to use. $\mathrm{Cp^*_2HfH_2}$ and $\mathrm{Cp^*_2HfD_2}$ as well as *trans*-1-deuterio-3,3-dimethyl-1-butene and *cis*-1,2-dideuterio-3,3-dimethyl-1-butene were prepared following literature procedures.[11,12] *tert*-Butyl hydroperoxide (Aldrich, 90%) was dried by azeotropic distillation with benzene and stored as an approximate 4 M solution over 4-Å molecular sieves.[13] All other reagents were either degassed and vacuum distilled from 4-Å molecular sieves or used as received. Alkyl migration reactions of **2a** and **2b** were performed in sealed NMR tubes; spectra were recorded on a JOEL GX-400 spectrometer.

$\mathbf{Cp^*_2Hf(H)(CH_2CH_2CMe_3)}$ (1). A 2.00-g (4.43-mmol) sample of $\mathrm{Cp^*_2HfH_2}$ was dissolved in 25 mL of toluene. 3,3-Dimethyl-1-butene (5 mL, 38.8 mmol) was added by vacuum transfer; the reaction was stirred at ambient temperature for 10 h. The volatiles were removed in vacuo, and the residue was taken up in petroleum ether and filtered. The filtrate was concentrated and cooled to −78 °C, precipitating a white crystalline solid. Yield: 1.29 g (54.4%). ^{1}H NMR (benzene-d_6): δ 12.85 (s, $\mathrm{Hf}H$), 1.94 (s, Cp^*), 1.02 (s, $\mathrm{HfCH_2CH_2C}Me_3$), 0.86 (AA′XX′, $\mathrm{HfCH_2C}H_2\mathrm{CMe_3}$), −0.16 (AA′XX′, $\mathrm{HfC}H_2\mathrm{CH_2CMe_3}$). Anal. Calcd for $\mathrm{C_{26}H_{44}Hf}$ (found): C, 58.36 (58.41); H, 8.29 (7.62).

***threo*-$\mathbf{Cp^*_2Hf(H)(CHDCHDCMe_3)}$ (1a).** In a procedure analogous to the preparation of 1, a 2.51-g (5.57-mmol) sample of $\mathrm{Cp^*_2HfH_2}$ and 0.90 mL (6.98 mmol) of *cis*-1,2-dideuterio-3,3-dimethyl-1-butene gave, after workup, 1.29 g (43%) of a white sample of **1a**. ^{1}H NMR (benzene-d_6): δ 12.85 (s, $\mathrm{Hf}H$), 1.95 (s, Cp^*), 1.02 (s, $\mathrm{HfCHDCHDC}Me_3$), 0.86 (br, $^3J_{\mathrm{HH}} < 2.5$ Hz, $\mathrm{HfCHDC}H\mathrm{DCMe_3}$), −0.22 (br, $^3J_{\mathrm{HH}} < 2.5$ Hz, $\mathrm{HfC}H\mathrm{DCHDCMe_3}$).

***erythro*-$\mathbf{Cp^*_2Hf(D)(CHDCHDCMe_3)}$ (1b).** In a procedure analogous to the preparation of 1, a 1.23-g (2.72-mmol) sample of $\mathrm{Cp^*_2HfD_2}$ and 0.38 mL (2.95 mmol) of *trans*-1-deuterio-3,3-dimethyl-1-butene gave, after workup, 0.82 g (56%) of **1b** as a white solid. ^{1}H NMR (benzene-d_6): δ 1.96 (s, Cp^*), 1.05 (s, $\mathrm{HfCHDCHDC}Me_3$), 0.83 (d, $\mathrm{HfCHDC}H\mathrm{DCMe_3}$, $^3J_{\mathrm{HH}} = 14.6$ Hz), −0.22 (d, $\mathrm{HfC}H\mathrm{DCHDCMe_3}$, $^3J_{\mathrm{HH}} = 14.6$ Hz).

