $Tp'(CO)_2W(NHR)$ (**1**, R = Bu^t; **2**, R = C_6H_5) (eq 1). Analytically pure products

$$Tp'(CO)_3WI + \underset{\substack{R = Bu^t,\\ C_6H_5}}{NH_2R} \xrightarrow[\Delta]{THF} Tp'(CO)_2W(NHR) \quad (1)$$

were obtained following chromatography on alumina. These complexes display infrared absorptions at 1910 and 1782 cm^{-1} for **1** and at 1900 and 1786 cm^{-1} for **2**, assigned to the cis-carbonyl vibrations, and low-field ^1H NMR signals [**1**, 14,2 ppm; **2**, 15.4 ppm (major isomer)] assigned to the amido proton.[5]

Oxidation of either of the amido complexes with iodine produces a cationic nitrene monomer with iodide as the counterion (eq 2).

$$\underset{R = Bu^t,\ C_6H_5}{Tp'(CO)_2W(NHR)} + I_2 \xrightarrow{CH_2Cl_2} [Tp'(CO)_2W(NR)][I] + HI \quad (2)$$

The net result of this oxidation is simple hydride abstraction from the amido nitrogen; alternate syntheses of the nitrene cations are accessible with trityl hexafluorophosphate as the oxidant (eq 3).

$$\underset{R = Bu^t,\ C_6H_5}{Tp'(CO)_2W(NHR)} + [Ph_3C][PF_6] \xrightarrow{CH_2Cl_2} [Tp'(CO)_2W(NR)][PF_6] + Ph_3CH \quad (3)$$

Formation of the cation $[Tp'(CO)_2W(NR)^+]$ is evident in the high-frequency carbonyl stretches (**3**, 2081 and 2003 cm^{-1}; **4**, 2079 and 2006 cm^{-1}). Tungsten couplings to the ipso carbon of the nitrene phenyl substituent and to the tertiary carbon of the nitrene *tert*-butyl substituent are observed (**3**, $^2J_{WC}$ = 23 Hz; **4**, $^2J_{WC}$ = 31 Hz); similar couplings have been noted in tungsten carbyne complexes.[14] Structures of **3** and **4** (Figures 1 and 2) reveal nearly linear W–N–C linkages with typical W–N multiple bond lengths [**3**, 172 (1)° and 1.72 (1) Å; **4**, 171.6 (6)° and 1.755 (7) Å].[15]

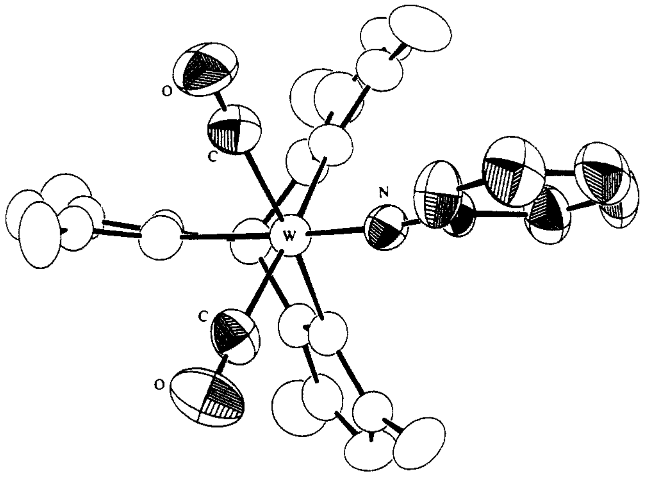
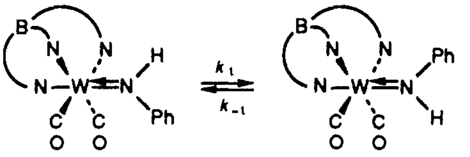


Figure 2. ORTEP drawing of $Tp'(CO)_2W(NPh)^+$.

^1H NMR measurements establish that the amido complex **2** exists as a pair of equilibrating isomers in solution. Dissolution of crystalline **2** in CD_2Cl_2 at 22 °C reveals a single amido species with an NH signal at 15.3 ppm. Over 5 days a new NH at 13.0 ppm grows in to account for 12% of the total material. Kinetic measurements at 22 °C establish first-order rates of interconversion with $k_1 = 7.0 \times 10^{-6}\ s^{-1}$, $\Delta G^\ddagger$ = 24.2 kcal/mol, $k_{-1} = 5.2 \times 10^{-5}\ s^{-1}$, $\Delta G^\ddagger$ = 23.0 kcal/mol, K_{eq} = 0.13. As summarized in eq 4, we propose that this isomerism reflects restricted rotation



(4)

around the tungsten–amide multiple bond, the major isomer being the sterically favored one in which the phenyl group is oriented away from Tp′. Optimal π-bonding occurs when the amide lies in the mirror plane and is independent of whether the phenyl group is syn or anti.[16] No minor isomer was detected in the bulkier *tert*-butyl-substituted complex **1**.

