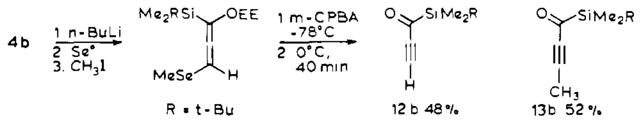
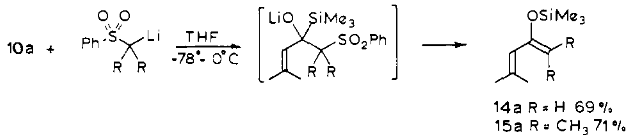
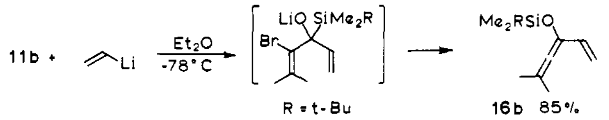
by [2,3]-sigmatropic rearrangement of an allenyl selenoxide.[3d] Introduction of the required organoseleno group was most effectively carried by the two-step procedure shown;[9] direct reaction of the allenyllithium with Ph_2Se_2 gave lower yields. Compound **13b** was prepared simlarly in 52% overall yield by methylation of **4b** followed by the selenation–oxidation sequence. Compounds **12b** and **13b**[6c] are canary-yellow liquids which, like the silyl enones discussed earlier, can be distilled and handled without special precautions,[10] although they are somewhat light sensitive and are best stored in the freezer.

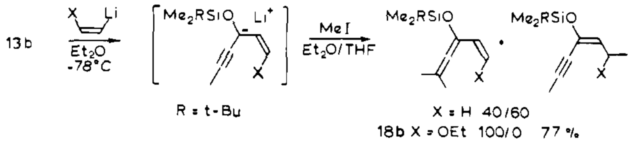


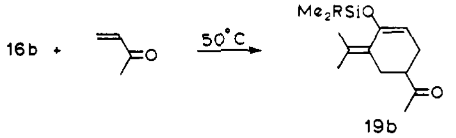
The availability of these new types of silyl ketones allows us to extend our synthetic applications of the Brook rearrangement.[1] Reaction of α-heterosubstituted lithium reagents with enone **10a** gives the siloxydienes **14a** and **15a**, the expected products of β elimination from the carbanion formed by [1,2] silicon rearrangement. The advantage of this approach to siloxydienes is its regiospecificity. The usual route involves enolization–silylation of enones, a reaction which cannot always be adequately controlled.



Unlike siloxydienes, siloxyvinylallenes, such as **16**, cannot be prepared by the enolization–silylation of carbonyl compounds.[11] In a reaction similar to the siloxydiene synthesis described above, addition of alkenyllithium reagents to α-halo silyl enones gives siloxyvinylallenes. We have also prepared compounds of this type by alkylation of pentaenynyl anions formed by addition of alkenyllithium reagents to α,β-acetylenic silyl ketones. The first method, illustrated by the preparation of **16b**, may be more general than the second, since success in the latter depends on the regioselectivity of an alkylation. Pentaenynyl anions are usually alkylated at one or both of the termini, depending on substitution.[12] This is illustrated in the two cases shown below. Reaction of vinyllithium with **13b** followed by [1,2]-silicon migration gives an anion (**17**, X = H) which is alkylated by methyl iodide to form two products. *cis*-2-Ethoxyvinyllithium,[13] on the other hand, gave only vinylallene **18b**.[6d] Furthermore, the cis stereochemistry of the lithium reagent was retained in the product.







We feel that siloxyvinylallenes may have some interesting uses as Diels–Alder dienes for the synthesis of alkylidenecyclohexanone derivatives (e.g., **19b**, the only regioisomer detected).[14] Work on the chemistry of the various polyfunctional compounds described here is continuing.

Acknowledgment. We thank the National Science Foundation and the Wisconsin Alumni Research Foundation for support of this research.