$\mathbf{Cp^*_2Hf(CH_2CH_2CMe_3)(OOCMe_3)}$ (2). A 0.91-g (1.7-mmol) sample of 1 was dissolved in 40 mL of toluene and the solution cooled to 0 °C. *tert*-Butyl hydroperoxide (0.62 mL, 1.86 mmol) was added via syringe against an argon counterflow. The reaction was stirred for 30 min. The volatiles were removed in vacuo and the residuals taken up in acetone, filtered, concentrated, and cooled to −78 °C. The white crystalline product was collected by filtration and dried in vacuo. Yield: 0.53 g (49.8%). ^{1}H NMR (benzene-d_6): δ 1.90 (s, Cp^*), 1.50 (AA′XX′, $\mathrm{HfCH_2C}H_2\mathrm{CMe_3}$), 1.21 (s, $\mathrm{HfOOC}Me_3$), 1.16 (s, $\mathrm{HfCH_2CH_2C}Me_3$), 0.25 (AA′XX′, $\mathrm{HfC}H_2\mathrm{CH_2CMe_3}$). Anal. Calcd for $\mathrm{C_{30}H_{52}O_2Hf}$ (found): C, 57.82 (57.60); H, 8.41 (8.06).

***threo*-$\mathbf{Cp^*_2Hf(CHDCHDCMe_3)(OOCMe_3)}$ (2a).** In a procedure analogous to the preparation of **2**, a 0.63-g (1.18-mmol) sample of **1a** and 0.6 mL of *tert*-butyl hydroperoxide (2.4 mmol) gave, after workup, 0.26 g (34%) of **2a** as a white solid. ^{1}H NMR (benzene-d_6): δ 1.90 (s, Cp^*), 1.47 (d, $\mathrm{HfCHDC}H\mathrm{DCMe_3}$, $^3J_{\mathrm{HH}} = 2.7$ Hz), 1.21 (s, $\mathrm{HfOOC}Me_3$), 1.15 (s, $\mathrm{HfCHDCHDC}Me_3$), 0.17 (d, $\mathrm{HfC}H\mathrm{DCHDCMe_3}$, $^3J_{\mathrm{HH}} = 2.7$ Hz).

***erythro*-$\mathbf{Cp^*_2Hf(CHDCHDCMe_3)(OOCMe)}$ (2b).** In a procedure analogous to the preparation of **2**, a 0.65-g (1.2-mmol) sample of **1b** and 0.61 mL (2.4 mmol) of *tert*-butyl hydroperoxide gave, after normal workup, 0.34 g (45%) of **2b** as a white microcrystalline solid. ^{1}H NMR (benzene-d_6): δ 1.90 (s, Cp^*), 1.46 (d, $\mathrm{HfCHDC}H\mathrm{DCMe_3}$, $^3J_{\mathrm{HH}} = 14.7$ Hz), 1.21 (s, $\mathrm{HfOOC}Me_3$), 1.15 (s, $\mathrm{HfCHDCHDC}Me_3$), 0.18 (d, $\mathrm{HfC}H\mathrm{DCHDCMe_3}$, $^3J_{\mathrm{HH}} = 14.7$ Hz).

$\mathbf{Cp^*_2Hf(OCH_2CH_2CMe_3)(OCMe_3)}$ (3). A 0.30-g (0.48-mmol) sample of **2** was dissolved in 25 mL of toluene; the solution was allowed to stand at ambient temperature for 4 days. Removal of solvent in vacuo and crystallization from acetone gave a white solid. Yield: 0.21 g (70%). ^{1}H NMR (benzene-d_6): δ 4.34 (AA′XX′, $\mathrm{HfOC}H_2\mathrm{CH_2CMe_3}$), 2.00 (s, Cp^*), 1.76 (AA′XX′, $\mathrm{HfOCH_2C}H_2\mathrm{CMe_3}$), 1.42 (s, $\mathrm{HfOC}Me_3$), 1.00 (s, $\mathrm{HfOCH_2CH_2C}Me_3$). Anal. Calcd for $\mathrm{C_{30}H_{52}O_2Hf}$ (found): C, 57.82 (57.95); H, 8.41 (8.14).

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Registry No. **1**, 137394-32-2; **1a**, 137394-33-3; **1b**, 137394-34-4; **2**, 137394-35-5; **2a**, 137394-36-6; **2b**, 137394-37-7; **3**, 137394-38-8; $\mathrm{Cp^*_2HfH_2}$, 81956-87-8; 3,3-dimethyl-1-butene, 558-37-2; *cis*-1,2-dideuterio-3,3-dimethyl-1-butene, 84784-40-7; *trans*-1-deuterio-3,3-dimethyl-1-butene, 57002-05-8.