Electrophilic character at the nitrogen atoms in the nitrene complexes **3** and **4** is suggested by their facile hydride reductions. Treatment of the phenyl nitrene complex **4** with lithium borohydride in CH_3CN at −40 °C followed by warming to 25 °C results in spectroscopically pure amido complex **2** (80% isolated

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(15) (a) Data for both structures were collected on an Enraf-Nonius CAD4 diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) using an ω–2θ scan. Reflections with $I > 2.5\sigma(I)$ were considered observed and included in subsequent calculations. The structures were solved by direct methods, and an empirical absorption correction was applied using DIFABS (Walker, N.; Stuart, D. *Acta Crystallogr.* **1983**, *A39*, 158). Refinement was by full-matrix least squares. Hydrogen atoms were included in calculated positions with thermal parameters derived from the atom to which they were bonded. All computations were performed by using the NRCVAX suite of programs: Gabe, E. J.; Le Page, Y.; Charland, J.-P.; Lee, F.; White, P. S. *J. Appl. Crystallogr.* **1989**, *22*, 384. (b) Crystal data for **3**: triclinic, $P\bar{1}$; a = 10.956 (2) Å, b = 12.521 (7) Å, c = 12.823 (3) Å, α = 84.85 (3)°, β = 76.91 (2)°, γ = 74.03 (3)°; Z = 2. Diffraction data: 4283 unique reflections with $2\theta < 45°$ with 3096 considered observed, μ = 3.86 mm^{-1}, transmission factors 0.47–0.76. Refinement data: R = 0.057, R_W = 0.061, goodness of fit = 1.50; highest final peak, 1.34 e/Å^3; deepest hole, −1.30 e/Å^3. (c) Crystal data for **4**: monoclinic, $P2_1/n$; a = 10.301 (2) Å, b = 10.389 (2) Å, c = 27.816 (7) Å, β = 97.69 (2)°; Z = 4. Diffraction data: 3827 unique reflections with $2\theta < 45°$ with 2885 considered observed, μ = 4.12 mm^{-1}, transmission factors 0.39–0.49. Refinement data: R = 0.036, R_w = 0.045, goodness of fit = 1.49; highest final peak, 0.68 e/Å^3, deepest hole, −0.63 e/Å^3.

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yield). Similarly, the *tert*-butyl-substituted nitrene complex **3** yields only amido complex **1**.

This system provides convenient access to gram quantities of electrophilic nitrene monomers of tungsten(IV). Efforts to utilize the reagents for nitrene transfer reactions are underway.

Acknowledgment. We are grateful to the National Institutes of Health (GM28938) and the Department of Energy (DE-FG05-85ER13430) for financial support.

Supplementary Material Available: Full experimental details, including preparations and spectral and analytical data (infrared, ^{1}H NMR, ^{13}C NMR, elemental analyses) for complexes **1–4**, and X-ray diffraction data for **3** and **4**, including tables of crystal data, bond distances and angles, fractional atomic coordinates, and anisotropic thermal parameters (30 pages); tables of observed and calculated structure factors for **3** and **4** (26 pages). Ordering information is given on any current masthead page.

Reduction of Phenylacetylene in $[Tp'(CO)_2W(PhC_2H)][BF_4]$ To Form a β-Agostic Methylphenylcarbene Ligand

S. G. Feng, P. S. White, and J. L. Templeton*

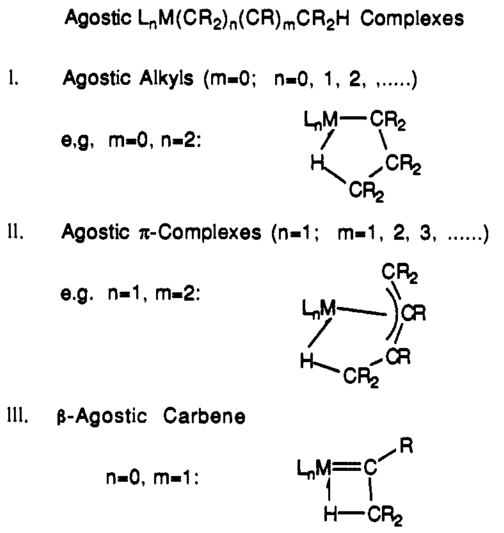
Department of Chemistry, University of North Carolina
Chapel Hill, North Carolina 27599

Received July 2, 1990

Agostic bonds have proliferated[1] since the first insightful review by Brookhart and Green in 1983.[2] Numerous α-agostic carbenes, alternatively described as protonated carbynes, of both groups V[3] and VI[4] have been reported by Schrock and co-workers. The paradigm for olefin insertion and polymerization reactions involves β-agostic alkyls.[5] Four-electron-donor alkyne ligands, common for group VI,[6] provide access to η^2-vinyl ligands[7] which are precursors to β-agostic carbene products as reported here.

