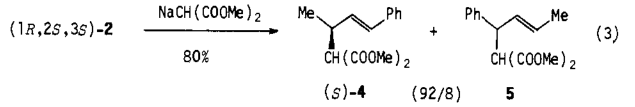
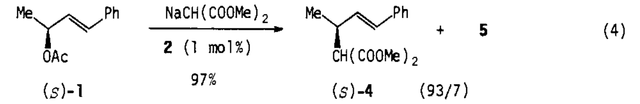
from the acetate (*S*)-**1** is a 1*R*,2*S*,3*S* isomer of 47% ee, indicating that the oxidative addition proceeded with inversion in 81% stereoselectivity.[27]

The π-allylpalladium 1,2-bis(diphenylphosphino)ethane complex (1*R*,2*S*,3*S*)-**2** (47% ee) was allowed to react with dimethyl sodiomalonate in THF to give 74% yield of (*S*)-dimethyl[1-((*E*)-styryl)ethyl]malonate[28] (**4**) ($[\alpha]^{20}_D$ −26° (*c* 0.8, chloroform), 38% ee) and 6% yield of its regioisomer **5** (eq 3). Use of the complex **2** as a catalyst (1 mol %) for the reaction of (*S*)-**1** (58% ee) with dimethyl sodiomalonate brought about the formation of (*S*)-**4**[28] ($[\alpha]^{20}_D$ −40° (*c* 1.0, chloroform), 58% ee) in a high yield (eq 4). These stereochemical results, inversion and retention obtained here for the stoichiometric and catalytic alkylation, respectively, confirm those reported by use of diasteromeric π-allylpalladium complexes[2-4] and allylic acetates.[10-13]



(3)



(4)

Thus, the stereochemistry in each step of the catalytic cycle has been verified in the enantiomeric system: inversion at the oxidative addition and inversion at the alkylation of π-allylpalladium with dimethyl sodiomalonate, leading to net retention in catalytic alkylation.

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[27] The loss of enantiomeric purity may be attributable to attack of acetate anion on the π-allyl moiety from the same side as palladium, which generates (*R*)-**1**. See ref 10 and: Bäckvall, J.-E.; Nordberg, R. E. *J. Am. Chem. Soc.* **1981**, *103*, 4959.

[28] The maximum specific rotation of (*R*)-**4** is $[\alpha]^{20}_D$ +68.9° (*c* 1, CHCl$_3$): Hayashi, T.; Konishi, M.; Kumada, M., unpublished results.

Low-Temperature Photochemistry of (η^5-C$_5$R$_5$)W(CO)$_3$CH$_2$SiMe$_2$H: Loss of Carbon Monoxide Followed by β-Hydrogen Transfer

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We report that near-UV irradiation of (η^5-C$_5$R$_5$)W(CO)$_3$CH$_2$SiMe$_2$H (R = H, Me) at 77 or 196 K in hydrocarbon media results in loss of CO and the Si–H bond to give *cis*-(η^5-C$_5$R$_5$)W(CO)$_2$(H)(CH$_2$SiMe$_2$). Such a species has been proposed as an intermediate in the conversion represented by eq 1.[1] We

$$(\eta^5\text{-C}_5\text{H}_5)\text{Fe(CO)}_2\text{CH}_2\text{SiMe}_2\text{H} \rightarrow (\eta^5\text{-C}_5\text{H}_5)\text{Fe(CO)}_2\text{SiMe}_3 \qquad (1)$$

undertook this study with the knowledge[2] that (η^5-C$_5$R$_5$)W(CO)$_3$(alkyl) complexes undergo light-induced loss of CO in rigid hydrocarbon media at low temperature to give spectroscopically detectable 16e$^-$ (η^5-C$_5$R$_5$)W(CO)$_2$(alkyl) complexes. For alkyl groups having β-H's, warm-up of the 16e$^-$ species yields *trans*-(η^5-C$_5$R$_5$)W(CO)$_2$(H)(alkene) complexes.[2ab,e] We hoped to observe the related transfer of a β-H from a Si to the W atom. We chose to examine the W and not the Fe system in eq 1, because the alkene complex from (η^5-C$_5$H$_5$)Fe(CO)$_2$C$_2$H$_5$ is exceedingly labile.[3]