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Correlation of ^{103}Rh NMR Shielding and Rate Constants of CO Displacement Reactions in $(\eta^5\text{-}C_5H_4X)Rh(CO)_2$ Complexes[†]

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Summary: ^{103}Rh chemical shifts of $(\eta^5\text{-}C_5H_4X)Rh(CO)_2$ complexes (X = H, CH_3, $N(CH_3)_2$, CF_3, Cl, NO_2) show a linear correlation ($R^2 = 0.973$) with rate constants log k_{obsd} of the CO/PPh_3 displacement reaction reported by Basolo and Cheong. Increasing reactivity is reflected in lower shielding of the ^{103}Rh nucleus, and substituents with lone-pair electrons ($N(CH_3)_2$, Cl) do not disturb the correlation.

Seven years ago we reported together with Bönnemann et al. a linear relationship between the ^{59}Co chemical shift of several $(\eta^5\text{-}C_5H_4R)Co(COD)$ complexes and their catalytic activity.[2] In 1989 it was shown by DeShong et al. and our group that the ^{55}Mn resonance of a series of manganacycles correlated linearly with the logarithm of the observed rate constants for a demetalation reaction.[3] More recently, ^{59}Co shielding in alkylcobaloximes and related complexes has proven to be very sensitive to steric

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Scheme I

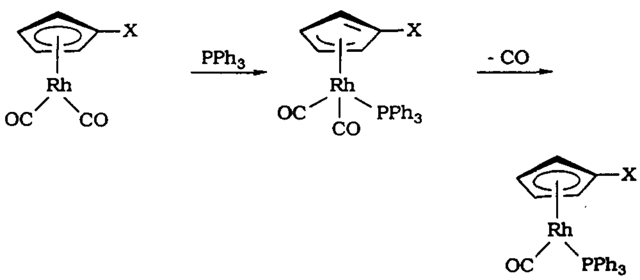


Table I. ^{103}Rh Chemical Shifts[a] of (η^5-C_5H_4X)Rh$(CO)_2$ Complexes and Rate Constants for the Carbonyl Displacement Reaction with PPh_3[b]

X	δ(^{103}Rh), ppm	k_{obsd}, M^{-1} s^{-1}
NO_2	-1117[c]	1.26
Cl	-1166	5.18 × 10^{-2}
CF_3	-1220.5[d]	1.58 × 10^{-2}
$N(CH_3)_2$	-1248	1.05 × 10^{-3}
CH_3	-1310.5[c]	9.98 × 10^{-5}
H	-1321.5[e]	1.30 × 10^{-4}

[a] In C_6D_6 at 298 ± 1 K. [b] In decalin at 298 K.[5] [c] See also ref 1. [d] J(Rh,F) = 1.1 Hz. [e] Reference 6.

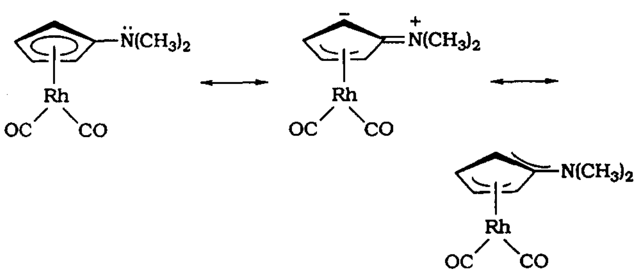
effects in cobalt–carbon coordination.[4] These results demonstrate that the large chemical shifts of transition-metal nuclei not only are sensitive to structural changes but also seem to be a useful parameter to probe electronic substituent effects, steric effects, and, most important, reactivity. An extension to ^{103}Rh NMR spectroscopy is attractive, inasmuch as many organorhodium compounds show catalytic activity, and such investigations are in progress. Here we wish to report a linear correlation between the ^{103}Rh chemical shifts of (η^5-C_5H_4X)Rh$(CO)_2$ complexes (X = H, CH_3, $N(CH_3)_2$, CF_3, Cl, NO_2) and the specific rates log k_{obsd} for the carbonyl displacement reaction with triphenylphosphine, which were measured by Basolo and Cheong.[5]

For this reaction an associative η^5–η^3–η^5 "ring slippage" mechanism has been postulated, which is supported by relatively small $\Delta H^{\ddagger}$ (9.5–15.6 kcal/mol) and large negative $\Delta S^{\ddagger}$ (−18 to −40 eu) values (Scheme I).

