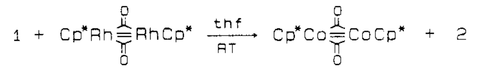
rings are accounted for in these signals (Cp(H):Cp*(H) = 1:6 for 1 and 2 and 2:3 for 3 and 4). This behavior is consistent with the carbonyl ligands engaging in a fast fluxional shuttling around the edges of the triangular core.[9]

To more fully characterize the paramagnetism of these complexes, we investigated the temperature dependence of their proton NMR spectra. As has been observed previously,[1a] complex 1 exhibits a strict linear relationship between the shift and inverse temperature. This type of behavior is consistent with a $S = 1$ triplet formulation for the complex over the temperature range studied (±90 °C). Plots of ^1H shift versus $1/T$ for complexes 2 and 4 are shown in Figure 1. For the rhodium–dicobalt cluster 4, as the temperature is lowered, the paramagnetically derived shift goes asymptotically to zero (diamagnetic position) consistent with a ground-state singlet formulation with low-lying, thermally available triplet states. Clusters 2 and 3 exhibit similar though somewhat unusual shift versus $1/T$ behaviors as shown for 2. A maximum paramagnetic shift is observed in both cases with the shift falling off at both higher and lower temperatures. Although a detailed description of the electronic structures for 1–4 cannot be made with the data in hand, their complex and seemingly incongruent properties may all be explained by a ground-state singlet formulation with thermally accessible, low-lying triplet states.[10] Detailed magnetic susceptibility studies are in progress to determine the precise origin of these interesting electronic effects.

Preliminary investigations into the reactions of these cluster complexes with various ligands and organometallic complexes reveal the following patterns: (1) One CpCo fragment is easily lost upon exposure to dioxygen with the concomitant formation of the parent dinuclear complex in the case of 1 and 2. (2) The trinuclear framework remains intact when complexes 1–4 are exposed to carbon monoxide. The resulting tricarbonyl adducts 5–8 conform to one of the known structure types for these complexes.[11]

Finally, complexes 1–4 appear to function as CpCo transfer agents when exposed to appropriate organometallic acceptors. For example, complex 1 cleanly transfers a CpCo fragment to $[Cp^*Rh(\mu\text{-}CO)]_2$ in THF at room temperature over several hours as shown in eq 5.



$$1 + Cp^*Rh\overset{\overset{O}{\|}}{\underset{\underset{O}{\|}}{\equiv}}RhCp^* \xrightarrow[RT]{thf} Cp^*Co\overset{\overset{O}{\|}}{\underset{\underset{O}{\|}}{\equiv}}CoCp^* + 2 \qquad (5)$$

These reactions appear to be under equilibrium control. The equilibrium constant for the reaction of 1 with $[Cp^*Rh(\mu\text{-}CO)]_2$ is 4.2 at room temperature in THF-d_8. Equilibrium and rate studies are underway to characterize this CpCo transfer reaction.[12]

From the results reported here both the underlying electronic structures and the reactivities exhibited by this class of trinuclear complexes appear to be a sensitive function of several factors including the metals involved, the number of Cp and Cp* ligands associated with the cluster, the type of incoming ligand, and the reaction conditions. With the synthetic approach described herein we are currently exploring other combinations of metals and ligands within the trinuclear framework as well as the diverse types of reactivity these clusters exhibit in an attempt to more fully understand their chemistry.

Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, and the Research Corp. for partial support of this research. We acknowledge generous loans of rhodium metal starting materials from Johnson Matthey, Inc.

Registry No. 1, 112532-12-4; 2, 112532-13-5; 3, 112532-14-6; 4, 112532-15-7; $CpCo(C_2H_4)_2$, 69393-67-5; $Cp^*Co(\mu\text{-}CO)_2CoCp^*$, 69657-52-9; $Cp^*Rh(\mu\text{-}CO)_2RhCp^*$, 69728-34-3; $Cp^*Co(CO)_2$, 12129-77-0; $Cp^*Rh(CO)_2$, 32627-01-3.

