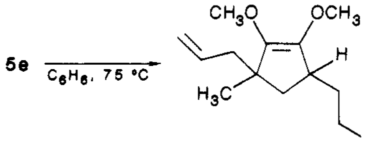
addition of Me_3SiCl and purification on silica gel to give **5a** in 52% yield. A possible intermediate in the formation of **5a**, which we did not attempt to isolate nor detect, would be **5** (R^1-R^3 = H, R^4 = R^5 = $SiMe_3$).

Whenever possible (**5c-f,h-l**) the reactions described in Scheme I proceeded in a highly stereoselective manner with the formation of complexes **5** as one major diastereomer. For example, *meso*-**5c** (eq 1) was formed diastereoselectively (>90%, as determined by ^1H NMR spectroscopy)[13] in the reaction between **2** (R^1 = CH_3, R^2 = H) and **3** (R^3 = H, R^4 = CH_3) and Me_3SiCl as the electrophilic quenching agent. Moreover, a single-crystal X-ray structure of **5j** (Figure 1) clearly revealed that this isomer, which was the only one formed, had the two large tungsten carbene fragments on the same side of cyclopentane ring. No doubt, **5j** arises by $CH_2{=}CHCH_2Br$ adding to the sterically less hindered side of **4j**.[14]

The solid-state structure of **5j** (Figure 1) contains two $(CO)_5W$ fragments bridged by a μ-1,5-bis(pentylidene) ligand.[15] To our knowledge compound **5j** is only the second (μ-bis(carbene))ditungsten complex that has been characterized by X-ray crystallography.[2k] The structural features associated with the $(CO)_5W$-carbene portions of **5j** are similar and are typical of other Fischer-type pentacarbonyl tungsten carbene complexes.[2k,l,16] For example, the W(1)-C(11) and W(2)-C(19) carbene bond distances of 2.10 (2) and 2.16 (2) Å, respectively, are similar to distances in other tungsten carbene complexes in which an alkoxy group and a carbon are directly bonded to the carbene carbon. The two tungsten carbene fragments have different orientations with respect to the cyclopentane ring; the angle between the respective mean planes through C(12), O(11), C(11), W(1), C(13) and C(20), O(12), C(19), W(2), C(18) is 83.1°.

Initial studies on the reactivity of these (μ-bis(carbene))ditungsten complexes **5** have revealed an interesting intramolecular carbene-carbene coupling reaction (eq 2).[17]



7

(2)

Thus, heating a solution of **5e** in benzene for 3 h cleanly produced the 1,2-dimethoxycyclopentene derivative **7** in 90% yield. The organometallic products formed in this reaction, as determined by an $^{13}C\{^1H\}$ NMR experiment conducted in C_6D_6, were $W(CO)_6$ and $(\eta^6\text{-}C_6D_6)W(CO)_3$.[18] The formation of **7** is significant, in that, the parent compound 1,2-dimethoxycyclopentene appears to be unknown;[20] furthermore, a new, general route to highly substituted five-membered carbacycles could be developed starting with complexes **5**.[21]

Acknowledgment. We wish to thank the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research and the NSF Chemical Instrumentation Program for funds used to purchase the diffractometer.

Registry No. **2** (R^1 = R^2 = H), 115677-70-8; **2** (R^1 = H, R^2 = CH_3), 115677-71-9; **2** (R^1 = H, R^2 = *n*-C_3H_7), 115677-72-0; **2** (R^1 = R^2 = CH_3), 115677-73-1; **3** (R^3 = R^4 = H), 83801-34-7; **3** (R^3 = H, R^4 = CH_3), 108104-17-2; **3** (R^3 = R^4 = CH_3), 115792-67-1; **3** (R^3, R^4 = $(CH_2)_3$), 111772-18-0; **3** (R^3, R^4 = $(CH_2)_4$), 111772-19-1; 3 (R^3, R^4 = $(CH_2)_6$), 111772-20-4; **5a**, 115677-58-2; **5b**, 115677-59-3; **5c**, 115677-60-6; **5d**, 115677-61-7; **5e**, 115677-62-8; **5f**, 115677-63-9; **5g**, 115677-64-0; **5h**, 115677-65-1; **5i**, 115677-66-2; **5j**, 115677-67-3; **5k**, 115677-68-4; **5l**, 115677-69-5; Me_3SiCl, 75-77-4; $CH_2{=}CHCH_2Br$, 106-95-6; $PhCH_2Br$, 100-39-0; $CH_3OSO_2CF_3$, 333-27-7.

