through the cyclohexadienyl complex shown in Scheme I. We were able to isolate and characterize this species by the reaction of exactly 1.0 equiv of CF_3SO_3H with a suspension of $Ru(C_6Me_6)[C_6Me_4(CH_2)_2]$ in acetonitrile. The solvent was removed from the resulting clear yellow solution, leaving a yellow residue which was triturated with diethyl ether. The triflate salt was redissolved in acetonitrile and mixed with $NaBPh_4$. Addition of ether resulted in the formation of orange microcrystals of $[Ru(C_6Me_6)(C_6Me_5CH_2)]BPh_4$.[14] The NMR spectral features, particularly of the exocyclic methylene group, allow positive identification of this species. The ^1H NMR resonance at 3.57 ppm is nearly identical with that in $Fe(\eta^5\text{-}C_5H_5)(C_6Me_5CH_2)$ which appears at 3.60 ppm,[11] and the ^{13}C NMR spectrum exhibits a methylene carbon resonance at 86.3 ppm. $[Ru(C_6Me_6)(C_6Me_5CH_2)]^+$ can also be prepared and isolated by the deprotonation of $[Ru(C_6Me_6)_2]^{2+}$ with exactly 1.0 equiv of *tert*-butoxide.

Both of the methylene groups in $Ru(C_6Me_6)[C_6Me_4(CH_2)_2]$ can also be methylated by using $CF_3SO_3CH_3$, giving $[Ru(C_6Me_6)(C_6Me_4Et_2)](CF_3SO_3)_2$.[15] The ^1H NMR spectrum readily confirms that the two ethyl groups are adjacent to each other. That this is not required by the structure of the initial *o*-xylylene complex is a consequence of the intermediate in this reaction which is analogous to that in the protonation reaction. The complex $[Ru(C_6Me_6)(C_6Me_5CH_2)]^{1+}$ is amphoteric. Therefore, it may be capable of undergoing degenerate intramolecular proton shifts. The search for this exchange as well as the further characterization of the reactions of this new *o*-xylylene complex are currently underway.

Acknowledgment. We gratefully acknowledge the Research Corp. and the National Science Foundation (Grant CHE 8106096) for support of this work and Mr. Robert Stevens for assistance with the x-ray crystallographic study.

Registry No. $Ru(C_6Me_6)[C_6Me_4(CH_2)_2]$, 83005-40-7; $Ru(C_6Me_6)[C_6Me_5CH_2)]BPh_4$, 83005-42-9; $Ru(C_6Me_6)(C_6Me_4Et_2)](CF_3SO_3)_2$, 83005-44-1; $[Ru(C_6Me_6)_2](PF_6)_2$, 83005-45-2.

Supplementary Material Available: A listing of the structure factor amplitudes and a table of positional and thermal parameters (11 pages). Ordering information is given on any current masthead page.

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[14] Data for $[Ru(C_6Me_6)(C_6Me_5CH_2)]BPh_4$: ^1H NMR (CD_3CN) δ 7.28 (m, 8 H), 6.97 (t, 8 H), 6.84 (t, 4 H), 3.57 (s, 2 H), 2.08 (s, 3 H), 2.00 (s, 18 H), 1.83 (s, 6 H), 1.47 (s, 6 H); ^{13}C NMR (CD_3CN, undecoupled) δ 164.7 (q, BPh_4^-), 136.7 (d, BPh_4^-), 126.5 (d, BPh_4^-), 122.7 (d, BPh_4^-), 144.8 (s), 102.2 (s), 99.1 (s), 93.1 (s), 86.3 (t), 71.5 (s), 16.0 (s), 15.4 (s), 15.2 (s), 14.8 (s). Anal. Calcd for $C_{48}H_{55}BRu$: C, 77.51; H, 7.45. Found: C, 76.50; H, 7.74 (a small amount of the triflate salt from the metathesis contaminated this sample).