Supplementary Material Available: Tables of observed ^1H NMR shifts versus temperature for complexes 1 through 4 and elemental analysis data for complexes 1 through 8 (2 pages). Ordering information is given on any current masthead page.

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[9] For a more detailed discussion of this type of behavior, cf. ref 1a.

[10] Behavior similar to **4** has been reported by Dahl and co-workers.[2c] It is also possible that these complexes exhibit both high- and low-spin ground-state electron configurations. See ref 8 for a discussion of the possible ground states for these clusters.

[11] Three distinct structural types for $Cp_3M_3(CO)_3$ (M = Co, Rh) complexes have been described. Following the designations of R. J. Lawson and J. R. Shapley (*Inorg. Chem.* **1978**, *17*, 772–774), these are C_{3v} $(MCp)_3(\mu\text{-}CO)_3$, C_s $(MCp)_3(\mu_3\text{-}CO)(\mu\text{-}CO)_2$, and C_s $(MCp)_3(\mu\text{-}CO)_2(CO)$. Complexes 1 through **4** form the C_s $(MCp)_3(\mu_3\text{-}CO)(\mu\text{-}CO)_2$ with CO: **5** (from 1), IR (CH_2Cl_2) 1815 (s), 1765 (m), 1665 (s) cm^{-1}, ^1H NMR (C_6D_6, 90 MHz) δ 4.703 (s, 5 H), 1.466 (s, 30 H); **6** (from **2**) IR (CH_2Cl_2) 1815 (s), 1770 (m), 1730 (w), 1655 (s) cm^{-1}, ^1H NMR (C_6D_6) δ 4.689 (s, 5 H), 1.627 (s, 30 H); **7** (from **3**) IR (KBr) 1830 (s), 1780 (s), 1685 (sh), 1675 (s) cm^{-1}, ^1H NMR (C_6D_6) δ 4.62 (s, 10 H), 1.390 (s, 15 H); **8** (from **4**) IR (KBr) 1800 (s), 1760 (s), 1650 (s) cm^{-1}, ^1H NMR (C_6D_6) δ 4.62 (s, 10 H), 1.56 (s, 15 H.

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Transition-Metal Silyl Complexes. 22.[1] Anionic Silyl Complexes, $L_nM\text{-}SiR_3^-$, as Equivalents to L_nM^{2-} in Synthesis. A Novel Route to Carbene Complexes

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Summary: Cyclopropenylidene complexes are obtained by reaction of anionic silyl complexes $[(\eta^5\text{-}CH_3C_5H_4)(CO)_2MnSiMePh_2]^-$ or $[(Ph_3P)(CO)_3FeSiMePh_2]^-$ with 3,3-dichloro-1,2-diphenylcyclopropene. The structure of $(\eta^5\text{-}CH_3C_5H_4)(CO)_2Mn{=}\overline{C{-}CPh{=}C}Ph$ (1) has been determined by X-ray diffraction.

Whereas the chemistry of simple metal carbonyl anions is well-established, the reactivity of anionic transition-metal silyl complexes has only recently attracted some attention. They readily react with organic and inorganic halides and therefore are useful precursors to silyl-substituted complexes with metal–carbon, metal–metal, or metal–metalloid bonds.[2,3] Beyond such substitution reactions, the reactivity of the silyl ligand renders additional preparative applications possible. When $[MeCp(CO)_2MnSiR_3]^-$ (MeCp = $\eta^5\text{-}CH_3C_5H_4$) is reacted with Ph_3PAuCl or $(picoline)_2PtCl_2$ in the presence of moist picoline, binuclear complexes with hydrogen-bridged metal–metal bonds are formed,[4] the anionic silyl complex

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[4] Kunz, E.; Müller, J.; Schubert, U. *J. Organomet. Chem.* **1987**, *320*, C11.

