**Table V**

Weighted Least-Squares Planes and Atomic Displacements (Å × $10^3$)[b]

| atom | plane 1 | plane 2 |
|---|---|---|
| Pt | 0[a] | 0[a] |
| O(1) | 2[a] | 20[a] |
| C(1) | −20[a] | −19[a] |
| MP | 86 | 69 |
| Pt* | −18 | 0[a] |
| O(1)* | −20[a] | −20[a] |
| C(1)* | 3 | 19[a] |
| MP* | −104 | −69 |
| C(9)* | −495 | −505 |

Equations of the Planes Referred to Crystallographic Axes
plane 1: $5.371x - 8.434y - 3.367z = -5.892$
plane 2: $5.341x - 8.515y - 3.285z = -5.900$
plane 3: $2.443x + 3.551y + 8.545z = 7.156$

Dihedral Angles (Deg) between the Planes:
1-2, 0.5; 1-3, 78.9; 2-3, 78.8

[a] Distances refer to atoms defining the plane. [b] See footnote to Table IV. Plane 3 is defined by Pt, C(4), and C(5).

influence of a σ-bonded carbon atom.[12,13] The difference of 0.12 Å in the platinum–oxygen bond lengths is comparable to that between the two platinum–chlorine bond lengths trans to a σ-bonded carbon and to a π-bonded olefin in other platinum(II) complexes.[14]

The cyclo-olefinic ring has the same distorted boat conformation found in the complex chloro(2-methoxycycylo-octa-1,5-dienyl)(pyridine)platinum(II).[12] The slight lengthening of the double bond (1.411 Å) is a common feature observed upon coordination and compares well with the values quoted by Mason et al.[11,15] The Pt–C(1) σ-bond length (2.038 Å) and the Pt–C(olef) distances (mean value 2.10 Å) are among the shortest observed in other σ-alkyl[13] and π-olefin[16] complexes. The C═C bond makes an angle of 78.4° with the metal coordination plane and its midpoint is 0.07 Å out of it. As the dihedral angle between the coordination plane and the plane containing Pt and the olefinic carbon atoms has nearly the same value, the deviation of the double bond from the perpendicularity to the coordination plane consists in a tilt around the Pt-$\substack{\mathrm{C}\\ \mathrm{C}}$ axis, as found for similar compounds.[17] This distortion optimizes the nonbonding distances (3.57 and 3.52 Å, respectively) between the olefinic carbon atoms and the methyl C(9). All the intermolecular contacts fall in the normal range.

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**Registry No.** $[Pt(OMe)(C_8H_{12}OMe)]_2$, 75534-44-0; $PtCl_2COD$, 12080-32-9.

**Supplementary Material Available:** Table of the observed and calculated structure factors (6 pages). Ordering information is given on any current masthead page.

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# Preparation and Properties of Some New Cobalt and Chromium Carbonyl Derivatives of 1,4-Bis(dimethylsilyl)benzene

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Although there are numerous silyltetracarbonylcobalt compounds known, very few of these contain more than one cobalt center.[2] A relatively stable derivative containing two tetracarbonylcobalt entities joined by a bridging $SiH_2$ group has been reported;[3] however systems in which two $SiMe_2Co(CO)_4$ moieties are joined by a bridging oxygen (O),[4] nitrogen, (NH),[5] or alkyl ($CH_2$ or $CH_2CH_2$)[4,6] grouping are much less stable than comparable compounds that contain one tetracarbonylcobalt grouping. For example, solid $Me_3SiCo(CO)_4$ decomposes slowly at room temperature (87% of a sample held at room temperature in vacuo for 3 months was recovered unchanged)[7] whereas the bridged bis((tetracarbonylcobaltio)dimethylsilyl) systems $(CO)_4CoSiMe_2$–X–$SiMe_2Co(CO)_4$ (X = O, $CH_2$) decompose readily at −78 °C in a nitrogen atmosphere.[4] In the present project we have undertaken a study of the synthesis and properties of some bis((tetracarbonylcobaltio)dimethylsilyl) derivatives which contain an aromatic bridging group.

## Experimental Section

All synthetic work was done in nitrogen-purged drybags and/or glassware suitably modified for inert-atmosphere work. Infrared spectra of complexes were obtained on Perkin-Elmer spectrometers (Models 457 or 580). Cyclohexane was used as a solvent. Proton NMR spectra were recorded with use of a Hitachi-Perkin-Elmer R20 spectrometer. Spectra were obtained at ambient temperature on dilute solutions (ca. 5%) in deuteriochloroform with a cyclohexane reference. 1,4-Bis(dimethylsilyl)benzene (Silar Laboratories) and fresh samples of $Co_2(CO)_8$ and $Cr(CO)_6$ (Pressure Chemical Co.) were obtained commercially and used as received. Analyses were performed by Geller Microanalytical Laboratory.