[15] Data for $[Ru(C_6Me_6)(C_6Me_4Et_2)](CF_3SO_3)_2$: ^1H NMR (CD_3CN) δ 2.49 (q, J = 8 Hz, 4 H), 2.16 (s, 6 H), 2.13 (s, 18 H), 2.12 (s, 6 H), 1.14 (t, J = 8 Hz, 6 H). Anal. Calcd for $C_{28}H_{40}F_6O_6RuS_2$: C, 44.73; H, 5.36. Found: C, 43.88; H, 5.37.

Metallametallocenes: Ferracobaltocene and Ferrarhodocene. New Aromatic Species[†,1]

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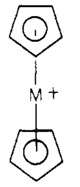
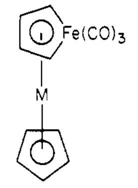
Received September 20, 1982

Summary: The novel mixed binuclear compounds (tricarbonylferracyclopentadiene)cyclopentadienylcobalt and (tricarbonylferracyclopentadiene)cyclopentadienylrhodium have been synthesized by two different pathways. X-ray crystallographic data obtained for the rhodium complex and a benzo derivative of the cobalt complex establish a sandwichlike or metallocene structure for these compounds.

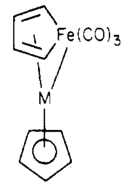
The concept of isolobal species introduced by Hoffman[2] suggests that electronicly similar species could be obtained by the exchange of a $Fe(CO)_3$ moiety for ^+CH, by virtue of the similarity of the symmetry and nodal characteristics of their frontier orbitals. Application of this isolobal interchange to the "aromatic" metallocenium species **1** leads to the formulation of the metallametallocene species **2** which might be similarly stabilized or exhibit "aromaticity".[3] We now report the syntheses of **2a,b** by two different routes and evidence pointing to the aromatic-like character of these species.



1a, M = Co
b, M = Rh

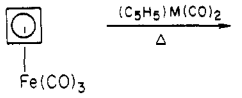


2a, M = Co
b, M = Rh



2′

Reaction of (cyclobutadiene)tricarbonyliron with excess $C_5H_5M(CO)_2$, M = Co, Rh, in hydrocarbon solvents at 110–130 °C led to metal insertion affording the air-stable complexes $(C_5H_5M)C_4H_4Fe(CO)_3$ (**2a**, M = Co; **2b**, M = Rh) in 15–30% yield (eq 1).[13] Solutions of the brick-red cobalt or yellow-orange rhodium complex in nondegassed solvents were quite stable, showing little evidence of oxidative decomposition even over extended time periods.[4] These complexes were characterized by IR, NMR, and high-resolution mass spectroscopy.[5]



2a,b (1)

M = Co, Rh

[†] Dedicated to the memory of Professor Rowland Pettit (Rolly), a treasured mentor and friend.

[*] To whom correspondence should be addressed at the Department of Chemistry, University of Texas at Austin, Austin, TX 78712.

[1] Portions of this work have been presented at the 25th Pentasectional American Chemical Society Meeting, Tulsa, Okla., April 1978, and at the 181st National American Chemical Society Meeting, Atlanta, Georgia, March, 1981.

[2] Elian, M.; Hoffmann, R. *Inorg. Chem.* **1975**, *14*, 1058. Elian, M. Chen, M. M. L.; Mingos, D. M. P.; Hoffmann, R. *Ibid.* **1976**, *15*, 1148.

[3] (a) In analogy to ferrocene the cobaltocenium and rhodocenium cations 1 with "electron imbalances" of zero or with closed-shell electron configurations can be regarded as "aromatic", see e.g.: Haaland, A. *Acc. Chem. Res.* **1979**, *12*, 415. Gard, E.; Haaland, A.; Novak, D. P.; Seip, R. *J. Organomet. Chem.* **1975**, *88*, 181 and references therein. (b) For a discussion of delocalization or "aromaticity" in metallocycles, see: Thorn, D. L.; Hoffmann, R. *Nouv. J. Chim.* **1979**, *3*, 39.