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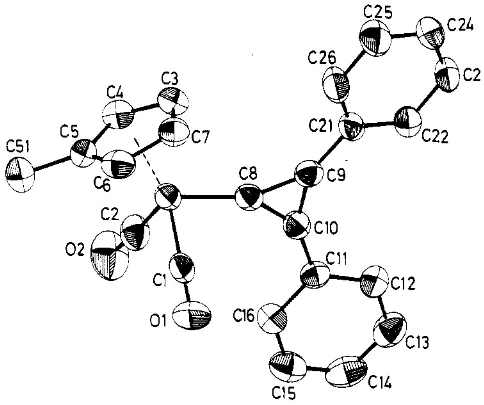
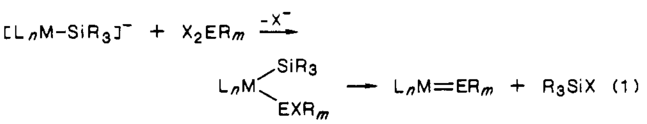


Figure 1. ORTEP diagram of 1. Hydrogen atoms have been omitted for clarity. Important bond lengths (pm) and angles (deg) are as follows: Mn–C8 = 189.6 (4), Mn–C1 = 176.2 (5), Mn–C2 = 177.4 (5), C8–C9 = 142.4 (6), C8–C10 = 142.4 (6), C9–C10 = 134.9 (6), C1–O1 = 116.2 (6), C2–O2 = 115.4 (6); C1–Mn–C2 = 90.0 (2), C1–Mn–C8 = 88.6 (2), C2–Mn–C8 = 96.5 (2), Mn–C1–O1 = 178.7 (4), Mn–C2–O2 = 175.6 (4), Mn–C8–C9 = 152.6 (3), Mn–C8–C10 = 150.8 (3), C9–C8–C10 = 56.6 (3).

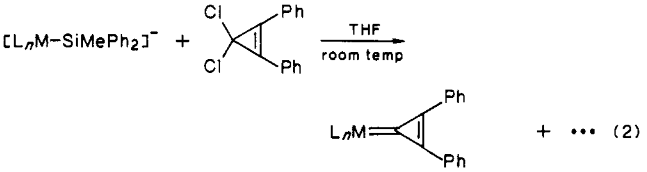
acting as a substitute for the unknown $[MeCp(CO)_2MnH]^-$. Gladysz et al. found that acylation of $[(CO)_4FeSiMe_3]^-$ is followed by a rapid 1,3-silatropic shift from iron to the acyl oxygen to give siloxycarbene complexes.[3]

Another preparative route to compounds with metal–element double bonds involving anionic silyl complexes, which is generalized in eq 1, has no precedent so far. In



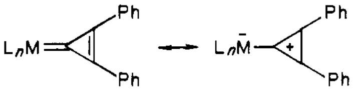
$$[L_nM\text{-}SiR_3]^- + X_2ER_m \xrightarrow{-X^-} L_nM\begin{matrix}SiR_3\\ EXR_m\end{matrix} \longrightarrow L_nM{=}ER_m + R_3SiX \quad (1)$$

this reaction anionic silyl complexes should be equivalent to L_nM^{2-} but offer the advantage that they can be used when L_nM^{2-} is unknown or unstable. To test this hypothesis, we treated $[MeCp(CO)_2Mn\text{-}SiMePh_2]^-$[2b,5] and $[(Ph_3P)(CO)_3Fe\text{-}SiMePh_2]^-$[2d] with 3,3-dichloro-1,2-diphenylcyclopropene, a compound which is known to form a cyclopropenylidene complex on reaction with $[(CO)_5Cr]^{2-}$.[6,7] The anticipated carbene complexes 1[8] and 2[9] are in fact obtained by this route (eq 2).