**Synthesis of $\eta^6$-[1,4-Bis(dimethylsilyl)benzene]chromium Tricarbonyl.** A mixture of 1,4-bis(dimethylsilyl)benzene (17.2 g, 88 mmol) and $Cr(CO)_6$ (6.3 g, 29 mmol) in a glyme (15 mL)/diglyme (30 mL) solution was allowed to reflux for 28 h in a Strohmeier-type reactor.[8] After removal of the solvents and unreacted starting materials by vacuum distillation, a black oil remained. This oil was passed through a 3 × 1$^1/_2$ in. alumina column with pentane as eluant. Upon cooling of the product-containing eluant to −78 °C, crude $\eta^6$-[1,4-$(HSiMe_2)_2C_6H_4$]$Cr(CO)_3$ (4.0 g, 12 mmol, 41% yield) precipitated. The pure product, obtained as yellow crystals (mp 46.5–48.5 °C), was obtained after further purification by recrystallization from pentane and sublimation onto a cold finger held at 0 °C. Anal. Calcd for $C_{13}H_{18}O_3Si_2Cr$: C, 47.2; H, 5.5. Found: C, 47.1; H, 5.5. Spectral characterization data are given in the discussion.

**Synthesis of $\eta^6$-[1,4-Bis((tetracarbonylcobaltio)dimethylsilyl)benzene]chromium Tricarbonyl.** A solution of $\eta^6$-[1,4-$(HSiMe_2)_2C_6H_4$]$Cr(CO)_3$ (2.27 g, 6.87 mmol) in methylene chloride

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(10 mL) was added to a precooled (0 °C) methylene chloride solution of $Co_2(CO)_8$ (1.61 g, 4.71 mmol), and the mixture was allowed to react at 0 °C for 1 h with stirring. The solution was then allowed to gradually warm to room temperature. After an additional 12-h reaction period, the solvent was pumped away, and the remaining solid was washed with pentane until the washings were essentially colorless. The solid was redissolved in methylene chloride and the resulting solution centrifuged to remove any insoluble material. Upon addition of pentane to the product-containing methylene chloride solution, a yellow solid precipitated. A second precipitation yielded a bright yellow solid identified as $\eta^6$-[1,4-$(CO)_4CoSiMe_2$–$C_6H_4$–$SiMe_2Co(CO)_4$]$Cr(CO)_3$ (1.01 g, 1.5 mmol, 32% yield) by chemical and spectral analyses. The compound melts at 102 °C dec. Anal. Calcd for $C_{21}H_{16}O_{11}Si_2CrCo_2$: C, 37.6; H, 2.4. Found: C, 37.8; H, 2.7.

**Synthesis of 1,4-Bis((tetracarbonylcobaltio)dimethylsilyl)benzene.** 1,4-Bis(dimethylsilyl)benzene (1.5 g, 7.7 mmol) was added slowly at room temperature with stirring to freshly purified orange crystals of $Co_2(CO)_8$ (2.6 g, 7.6 mmol) in a nitrogen-filled 100-mL reaction vessel that was fitted with a mercury bubbler. The reaction was exothermic, and a gas, presumed to be hydrogen, was evolved. During the reaction a white solid was observed in the pasty mixture, and after about 30 min, 10 mL of dry pentane was added, and the system was allowed to stir for an additional 1 h. The white precipitate was filtered and washed 5 times with 10-mL portions of pentane. During each washing, the solid remained an off-white color, but the filtrate acquired a dark purple color. (Even after washing the solid with 300 mL of pentane in 10-mL portions, this coloring appeared in the filtrate.) The off-white solid was identified as 1,4-Bis((tetracarbonylcobaltio)dimethylsilyl)benzene (1.8 g, 3.4 mmol, 45% yield) by its chemical and spectral analyses. Anal. Calcd for $C_{18}H_{16}Co_2O_8Si_2$: C, 40.5; H, 3.0. Found: C, 39.5; H, 3.0. IR ($\nu$(CO)): 1993, 2032, 2082 $cm^{-1}$. NMR: $\delta$ 0.87 ($CH_3$), 7.21 ($C_6H_4$).

