been observed in saturated[4] and unsaturated dinuclear carbonyls.[8,9] The bond distance Ru(1)–Ru(2) = 2.936 (1) Å. Ru–C distances are 1.938 (9)–1.836 (10) Å, and the Ru–C–O angles are 179.4 (8)–172.7 (1)°. The closest intermolecular distance observed is 3.075 (6) Å, between the methyl carbon of CH_3CN and O(12).

The structure of the $[Os_2(CO)_8]^{2-}$ dianion is like that of $[Fe_2(CO)_8]^{2-}$.[4] It can be viewed as two $Os(CO)_4$ units with CO's at four vertices of a trigonal bipyramid with the fifth vertex being an axial site common to both $Os(CO)_4$ units. The equatorial planes are staggered with respect to each other. As in $[Fe_2(CO)_8]^{2-}$, each equatorial CO is tilted toward the opposite metal.[4] The angle of tilt, M–M–C, is in the range 80.5 (2)–87.0 (2)°. Metal–metal distances for the independent molecules in the unit cell are Os(1)–Os(2) = 2.992 (1) Å and Os(3)–Os(3′) = 2.985 (1) Å. The Os–C distances are 1.917 (6)–1.867 (6) Å, and the Os–C–O angles are 178.8 (6)–174.9 (6)°. The closest intermolecular distance observed is 3.042 (10) Å, between a phenyl carbon of the cation (C(246)) and O(14).

The salt $Na[Ru_2(CO)_8]$ was prepared from the reaction of a 1:1 molar ratio of $Na_2[Ru(CO)_4]$ (264.3 mg) and CO_2 (1.02 mmol) in THF (15 mL), stirred overnight at −78 °C and then for 4–5 h at room temperature. A pale red solution formed, and the theoretical amount of CO was produced. The resultant $Na_2[Ru_2(CO)_8]$ was converted to $[P(C_6H_5)_4]_2[Ru_2(CO)_8]$, a pale yellow solid, in about 90% yield by metathesis of $Na_2[Ru_2(CO)_8]$ with $[P(C_6H_5)_4]Br$ in THF. $[P(C_6H_5)_4]_2[Ru_2(CO)_8]$: IR ($CH_3CN$) ν(CO) 1930, 1865 cm^{-1}; ^{13}C NMR (CH_3CN, +30 to −35 °C) 215.1 ppm.

Reaction of $K_2[Os(CO)_4]$ with CO_2 in a 1:1 molar ratio in THF overnight at −78 °C and then at 60 °C for 48 h produced a solution of $K_2[Os_2(CO)_8]$. The salt $[P(C_6H_5)_4]_2[os_2(CO)_8]$ was obtained as a yellow solid in 55% yield in the same manner as indicated above. $[P(C_6H_5)_4]_2[Os_2(CO)_8]$: IR ($CH_3CN$) ν(CO) 1940, 1865 cm^{-1}; ^{13}C NMR (CH_3CN, 30 to −35 °C) 193.5 ppm.

Acknowledgment. We thank the National Science Foundation for support of this work through Grant CHE 84-11630.

Supplementary Material Available: Listing of positional parameters, anisotropic thermal parameters of non-hydrogen atoms, selected bond distances and bond angles, least squares planes and dihedral angles, and structure factor amplitudes (113 pages). Ordering information is given on any masthead page.

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Preparation and Reactivity of Mononuclear (η^5-Cyclopentadienyl)cobalt Carbene Complexes

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Received April 23, 1985

Summary: $(\eta^5\text{-}C_5H_5)Co(CO)_2$ reacts with bis(1,3-dimethylimidazolin-2-ylidene) to furnish the first mononuclear cyclopentadienylcobalt carbene complex **1**. Complex **1** reacts with several electrophilic reagents including Ph_2S_2 to produce the structurally characterized paramagnetic cyclopentadienylcobalt carbene complex **4**.