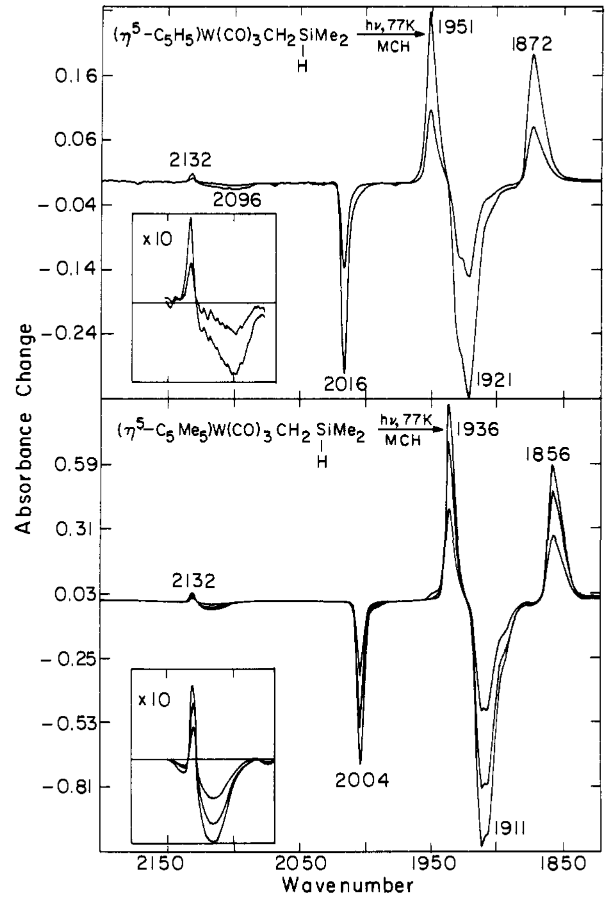

Figure 1. IR difference spectra (Nicolet 7199 FTIR) accompanying near-UV irradiation of ~1 mM (η^5-C$_5$R$_5$)W(CO)$_3$CH$_2$SiMe$_2$H (top, R = H; bottom, R = Me) at 77 K in methylcyclohexane. The successive scans (greater optical changes) are for additional irradiation times to give ultimately ~50% consumption of starting material.

Figure 1 shows IR spectral changes accompanying near-UV irradiation of (η^5-C$_5$R$_5$)W(CO)$_3$CH$_2$SiMe$_2$H at 77 K in a deoxygenated methylcyclohexane matrix.[4] Disappearance of the starting tricarbonyl is rapid and is associated with the dissociative loss of CO, as evidenced by the growth of absorption at 2132 cm^{-1} due to uncomplexed CO.[5] The absorbance change at 2132 cm^{-1} compared to the absorbance changes of the two bands in the CO stretching region of the starting tricarbonyl shows that one CO is generated per molecule of tricarbonyl consumed, within an experimental error of ~±20%.[6] It is noteworthy that the absorption associated with the Si–H bond (~2100 cm^{-1}) of the starting complex declines in a manner consistent with loss of one Si–H bond per CO molecule generated. In addition to the growth

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[1] (a) Pannell, K. H. *J. Organomet. Chem.* **1970**, *21*, P17. (b) β-H transfer was also proposed for a RhCH$_2$SiMe$_2$H complex: Cundy, C. S.; Lappert, M. F.; Pearce, R. *Ibid.* **1973**, *59*, 161.

[2] (a) Wrighton, M. S.; Kazlauskas, R. J. *J. Am. Chem. Soc.* **1982**, *104*, 6005; **1980**, *102*, 1727. (b) Alt, H. F.; Eichner, M. E. *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 78. (c) Mahmoud, K. A.; Narayanaswamy, R.; Rest, A. J. *J. Chem. Soc., Dalton Trans.* **1981**, 2199. (d) Hitam, R. B.; Hooker, R. H.; Mahmoud, K. A.; Narayanaswamy, R.; Rest, A. J. *J. Organomet. Chem.* **1981**, *222*, C9. (e) Klein, B.; Kazlauskas, R. J.; Wrighton, M. S. *Organometallics* **1982**, *1*, 1338.