[4] The observed stability stands in strong contrast to that for solutions of the isolobal cobalta derivative, $(C_5H_5Co)C_4H_4CoC_5H_5$: Rosenblum, M.; North, B.; Wells, D.; Giering, W. P. *J. Am. Chem. Soc.* **1972**, *94*, 1239.

[5] **2a**: mp 50–52 °C; IR (Skelly B) 1965 (vs), 2035 (vs) cm^{-1}; ^1H NMR (CS_2) δ 4.88 (s, C_5H_5), 5.88 ("dd", $H_2 = H_3$), 7.39 ("dd", $H_1 = H_4$); high resolution MS, *m/e* 315.9218 (P^+) (calcd 315.9232). **2b**: mp 69.5–71.5 °C; IR (Skelly B) 1965 (vs), 2035 (vs) cm^{-1}; ^1H NMR (CS_2) δ 5.30 (s, C_5H_5), 6.2 (m, $H_2 = H_3$), 7.1 ("dd", $H_1 = H_4$); high-resolution MS, *m/e* 359.8953 (P^+) (calcd 359.8944).

0276-7333/82/2301-1718$01.25/0

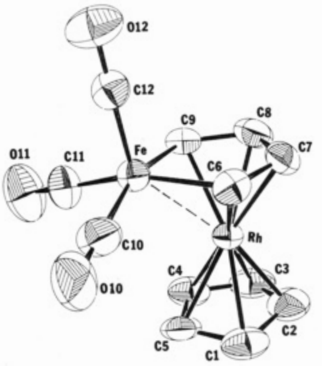


Figure 1. ORTEP plot of complex **2b**. Selected interatomic distances (Å): Fe–C_6 = 1.922 (10); Fe–C_9 = 1.930 (10); Fe–Rh = 2.557 (2); C_6–C_7 = 1.41 (2); C_7–C_8 = 1.38 (2); C_8–C_9 = 1.42 (2); average $C_{ferrole}$–Rh 2.141 (10); average C_{Cp}–Rh = 2.176 (10) Å.

The ^1H and ^{13}C NMR spectral data for the insertion products, which closely resemble that observed for the (ferrole)tricarbonyliron complex **5**,[6] unfortunately did not permit convenient structural assignment. Although molecular orbital arguments can be advanced in favor of structure **2**,[7] confirmation of the "sandwich" or metallocene structure **2** was achieved by X-ray crystalographic analysis of the cyclopentadienylrhodium insertion product **2b** (see Figure 1) as well as of a derivative of **2a**.[8] In **2b** the ferrole ring is approximately planar with the iron atom only slightly displaced (0.188 Å or 7°) outward from the plane described by the four carbon atoms of the ring. The rhodium atom is sandwiched between two approximately parallel and eclipsed five-membered rings. The C–C bond distances in the ferrole ring are remarkably similar (1.40 Å, within experimental error) with the inner C–C bond perhaps being somewhat shorter. Most importantly with regard to the possible "aromaticity" of the metallametallocene, the relatively short Fe–C bond distances (average 1.926 Å) are consistent with some multiple-bond character in these bonds,[9] as represented by structures **2a** and **2b**. A formal valence-bond representation involving a coordinate covalent M–Fe bond can be drawn as **2′**.[10]

Even deactivated (cyclobutadiene)tricarbonyliron complexes exhibit the C_5H_5M insertion reaction.[11] Reactions of (benzoyl- and acetylcyclobutadiene)tricarbonyliron with $(C_5H_5)Co(CO)_2$ yielded the 2- and 3-acylferracobaltocene derivatives in ratios of 1:2.8 and 1:3, respectively.