## Results and Discussion

Most of the (arylsilane)chromium tricarbonyl complexes that have been reported contain only one silicon center.[9] Organic groups are by far the most common substituent on silicon in these systems, although several silicon-functional derivatives which contain silicon–chlorine bonds are known.[10] A few arylsilane complexes which contain more than one organosilicon grouping have been investigated;[11,12] however, derivatives of this latter type with functional groups on silicon have not been studied in spite of their important potential use as metal-complexed cross-linking reagents or bridging centers. In the present work, we have prepared the chromium tricarbonyl complex of the bifunctional derivative 1,4-$(HSiMe_2)_2C_6H_4$ and have used it as a precursor in the synthesis of the bridging bis(cobalt) trimetal complex $\eta^6$-[1,4-$(CO)_4CoSiMe_2$–$C_6H_4$–$SiMe_2Co(CO)_4$]$Cr(CO)_3$. The successful preparation of this compound and its uncomplexed analogue 1,4-$(CO)_4CoSiMe_2$–$C_6H_4$–$SiMe_2Co(CO)_4$ gave us the opportunity to observe the thermal stability of some bis((tetracarbonylcobaltio)dimethylsilyl) derivatives containing bridging aromatic and modified-aromatic groups.

The synthesis of the chromium tricarbonyl complex of 1,4-(dimethylsilyl)benzene was achieved by refluxing the latter compound with $Cr(CO)_6$ for 28 h in a glyme/diglyme mixture.

$$\text{1,4-}(HSiMe_2)_2C_6H_4 + Cr(CO)_6 \rightarrow \eta^6\text{-}[\text{1,4-}(HSiMe_2)_2C_6H_4]Cr(CO)_3 + 3CO$$

The solid produced in the reaction had the characteristic yellow color of $\pi$-aryl $Cr(CO)_3$ complexes. The survival of the Si–H bonds and further proof of the addition of the $Cr(CO)_3$ grouping were confirmed by the compound's infrared and proton NMR spectra. In the infrared spectrum, $\nu$(Si–H) is present at 2130 $cm^{-1}$, shifted very slightly from its position at 2136 $cm^{-1}$ in the uncomplexed derivative. The $\nu$(CO) absorptions for the new complex appear as an $A_1$ and an unsplit E modes at 1974 and 1910 $cm^{-1}$, respectively.[13] The proton NMR spectrum of the complex shows a Si–H septet at $\delta$ 4.44, shifted only slightly from the Si–H septet at $\delta$ 4.40 for the uncomplexed material. The doublet ($J_{H-H}$ = 4.2 Hz) silicon–methyl resonance is at $\delta$ 0.40 in both the free and complexed derivatives. As expected, the protons of the $C_6H_4$ grouping are more sensitive than the other protons to the incorporation of the $Cr(CO)_3$ entity as a $\pi$ complex and a characteristic 2.2-ppm upfield shift is observed on going from the free derivative ($\delta$ 7.49) to the complexed derivative ($\delta$ 5.27).[10] As in the case of other metal-complexed 1,4-bissubstituted benzene systems, the $C_6H_4$ protons appear as a sharp singlet, indicating nonrestricted rotation of the chromium tricarbonyl grouping.[11]

Purified samples of solid $\eta^6$-[1,4-$(HSiMe_2)_2C_6H_4$]$Cr(CO)_3$ are very stable thermally at room temperature, particularly when they are kept in the dark in a nitrogen atmosphere. Under these conditions a sample of the compound remained unchanged after 24 months. However, if the solid compound under a nitrogen atmosphere is exposed to laboratory lights, it slowly changes to a yellow oil over a period of about 1 week. It will also decompose to a yellow oil, $Cr(CO)_6$, and a black solid when heated to 100 °C for 2 h in an evacuated tube. A pure sample could be exposed to air for up to 2 days without any observable color change; however after 2 days, the compound decomposes to form an unidentified yellow oil and a green solid. The decomposition in air is accelerated by light. Solutions of the compounds in methylene chloride or pentane decompose rapidly when exposed to air.