[3] Kazlauskas, R. J.; Wrighton, M. S. *Organometallics* **1982**, *1*, 602.

[4] The (η^5-C$_5$R$_5$)W(CO)$_3$CH$_2$SiMe$_2$H complexes were synthesized in a manner analogous to that for (η^5-C$_5$H$_5$)Fe(CO)$_2$CH$_2$SiMe$_2$H: Bulkowski, J. E.; Miro, N. D.; Sepelak, D.; Van Dyke, C. H. *J. Organomet. Chem.* **1975** *101*, 267. Low-temperature irradiations were carried out as described in ref 2a, 2e, and 3.

[5] Leroi, G. E.; Ewing, G. E.; Pimentel, G. C. *J. Chem. Phys.* **1964**, *40*, 2298.

[6] A variety of metal carbonyls known to lose CO upon irradiation in rigid media have been examined under the same conditions to establish an extinction coefficient of ~300 M^{-1} cm^{-1} for CO in an alkane matrix. These results will be reported in a full paper.

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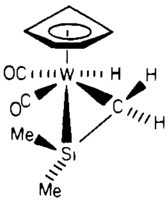
Table I. NMR Data for Relevant Complexes[a]

complex	^{1}H, δ[b]	^{13}C, δ[b]
$(\eta^5\text{-}C_5H_5)W(CO)_3\text{-}CH_2SiMe_2H$	C_5H_5, 4.19 (s, 5) $SiMe_2$, 0.36 (d, 6) CH_2, −0.26 (d, 2) SiH, 4.36 (m, 1)	
cis-$(\eta^5\text{-}C_5H_5)W(CO)_2\text{-}(H)(CH_2SiMe_2)$	C_5H_5, 4.31 (s, 5) $SiMe_2$, 0.27 (s, 3), −0.04 (s, 3) CH_2, 0.48 (dd, 1),[c] −0.11 (d, 1) (^1J = 10 Hz) WH, −11.48 (s, 1) ($J_{^{183}W^1H}$ = 55 Hz)[d]	
$(\eta^5\text{-}C_5Me_5)W(CO)_3\text{-}CH_2SiMe_2H$	C_5Me_5, 1.42 (s, 15) $SiMe_2$, 0.51 (d, 6) CH_2, −0.64 (d, 2) SiH, 4.89 (m, 1)	CO, 235.0, 224.3 C_5, 102.7 Me_5, 10.1 $SiMe_2$, 1.3 CH_2, −29.0
cis-$(\eta^5\text{-}C_5Me_5)W(CO)_2\text{-}(H)(CH_2SiMe_2)$	C_5Me_5, 1.65 (s, 15) $SiMe_2$, 0.29 (s, 3), 0.03 (s, 3) CH_2, 1.03 (dd, 1),[c] −0.87 (d, 1) (^1J = 10 Hz) WH, −10.78 (s, 1) ($J_{^{183}W^1H}$ = 54 Hz)[d]	CO, 242.7, 232.5 C_5, 101.5 Me_5, 11.3 CH_2SiMe_2, −1.1, −8.0, −27.6

[a] All data are for toluene-d_8 solutions at 200 K. [b] Chemical shifts vs. $Si(CH_3)_4$; peak multiplicity (s = singlet, d = doublet, dd = doublet of doublets, m = multiplet) and integration are given in parentheses for ^{1}H NMR. [c] This proton is weakly coupled, ~4 Hz, to the proton on W, as evidenced by homonuclear decoupled spectra. [d] The proton on W is weakly coupled to one of the CH_2 protons. In the coupled spectrum, the peak width at half height is broader by ~3 Hz compared to the homonuclear decoupled spectrum.