Since the discovery of the first transition-metal carbene complex $(CO)_5W[C(OCH_3)CH_3]$ by Fischer and Maasböl in 1964,[1] there has been an ever increasing interest in the preparation, structure, and reactivity of new transition-metal carbene complexes.[2] Diamagnetic carbene complexes are known for nearly all the transition metals; in contrast, there are relatively few paramagnetic carbene complexes including derivatives of iron,[3] chromium,[4] and ruthenium.[5] This report describes the preparation and reactivity of several mononuclear cyclopentadienylcobalt carbene complexes including the isolation of a paramagnetic cobalt carbene complex (**4**) which has been characterized by single-crystal X-ray diffraction methods.

Several mononuclear cobalt carbene complexes are known,[6] but surprisingly, no carbene complexes derived from $(\eta^5\text{-}C_5H_5)Co(CO)_2$ have been reported, even though this compound has been known for over 30 years.[7] Dinuclear bridging carbene complexes of cobalt have, however, been prepared from the reaction of $(\eta^5\text{-}C_5H_5)Co(CO)_2$ with diazo compounds.[8]

Treatment of $(\eta^5\text{-}C_5H_5)Co(CO)_2$ with bis(1,3-dimethylimidazolindin-2-ylidene) in boiling methylcyclohexane produced **1** as red-brown crystals.[9] Quantities up to 5 g have been prepared in yields ranging from 60 to 75%. In most cases complex **1** crystallizes analytically pure from the reaction mixture. This cobalt carbene is extremely air-sensitive as a solid and in solution but exhibits good thermal stability in a nitrogen atmosphere. No reaction occurred between **1** and Ph_3P in boiling toluene during a 24-h period (>90% recovery of **1**). Complex **1** shows a very low-energy metal carbonyl band in the infrared spectrum at 1915 cm^{-1} (pentane). This value is one of the lowest metal carbonyl bands observed for complexes of the type $(\eta^5\text{-}C_5H_5)Co(CO)L$. Complexes with similar values include $(\eta^5\text{-}C_5H_5)Co(CO)(PMe_3)$ (1923 cm^{-1}, pentane)[10] and $(\eta^5\text{-}C_5Me_5)Co(CO)(PMe_3)$ (1915 cm^{-1}, hexane).[11] This sug-

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(9) Anal. Calcd for $C_{11}H_{15}CoN_2O$: C, 52.81; H, 6.04; N, 11.20. Found: C, 53.08; H, 6.06; N, 11.14. ^1H NMR (C_6D_6): δ 2.54 (s, 4 H), 3.05 (s, 6 H), 4.77 (s, 5 H). $^{13}C\{^1H\}$ NMR (C_6D_6): δ 38.44, 50.62, 81.24, 207.38, 218.46.

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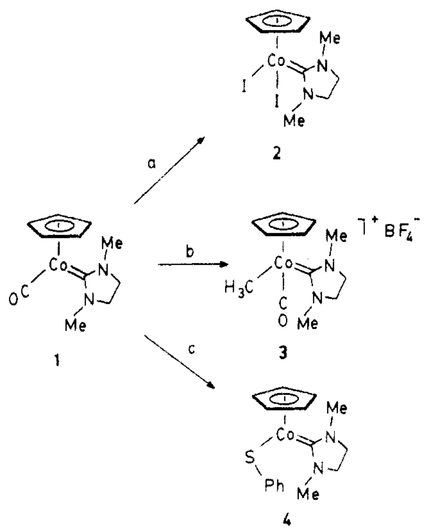
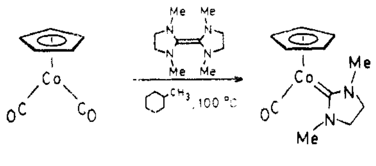
Scheme I[a]



[a] Reagents: (a) I_2, Et_2O; (b) CH_3I, MeOH, $AgBF_4$; (c) Ph_2S_2, toluene.

gests that the carbene fragment in 1 is an extremely strong σ-donor ligand.[12]