The usefulness of $\eta^6$-[1,4-$HSiMe_2$–$C_6H_4$–$SiMe_2H$]$Cr(CO)_3$ as a synthetic precursor was demonstrated by the compound's reaction with $Co_2(CO)_8$.

$$\eta^6\text{-}[\text{1,4-}HSiMe_2\text{–}C_6H_4\text{–}SiMe_2H]Cr(CO)_3 + Co_2(CO)_8 \rightarrow \eta^6\text{-}[\text{1,4-}(CO)_4CoSiMe_2\text{–}C_6H_4\text{–}SiMe_2Co(CO)_4]Cr(CO)_3 + H_2$$

The identification of this new bis((tetracarbonylcobaltio)dimethylsilyl) derivative containing a $Cr(CO)_3$-complexed bridging aromatic group was confirmed by infrared and proton NMR measurements. The chemical shift of the $C_6H_4$ protons in the mixed-metal complex remained in the upfield position ($\delta$ 5.55) as noted for the $Cr(CO)_3$-complexed dihydride precursor while the Si–H signal was completely absent and the silicon methyl signal appeared as a single absorption at $\delta$ 0.94. There was no Si–H absorption in the compound's infrared spectrum, while the two $A_1$ and E $\nu$(CO) absorptions of the $Cr(CO)_3$ grouping remained at 1971 and 1909 $cm^{-1}$, respectively. Three additional $\nu$(CO) absorptions were observed at 1996, 2028, and 2086 $cm^{-1}$ which can be attributed to the presence of two noninteracting $Co(CO)_4$ entities. The positions and intensities of these cobalt carbonyl absorptions correspond well with those reported for other similar bis(tetracarbonylcobalt) derivatives. For example, in $(CO)_4CoSiMe_2CH_2CH_2SiMe_2Co(CO)_4$ there are three $\nu$(CO) absorptions at 1995, 2028, and 2089 $cm^{-1}$ with a weak unassigned shoulder on the high-energy side of the 2089-$cm^{-1}$ band.[6] The main $\nu$(CO) absorptions for $(CO)_4CoSiMe_2$–O–$SiMe_2Co(CO)_4$ appear at 2000, 2020, and 2080 $cm^{-1}$ with an additional weak band at 1960 $cm^{-1}$.[4]

When purified, $\eta^6$-[1,4-$(CO)_4CoSiMe_2$–$C_6H_4$–$SiMe_2Co(CO)_4$]$Cr(CO)_3$ was considerably more stable than other bis((tetracarbonylcobaltio)dimethylsilyl) derivatives that contain oxygen- or alkyl-bridging groups.[4,6] Whereas samples

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of $(CO)_4CoSiMe_2–X–SiMe_2Co(CO)_4$ (X = O, $CH_2$) decompose at −78 °C, purified samples of $\eta^6$-[1,4-$(CO)_4CoSiMe_2–C_6H_4–SiMe_2Co(CO)_4$]$Cr(CO)_3$ could be stored for several weeks under a nitrogen atmosphere at room temperature in the dark without any apparent decomposition. Likewise, solutions of the compound in methylene chloride were stable for several hours in a nitrogen atmosphere, although when the solutions were exposed to air, decomposition was accelerated appreciably.

In order to see if the coordination of the $Cr(CO)_3$ entity had a part in the improved stability, we undertook the preparation of the uncomplexed analogue 1,4-$(CO)_4CoSiMe_2–C_6H_4–SiMe_2Co(CO)_4$. This compound was obtained by the reaction of 1,4-$HSiMe_2–C_6H_4–SiMe_2H$ with $Co_2(CO)_8$. The infrared and proton NMR spectra of the compound were very similar to those of the analogous $Cr(CO)_3$ complex described above except that the two $A_1$ and E $Cr(CO)_3$ absorptions were absent in the infrared spectrum and the position of the $C_6H_4$ protons in the NMR spectrum was at a lower field position ($\delta$ 7.21) than in the spectrum of the complex. The compound was reasonably stable thermally but not nearly as stable as the analogous $Cr(CO)_3$ complex. It slowly acquired a violet color when stored as a solid in a nitrogen atmosphere at room temperature overnight. After 2 weeks under these conditions, the material was a gray-brown powder. The pure compound dissolves readily in cyclohexane, and there was no obvious color change when fresh solution samples were taken for infrared analysis. However, after several hours under these conditions, the solution acquired a purple color. New absorptions appeared in the spectrum which corresponded to the major bands found in $Co_2(CO)_8$. The solution darkened further when the sample was held in solution at room temperature for longer time periods. Samples of the pure compound and solutions of the material rapidly acquired a dark brown color when exposed to air.