of the 2132-cm^{-1} absorption, two strong absorptions appear in the CO stretching region that are assigned to a dicarbonyl product. Surprisingly, irradiation of $(\eta^5\text{-}C_5R_5)W(CO)_3CH_2SiMe_2H$ at 77 K in 1-pentene gives nearly the same IR spectral changes as in a methylcyclohexane matrix, including the loss of the ~2100-cm^{-1} band attributed to the Si–H bond. This result is significant because the 1-pentene is a potential ligand and apparently cannot intercept the presumed 16e$^-$ intermediate formed upon loss of CO. In the case of $(\eta^5\text{-}C_5H_5)W(CO)_3$(alkyl) (alkyl = CH_3 or C_2H_5), photolysis in a 1-pentene matrix at 77 K yields $(\eta^5\text{-}C_5H_5)W(CO)_2$(alkyl)(1-pentene). UV–vis spectral changes accompanying the near-UV irradiation of $(\eta^5\text{-}C_5H_5)W(CO)_3CH_2SiMe_2H$ at 77 K in methylcyclohexane have been monitored and reveal the loss of the 310 and 360 (sh) nm absorption of the starting tricarbonyl and the growth of an absorption at 380 nm. Similar UV–vis changes occur when the $\eta^5\text{-}C_5Me_5$ complex is irradiated at 77 K. Under the same conditions, $(\eta^5\text{-}C_5R_5)W(CO)_3$(alkyl) yields $(\eta^5\text{-}C_5R_5)W(CO)_2$(alkyl) having an absorption at 555–600 nm, significantly lower in energy than the ~310-nm absorption maximum of the starting tricarbonyl.[2a] The lack of a large red shift of the lowest energy UV–vis absorption upon irradiation of $(\eta^5\text{-}C_5R_5)W(CO)_3CH_2SiMe_2H$ at 77 K indicates that the resulting dicarbonyl is not coordinatively unsaturated. The small UV spectral changes, the lack of spectral differences in a methylcyclohexane and a 1-pentene matrix, and the disappearance of the ~2100-cm^{-1} band associated with the Si–H bond suggest that the photoproduct at 77 K is *cis*-$(\eta^5\text{-}C_5R_5)W(CO)_2(H)(CH_2SiMe_2)$. A cis geometry for the dicarbonyl is based on the relative intensity of the two absorptions in the CO stretching region.[7] ^{1}H and ^{13}C NMR data, vide infra, substantiate the formulation of the product.

Warming *cis*-$(\eta^5\text{-}C_5R_5)W(CO)_2(H)(CH_2SiMe_2)$ in an alkane matrix from 77 to 200 K yields essentially no IR spectral changes in the CO stretching region, allowing the measurement of ^{1}H and ^{13}C NMR spectra in fluid hydrocarbon solutions[8] (Table I). These data, along with the IR data, support the formulation of the photoproduct as depicted in I. The low solubility of the $\eta^5\text{-}C_5H_5$ complex has prevented acquisition of the ^{13}C NMR for the product, but the ^{13}C NMR for the starting $\eta^5\text{-}C_5Me_5$ complex and the photoproduct can be obtained at 200 K (Table I). The long acquisition time (~6 h for the ^{1}H-decoupled spectrum) has precluded the determination of the nondecoupled spectrum. The product spectrum is consistent with the formation of one cis dicarbonyl product: the $\eta^5\text{-}C_5Me_5$ resonances are singlets, but there are two CO resonances. Additionally, in the 0 to −30 ppm region there are three resonances showing that the three carbons of the CH_2SiMe_2 are inequivalent. The ^{1}H NMR of both the $\eta^5\text{-}C_5H_5$ and the $\eta^5\text{-}C_5Me_5$ product have been obtained (Table I) and show a single hydride resonance coupled to ^{183}W. The Si–CH_3 protons appear as two singlets, consistent with the ^{13}C NMR that shows the methyl carbons to be inequivalent. Each of the CH_2 protons appears as a doublet. The low-field doublet is further split into a doublet of doublets by weak coupling, ~4 Hz, to the proton on the W. The hydride resonance appears as a singlet but is broadened slightly by the coupling to the low field CH_2 proton. The integrations are correct, ±10%, for the product as formulated in I.