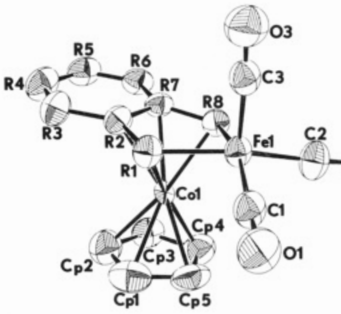
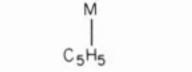
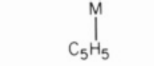


Figure 2. ORTEP plot of complex **3a**. Selected interatomic distances (Å): Fe–R_1 = 1.945 (3); Fe–R_8 = 1.941 (3); Fe–Co = 2.482 (1); R_1–R_2 = 1.429 (3); R_7–R_8 = 1.416 (4); R_2–R_7 = 1.438 (4); R_2–R_3 = 1.435 (4); R_3–R_4 = 1.352 (4); R_4–R_5 = 1.415 (6); R_5–R_6 = 1.352 (5); R_6–R_7 = 1.443 (4) Å; bond between Fe and Co missing.

Insertion into the tricarbonyliron complex of benzocyclobutadiene was also facile, giving the *sym*- and *unsym*-benzoferrametallocenes **3** and **4**. The ^1H and ^{13}C NMR spectra and their comparison with those for the analogous (benzoferrole)tricarbonyliron complexes are consistent with the assigned structure. X-ray crystallographic analysis of crystals of **3a** has confirmed the metallocenyl or sandwichlike structure (Figure 2).[12]

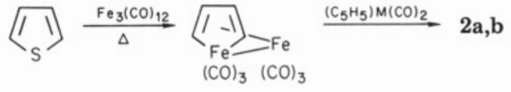


3a, M = Co
b, M = Rh



4a, M = Co
b, M = Rh

A more convenient and less expensive route[13] to the ferrametallocenes involves the two-step sequence in eq 2.



5

The desulfurization of thiophene was realized by using the procedure of Weiss[14] in yields of only 15–20%. The ligand exchange step, on the other hand, proceeded quite well. For example, in the cobalt case, the yield of the ferrametallocene was 88%, on the basis of recovered starting

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[6] (a) Our ^1H and ^{13}C NMR data for **5** agree well with the published values, although we believe that the published proton assignments should be reversed: ^1H NMR: Dettlaf, G.; Weiss, E. *J. Organomet. Chem.* **1976**, *108*, 213. ^{13}C NMR: Grevels, F. W.; Schulz, D.; von Gustorf, E. K.; Bunberg, D. St. P. *Ibid.* **1975**, *91*, 341. Todd, L. J.; Hickey, J. P.; Wilkinson, J. R.; Huffman, J. C.; Folting, K. *Ibid.* **1976**, *112*, 167. (b) The ^1H NMR data for **2a** are also similar to that reported for the related dicobalt derivative[4] if the reported assignments are reversed.

[7] Arguments involve PMO extrapolation from the approach used in Hoffmann's MO structural study of ferrole **5**. Thorn, D. L.; Hoffmann, R. *Inorg. Chem.* **1977**, *17*, 126. Yamazaki has reached a similar conclusion.[13] Simple inductive arguments based upon a valence bond representation (viz. **2′**) would appear to also favor structure **2** over one in which the two metal moities have exchanged positions.

[8] (a) Single-crystal X-ray data for **2b** were collected at room temperature (Syntex P_3 automated diffractometer and molybdenum radiation): a = 14.453 (11), b = 12.060 (6), c = 14.016 (6) Å; $\alpha = \beta = \gamma = 90°$, orthorhombic P_{bca}, Z = 8, d_{calcd} = 1.956 g cm^{-3}; R = 0.057 (2491 observed reflections). (b) X-ray crystallographic analysis has confirmed the metallocene structure of 2,5-diphenyl-3,4-dicarbomethoxyferracobaltocene.[13]

[9] (a) Although it is difficult to find a reference value for a Fe–C(sp^2) single bond length, a value of 2.03 Å appears to be reasonable on the basis of the model compounds: e.g., Mills, O. S.; Redhouse, A. D. *J. Chem. Soc. A* **1968**, 1282 and references therein. (b) Hoffmann has used the near equality of the C–C bond lengths in metallocycles as a criterion for electronic delocalization or "aromaticity".[3b]

[10] The variable-temperature ^1H NMR spectra of the ferrametallocenes from 80 to 160 °C remained invariant (no evidence for line broadening), strongly suggesting the absence of any appreciable metal–metal interchange or libration about the C_4H_4 moiety, in contrast to that observed in related binuclear metallocycles in which both metals were identical: cf. ref 4. Lee, W.-S.; Brintzinger, H. H. *J. Organomet. Chem.* **1977**, *127*, 93 and references therein.