1

Complex 1 undergoes oxidative addition reactions with several substrates (Scheme I). Treatment of 1 with I_2 in diethyl ether produces complex 2 in 59% yield as black air-stable crystals.[13] Furthermore, treatment of 1 with CH_3I in methanol followed by $AgBF_4$ produced the cationic carbene complex 3 in 58% yield as orange crystals.[14] Complex 3 shows a metal carbonyl band in the infrared spectrum at 2030 cm^{-1} (acetone-d_6). A similar compound, $[(\eta^5\text{-}C_5H_5)Co(CO)(PMe_3)Me]PF_6$, shows a carbonyl band at 2040 cm^{-1}.[10] When a toluene solution of 1 was treated with Ph_2S_2, the solution gradually changed color from red-brown to dark green. Concentrating this solution followed by cooling to −30 °C produced dark green crystals of 4 in 60% yield.[15]

Complex 4 arises from a one-electron oxidative addition reaction. This result is somewhat surprising because $(\eta^5\text{-}C_5H_5)Co(CO)(PMe_3)$ reacts with Ph_2S_2 to give $(\eta^5\text{-}C_5H_5)Co(SPh)_2(PMe_3)$, a product arising from a two-electron oxidative addition process.[16] Steric factors may prevent the formation of $(\eta^5\text{-}C_5H_5)Co(SPh)_2[C(NMe)CH_2CH_2(NMe)]$ in the reaction of 1 with Ph_2S_2.[17] The d^7 Co(II) complex 4 is paramagnetic, $\chi = 5.76 \times 10^{-6}$ ($\mu_{eff} = 2.2\ \mu_B$) as determined by Evans's NMR method,[18] and is formally a 17-electron complex. Magnetic moments for similar Co(II) compounds include $(\eta^5\text{-}C_5H_5)_2Co$ ($\mu_{eff} = 1.76\ \mu_B$)[19] and $(\eta^5\text{-}C_5H_5)Co(PPh_3)Cl$ ($\mu_{eff} = 1.8\ \mu_B$).[20]

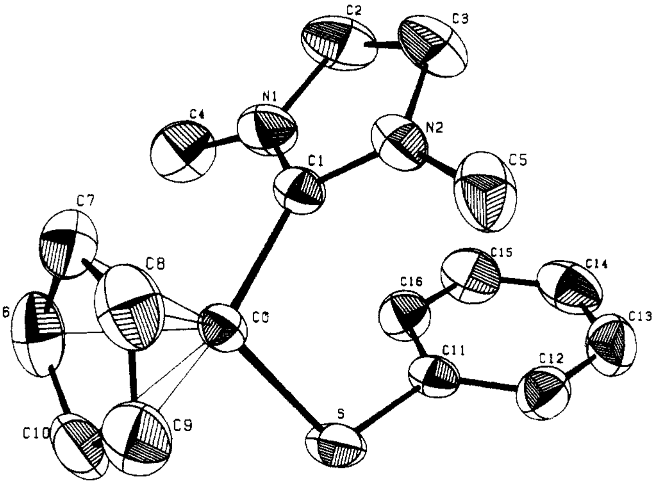


Figure 1. Molecular structure and atom labeling scheme for $(\eta^5\text{-}C_5H_5)Co(SPh)[C(NMe)CH_2CH_2(NMe)]$ (4) with the atoms represented by their 50% probability ellipsoids for thermal motion. Bond distances and angles involving the centroid of the cyclopentadienyl ligand are Co–Cent = 1.73 Å, Cent–Co–S = 130.3°, and Cent–Co–C(1) = 131.9°.