1, L_nM = $MeCp(CO)_2Mn$
2, L_nM = $(Ph_3P)(CO)_3Fe$

From the single ν(CO) band in the IR spectrum of 2 and only one signal for the carbonyl ligands in the ^{13}C NMR spectrum, we conclude that the geometry of 2 is a trigonal bipyramid with the PPh_3 and the carbene ligand in the axial positions, as in $(Et_3P)(CO)_3Fe{=}\overline{C{-}NMe{-}CH_2CH_2NMe}$.[10] The structure of 1 was determined by an X-ray structure analysis (Figure 1).[11] In $Cp(CO)_2MnCR_2$ complexes the confirmation is electronically favored, in which the plane of the atoms bonded to the carbene carbon coincides with the mirror plane of the $Cp(CO)_2Mn$ fragment.[12] In 1 the angle between both planes is only 14°. One of the phenyl substituents (C11–C16) is coplanar with the cyclopropene ring, and the other is twisted by 11°. Probably due to by steric interactions between the Cp ligand and the phenyl group C21–C26, the cyclopropene ring is slightly tilted, the angle Mn–C8–C9 (152.6°) being 1.8° larger than Mn–C8–C10 (150.8°). The Mn–C8 bond length is of the same magnitude as that in other $Cp(CO)_2MnCR_2$ complexes.[14] The C–C distances within the three-membered ring, the ^{13}C resonances of the cyclopropenyl carbon atoms, and IR data indicate that the bonding situation must be described by the two resonance forms A and B. The C9–C10 distance is distinctly shorter



A B

than C8–C9 and C8–C10;[13] however, the difference is not as pronounced as in cyclopropenes.[15] Both the ^{13}C chemical shift for the phenyl-substituted carbon atoms of the cyclopropenyl moiety at rather low field (178.9 ppm in 1)[16] and the ν(CO) bands in the IR spectrum of 1, which are shifted by about 45 cm^{-1} to lower wavenumbers relative to $MeCp(CO)_2MnCPh_2$,[17] indicate that electron density is to some extend withdrawn from the cyclopropene unit, according to B.

1,2-Elimination of R_3SiX has repeatedly been used in main-group chemistry to generate double bonds. Our results show that it can also be applied to the synthesis of transition-metal complexes containing metal–carbon double bonds. We assume that the first step of the reaction of anionic silyl complexes with 3,3-dichloropropene involves substitution of one chlorine substituent and formation of a metal complex that contains both a silyl group

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(8) Anal. Found (Calcd): C, 73.06 (72.63); H, 4.50 (4.50). Yield of isolated 1: 24%. IR (toluene, ν(CO)): 1935 (vs), 1868 (vs) cm^{-1}. ^1H NMR (200 MHz, C_6D_6, δ): 2.0 (s, 3 H, C_5H_4Me), 4.75 (m, 4 H, C_5H_4), 8.1 and 7.2 (m, 10 H, C_6H_5). $^{13}C\{^1H\}$ NMR (200 Hz, C_6D_6, δ): 14.3 (CH_3), 83.6, 84.1, 102.0 (C_5H_4), 125.7–132.5 (C_6H_5), 178.9 (*C*Ph), 234.1, 233.5 (*C*O and *C*(carbene)). Melting point: 131 °C.

(9) Anal. Found (Calcd): C, 72.92 (72.97); H, 4.54 (4.22). Yield of isolated 2: 22%. IR (toluene, ν(CO)): 1888 (vs, br) cm^{-1}. ^{31}P NMR (90 MHz, C_6D_6, δ, external H_3PO_4): 79.20. ^1H NMR (200 MHz, C_6D_6, δ): 7.2 (m), 8.1 (m), 8.3 (m). $^{13}C\{^1H\}$ NMR (200 Hz, C_6D_6, δ): 125–138 (C_6H_5), 177.5 (s, *C*Ph), 217.5 (d, CO, *J*(PFeC) = 27.0 Hz), 219.4 (d, C(carbene), *J*(PFeC) = 11.7 Hz). Melting point: 85 °C.