Supplementary Material Available: Spectral and analytical data for new compounds, particulars of data collection, and tables of bond distances and angles, positional and thermal parameters, and least-squares planes results for **5j** (12 pages); a listing of observed and calculated structure factor amplitudes for **5j** (6 pages). Ordering information is given on any current masthead page.

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(13) ^1H NMR of *meso*-**5c** ($CDCl_3$): δ 4.58 (s, 6 H), 4.02 (sextet, *J* = 6.7 Hz, 2 H), 1.95 (dt, *J* = 13.9, 6.9 Hz, 1 H), 1.12 (dt, *J* = 13.8, 7.0 Hz, 1 H), 1.06 (d, *J* = 6.7 Hz, 6 H).

(14) We are currently investigating the origins of this observed diastereoselectivity and will report these findings in a more complete form at a later date.

(15) Complex **5j** crystallizes in the centric *Pbca* space group with cell constants (at 20 °C) a = 11.931 (6) Å, b = 14.404 (8) Å, c = 31.496 (9) Å, and D_{calcd} = 2.10 g cm^{-3} for Z = 8 (Mo Kα, λ = 0.710 73 Å). Data were collected on a Enraf-Nonius CAD4 diffractometer. Least-squares refinement based on 2868 observed reflections led to a final R value of 0.064 (314 parameters varied in the final cycles). All but the methyl (C(12), C(20)) and C(23) hydrogen atoms were placed in calculated positions 0.95 Å from the bonded carbon atom and allowed to ride on that atom with B fixed at 5.5 Å^2. All non-hydrogen atoms except C(2), C(7), C(18), and C(11) were refined with anisotropic thermal parameters.

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(18) $^{13}C\{^1H\}$ NMR (C_6D_6): $W(CO)_6$, δ 191.11; $(\eta^6\text{-}C_6D_6)W(CO)_3$, δ 215.73, 89.04 (t).[19]

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Chemistry of the 17-Electron Compound $[(\eta^5\text{-}C_5Me_5)Cr(CO)_3]$

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Summary: The compound $[(\eta^5\text{-}C_5Me_5)Cr(CO)_3]_2$ has a very weak metal-metal bond and dissociates extensively in solution. The resulting monomer $[(\eta^5\text{-}C_5Me_5)Cr(CO)_3]$ takes part in a variety of reactions characteristic of 17-electron compounds.

The compound $[(\eta^5\text{-}C_5H_5)Cr(CO)_3]_2$ (I) is unusual among 18-electron, dimeric metal carbonyls in that it has a very weak metal-metal bond and dissociates thermally in solution to an extent of about 10%.[1] We have utilized this fact to study the chemistry of the monomeric $[(\eta^5\text{-}C_5H_5)Cr(CO)_3]$ (II),[2] with which I is in facile equilibrium, veri-

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Table I. IR and (for Diamagnetic Compounds) ^{1}H NMR Data

compound	ν (hexane), cm^{-1}	δ (toluene-d_8)
[$Cp^*Cr(CO)_3$][a]	1999 (vs), 1849 (s, br)	
[$Cp^*Cr(CO)_2PMe_3$]	1910 (vs), 1797 (s, br)	
[$Cp^*Cr(CO)_2PMe_2Ph$]	1909 (vs), 1793 (s, br)	
[$Cp^*Cr(CO)_2P(OMe)_3$]	1919 (vs), 1841 (s, br)	
[$Cp^*Cr(CO)_2$(*t*-BuNC)]	1920 (vs), 1828 (s, br)[b]	
$Cp^*Cr(CO)_3I$	2018 (s), 1967 (vs), 1934 (s)	1.72 (s, Cp*)
$Cp^*Cr(CO)_3Br$	2032 (s), 1979 (vs), 1935 (s)	1.40 (s, Cp*)
$Cp^*Cr(CO)_3Cl$	2034 (s), 2027 (s), 1975 (vs), 1965 (vs), 1927 (s)	1.36 (s, Cp*)
$Cp^*Cr(CO)_3H$	2002 (vs), 1951 (s), 1928 (vs), 1921 (vs)	1.57 (s, 15 H, Cp*), −5.58 (s, 1 H, CrH)
$Cp^*Cr(CO)_3Me$	1998 (s), 1926 (vs), 1918 (s)	1.41 (s, 15 H, Cp*), 0.36 (s, 3 H, CrMe)
$Cp^*Cr(CO)_3CH_2Ph$	1994 (s), 1924 (vs), 1919 (sh)	1.39 (s, 15 H, Cp*), 2.43 (s, 2 H, CH_2), 6.9–7.5 (m, 5 H, Ph)
$Cp^*Cr(CO)_3(\eta^1\text{-}C_3H_5)$	1995 (s), 1929 (vs), 1916 (s)	1.36 (s, 15 H, Cp*), 1.93 (d, J = 8.7 Hz, 2 H), 4.78 (d, J = 8.7 Hz, 1 H), 5.27 (d, J = 16.6 Hz, 1 H), 6.26 (m, 1 H)
$Cp^*Cr(CO)_3SnBu_3$	1964 (vs), 1898 (s), 1880 (vs)	1.61 (s, Cp*), 0.9–1.7 (m, Bu)