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Pentadienyl Compounds of Vanadium, Chromium, and Manganese

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We recently reported the synthesis and characterization of several methylated bis(pentadienyl)iron complexes for which the general classification of "open ferrocenes" was shown appropriate.[2] These reddish compounds mimicked their ferrocene analogues in many regards such as solubility, volatility, air stability, and their general structural natures as demonstrated by ^1H NMR spectroscopy and an X-ray diffraction study of bis(2,4-dimethylpentadienyl)iron. Several considerations have indicated to us that a relatively large class of stable pentadienyl compounds ought to exist. The first, as previously described,[2] is that the π molecular orbitals of a pentadienyl ligand[3] in a "u" conformation are analogous in symmetry, orientation, and nodal properties to those of the closed cyclopentadienyl ligand. Further, the energies of the potential donor and acceptor orbitals of the pentadienyl ligand are, respectively, higher and lower than those of the cyclopentadienyl system,[3] suggesting the possibility of even stronger metal–ligand interactions. These first observations, however, do not take into account the cyclic nature of the cyclopentadienyl ligand, which likely imparts further kinetic stabilization nor do they take into account the larger size of the pentadienyl ligands, which may cost some orbital overlap. Here, however, one can make a comparison between alkyl, allyl, and pentadienyl ligands. The fact that allyl compounds generally tend to be more stable than alkyl compounds would suggest that (at least pentahapto bound) pentadienyl compounds should be even more stable, essentially through chelation and delocalization influences.[4] Indeed, these considerations seem to be borne out by subsequent theoretical calculations and Mössbauer data.[5] In order to test the validity

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(1) NSF Predoctoral Fellow, 1980–present.

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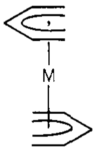
of these hypotheses, and to demonstrate the versatility of pentadienyl ligands in general, we have extended our initial "open ferrocene" studies to complexes of vanadium, chromium, and manganese.

The reactions of VCl_2 or $CrCl_2$ with 2 mol of the potassium salt of the 2,4-dimethylpentadienyl anion[6] in THF have been found to lead to the formation of deep green solutions, from which deep green crystalline products have been isolated following pentane extraction and purification by recrystallization and/or sublimation.[7] Similar products have also been isolated and mostly characterized for the 3-methylpentadienyl ligand. The infrared spectra of these compounds are quite similar to those of their corresponding iron analogues. Analytical data for these compounds fit reasonably well with that expected for the formulations $V(2,4\text{-}C_7H_{11})_2$ [$2,4\text{-}C_7H_{11} = 2,4\text{-}(CH_3)_2C_5H_5$] and $Cr(2,4\text{-}C_7H_{11})_2$,[8] which have also been confirmed by mass spectral data.[9] In the case of the d^4 chromium compound, magnetic susceptibility measurements by the Evans method[10] demonstrate the presence of two unpaired electrons, quite analogous to chromocene itself[11] and to the previously reported bis(pentadienyl)chromium.[12] Preliminary unit cell data for this compound demonstrate a volume increase of ca. 7% per molecule compared to $Fe(2,4\text{-}C_7H_{11})_2$.[13] This increase can be compared to the similar cell volume increase of 6.5% in going from $Fe(C_5H_5)_2$ to $Cr(C_5H_5)_2$[14] (during which a 5% increase in the metal–carbon bond distance takes place).[15] Thus, in the case of chromium, there seems to be a clear correspondence between the cyclopentadienyl and pentadienyl systems with regard to the effects of electron configurations on bond strengths (the "electron imbalance" relationship).[16] We therefore tentatively formulate this and other bis(pentadienyl)chromium compounds as "open sandwiches" (i.e., I, M = Cr). The situation with regard to the vanadium compound is less clear, however. Solution magnetic susceptibility measurements indicate in various solvents the presence of only one unpaired electron,[17] compared to vanadocene's three, for what is formally divalent vanadium (d^3 configuration). The source and implications of this observation are presently under investigation, including an X-ray diffraction study, in the hope of gaining a better understanding of this unusual compound.[18]



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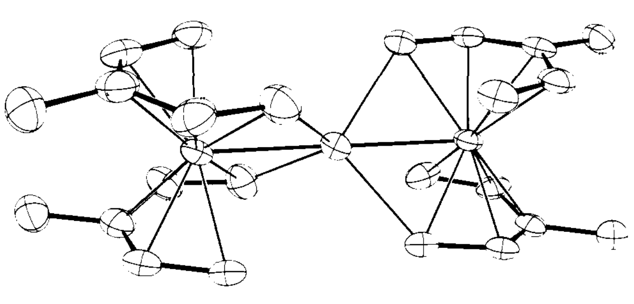

Figure 1.

