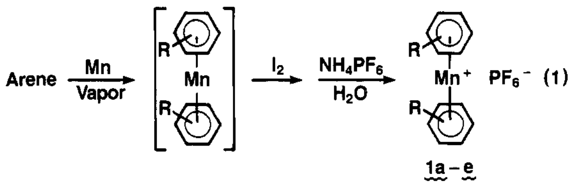
workup and ion exchange.[5] Similar reactions with *o*-, *m*-, and *p*-xylene and mesitylene also yielded the corresponding (η^6-arene)$_2$Mn$^+$PF$_6^-$ complexes (**1b**–**e**). The bis(arene)manganese cations may be isolated as their I$^-$ salts, but these are considerably less stable than the hexafluorophosphate salts. We have been unable to isolate a stable (η^6-C$_6$H$_6$)$_2$Mn$^+$ complex. The (η^6-arene)$_2$Mn$^+$PF$_6^-$ complexes are soluble in polar organic solvents and are stable in these solvents in the absence of air and light. The crystalline solids may be handled in air for short periods of time. Upon decomposition, the (η^6-arene)$_2$Mn$^+$PF$_6^-$ complexes release the free arene ligand.

The new complexes **1a**–**e** have been thoroughly characterized; ^{1}H and ^{13}C NMR and FAB MS data clearly support their formulation as cationic bis(arene) sandwich compounds.[6] To the best of our knowledge, the only other documented (η^6-arene)$_2$Mn$^+$ complex is (η^6-C$_6$H$_6$)Mn(η^6-C$_6$(CH$_3$)$_6$)$^+$PF$_6^-$ which was isolated in 2% yield from the cyclotrimerization of 2-butyne by (C$_6$H$_5$)$_2$Mn.[7] The properties of **1a**–**e** are in good agreement with those reported for (η^6-C$_6$H$_6$)Mn(η^6-C$_6$(CH$_3$)$_6$)$^+$PF$_6^-$.[7]

In the absence of I$_2$, we are unable to isolate any Mn-containing organometallic species from the condensation of Mn atoms and arenes. Dark gray to black matrices are observed which yield finely divided Mn metal on warmup; similar results have been reported by other laboratories.[8] This may be taken as good evidence that incorporation of I$_2$ into the reaction matrix traps an unstable intermediate. We thus envision the formation of **1a**–**e** proceeding via an unstable 19-electron sandwich complex which is oxidized by I$_2$ in the reaction matrix to yield the observed 18-electron (η^6-arene)$_2$Mn$^+$ complex (eq 1). Precedent for such an oxidation exists in the well-known reaction of (η^6-arene)$_2$Cr complexes with I$_2$ to form the 17-electron (η^6-arene)$_2$Cr$^+$ complexes.[9] The presence of I$_2$ does create a potential problem in that it may compete with arene for Mn atoms by forming MnI$_2$; reactor design and control over reactant ratios should minimize this problem.



Arene = toluene (1a), *o*-xylene (1b), *m*-xylene (1c), *p*-xylene (1d), mesitylene (1e)

In conclusion, we have successfully demonstrated the use of in situ oxidations in metal vapor synthesis. The preparation of (η^6-arene)$_2$Mn$^+$ complexes from the reaction of Mn atoms, arenes, and iodine lends support to the postulate that the low yields typically observed in the reactions of Mn atoms with organic ligands[2,3] are related to the instability of the 19-electron Mn(0) intermediates formed. Although the overall yields of (η^6-arene)$_2$Mn$^+$ complexes are disppointingly low (1–2%, based on I$_2$ or Mn), significant amounts may be obtained in a single run. Thus, a series of homoleptic (η^6-arene)$_2$Mn$^+$ complexes are, for the first time, available in quantities that will allow elucidation of their chemcal and physical properties.

Acknowledgment is made to the 3M Company by W.E.B. for its generous research support.

Registry No. **1a**, 116301-18-9; **1b**, 116301-20-3; **1c**, 116301-22-5; **1d**, 116301-24-7; **1e**, 116301-26-9; Mn, 7439-96-5.

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(5) **Synthesis of 1a.** All manipulations were performed under inert-atmosphere conditions. Manganese metal (3.72 g, 67.7 mmol), iodine (3.45 g, 27.2 mmol), and toluene (180 mL, 1.69 mol) were cocondensed at −196 °C over a 2-h period. The reactor was warmed to room temperature, vented with nitrogen, and the reaction mixture was syphoned into a Schlenk flask. The reactor walls were washed with THF; the reaction mixture and THF washes were filtered through Celite and evaporated to dryness under vacuum. The residue was dissolved in 25 mL of acetone and filtered into 30 mL of water containing 0.25 g of ammonium hexafluorophosphate. A pink precipitate formed and was collected by filtration. The precipitate was dissolved in 15 mL of THF, and the solution was cooled to −15 °C to afford (η^6-C$_6$H$_5$CH$_3$)$_2$Mn$^+$PF$_6^-$ (0.11 g, 0.286 mmol, 1.0% based on iodine) as orange-pink crystals.