[a] Cp* = η^5-C_5Me_5. [b] ν_{CN} = 2091 (w), 2064 (w) cm^{-1}.

fying that the chemistry of solutions of I/II actually reflects the reactivity of the monomer. It was also found that II takes part in a variety of atom abstraction reactions and is very substitution-labile, as anticipated for a 17-electron, metal-centered radical.[3]

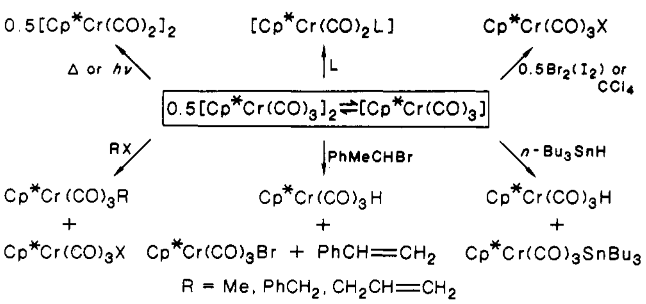
Since the presence of bulky ligands L inhibits the dimerization of monomers of the type [$(\eta^5\text{-}C_5H_5)Cr(CO)_2L$],[2] it was anticipated that the presence of alkyl substituents on the ring in a compound such as [$(\eta^5\text{-}C_5Me_5)Cr(CO)_3$]$_2$ (III) might also stabilize the monomeric [$(\eta^5\text{-}C_5Me_5)Cr(CO)_3$] (IV) with respect to dimerization. We report here the initial results of a successful investigation into the physical and chemical properties of IV.

Although III has been reported to be formed via the high-temperature carbonylation of the triple-bonded compound [$(\eta^5\text{-}C_5Me_5)Cr(CO)_2$]$_2$,[4] a much superior route involves dehydrogenation with isoprene[5] of the hydride $(\eta^5\text{-}C_5Me_5)Cr(CO)_3H$, i.e.

$$2(\eta^5\text{-}C_5Me_5)Cr(CO)_3H + \text{isoprene} \rightarrow \text{III} + \text{monoolefins} \tag{1}$$

The hydride is formed in high yield by treating $Cr(CO)_3(MeCN)_3$ with C_5Me_5H,[6] and the dimer that forms in (1) can be purified by recrystallization from 4:1 hexane/benzene at −30 °C or by sublimation at 50 °C (0.01 mmHg).[6] Compound III, which can be isolated in yields of 70% based on $Cr(CO)_6$, forms black, air-stable (for days, at least, in the solid state) crystals that have been characterized by X-ray crystallography.[7] The dimer is found

Scheme I[a]



[a] Cp* = η^5-C_5Me_5.

to be isostructural with the recently reported molybdenum analogue[8] and has a chromium–chromium bond length of 3.3107 (7) Å, significantly longer and therefore weaker than the metal–metal bonds of both I (3.281 (1) Å)[1a] and [$(\eta^5\text{-}C_5Me_5)Mo(CO)_3$]$_2$ (3.278 (14) Å).[8]

Consonant with the latter suggestion, III dissolves in solvents such as hexane, benzene, and toluene to give bright red, extremely air-sensitive solutions containing IV. Magnetic moment measurements by the Evan's method[9] show that the magnetic moment of the solute is 1.70 (±0.05) μ_B/chromium atom, very close to the spin-only value for one unpaired electron and suggesting extensive dissociation to monomer at room temperature at the concentration studied ([Cr] ≈ 0.04 M). However, the degree of dissociation is temperature- and concentration-dependent, and concentrating or cooling a toluene solution results in the magnetic moment being lowered, the ν_{CO} attributed to IV at 1994 (vs) and 1895 (vs, br) cm^{-1} decreasing in intensity[10] and new ν_{CO} at 1918, 1877, and 1848 cm^{-1} attributed to III[11] appearing. Accompanying these changes, which are completely reversible, the solution turns deep green, the color of III.