I

No NMR evidence is found for the disilacyclobutane that would be expected for loss of the CH_2SiMe_2,[9] and no evidence is found for $[(\eta^5\text{-}C_5R_5)W(CO)_2(H)]_2$ that would be the expected[10] organometallic product from the resulting $(\eta^5\text{-}C_5R_5)W(CO)_2H$. In support of the hypothesis[1] that cis-$(\eta^5\text{-}C_5H_5)W(CO)_2(H)(CH_2SiMe_2)$ is an intermediate in a process analogous to eq 1, we find that warm-up of the photoproduct to 298 K under CO yields a significant amount of $(\eta^5\text{-}C_5H_5)W(CO)_3SiMe_3$ (~30–50%), along with $[(\eta^5\text{-}C_5H_5)W(CO)_3]_2$ and some $(\eta^5\text{-}C_5H_5)W(CO)_3H$. The formation of the $SiMe_3$ species confirms that the CH_2SiMe_2 unit remains in the coordination sphere. The *cis*-$(\eta^5\text{-}C_5Me_5)W(CO)_2(H)(CH_2SiMe_2)$ is qualitatively more inert than the C_5H_5 analogue, but it does react and a pure solid has not yet been isolated.

The metallasilacyclopropane structure depicted in I is one extreme way to represent the W–CH_2SiMe_2 bonding. This formulation is most consistent with the ^{1}H and ^{13}C chemical shifts. The fact that the more electron-rich C_5Me_5 species is more inert than the C_5H_5 analogue is consistent with the metallasilacyclopropane bonding. Rotation about the W–C_2H_4 bond in $(\eta^5\text{-}C_5H_5)W(CO)_2(CH_3)(C_2H_4)$ has a large barrier, and the metallacyclopropane W–C_2H_4 extreme has been invoked.[11] The metallasilacyclopropane structure avoids a C–Si double bond in the CH_2SiMe_2 unit. Additional studies of the structure, bonding,

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(7) On the basis of arguments similar to those in: Cotton, F. A.; Wilkinson, G. "Advanced Inorganic Chemistry", 4th ed.; Wiley: New York, 1980; pp 1073–1075.

(8) Photolysis of $(\eta^5\text{-}C_5R_5)W(CO)_3CH_2SiMe_2H$ at 200 K in toluene, toluene-d_8, methylcyclohexane, or methylcyclohexane-d_{14} yields the same product as that generated by photolysis at 77 K followed by warm-up to 200 K.

(9) (a) Gusel'nikov, L. E.; Flowers, M. C. *Chem. Commun.* **1967**, 864; (b) *J. Chem. Soc. B* **1968**, 419 and 1396. We do, however, see low yields (<30%) of products having ^{1}H NMR resonances in the 0–0.5-ppm region. These products have not been identified.

(10) Alt, H. G.; Mahmoud, K. A.; Rest, A. J. *Angew. Chem.* **1983**, *95*, 569; *Angew. Chem., Int. Ed. Engl.* **1983**, *22*, 544.

(11) (a) Alt, H. G.; Schwarzel, J. A.; Kreiter, C. G. *J. Organomet. Chem.* **1978**, *153*, C7. (b) Kreiter, C. G.; Nist, K.; Alt, H. G. *Chem. Ber.* **1981**, *114*, 1845.

and chemistry of *cis*-(η^5-C_5R_5)W(CO)$_2$(H)(CH_2SiMe_2) and related species are in progress.

Acknowledgment. We thank the National Science Foundation for support of this research. We appreciate the valuable insights of Professor Alan Davision in the interpretation of the ^{1}H NMR of I.

Mechanistic Investigation of Olefin Insertion into the Rhodium-Hydrogen Bond

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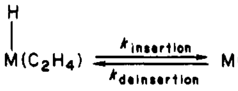
Olefin insertion into a metal-hydrogen bond is a key component of many catalytic cycles (e.g., hydroformylation, hydrogenation, and olefin isomerization)[1] and is of fundamental interest with respect to product distribution. In rhodium-catalyzed hydroformylation, for example, it is generally considered that the greater steric bulk of phosphine ligands compared to CO influences the ratio of linear:branched product by favoring insertion to linear alkylrhodium species. Despite the importance of olefin hydrides in catalytic cycles, such species have rarely been amenable to mechanistic study. In fact, the equilibrium between a transition-metal olefin hydride and the corresponding alkyl species (eq 1) has been observed only rarely[2-5] and experimental determination of the reaction coordinate for insertion is virtually unknown (cf. ref 5). This communication reports the detailed characterization of a hydridorhodium(I) olefin complex that permits evaluation of rate constants for insertion.