In the reaction of $MnCl_2$ with 2 equiv of the 3-methylpentadienyl anion,[6,19] a course is gauche conformation which superficially parallels that of the manganocene system.[20] Here, an amber-brown product has been isolated and characterized by elemental analysis as well as infrared and mass spectroscopy. In this case, the product is only slightly hydrocarbon soluble and cannot be sublimed. The mass spectrum (on heating), however, does exhibit a peak corresponding to $Mn(C_6H_9)_2^+$, as well as a number of related fragments,[21] and a magnetic moment indicative of substantial ionic (i.e., high spin) character was observed.[22] These observations at first suggested an ionic constitution which could lead to at least oligomeric if not polymeric structures, as in the case of manganocene itself.[23] For ascertaining the exact molecular constitution, an X-ray diffraction study was undertaken.[24] The structural result, depicted in Figure 1, reveals an unexpected and unusual trimetallic complex of formulation $Mn_3(3\text{-}C_6H_9)_4$ ($3\text{-}C_6H_9 = 3\text{-}CH_3C_5H_6$). The terminal manganese atoms are coordinated by two 3-methylpentadienyl ligands in pentahapto fashion and to the central manganese atom, the latter bond distance averaging 2.516 (1) Å. The two 3-methylpentadienyl ligands bonded to a given terminal manganese atom adopt nearly the same gauche conformation as observed in $Fe(2,4\text{-}C_7H_{11})_2$.[2] The average distances between the terminal manganese atom and the pentadienyl carbon atoms [C(1)–C(5)] are 2.129 (4), 2.075 (4), 2.149 (4), 2.058 (3), and 2.157 (4) Å, respectively, where C(5) is the terminal carbon atom (or its equivalent) which bridges a terminal manganese atom with the central manganese atom. Since the Mn–C(1) to Mn–C(4) bond

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(6) (a) Yasuda, H.; Ohnuma, Y.; Yamauchi, M.; Tani, H.; Nakamura, A. *Bull. Chem. Soc. Jpn.* **1979**, *52*, 2036. (b) Bates, R. B.; Gosselink, D. W.; Kaczynski, J. A. *Tetrahedron Lett.* **1967**, 199.

(7) The reactions are carried out by dropwise addition of the anion to the metal halide at −78 °C. The mixtures are then slowly warmed to room temperature for approximately 2 h after which the solvent is removed in vacuo. In general, good yields are obtained and the major loss of product appears due to the high solubility of the compounds in pentane even at −78 °C.

(8) (a) Anal. Calcd for $C_{14}H_{22}V$: C, 69.69; H, 9.19. Found: C, 69.66; H, 9.28. (b) Calcd for $C_{12}H_{18}Cr$: C, 67.27; H, 8.47. Found: C, 66.36; H, 8.63. (c) Calcd for $C_{14}H_{22}Cr$: C, 69.39; H, 9.15. Found: C, 67.93; H, 9.12.

(9) In both the vanadium and the chromium compounds, significant parent peaks were observed as well as peaks corresponding to the free metal and free ligand, and various other reasonable molecular fragments. The 3-methyl compound of chromium also yielded reasonable mass spectral and analytical data.[8b] These compounds seem to possess even higher solubilities and volatilities than the related $Fe(2,4\text{-}C_7H_{11})_2$ compound, and therefore appear to be monomeric, in accord with previously reported molecular weight data for $Cr(C_5H_7)_2$.

(10) Evans, D. F. *J. Chem. Soc.* **1959**, 2003.

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(13) $a = b = 8.089$ (1) Å, c 20.834(5) Å, $V = 1363.2$ Å^3. That this increase is accompanied by a similar increase in Cr–C bond distance has since been confirmed by a single-crystal study.

(14) Weiss, E.; Fischer, E. O. *Z. Anorg. Allg. Chem.* **1956**, *284*, 69.

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(17) $\mu = 1.7$ (THF), 1.8 (toluene), 2.0 (methylcyclohexane) μ_B by the Evans method.[10]

(18) An X-ray diffraction study has since demonstrated an open sandwich structure for this compound.

(19) The reaction is carried out in a similar fashion to that for the vanadium and chromium compounds. However, the crude product is extracted with hot hexane.

(20) (a) Wilkinson, G.; Cotton, F. A.; Birmingham, J. M. *J. Inorg. Nucl. Chem.* **1956**, *2*, 95. (b) Fischer, E. O.; Jira, R. *Z. Naturforsch.* B **1954**, *9B*, 618.