This behavior is accompanied by equally striking changes in the ^{1}H NMR spectrum of a dilute ([Cr] = 0.007 M), room-temperature toluene-d_8 solution of III/IV, which exhibits a single, broad methyl resonance at δ 31.3. On concentrating the solution such that [Cr] = 0.035 M, the methyl resonance shifts upfield to δ 24.4, consistent with rapid exchange between III and IV and the presence of

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[5] This type of reactivity has been observed for $(\eta^5\text{-}C_5H_5)Cr(CO)_3H$; see ref 2a and: Miyake, A.; Kondo, H. *Angew. Chem., Int. Ed. Engl.* **1968**, *7*, 631.

[6] Leoni, P.; Landi, A.; Pasquali, M. *J. Organomet. Chem.* **1987**, *321*, 365. The hydride was prepared essentially as in this paper and was purified by sublimation at 65–75 °C. The dimer III was prepared by stirring a hexane solution of the hydride with excess isoprene for 2 h. On standing overnight at −20 °C, dark crystals of the product precipitated and were collected by filtration.

[7] Kidd, C. E.; Jaeger, T. J.; Fortier, S.; Baird, M. C., unpublished results. Compound III crystallizes in space group $P2_1/n$ with a = 15.263 (5) Å, b = 8.970 (6) Å, c = 9.320 (2) Å, β = 95.87 (2)°, V = 1269.26 Å^3, Z = 4, D_{calcd} = 1.419 Mg m^{-3}, and μ = 8.715 cm^{-1}. Refinement resulted in R = 0.039 and R_w = 0.053. Note that the monoclinic cell was selected by using the normal convention $c < a$. The unit cell and space group of the molybdenum analogue[8] are readily obtained by using the transformation matrix

$$\begin{bmatrix} 0 & 0 & \bar{1} \\ 0 & 1 & 0 \\ 1 & 0 & 1 \end{bmatrix}$$

[8] Leoni, P.; Marchetti, F.; Pasquali, M.; Zanello, P. *J. Chem. Soc., Dalton Trans.* **1988**, 635.

[9] Evans, D. F. *J. Chem. Soc.* **1959**, 2003.

[10] The spectrum is very similar to that of II,[1d] the absorptions of IV being to lower frequency in keeping with the greater electron-donating properties of the η^5-C_5Me_5 ligand.

[11] IR spectrum of III: in Nujol mull, 1903 (vs), 1892 (vs), 1868 (s) cm^{-1}; in a KBr disk, 1906 (vs, br), 1869 (vs), 1851 (m) cm^{-1}.

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increased amounts of diamagnetic III.

On cooling the latter solution from 95 to 35 °C, the broad ($\Delta\nu_{1/2} \approx$ 180 Hz), averaged methyl resonance shifts downfield from δ ~29.0 to δ ~32; the correlation of chemical shift with temperature appears to be consistent with Curie law behavior.[12] Interestingly, however, on further cooling, the apparent Curie law behavior is lost, as the methyl resonance reverses the trend and shifts upfield, presumably because of an increasing contribution of the methyl resonance of the diamagnetic III to the averaged chemical shift. The spectrum remains invariant below -75 °C, a sharp singlet at δ 1.54 being attributable to III.

Solutions of III/IV exhibit pronounced chemical reactivity characteristic of metal-centered radicals, as shown in Scheme I. While benzene solutions seem indefinitely stable at room temperature under rigorous exclusion of air and light, conversion to $[(\eta^5\text{-}C_5Me_5)Cr(CO)_2]_2$[4b] occurs on protracted heating above 40 °C or on exposure to sunlight. As with the η^5-C_5H_5 analogue,[1d,2] IV is very substitution-labile and readily reacts with ^{13}CO to give ^{13}CO-enriched material, as shown by changes in the IR spectrum. Similarly, reactions with *t*-BuNC and smaller tertiary phosphines (PMe_3, PMe_2Ph, $P(OMe)_3$, but not the larger PPh_3) result in the formation of extremely air-sensitive, monosubstituted compounds of the type $[(\eta^5\text{-}C_5Me_5)Cr(CO)_2L]$. The substituted compounds are thermally labile and have not been successfully purified. However, they have been identified by their IR spectra (Table I), which are very similar to those of compounds of the type $[(\eta^5\text{-}C_5H_5)Cr(CO)_2L]$,[2] by their paramagnetism (one unpaired electron), by the presence of very broad resonances in the ^{1}H NMR spectra, and by the mass spectra of the PMe_3 and PMe_2Ph derivatives, which exhibit peaks at *m/z* values corresponding to the M^+, $(M - CO)^+$, and $(M - 2CO)^+$ ions. The steric restrictions to substitution reactions of IV are evident from the lack of reaction of PPh_3; both this ligand and tricyclohexylphosphine form derivatives in the η^5-C_5H_5 system.[2]