$$\overset{\displaystyle H}{\overset{|}{M}}(C_2H_4) \underset{k_{\text{deinsertion}}}{\overset{k_{\text{insertion}}}{\rightleftharpoons}} MCH_2CH_3 \qquad (1)$$

Hydrido(ethylene)bis(triisopropylphosphine)rhodium (**1**) forms readily in solution by displacement of N_2 when the known complex HRh(P-*i*-Pr$_3$)$_2$(N_2)[6] is treated with either a stoichiometric amount or a large excess of ethylene. The ethylene hydride has been characterized spectroscopically[7] but has not been isolated.

The significant ^{1}H NMR spectral features of **1** are invariant from −90 to 0 °C. A trans olefin hydride is suggested since the phosphines appear to be equivalent (cf. hydride coupling $^2J_{PH}$). The coordinated ethylene and hydride resonances broaden above 0 °C, but above 30 °C irreversible sample decomposition occurs. The observed exchange broadening arises from insertion to alkyl followed by M-C-C single-bond rotation and rapid β-hydride elimination. Further evidence for this insertion process is provided by deuterium-labeling experiments.[8] Treatment of HRh(P-*i*-Pr$_3$)$_2$(N_2) with C_2D_4 brings about loss of the ^{1}H NMR hydride resonance, while subsequent introduction of C_2H_4 into the same sample results in recovery of hydride intensity.

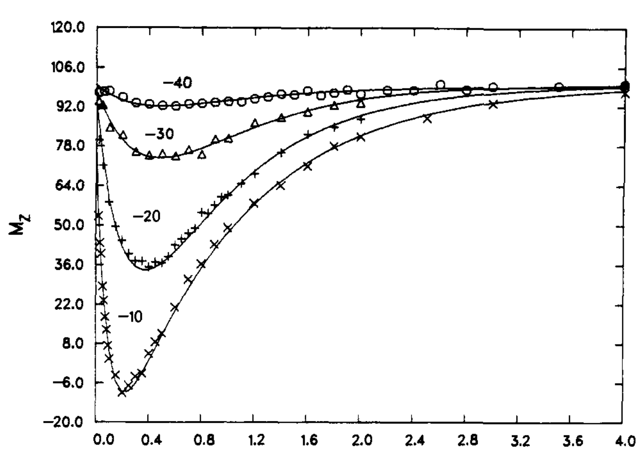


Figure 1. Response of hydride (M_z) to selective inversion of coordinated ethylene at indicated temperatures (°C). Data were acquired out to 8 s in each case. Data obtained at 0 °C are omitted for clarity.

Table I. Exchange Rate and Relaxation Parameters for HRh(C_2H_4)(P-*i*-Pr$_3$)$_2$

T, °C	k, s^{-1} [a]	T_1, s		k_2, s^{-1} [b]
		C_2H_4	H	
−40	0.29 (0.04)	0.44	0.63	1.1
−30	0.78 (0.06)	0.60	0.70	2.9
−20	2.62 (0.09)	0.67	1.01	9.8
−10	8.01 (0.24)	0.92	1.04	30.0
0	18.6 (0.8)	1.54	1.02	70.0

[a] Standard deviations in parentheses. T_1 values are considered to be accurate to no better than ±10%.[13] [b] See text (eq 4).

Attempts to quantify the insertion rate by trapping the alkyl complex once it is formed were not successful, although direct evidence for an ethylrhodium species was obtained.[9] Furthermore, the narrow temperature range in which line-shape changes were observable precluded complete band-shape analysis as the principal tool for quantification of the insertion rate.