(21) (a) Besides the $Mn(C_6H_9)_2^+$ peak, sizable Mn^+ and $C_6H_9^+$ peaks were observed. Also, peaks were observed for dimetallic and trimetallic ions up to $Mn_3(C_6H_9)_4^+$. However, such peaks were also present in some of the mass spectra of various other pentadienyl complexes and are not uncommon even for metal cyclopentadienyl compounds, either due to decomposition or gas-phase ion–molecule reactions.[21b,c] (b) Cais, M.; Lupin, M. S. *Adv. Organomet. Chem.* **1970**, *8*, 355. (c) Schumacher, E.; Taubenest, R. *Helv. Chim. Acta* **1964**, *47*, 1525.

(22) A moment of 5.7 μ_B was obtained in THF, compared to a calculated moment of 5.9 μ_B for five unpaired electrons. More careful and detailed studies are planned to ensure the absence of complications due to impurities.

(23) Bünder, W.; Weiss, E. *Z. Naturforsch. B 1978*, **33B**, 1235.

(24) (a) Single plate-like crystals of the product could be grown by slowly cooling hot, saturated solutions of the compound in hexane. A combination of oscillation and Weissenberg photographs, along with standard Syntex P$\bar{1}$ software programs, were used in the determination of the unit cell data. The space group is C_i^1–P1 (No. 2); $a = 6.937$ (1) Å, $b = 7.427$ (1) Å, $c = 23.940$ (3) Å, $\alpha = 83.54$ (1)° $\beta = 83.77$ (1)°, $\gamma = 64.15$ (1)°, $Z = 2$. Data were collected by using Mo Kα radiation ($\lambda = 0.71073$ Å) using standard θ–2θ scans. After correction for absorption, the structure was solved by Patterson, Fourier, and least-squares techniques. Anisotropic refinement led to convergence at a conventional R index[24b] of 0.050 for the 2431 unique reflection for which $I > 3\sigma(I)$. (b) $R = \sum||F_o| - |F_c||/\sum|F_o|$. The weighted R index, R_w, for the structure was 0.072, where $R_w = \sum(w(|F_o| - |F_c|)^2/\sum wF_o^2)^{1/2}$.

distances are on the average only ca. 0.025 Å longer than the corresponding distances in $Fe(2,4\text{-}C_7H_{11})_2$, it can be concluded that the "electron imbalances"[16] of the two systems are essentially equal at zero.[25] Thus, the terminal $Mn(3\text{-}C_6H_9)_2$ fragments must obtain the noble gas configuration by either forming formal single bonds to the central manganese atom or by each abstracting a single electron from the central manganese atom. The latter scheme seems more appropriate, and the valence designations for the complex would then involve Mn^+ for the terminal manganese atoms and Mn^{2+} for the central manganese atoms. In essence, the complex may then be regarded as the associated salt of Mn^{2+} and $Mn(3\text{-}C_6H_9)_2^-$. The ability of high spin Mn^{2+} to adopt such ionic configurations is, of course, well exemplified in manganocene itself. The formal positive valences of all three manganese atoms explains their mutual attractions to the pentadienyl ligands as well as the shorter metal–metal separation than that observed in the linear $Mn_3(CO)_{14}^-$ ion [2.895 (5) Å].[26] The coordination geometry of the central Mn^{2+} ion is particularly unusual in that it closely approximates an *edge*-bicapped tetrahedral geometry [the C(5)–Mn–C(5)′ angles ranging from 103.6 (3) to 118.4 (3)° with Mn–C(bridge) = 2.334 (4) Å]. An alternative description is that there are two nearly perpendicular trigonal planar sets of bonds surrounding Mn(2) for which the appropriate Mn(1 or 3)–Mn(2)–C(bridge) angles average 127.3 (1)° while the C(bridge)–Mn(2)–C(bridge) angles average 105.3 (2)°. The interaction of the apparent electrophile Mn^{2+} with both the terminal manganese atoms and the bridging C(5) atoms can be taken as evidence of both ligand and metal basicities in our systems. Such basicities have been proposed for ferrocene,[27] and the present structural result may even serve as a model for electrophilic substitution reactions of ferrocene and ferrocene-like molecules. Also of interest are the carbon–carbon bond distances, averaging 1.410 (3) Å for all but the C(4)–C(5) bonds, which averaged 1.441 (6) Å. The central C(2)–C(3)–C(4) angles average 121.1(4)°—significantly smaller than the C(1)–C(2)–C(3) or the C(3)–C(4)–C(5) angles at 125.2 (4) and 127.5 (3)°, respectively. Interestingly, the reverse order was observed in $Fe(2,4\text{-}C_7H_{11})_2$ with the C(2)–C(3)–C(4) angles averaging 125.5 (3)° while the others averaged 122.4 (2)°. In both cases, then, the methyl substituents serve to contract the C–C–C bond angles to ca. 122° from the value of ca. 126° when no methyl substituent is present. This contraction upon methylation may be responsible for the apparent stabilizing effect of methylation observed in the iron series by bringing about an increase in the metal–ligand overlap and perhaps also by allowing for increased separation between ligands as longer metal–ligand plane distances can still give rise to shorter metal–carbon distances.