The strong influence of the substituent X is reflected in the large range of the observed rate constants (~4 orders of magnitude) and the chemical shift range of the metal (~200 ppm), where the nucleophilic attack takes place. In a first approximation, the electron-withdrawing substituents NO_2, Cl, and CF_3 enhance the rates of substitution and deshield the Rh nucleus, whereas the electron-donating group $N(CH_3)_2$ decreases the rate and shields the nucleus. However, we expected to observe the ^{103}Rh chemical shift of the $N(CH_3)_2$-substituted compound at higher field (i.e. more negative δ(^{103}Rh) value), as $N(CH_3)_2$ is a powerful π-donor.

A plot of Hammett σ_p vs log k_{obsd} values for the CO displacement reaction had shown that the Cl- and N-$(CH_3)_2$-substituted complexes reacted much faster than expected.[5] Recent photoelectron spectroscopy studies indicate a stabilization of the η^3 intermediate in the photoexcited state through π-delocalization on the "slipped ring".[7] Such a p_π overlap in the transition state of the rate-determining first step (Scheme I) would increase the rates of substitution, which explains the deviations from linearity in the Hammett plot. We postulate that there is a similar resonance effect in the ground state or in a low-lying electronically excited state of the $N(CH_3)_2$-substituted complex (Scheme II). This would reduce the coordination of the rhodium atom and thus account for the relative deshielding of the ^{103}Rh nucleus.

Scheme II



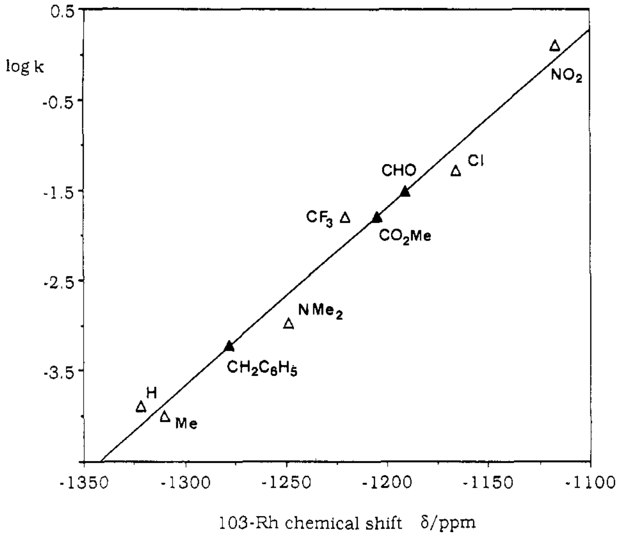


Figure 1. Plot of δ(^{103}Rh) vs log k for CO/PPh_3 displacement: Δ correlation points; (▲) ^{103}Rh shifts,[1] but rates unknown. R^2 = 0.973.

Similar deshielding effects have been reported for [16e]-allylmetal complexes of the type (η^3-allyl)(η^4-butadiene)(η^5-cyclopentadienyl)zirconium(0).[8]

For the six rhodium complexes, a linear relationship (R^2 = 0.973) is found between the δ(^{103}Rh) and the log k_{obsd} values for associative substitution (Figure 1). Included are the ^{103}Rh chemical shifts of the benzyl, carbomethoxy, and formyl derivatives,[1] for which rate constants are not available but which may have predictive value.

In conclusion, the ^{103}Rh chemical shift provides a highly sensitive measure of the electronic environment of the metal atom; it reflects both the inductive and resonance effects affecting the rate constants of the CO/PPh_3 displacement reaction and may prove to be a useful probe into organometallic reactivity.