Because of the lack of structural data for cobalt carbene complexes[6c,e] and monocyclopentadienylcobalt(II) compounds,[21] we determined the single-crystal X-ray structure of complex 4.[22]

The molecular structure and atom labeling scheme for complex 4 are presented in Figure 1. There is no crystallographically imposed symmetry in the molecule, which crystallizes with four monomers in the orthorhombic unit cell. The Co–C(1) carbene bond distance of 1.902 (3) Å is short and indicates a strong metal–carbene interaction. This value is marginally shorter than the 1.91-Å cobalt–carbene separation found for $(Ph_3Ge)(CO)_3Co[C(OEt)Et]$[6c] and significantly shorter than the 1.974 (15) Å observed in $Co(CO)(NO)(PPh_3)[C(NMe)CH(Me)CH_2(NMe)]$.[6e] The cobalt and atoms C(1), N(1), N(2), C(2), and C(3) of the carbene ligand are coplanar to 0.02 Å. The methyl

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[12] Similar observations have been made by Lappert and co-workers for other carbene complexes containing this ligand. See: ref 6d and Lappert, M. F. *J. Organomet. Chem.* **1975**, *100*, 139.

[13] Anal. Calcd for $C_{10}H_{15}CoI_2N_2$: C, 25.23; H, 3.18; N, 5.89. Found: C 25.37; H, 3.23; N, 5.79. ^1H NMR (acetone-d_6): δ 3.65 (s, 6 H), 3.86 (s, 4 H), 5.18 (s, 5 H). MS: *m/e* 349 (M^+ − I).

[14] Anal. Calcd for $C_{12}H_{18}BCoF_4N_2O$: C, 40.94; H, 5.15; N, 7.96. Found: C, 39.67; H, 5.21; N, 7.86. ^1H NMR (acetone-d_6): δ 1.03 (s, 3 H), 3.29 (s, 6 H), 3.80 (s, 4 H), 5.72 (s, 5 H). Compound 3 is somewhat thermally sensitive and two analytical determinations gave low percent carbon.

[15] Anal. Calcd for $C_{16}H_{20}CoN_2S$: C, 57.99; H, 6.08; N, 8.46. Found: C, 58.08; H, 6.09; H, 8.35. MS: *m/e* 331 (M^+).

[16] Werner, H.; Jutnani, B. *J. Organomet. Chem.* **1981**, *209*, 211.

[17] Using CPK molecular models it was determined that the carbene ligand $C(NMe)CH_2CH_2(NMe)$ was larger than PMe_3. The measured cone angle of 127° [maximum half angle = 75°; minimum half angle = 52°; Co–C(carbene) distance = 2.11 Å] for $C(NMe)CH_2CH_2(NMe)$ is somewhat larger than the cone angle of 118° for PMe_3. See: Tolman, C. A. *Chem. Rev.* **1977**, *77*, 313.

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[22] Complex 4 crystallizes in the orthorhombic space group $P2_12_12_1$ (D_2^4, No. 19) with cell constants a = 8.558 (2) Å, b = 12.375 (2) Å, c = 14.865 (2) Å, and D_{calcd} = 1.40 g cm^{-3} for Z = 4 (Mo Kα, λ = 0.71073 Å). Data were collected on an Enraf-Nonius CAD4 diffractometer. Least-squares refinement based on 1836 observed reflections led to a final R value of 0.037 (181 parameters varied in the final cycles). Investigation of the enantiomorphic form resulted in a higher R value (0.045) and higher standard deviations. Hydrogen atoms were included with fixed thermal parameters. Their positional parameters were refined for several least-squares cycles and were then fixed in the final refinement. All non-hydrogen atoms were refined with anisotropic thermal parameters. The data were corrected for Lorentz, polarization effects, and absorption.

groups C(4) and C(5) bend 0.04 and 0.08 Å out of this plane, respectively.

Despite the close approach of the carbene moiety, there does not appear to be any great strain on the other two ligands. The Co–S bond distance (2.175 Å) is just under the Co–S single bond value of 2.20 Å predicted from the sum of single bond radii.[23] The average Co–C(π) separation of 2.10 Å is essentially identical with the values of 2.096 (8) and 2.083 Å found for the average Co–C(cyclopentadienyl) distances in $(\eta^5\text{-}C_5H_5)_2Co$[24] and $[(\eta^5\text{-}C_5H_5)Co(PEt_3)_2]BF_4$,[21] respectively. The S–Co–C(1) bond angle of 97.8 (1)° is less than the P–Co–P angle of 101.21 (3)° observed in the more sterically demanding environment of $[(\eta^5\text{-}C_5H_5)Co(PEt_3)_2]BF_4$.[21] Smaller ligand–metal–ligand angles have been found for compounds of the type $(\eta^5\text{-}C_5Me_5)Co(CO)_2$ (CO–Co–CO = 93.7 (2)°)[25] and $(\eta^{10}$-fulvalene)$[Co(CO)_2]_2$ (CO–Co–CO = 92.7 (3)°).[26]