We conclude from these observations that the thermal stability of bis((tetracarbonylcobaltio)dimethylsilyl) derivatives is influenced by the nature of the bridging group. The derivatives containing the bridging aromatic groups (para positions are more stable than those that contain bridging oxygen, nitrogen (NH), or alkyl groupings. The chromium tricarbonyl complex of the bridged aromatic system is considerably more stable than the analogous uncomplexed derivative. All of the systems have a marked tendency to decompose in solution. We have not been able to establish the exact decomposition pathways of the decompositions observed, although $Co_2(CO)_8$ appears to be one of the initial decomposition products in solution.

**Registry No.** $\eta^6$-[1,4-$(HSiMe_2)_2C_6H_4$]$Cr(CO)_3$, 75830-40-9; $\eta^6$-[1,4-$(CO)_4CoSiMe_2–C_6H_4–SiMe_2Co(CO)_4$]$Cr(CO)_3$, 75830-41-0; 1,4-$(CO)_4CoSiMe_2–C_6H_4–SiMe_2Co(CO)_4$, 75830-42-1; $Cr(CO)_6$, 13007-92-6; $Co_2(CO)_8$, 10210-68-1.

# Correspondence

## Interpretation of $^{13}C$ NMR Relaxation Times in $K_2Ni(CN)_4$ and Other Diamagnetic Cyano Complexes

*Sir:*

Recently carbon-13 NMR relaxation times were reported for several diamagnetic cyano complexes.[1] Except for $Fe(CN)_6^{4-}$, which was thought to be contaminated by paramagnetic impurities, the carbon-13 spin–lattice relaxation times, $T_1$'s, for the cyano carbons of these complexes exceeded 30 s. Carbon-13 spin–spin relaxation times, $T_2$'s, were however much shorter, ranging between 0.11 and 0.64 s at 303 K. All reported measurements were carried out for potassium salts of the complexes in aqueous ($D_2O$) solution. The observed ratios of $T_1/T_2$ ($10^2$–$10^3$) and the generally observed increase in $T_1$ with increasing temperature led Pesek and Mason[1] to conclude that the mechanism for $^{13}C$ relaxation ($T_1$ and $T_2$) in these complexes is consistent with scalar coupling of the second kind to nitrogen-14.[2–4] In this correspondence we present evidence which indicates that the chemical shielding anisotropy (CSA) and $^{14}N$–$^{13}C$ dipole–dipole mechanisms are important for spin–lattice relaxation in $Ni(CN)_4^{2-}$ at the applied field used in ref 1 (2.35 T) and show that although scalar coupling of the second kind is important for spin–spin relaxation, its contribution is negligible for spin–lattice relaxation.

Carbon-13-enriched (50%) potassium tetracyanonickelate(II) was prepared from $K^{13}CN$ and $Ni(CN)_2$ as previously described.[5] A 0.5 M solution of $K_2Ni(CN)_4$ in deuterium oxide was degassed under vacuum by the freeze–pump–thaw technique. Spin–lattice relaxation times were measured at 300 ± 2 K with use of the inversion recovery sequence at 20 MHz (1.87 T), on a Varian CFT-20 spectrometer and at 100.6 MHz with use of Bruker WH-400 (9.39 T) spectrometer. The $^{13}C$ $T_1$'s calculated with the use of a nonlinear three-parameter least-squares fitting procedure[6] were 97 ± 9 s at 20 MHz and 11.4 ± 0.8 s at 100.6 MHz. The $\pi/2$ pulses at these frequencies were 16 and 20 $\mu$s, respectively.

Since chemical shielding anisotropy is the only mechanism for spin–lattice relaxation which can lead to shorter $T_1$'s at higher fields under extreme narrowing conditions, the above data imply that the CSA mechanism completely dominates at 100.6 MHz. Assuming that the rates of relaxation are additive (eq 1) and that $T_1$(other) ≥ 97 s at 100.6 MHz leads to $T_1$(CSA) ≤ 12.9 s. Since $1/(T_1(\mathrm{CSA}))$ depends on the

$$\frac{1}{T_1(\mathrm{obsd})} = \frac{1}{T_1(\mathrm{CSA})} + \frac{1}{T_1(\mathrm{other})} \tag{1}$$

square of the applied field, $T_1$(CSA) is less than or equal to 325 s at 20 MHz and 208 s at 25 MHz. Although the errors in these estimates of $T_1$(CSA) are at least 10%, it is apparent that the chemical shielding anisotropy mechanism plays a significant role in $^{13}C$ relaxation at 25 MHz (2.35 T).[8]

The effective rotational correlation time, $\tau_{\mathrm{eff}}$, for the Ni-

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