Experimental Section

The syntheses of the compounds have been reported.[5,9,10] All rhodium spectra were measured on a Bruker AM-400 spectrometer at 298 ± 1 K in 5- or 10-mm sample tubes, using 0.7–1 M C_6D_6 solutions. The ^{103}Rh resonances were directly observed at 12.6

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MHz under inverse-gated ^{1}H decoupling to avoid negative NOE. Typically 12000 points were sampled for a spectral width of 8000 Hz. A pulse width of 20 μs was used, which corresponds to a flip angle of 45°. The time intervals between the pulses typically were 4 s. The chosen relaxation delays were 5 times longer than the acquisition periods. The ^{103}Rh chemical shifts are reported in ppm versus the absolute frequency standard of $\Xi_{\mathrm{ref}} = 3.16$ MHz[11] and are expected to be reproducible within ±1–2 ppm.

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Registry No. (η^5-C$_5$H$_4$NO$_2$)Rh(CO)$_2$, 75862-04-3; (η^5-C$_5$H$_4$Cl$_2$)Rh(CO)$_2$, 94890-74-1; (η^5-C$_5$H$_4$CF$_3$)Rh(CO)$_2$, 115406-90-1; (η^5-C$_5$H$_4$N(CH$_3$)$_2$)Rh(CO)$_2$, 115406-91-2; (η^5-C$_5$H$_4$CH$_3$)Rh(CO)$_2$, 63544-85-4; (η^5-C$_5$H$_4$H)Rh(CO)$_2$, 12192-97-1; PPh$_3$, 603-35-0.

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Phosphine Complexes of Zirconium and Hafnium. Synthesis, Structure, and Fluxional Behavior of the Trimethyl Derivatives MMe$_3$[N(SiMe$_2$CH$_2$PR$_2$)$_2$] (R = Me, Pr^i, Bu^t). Evidence for a Bicapped-Tetrahedral Geometry

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Summary: The addition of 3 equiv of methyl Grignard (MeMgCl) to the amidodiphosphine derivatives MCl$_3$[N(SiMe$_2$CH$_2$PR$_2$)$_2$] (M = Zr, Hf; R = Me, Pr^i, Bu^t) generates the corresponding trimethyl complexes MMe$_3$[N(SiMe$_2$CH$_2$PR$_2$)$_2$]. The solution structures and fluxional behavior of these molecules along with the X-ray structure of HfMe$_3$[N(SiMe$_2$CH$_2$PMe$_2$)$_2$] are presented.

Because of the recent renewed interest in soluble models for Ziegler–Natta polymerization catalysts based on group 4 hydrocarbyl complexes,[1-9] the synthesis of such derivatives continues to be a worthy goal. The most well-studied group 4 hydrocarbyl systems utilize cyclopentadienyl or substituted cyclopentadienyl ligands as ancillary ligands.[10] Examples are Cp′MR$_3$ and Cp′$_2$MR$_2$ (where Cp′ = η^5-C$_5$H$_5$, η^5-C$_5$Me$_5$, η^5-C$_5$H$_4$Me; R = Me, Ph).[11-14] Of course many other examples are known and include simple homoleptic hydrocarbyl complexes MR$_4$,[15] phosphine-stabilized derivatives such as MMe$_4$(dmpe)$_2$[16] and M(CH$_2$Ph)$_4$(dmpe) (where M = Zr or Hf),[17] and the bis- and tris(amido) derivatives MR$_2$[N(SiMe$_3$)$_2$]$_2$ and MR[N(SiMe$_3$)$_2$]$_3$.[18-20]

Our approach to investigating the organometallic chemistry of the early metals and the lanthanides[21-28] has been through the use of the mixed-donor tridentate ligand $^-$N(SiMe$_2$CH$_2$Pr$_2$)$_2$. In this paper we present the preparation and solution behavior of a series of trimethyl derivatives of zirconium and hafnium of the formula MMe$_3$[N(SiMe$_2$CH$_2$PR$_2$)$_2$] (M = Zr, Hf; R = Me, Pr^i, Bu^t); in addition, the X-ray crystal structure of the hafnium derivative HfMe$_3$[N(SiMe$_2$CH$_2$PMe$_2$)$_2$] is described. Not surprisingly, the stability of these hydrocarbyl derivatives is directly related to the steric bulk of the phosphorus substituents on the tridentate ligand.

Experimental Section

General Procedures. All manipulations were performed under prepurified nitrogen in a Vacuum Atmospheres HE-553-2 glovebox equipped with a MO-40-2H purification system or in

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