[11] Metal insertion into other cyclobutadiene complexes by thermal or photochemical means is not without precedent. Thermal: (a) Davis, R. E.; Barnett, B. L.; Amiet, R. G.; Merk, W.; McKennis, J. S.; Pettit, R. *J. Am. Chem. Soc.* **1974**, *96*, 7108. (b) Bruce R.; Moseley, K.; Maitlis, P. M. *Can. J. Chem.* **1967**, *45*, 2011. Photochemical: (c) Ref 4. (d) Victor, R.; Ben-Shoshan, R. *J. Chem. Soc., Chem. Commun.* **1974**, 93. In contrast to its benzo derivative, the parent (cyclobutadiene)tricarbonyliron resists insertion by "$Fe(CO)_3$".

[12] Single-crystal X-ray data for **3a** were collected at room temperature: a = 13.772 (4), b = 6.449 (3), c = 17.004 (6) Å; β = 110.52(2)°, monoclinic $P2_1/m$, Z = 4, d_{calcd} = 1.718 g cm^{-1}; R = 0.048 (4652 observed reflections).

[13] Another route to ferracobaltocenes discovered independently by Yamazaki's group involves the reaction of $Fe_2(CO)_9$ with cobaltacyclopentadiene complexes prepared from acetylenes. Although the parent complex **2a** can be made in this manner (albeit in very low yield), this method clearly is most suitable for the preparation of tetra- and disubstituted derivatives: Yamazaki, H.; Yasufuku, K.; Wakatsuki, Y. 27th Symposium on Organometallic Chemistry in Japan, Abstract A118, Tokyo, October 1980. We thank Dr. Yamazaki for bringing his interesting work to our attention.

[14] Dettlaf, G.; Weiss, E. *J. Organomet. Chem.* **1976**, *108*, 213.

material, and 65%, with no recovery. Application of this ligand exchange to the *sym*- and *unsym*-(benzoferrole)tricarbonyliron complexes yielded the analogous benzoferrametallocenes **3** and **4**.

The isolobal analogy of the ferrametallocenes to the metallocenium ions **1**, as well as to Hogeveen's pyramidal dication,[15] suggested that they might possess interesting electrochemistry and exhibit relatively low reduction potentials. Indeed, both **2a** and **2b** readily add one-electron reversibly ($E_{1/2}{}^{I}$ = −1.39 and −1.76 eV, respectively),[16] although not as readily as for the isolobal complex (ferrole)tricarbonyliron **5** ($E_{1/2}{}^{I}$ = −1.30 eV).[17] .

At the same time, the complexes **2a** and **2b**, similar to other metallocenes, are sufficiently electronically rich to be conveniently acylated under conventional Friedel–Crafts conditions at room temperature. Acetylation and benzoylation using the corresponding acid chloride–aluminum chloride complex in either carbon disulfide or dichloromethane occurred in 30–55% yield.[18] Consideration of ^{1}H and ^{13}C NMR chemical shift data, which often correlates with relative rates of Friedel–Crafts acylation,[19] suggested preferential electrophilic attack at the C_5-cyclopentadienyl ring. Contrary to this expectation, however, acylation occurred exclusively at the carbon adjacent to the iron in the ferracyclopentadienyl ring, i.e., presumbaly at the least electronically rich carbon atom, yielding the 2-acetyl and 2-benzoyl derivatives. In the acetylation reactions, where diacylation as well as monoacylation was observed, even the attack by the second acyl group occurred exclusively at the unsubstituted carbon adjacent to the iron, to yield the 2,5-diacetyl derivative[20] (~15% yield). Interestingly, preliminary data also suggest that the second acylation step is competitive with the first. This and additional chemistry, as well as our efforts to prepare additional mixed metallametallocenes, will be the subject of forthcoming reports.