Naturally of interest is the origin of this unusual complex. Obviously, at this early stage only some speculation may be offered. One possibility is that during an initial formation of $Mn(3\text{-}C_6H_9)_2$, some of the complex may either function as, or become converted to, some source of "bare manganese" complex, analogous to Wilke's fascinating "bare nickel" chemistry.[28] Other molecules of (perhaps somewhat ionic) $Mn(3\text{-}C_6H_9)_2$ may function as ligands which then coordinate sequentially to the bare manganese source. Such a process could then involve an intermediate $Mn_2(3\text{-}C_6H_9)_2$ complex, perhaps akin to the known $Ni_2(C_5H_7)_2$ compound.[29] If this were the case, the formation of these two polynuclear complexes could well proceed along related mechanistic paths. Indeed, there is good precedent for the interaction of carbanions with zero-valent metals. One pertinent example is the unusual complex $[[(LiC_6H_5)_3Ni]_2N_2{\cdot}(ether)_2]_2$ which results from the interaction of LiC_6H_5 with a "bare nickel" complex.[30] A second possible mechanism could involve an association process in solution of $Mn(3\text{-}C_6H_9)_2$ molecules, such as must take place in the crystallization or condensation of the manganocene polymer. Methylpentadienyl fragments or dimers may then be homolytically lost from these associated species, ultimately leading to the observed product. Whether or not this or a similar process takes place for other manganese complexes of varying degrees of substitution remains to be determined. However, the formation of bis(3-methylpentadienyl)manganese anions can be compared to the recent characterization of the decamethylmanganocene anion.[31]

The present report demonstrates that pentadienyl groups should indeed find broad application as useful and interesting ligands. In this regard we have, in fact, already observed high catalytic activity for some such complexes.[32] It is clear in some cases (such as iron and perhaps chromium) that a great deal of similarity exists with metallocene or metal allyl chemistry, while in other cases (such as manganese and perhaps vanadium) completely new facets of behavior seem to be emerging. We are continuing to explore this new ligand system.

Acknowledgment. R.D.E. wishes to express his gratitude for partial support of this research through grants from the University of Utah Research Committee and the donors of the Petroleum Research Fund, administered by the American Chemical Society. We would also like to thank Professors E. L. Allred and F. W. Cagle, Jr. for their interest and helpful discussions.

Supplementary Material Available: A listing of positional coordinates for $Mn_3(3\text{-}C_6H_9)_4$ (2 pages). Ordering information is given on any current masthead page.

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[25] As noted later, the 3-methylpentadienyl ligand is larger in size than the 2,4-dimethylpentadienyl ligand, and hence slightly longer M–C distances might be expected due to a slight loss of orbital overlap.

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[29] (a) Rienacker, R.; Yoshiura, H. *Angew. Chem., Int. Ed. Engl.* **1969**, *8*, 677. (b) Krüger, C. *Ibid.* **1969**, *8*, 678.

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Direct Observation of Metal-Centered Radicals in an Oxidative-Addition Reaction

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Oxidative addition of organic halides by free-radical mechanisms is a widely accepted pathway in organometallic chemistry.[1-4] A variety of methods, including racemization of chiral substrates,[1,3] radical cyclizations,[1,2] observation of CIDNP effects,[1,2] and "spin trapping",[4] have been used to ascertain the existence of free *organic* radicals in these processes. While these techniques presuppose intermediary paramagnetic transition-metal counterparts, the *direct* observation of such species in radical-chain oxidative-addition processes has not been noted.[5] We now report that if the reaction between alkyl halides and $Cp_2Zr(PPh_2Me)_2$ (where Cp is cyclopentadienyl) is continuoulsy monitored by EPR spectroscopy, *direct* evidence for the presence of these previously

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