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(11) Crystals obtained by cooling a toluene solution of 1: monoclinic, space group $P2_1/a$, a = 1072.5 (4) pm, b = 1499.1 (6) pm, c = 1120.8 (5) pm, β = 95.85 (3)°; V = 1793 × 10^6 pm^3; Z = 4; d(calcd) = 1.41 g cm^{-3}; Mo K_α (λ = 71.069 pm). A total of 3480 independent reflections (ω scan, 2° ≤ 2θ ≤ 50°) were reduced to structure factors by correction for Lorentz and polarization effects and by an empirical absorption correction. Solution of the structure by the Patterson method. Anisotropic refinement of all non-hydrogen atoms by full-matrix least squares (fixed hydrogen parameters) resulted in R = 0.058 and R_w = 0.045 ($1/w = \sigma^2 + 0.0001F_o^2$) for 2226 reflections with $F_o \geq 3\sigma(F_o)$.

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and an α-chloro-substituted organic ligand. We have recently shown that the inverse situation, a chloro ligand at the metal and a silyl group at the metal-bonded carbon atom, also can be used to generate metal–carbon double bonds by R_3SiX elimination: on reaction of 1-chloro-1-(trimethylsilyl)alkenes with $Cp(CO)_2Mn$ fragments vinylidene complexes are formed, probably by Me_3SiCl elimination.[18]

Acknowledgment. We thank Johanna Kron for the NMR measurements and Jürgen Meyer and Wolfgang Hepp for the X-ray measurements.

Registry No. 1, 112533-44-5; 2, 112533-45-6; $[MeCp(CO)_2MnSiMePh_2]^-$, 112533-46-7; $[(Ph_3P)(CO)_3FeSiMePh_2]^-$, 112533-47-8; 1,1-dichloro-2,3-diphenylcyclopropene, 2570-00-5.

Supplementary Material Available: Details of the solution of the structure and listings of final atomic parameters, anisotropic *B*'s, and interatomic distances and angles (11 pages); a listing of observed and calculated structure factors (16 pages). Ordering information is given on any current masthead page.

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Reactions of Nucleophilic Reagents with Dianionic Hexacoordinated Germanium Complexes: A New Convenient Route to Functional Organogermanes from Germanium Dioxide

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Summary: Tetraorganogermanes and triorganogermanes can be prepared in two steps from GeO_2: the preparation of the anionic hexacoordinated germanium complexes followed by reaction of these with Grignard reagents to give the organogermanes.

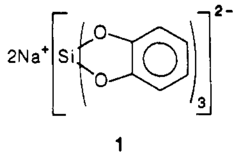
New routes for the preparation of functional organogermanes are attractive since the usual syntheses generally are long and difficult. For example, Ph_3GeH[1] is prepared as shown in Scheme I and other triorganogermanes are generally prepared in this way.[2]

Scheme I

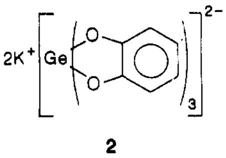
$$GeCl_4 \xrightarrow[\text{toluene}]{PhMgBr} GePh_4 \xrightarrow{Br_2} Ph_3GeBr \xrightarrow{LiAlH_4} Ph_3GeH$$

We previously have described[3] a novel synthesis of organosilicon compounds by reaction of organometallic reagents with the hexacoordinated silicon complex of catechol 1 which may be directly prepared from silica.

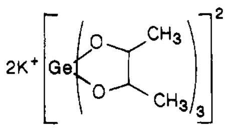
We now report that the germanium complexes 2 and 3 which can be prepared directly from GeO_2 under anhydrous conditions[4] can similarly be converted to germanes.