Registry No. **2a**, 83416-31-3; **2b**, 83416-32-4; **3a**, 83416-33-5; **3b**, 83416-34-6; **4a**, 83435-71-6; **4b**, 83435-72-7; $C_5H_5Co(CO)_2$, 12078-25-0; $C_5H_5Rh(CO)_2$, 12192-97-1; $Fe_3(CO)_{12}$, 17685-52-8; $Fe(CO)_3C_4H_4Fe(CO)_3$, 50277-83-3; (benzoylcyclobutadiene)tricarbonyliron, 67612-84-4; (acetylcyclobutadiene)tricarbonyliron, 52445-09-7; 2-benzoylferracobaltocene, 83435-73-8; 3-benzoylferrocobaltocene, 83416-35-7; 2-acetylferracobaltocene, 83416-38-0; 3-acetylferracobaltocene, 83416-36-8; (benzocyclobutadiene)tricarbonyliron, 12093-06-0; thiophene, 110-02-1; (cyclobutadiene)tricarbonyliron, 12078-17-0; 2,5-diacetylferracobaltocene, 83416-37-9.

Supplementary Material Available: Tables of crystal data, thermal parameters, positional parameters, and bond angles and distances for $C_{16}H_{11}O_3CoFe$ and $C_{16}H_{11}O_3RhFe$ (9 pages). Ordering information is given on any current masthead page.

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[15] Hogeveen, H.; Kwant, P. W. *J. Am. Chem. Soc.* **1974**, *96*, 2208; *Acc. Chem. Res.* **1975**, *8*, 413.

[16] Reference electrode: Ag/AgCl, NaCl saturated aqueous electrode: Tulyathan, B.; Rieke, R.; McKennis, J. S., unpublished results, manuscript in preparation.

[17] Zotti, G.; Rieke, R.; McKennis, J. S. *J. Organomet. Chem.* **1982**, *228*, 281.

[18] 2-Acetylferracobaltocene: mp 82–84 °C; IR (CS_2) 1673 (w), 1960 (vs), 1970 (vs, sh), 2035 (vs) cm^{-1}; ^{1}H NMR (CS_2) δ 2.16 (3, s, CH_3), 5.04 (5, s, Cp), 5.70 (1, "t", H_3), 5.84 (1, dd, H_2), 7.38 (1, dd, H_4); high-resolution MS, *m/e* 357.9541 (P^+) (calcd 357.9334). 2-Benzoylferracobaltocene: mp 130–131 °C; IR (CS_2) 1645 (w), 1960 (vs), 1972 (vs), 2035 (vs) cm^{-1}; ^{1}H NMR (CS_2) δ 5.14 (5, s, Cp), 5.75 (1, "t", H_3), 5.95 (1, dd, H_2), 7.24 and 7.67 (5, m, C_6H_5), 7.45 (1, dd, H_4); high-resolution MS, *m/e* 419.9481 (P^+) (calcd 419.9495).

[19] See ref 4 and references therein.

[20] 2,5-Diacetylferracobaltocene: mp 114.5–116 °C; IR (CS_2) 1675 (w), 1965 (vs), 1980 (vs), 2040 (vs)cm^{-1}; ^{1}H NMR (CS_2) δ 2.18 (6, s, CH_3), 5.18 (5, s, Cp), 5.68 (2, s, H_2H_3); high-resolution MS, *m/e* 399.9507 (P^+) (calcd 399.9444).