Magnetization-transfer techniques[10,11] for the study of slow NMR exchange processes are particularly suitable in the case of olefin hydrides.[12] An advantage is derived by selective inversion of the coordinated ethylene resonance (rather than the hydride) since the larger inverted population magnifies the perturbation experienced by the hydride. Application of this technique to **1** extends the temperature range in which exchange is observed to −40 °C. A compact description of the exchange is provided by eq 2 in which **M** represents a vector of magnetization intensities,

$$\dot{\mathbf{M}}(t) = \mathbf{K}\cdot\Delta\mathbf{M}(t) \qquad (2)$$

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(1) Parshall, G. W. "Homogeneous Catalysis"; Wiley: New York, 1980; pp 29-41.

(2) (a) Werner, H.; Feser, R. *Angew. Chem., Int. Ed. Engl.* **1979**, *18*, 157-158. (b) Werner, H.; Feser, R. *J. Organomet. Chem.* **1982**, *232*, 351-370.

(3) (a) Byrne, J. W.; Blaser, H. U.; Osborn, J. A. *J. Am. Chem. Soc.* **1975**, *97*, 3871-3873. (b) Chaudret, B. N.; Cole-Hamilton, D. J.; Wilkinson, G. *Acta Chem. Scand., Ser. A* **1978**, *A32*, 763-769. (c) Seiwell, L. P. *Inorg. Chem.* **1976**, *15*, 2560-2563.

(4) Klein, H. F.; Hammer, R.; Gross, J.; Schubert, U. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 809-810.

(5) McGrady, N. D.; McDade, C.; Bercaw, J. E. "Organometallic Compounds: Synthesis, Structure and Theory"; Texas A&M University Press: College Station, TX, 1983; pp 46-85.

(6) Yoshida, T.; Okano, T.; Thorn, D. L.; Tulip, T. H.; Otsuka, S.; Ibers, J. A. *J. Organomet. Chem.* **1979**, *181*, 183-201.

(7) ^{1}H NMR (0.011 M in C_7D_8 at −50 °C) δ −9.21 (dt, 1 H, $^1J_{RhH}$ = 5.3, $^2J_{PH}$ = 21.4 Hz), 1.11 (q, 36 H, $^3J_{HH}$ = 6.6, $^3J_{PH} + {}^5J_{PH}$ = 13.3 Hz), 1.93 (m, 6 H), 2.66 (s, 4 H); ^{31}P NMR (0.022 M in C_7D_8 at 23 °C) δ 67.99 ($^1J_{RhP}$ = 152.3 Hz); ^{13}C NMR (0.022 M in C_7D_8 at 23 °C) δ 20.92 (s, CH_3), 28.13 (dt, CH, $^2J_{RhC}$ = 1.5, $^1J_{PC} + {}^3J_{PC}$ = 19.6 Hz), 45.36 (d, C_2H_4, $^1J_{RhC}$ = 6.4 Hz). Gated decoupling ^{13}C experiments reveal $^1J_{HC}$ = 157.5 Hz for coordinated ethylene. IR in toluene solution shows ν(RhH) = 2005, 2042 cm^{-1}.

(8) Olefin exchange between free and coordinated ethylene occurs but is too slow to be observed on the NMR time scale.

(9) Addition of CD_3CN to a toluene-d_8 solution of **1** results in a complex mixture in which **1** still predominates. The other major species appear to be free phosphine and CH_3CH_2Rh(P-*i*-Pr$_3$)(C_2H_4)$_x$(CD_3CN)$_y$ (x, y = 1 or 2); the features of interest are ^{1}H NMR (ca. 0.01 M in C_7D_8/CD_3CN (3:2)) at −40 °C δ −0.40 (d quintet, CH_2, $^2J_{RhH} + {}^3J_{HH}$ = 7.7, $^3J_{PH}$ = 2.0 Hz), 0.20 (t, CH_3, $^3J_{PH}$ = 7.6 Hz).

(10) Forsen, S.; Hoffman, R. A. *J. Chem. Phys.* **1963**, *39*, 2892-2901; **1964**, *40*, 1189-1196.

(11) Alger, J. R.; Prestegard, J. H. *J. Magn. Reson.* **1977**, *27*, 137-141.

(12) All spectra were acquired on a Nicolet NT-360WB spectrometer. Suitable attenuation of the low-power transmitter resulted in selective inversion pulses that were typically 10-15 ms.

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