We are continuing to study the preparation and reactivity of other cobalt carbene complexes as well as new rhodium carbene complexes.[27]

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Supplementary Material Available: Tables of bond distances and angles, positional and thermal parameters, and observed and calculated structure factor amplitudes (12 pages). Ordering information is given on any current masthead page.

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(25) Byers, L. R.; Dahl, L. F. *Inorg. Chem.* **1980**, *19*, 277.

(26) Rogers, R. D., unpublished results.

(27) For example, $(\eta^5\text{-}C_5Me_5)Co(CO)[\overline{C(NMe)CH_2CH_2(N}Me)]$ has been prepared from $(\eta^5\text{-}C_5Me_5)Co(CO)_2$ and bis(1,3-dimethylimidazolindin-2-ylidene) in boiling methylcyclohexane. Anal. Calcd for $C_{16}H_{25}CoN_2O$: C, 59.99; H, 7.87; N, 8.75. Found: C, 60.16; H, 8.01; N, 8.59. ^1H NMR (C_6D_6): 1.92 (s, 15 H), 2.47 (m, 2 H), 2.53 (m, 2 H), 3.14 (s, 6 H). $^{13}C\{^1H\}$ NMR (C_6D_6) δ 11.31, 38.08, 50.80, 90.98, 210.17, 224.02 IR (Pentane): ν_{CO} 1900 cm^{-1}. Likewise, $(\eta^5\text{-}C_5H_5)Rh(CO)[\overline{C(NMe)\text{-}CH_2CH_2(N}Me)]$ was prepared in a manner similar to 1. Anal. Calcd for $C_{11}H_{15}N_2ORh$: C, 44.91, H, 5.14; N, 9.53. Found: C, 44.92; H, 5.14; N, 9.55. ^1H NMR (C_6D_6): δ 2.46 (s, 4 H), 2.98 (s, 6 H), 5.40 (s, 5 H). $^{13}C\{^1H\}$ NMR (C_6D_6): δ 39.25, 50.38, 85.60, 196.84 (d, $J_{^{103}Rh-^{13}C}$ = 92.9 Hz), 207.72 (d, $J_{^{103}Rh-^{13}C}$ = 62.9 Hz). IR (C_6D_6): ν_{CO} 1920 cm^{-1}. MS: *m/e* 294 (M^+).