Two-Coordinate Phosphorus Compounds with Bis(trimethylsilyl)methyl Ligands: Phosphaalkene and Phosphide Anion[†]

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Richard A. Kemp,[1a] **David W. H. Rankin,**[1b] **and**
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Received September 24, 1982

Summary: The reaction of $[(Me_3Si)_2CH]_2PCl$ with Na in *n*-hexane at 65 °C results in the formation of $[(Me_3Si)_2CH]_2PH$ (**3**) and $(Me_3Si)_2C{=}PC(H)(SiMe_3)_2$ (**4**), in addition to the phosphinyl radical $[\{(Me_3Si)_2CH\}_2P]\cdot$ (**1**) and the corresponding dimer $[(Me_3Si)_2CH]_4P_2$ (**2**). Compounds **3** and **4** have been synthesized by independent routes. The reaction of a mixture of **1** and **2** with Na in *n*-hexane produces **3** and the phosphide anion $[\{(Me_3Si)_2CH\}_2P]^-$ (**5**).

Lappert, Goldwhite, and co-workers[2] have established that stabilized phosphinyl radicals are produced when chlorophosphines bearing bulky groups are photolyzed in the presence of an electron-rich olefin. The $[\{(Me_3Si)_2CH\}_2P]\cdot$ radical, 1, which serves as a novel ligand,[3] can also be prepared by the action of Na on $[(Me_3Si)_2CH]_2PCl$ in *n*-hexane.[4,5] In the course of monitoring this reaction by $^{31}P\{^1H\}$ NMR spectroscopy, it is apparent that two further products arise in addition to the phosphinyl radical, 1, and the corresponding dimer $[(Me_3Si)_2CH]_4P_2$ (**2**).

A singlet at −72.2 ppm[6] in the 36.43 MHz $^{31}P\{^1H\}$ spectrum which becomes a doublet in a coupled spectrum (J_{PH} = 195.3 Hz) is due to the secondary phosphine, $[(Me_3Si)_2CH]_2PH$ (**3**). The identity of **3** was established via two independent routes: (i) the $LiAlH_4$ reduction of $[(Me_3Si)_2CH]_2PCl$ and (ii) reaction of the phosphinyl radical, 1, with C_6H_5SH. The yields of **3** (bp 65–75 °C at 0.04 torr) were 82% and ~100% for routes i and ii, respectively. High-resolution mass spectrum (HRMS), for **3**: calcd 350.1865; found 350.1874.

The second product exhibits a 36.43-MHz $^{31}P\{^1H\}$ chemical shift of +404 ppm,[6] thus suggesting that it is a two-coordinate phosphorus compound.[7] Identification of this material as the new phosphaalkene $(Me_3Si)_2C{=}PC(H)(SiMe_3)_2$ (**4**) is based on its independent synthesis by the following two-step procedure: (i) dehydrochlorination of $(Me_3Si)_2CHPCl_2$ with 1,4-diazabicyclo[2.2.2]octane

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[†] Dedicated to the memory of Rowland Pettit, whose research contributed so much to the fields of organic and organometallic chemistry.

[1] (a) University of Texas at Austin. (b) University of Edinburgh.

[2] (a) Gynane, M. J. S.; Hudson, A.; Lappert, M. F.; Power, P. P.; Goldwhite, H. *J. Chem. Soc., Chem. Commun.* **1976**, 623–624. (b) Gynane, M. J. S.; Hudson, A.; Lappert, M. F.; Power, P. P.; Goldwhite, H. *J. Chem. Soc., Dalton Trans.* **1980**, 2428–2433. (c) Cetinkaya, B.; Hudson, A.; Lappert, M. F.; Goldwhite, H. *J. Chem. Soc., Chem. Commun.* **1982**, 609–610.

[3] Cowley, A. H.; Kemp, R. A.; Wilburn, J. C. *J. Am. Chem. Soc.* **1982**, *104*, 331–332.

[4] Cowley, A. H.; Kemp, R. A. *Inorg. Chem.*, in press.

[5] This procedure was also discovered by: Power, P. P. Ph.D. Thesis, University of Sussex, 1977.

[6] Positive ^{31}P chemical shifts are in ppm downfield from external 85% H_3PO_4 and vice versa.

[7] For a summary of ^{31}P NMR data on phosphaalkenes, see: Appel, R.; Knoll, F.; Ruppert, I. *Angew. Chem., Int. Ed. Engl.* **1981**, *20*, 731–744 and references therein.

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