(6) **Characterization of 1a**: ^{1}H NMR (90 MHz, acetone-*d*$_6$, δ) 5.74 (br s, C$_6$*H*$_5$CH$_3$), 2.39 (s, C$_6$H$_5$C*H*$_3$); ^{13}C{^{1}H} NMR (22.5 MHz, acetone-*d*$_6$, ppm) 98.1, 84.2, 83.2, and 82.3 (s, 1:2:2:1, *C*$_6$H$_5$CH$_3$), 19.4 (s, C$_6$H$_5$*C*H$_3$); FAB MS, *m/z* (relative intensity) 240 ([M + H]$^+$, 17), 239 ([M]$^+$, 100), 147 ([M − C$_6$H$_5$CH$_3$]$^+$, 15). Anal. Calcd for C$_{14}$H$_{16}$F$_6$MnP: C, 43.77; H, 4.20. Found: C, 43.60; H, 3.87. **1b**: ^{1}H NMR (90 MHz, acetone-*d*$_6$, δ) 5.68 (d, *o*-C$_6$*H*$_4$(CH$_3$)$_2$), 2.42 (s, *o*-C$_6$H$_4$(C*H*$_3$)$_2$); ^{13}C{^{1}H} NMR (22.5 MHz, acetone-*d*$_6$, ppm) 97.9, 85.7, and 83.9 (s, 2:2:2, *o*-*C*$_6$H$_4$(CH$_3$)$_2$), 18.1 (s, *o*-C$_6$H$_4$(*C*H$_3$)$_2$); FAB MS, *m/z* (relative intensity) 267 ([M]$^+$, 100%). **1c**: ^{1}H NMR (90 MHz, acetone-*d*$_6$, δ) 5.63 (s, *m*-C$_6$*H*$_4$(CH$_3$)$_2$), 2.42 (s, *m*-C$_6$H$_4$(C*H*$_3$)$_2$); ^{13}C{^{1}H} (22.5 MHz, acetone-*d*$_6$, ppm) 98.9, 86.1, and 84.1 (s, 2:1:3, *m*-*C*$_6$H$_4$(CH$_3$)$_2$), 19.8 (s, *m*-C$_6$H$_4$(*C*H$_3$)$_2$); FAB MS, *m/z* (relative intensity) 268 ([M + H]$^+$, 25), 267 ([M]$^+$, 100), 161 ([M − C$_8$H$_{10}$]$^+$, 23). **1d**: ^{1}H NMR (90 MHz, acetone-*d*$_6$, δ) 5.62 (s, *p*-C$_6$*H*$_4$(CH$_3$)$_2$), 2.42 (s, *p*-C$_6$H$_4$(C*H*$_3$)$_2$); ^{13}C{^{1}H} NMR (22.5 MHz, acetone-*d*$_6$, ppm) 97.9 and 85.1 (s, 2:4, *p*-*C*$_6$H$_4$(CH$_3$)$_2$), 19.3 (s, *p*-C$_6$H$_4$(*C*H$_3$)$_2$); FAB MS, *m/z* (relative intensity) 267 ([M]$^+$, 100). **1e**: ^{1}H NMR (90 MHz, acetone-*d*$_6$, δ) 5.42 (s, C$_6$*H*$_3$(CH$_3$)$_3$), 2.47 (s, C$_6$H$_3$(C*H*$_3$)$_3$); ^{13}C{^{1}H} NMR (22.5 MHz, acetone-*d*$_6$, ppm) 99.1 and 85.3 (s, 3:3, *C*$_6$H$_3$(CH$_3$)$_3$), 19.5 (s, C$_6$H$_3$(*C*H$_3$)$_3$); FAB MS, *m/z* (relative intensity) 296 ([M + H]$^+$, 100), 178 ([M − C$_9$H$_{10}$]$^+$, 29), 175 ([M − C$_9$H$_{12}$]$^+$, 33).