Reaction of a Silene with a Silacyclopropane To Yield a Disilacyclopropane

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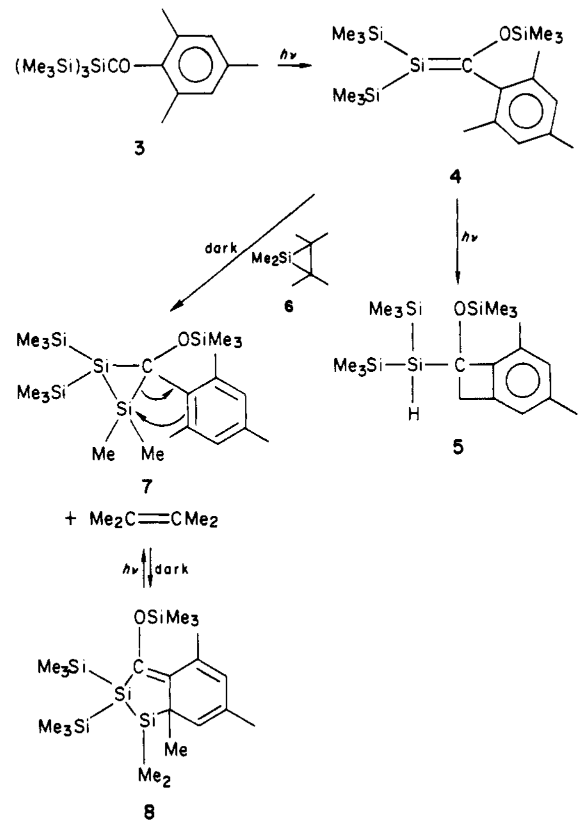
Summary: Photolysis of mesitoyltris(trimethylsilyl)silane initially yields the isomeric silene which subsequently inserts photochemically into one of the C–H bonds of an *o*-Me group to yield a substituted dihydrobenzocyclobutene. In the presence of hexamethylsilirane the silene adds Me_2Si: to yield a disilacyclopropane, the first such addition to a stable silene reported. In the dark the disilacyclopropane undergoes a 1,3-silyl carbon-to-aromatic carbon shift to give a bicyclo [4.3.0] system which photochemically isomerizes back to the disilacyclopropane.

Scheme I



As part of a program studying the behavior of relatively stable silenes, we wished to see if silylenes would add to the silicon–carbon double bond. Reasoning that the double bond of our previously made stable silenes 2, R = 1-adamantyl or CEt_3[1] would probably be too crowded (as

$$\underset{\textbf{1}}{(Me_3Si)_3SiCOR} \xrightarrow{h\nu} \underset{\textbf{2}}{(Me_3Si)_2Si{=}C(OSiMe_3)R}$$

preliminary experiments appear to confirm), we chose to investigate the behavior of the system where R = mesityl and have observed several unusual reactions including the first reported addition of a silylene to a stable silene to yield a disilacyclopropane.[2]

Photolysis of the mesitoylpolysilane 3 (see Scheme I) (prepared in the usual manner from (tris(trimethylsilyl)silyl)lithium and mesitoyl chloride[3,4]) gave rise to the expected silene 4 as evidenced by the growth of new NMR signals, in particular a ^{13}C signal at 197.6 ppm and a ^{29}Si signal at 37.7 ppm characteristic of the sp^2-hybridized carbon and silicon atoms, respectively, of the double bond.[5,6] During extended photolysis (15 h) to isomerize

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(2) A few disilacyclopropanes have been described previously: (a) Seyferth, D.; Duncan, D. P. J. *J. Organomet. Chem.* **1976**, *111*, C21. (b) Masamune, S.; Murakami, S.; Tobita, H.; Williams, D. J. *J. Am. Chem. Soc.* **1983**, *105*, 7776. (c) Ishikawa, M.; Sugisawa, H.; Kumada, M.; Higuchi, T.; Matsui, K.; Hirotsu, K.; Iyoda, J. *Organometallics* **1983**, *2*, 174.

(3) All NMR spectra were run in C_6D_6 with H-coupled and DEPT spectra being employed as appropriate to making assignments.

(4) Properties of **3**: mp 86–88 °C; IR(KBr) 1610 cm^{-1} (C=O); UV λ_{max} (ϵ) 373 (115), 387 (156), 404 (128) nm; ^1H NMR δ 0.23 (27 H, s, Me_3Si), 2.04 (3 H, s, *p*-Me), 2.15 (6 H, s, *o*-Me), 6.57 (2 H, br s, aromatic); ^{13}C NMR δ 1.66 (Me_3Si), 19.75 (*o*-Me), 20.98 (*p*-Me), 147.68, 137.56, 128.97, 131.07 (C_{ipso}, *o*, *m*, *p* of mesityl), 247.10 (C=O); ^{29}Si NMR δ −75.3 (Si), −11.4 (Me_3Si). Anal. Calcd for $C_{19}H_{38}Si_4O$: C, 57.80; H, 9.70. Found: C, 57.61; H, 9.81.

0276-7333/85/2304-1487$01.50/0