Addition of a nucleophile (H^-, Me^-) to the terminal carbon of the phenylacetylene ligand in $[Tp'(CO)_2W(PhC{\equiv}CH)][BF_4]$ [Tp' = hydridotris(3,5-dimethylpyrazolyl)borate] forms an η^2-vinyl ligand which can be protonated to form an alkylphenylcarbene ligand. The agostic bond present in $[Tp'(CO)_2W{=}C(Ph)CH_2R][BF_4]$ (R = H, Me), described in detail below, complements the range of saturated and unsaturated agostic ligands represented in Chart I. In a sense, the β-agostic carbene resembles both agostic π-complexes with unsaturation in the organic ligand and Schrock α-agostic carbenes with unsaturation in the metal–carbon bond.

Chart I

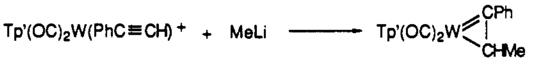


Oxidation of $[NEt_4][Tp'W(CO)_3]$[8] with iodine provides access to d^4 metal chemistry via the $Tp'W(CO)_3I$ monomer (eq 1).

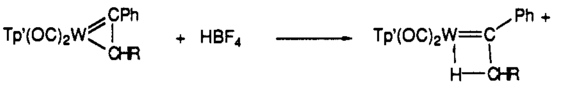
$$Tp'W(CO)_3^- + I_2 \longrightarrow Tp'W(CO)_3I + I^- \qquad (1)$$

$$Tp'(CO)_3I + AgBF_4 + PhC{\equiv}CH \longrightarrow [Tp'(OC)_2W(PhC{\equiv}CH)][BF_4] \qquad (2)$$

$$Tp'(OC)_2W(PhC{\equiv}CH)^+ + LiHBEt_3 \longrightarrow Tp'(OC)_2W(\eta^2\text{-}CPh{=}CH_2) \qquad (3)$$



$$Tp'(OC)_2W(PhC{\equiv}CH)^+ + MeLi \longrightarrow Tp'(OC)_2W(\eta^2\text{-}CPh{=}CHMe) \qquad (4)$$



$$Tp'(OC)_2W(\eta^2\text{-}CPh{=}CHR) + HBF_4 \longrightarrow [Tp'(OC)_2W{=}C(Ph)CHR(H)]^+ \qquad (5)$$

Iodide removal with silver tetrafluoroborate in the presence of phenylacetylene yields a dark forest green cationic alkyne complex, $[Tp'(CO)_2W(PhC{\equiv}CH)][BF_4]$ (eq 2). This dicarbonyl derivative (ν_{CO} = 2057 and 1970 cm^{-1}) displays classic four-electrn-donor alkyne properties[6] (^{1}H NMR, δ = 14.0 ppm, $\equiv CH$; ^{13}C NMR, 197 ppm, d, $^1J_{CH}$ = 223 Hz, $\equiv CH$, 225 ppm, $\equiv CPh$).

Nucleophilic addition at the terminal acetylene carbon can be achieved with either $Li[Et_3BH]$ (eq 3) or MeLi (eq 4) to form neutral η^2-vinyl complexes. The carbenoid character of C_α, bearing the phenyl group, is evident in the low-field ^{13}C chemical shift (η^2-$CPh{=}CH_2$, 234 ppm; η^2-$CPh{=}CHMe$, 265 ppm). NMR data for these complexes is similar to data reported by Green and co-workers for η^2-vinyl ligands in a series of $(\pi\text{-}C_5H_5)[P(OMe)_3]_2Mo(\eta^2\text{-}CR{=}CR_2)$ complexes.[9]

Protonation of the original alkyne terminal carbon results from addition of tetrafluoroboric acid to the neutral η^2-vinyl monomers (eq 5). The net result of sequential H^-, H^+ addition to the terminal alkyne carbon is conversion of $PhC{\equiv}CH$ to $Ph\ddot{C}CH_3$. While electrophilic addition to the C_β site of η^1-vinyl ligands to form nonagostic carbenes maintains the metal electron count,[10] a similar

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