Solutions of III/IV also take part in atom abstraction reactions typical of other 17-electron compounds,[3] reacting with bromine and iodine to form the known halo compounds $(\eta^5\text{-}C_5Me_5)Cr(CO)_3X$ (X = Br, I)[13] and with carbon tetrachloride to form the chloro analogue (Scheme I). As has been reported,[13] the bromo and iodo compounds are very labile and cannot be purified satisfactorily; the chloro compound is similarly difficult to work with. However, all three compounds were readily identified on the basis of comparisons of their spectroscopic properties, as shown in Table I. Interestingly, $(\eta^5\text{-}C_5Me_5)Cr(CO)_3I$ takes part in an iodine atom exchange process with IV, as evidenced by coalescence of the methyl resonances of the two compounds. This type of exchange does not occur for the compounds $(\eta^5\text{-}C_5Me_5)Cr(CO)_3X$ (X = H, Cl, Br), although it is a common feature of the chemistry of II.[2]

Compound IV also takes part in atom abstraction processes, reacting with methyl iodide, benzyl bromide, allyl bromide, and tri-*n*-butyltin hydride, as shown in Scheme I. In all cases, the products have been difficult to separate and/or have proven to be too labile to purify, and all have been of necessity identified spectroscopically. Thus the halo products were characterized on the basis of the spectroscopic data discussed above and presented in Table I, while the methyl,[14] benzyl, and allyl compounds were characterized by their IR and NMR properties (Table I) (and by the similarities of these properties to those of analogous η^5-C_5H_5 compounds[2]) following synthesis by other routes, such as reaction of the anion $[(\eta^5\text{-}C_5Me_5)Cr(CO)_3]^{-}$[13] with the appropriate organic halide or of $(\eta^5\text{-}C_5Me_5)Cr(CO)_3I$ with alkyllithium reagents.

Interestingly, while the primary alkyl compound 2-phenethyl bromide does not react with IV, the benzylic isomer 1-phenethyl bromide does, albeit in a different fashion than anticipated. As shown in Scheme I, the products are $(\eta^5\text{-}C_5Me_5)Cr(CO)_3Br$, $(\eta^5\text{-}C_5Me_5)Cr(CO)_3H$, and styrene. Evidently bromine abstraction does occur, but the thus formed secondary alkyl radical undergoes hydrogen abstraction by a second molecule of IV.

The hydride product of the reaction of IV with *n*-Bu_3SnH was characterized by its spectroscopic properties (Table I), the oily tin-containing compound by comparison of its spectroscopic properties with those of a sample made by treating $[(\eta^5\text{-}C_5Me_5)Cr(CO)_3]^{-}$[13] with *n*-$Bu_3SnCl$.

Acknowledgment. We are indebted to the National Science and Engineering Research Council for financial support.

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Kinetics and Mechanism of the Pyrolysis of 1,1-Dimethylsilacyclopent-3-ene

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Summary: Experimental kinetic data for the pyrolysis of the title compound and the formation of it and an isomer by addition of dimethylsilylene to butadiene are reported and shown to be consistent with a nonconcerted mechanism involving vinylsilacyclopropane intermediates. The mechanism closely approximates to steady-state conditions, with no single rate-determining step.

When we used buta-1,3-diene as a trap for dimethylsilylene during our studies of the silene $\rightleftharpoons$ silylene isomerization, $HMeSi{=}CH_2 \rightleftharpoons :SiMe_2$, we noted that silylene addition to form the principal adduct 1,1-dimethylsilacyclopent-3-ene (1) was reversible.[1] Lei and Gaspar[2] subsequently investigated the vacuum flow pyrolysis of 1 at 700 °C, confirming that extrusion of dimethylsilylene was the major reaction, accompanied by some isomerization of 1 to 1,1-dimethylsilacyclopent-2-ene (2). Very reasonably, they suggested that pyrolysis of 1 and the reverse addition of dimethylsilylene to butadiene might follow nonconcerted mechanisms involving a vinylsilacyclopropane intermediate; they have obtained good evidence for such mechanisms from the product composition of the pyrolysis of 1,1,2-trimethylsilacyclopent-3-ene[2] and from the addition of dimethylsilylene to methyl-substituted butadienes.[3]

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