1



2



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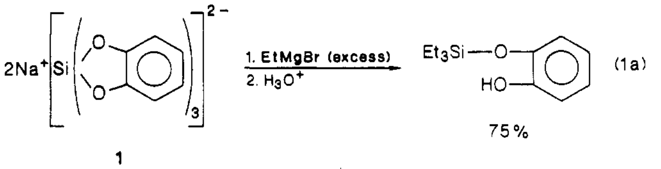
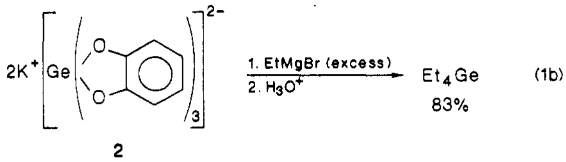
The results obtained with 2 are reported in Table I. Whatever the organometallic compound and the reaction conditions (solvent, temperature, and stoichiometry), only the tetrasubstituted organogermane is obtained. It is worth noting that the reactivity of 2 is greater than that of the analogous silicon complex 1[3] since in the latter case an excess of alkyl Grignard reagent leads to the formation of only three Si–C bonds (eq 1a and 1b).





Complex 2 also is more reactive than $GeCl_4$ or $GeBr_4$. Indeed, in ether a large excess of PhMgBr is necessary to prepare Ph_4Ge from $GeCl_4$ or $GeBr_4$ (eq 2 and 3).[5] To obtain a good yield of $GePh_4$ from $GeCl_4$ or $GeBr_4$, it is necessary to use THF[6] or toluene[7] as the solvent and to reflux the mixture.

$$GeBr_4 + 36 \text{ equiv of } PhMgBr \xrightarrow[35\ °C]{2\ h} \underset{40\%}{Ph_4Ge} \qquad (2)$$

$$GeBr_4 + 5 \text{ equiv of } PhMgBr \xrightarrow[20\ °C]{2\ h} \underset{33\%}{Ph_3GeBr} \qquad (3)$$

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(4) Preparation of Potassium Tris(1,2-benzenediolato)germanate (**2**). The reaction was carried out under nitrogen. A solution of MeOK (10.5 g, 0.15 mol) in methanol (75 mL) was added to a suspension of GeO_2 (7.83 g, 0.075 mol) in methanol (40 mL). Then a solution of catechol (24.75 g, 0.225 mol) in methanol (75 mL) was added at room temperature. The resulting mixture was stirred and heated at reflux for 72 h. The methanol was removed under vacuum and the solid residue washed twice with ether; 33.9 g of complex **2** was isolated as a white powder (95% yield): ^1H NMR (CD_3OD) δ 5.80–6.35 (m); ^{13}C NMR (CD_3OD) δ 151.2; 118.2, 112.8; IR (KBr) 1615 (m), 1580 (m), 1475 (F), 1325 (m), 1240 (F), 1090 (m), 1010 (m), 910 (f), 870 (m), 860 (m), 790 (F), 740 (F), 640 (F) cm^{-1}. Preparation of Potassium Tris(2,3-butanediolato)germanate (**3**). The reaction was carried out under nitrogen. A solution of MeOK (2.8 g, 0.04 mol) in 40 mL of methanol was added to a suspension of GeO_2 (2.09 g, 0.020 mol) in 20 mL of methanol. Then a solution of 5.4 g of 2,3-butanediol in 20 mL of methanol was added at room temperature. The resulting mixture was stirred and refluxed for 1 h. The methanol then was removed under vacuum and the solid residue washed twice with ether; 8.12 g of complex **3** was isolated as a white powder (97% yield): ^1H NMR (CD_3OD) δ 0.60 (d, 6 H, CH_3), 2.5–3.18 (m, 2 H, CH); ^{13}C NMR (CD_3OD) δ 73.8, 20.5; IR (KBr) 2940 (m), 1650 (m), 1440 (m), 1365 (F), 1120 (m), 1075 (F), 920 (F), 815 (F), 670 (m), 620 (m) cm^{-1}.

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