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Reactivity of Rh(PMe$_3$)$_4$Cl with Lithium Derivatives of Phosphorus-Substituted Diazomethanes. First Evidence for a Transient Nitrogen–Transition-Metal Nitrile Imine. X-ray Structure of $(\mathrm{PMe_3})_2\overline{\mathrm{RhNBuNCHP}}(\mathrm{N}\text{-}i\text{-}\mathrm{Pr_2})_2$

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Summary: Reaction at the diazo carbon atom occurred when the lithium derivative of [bis(diisopropylamino)thioxophosphoranyl]diazomethane and Rh(PMe$_3$)$_4$Cl were allowed to react. The 16-electron Rh(I)–C bonded diazo complex $(\mathrm{PMe_3})_2\overline{\mathrm{RhC(N_2)P(N}\text{-}i\text{-}\mathrm{Pr_2)_2(S)}}$ (**1**) containing a rhodium–sulfur bond was obtained. In contrast, when the lithium derivative of [bis(diisopropylamino)phosphino]diazomethane was used, the reaction took place at the terminal diazo nitrogen atom. The formation of $(\mathrm{PMe_3})_2\overline{\mathrm{RhN}\text{-}n\text{-}\mathrm{BuNCHP}}(\mathrm{N}\text{-}i\text{-}\mathrm{Pr_2})_2$ (**2**) is explained by the transient formation of a nitrogen–rhodium nitrile imine.

We have recently found that the lithium derivative of (trimethylsilyl)diazomethane reacts with Rh(PMe$_3$)$_4$Cl to

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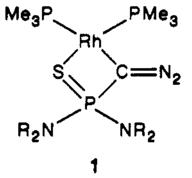
give stable Rh(I)–C and Rh(III)–C bonded diazo complexes.[1] These derivatives are potential precursors for rhodium carbene or carbyne complexes, the chemistry of which is still underdeveloped due in part to the lack of synthetic methods.[2] In order to evaluate the influence of the diazo substituent on the course of the reaction, we have extended this study to thioxophosphorane- and phosphine-substituted diazo anions $[C(N_2)[(S{=}){P}(N\text{-}i\text{-}Pr_2)_2]^-$ and $[C(N_2)[P(N\text{-}i\text{-}Pr_2)_2]^-$. The importance of the diazo substituents has been pointed out previously since the only isolable N-bonded diazomethane complexes possess electron-withdrawing groups or groups able to stabilize negative charge by resonance.[3]

We report the synthesis of a new Rh(I)–C bonded diazo complex, 1, and of a new rhodium(I) metallacycle: the 5-rhoda-4-phospha-Δ^2-pyrazoline complex 2, the formation of which suggests the existence of a transient N–Rh nitrile imine intermediate.

To a suspension of $Rh(PMe_3)_4Cl$[4] (580 mg, 1.3 mmol) in ether (10 mL) at −70 °C was added under argon a stoichiometric amount of $LiC(N_2)[(S{=})P(N\text{-}i\text{-}Pr_2)_2]$.[5] The solution was stirred for 1 h at −70 °C. Then the temperature was raised slowly, and the solution was stirred again at room temperature for 1 h. Elimination of ether and extraction with pentane (20 mL) gave a red solution from which red crystals of 1 (85% yield) precipitated at −30 °C.[6] The IR spectrum (Nujol mull) showed a sharp absorption at 1990 cm^{-1}, characteristic of the C-coordinated diazo ligand (in free $HC(N_2)[(S{=})P(N\text{-}i\text{-}Pr_2)_2]$, $\bar{\nu}(CN_2)$ is found at 2090 cm^{-1}). $^{31}P\{^1H\}$ NMR[7] presented an ABMX pattern, indicating two inequivalent PMe_3 (P2 and P3) bonded to rhodium and the $S{=}P(N\text{-}i\text{-}Pr_2)_2$ entity not directly bonded to the metal [+72 (P1), +3.5 (P2), +7.5 ppm (P3); $^2J_{P1Rh}$ = 24 Hz, $^1J_{P2Rh}$ = 183 Hz, $^1J_{P3Rh}$ = 158 Hz, $^3J_{P1P3}$ = $^3J_{P1P2}$ = 7 Hz, $^2J_{P2P3}$ = 48 Hz]. ^1H NMR spectrum [δ 1.29 (d, J_{HP} = 7.8 Hz, 9 H, PMe), 1.41 (d, J_{HP} = 7.5 Hz, 9 H, PMe), 1.49 (d, J_{HH} = 7 Hz, 12 H, CMe), 1.58 (d, J_{HH} = 7 Hz, 12 H, CMe), 3.81 (d sept, J_{HH} = 7 Hz, J_{HP} = 18 Hz, 4 H, CH] confirmed the structure of 1 as a 16-electron rhodium(I) complex containing a Rh–S bond.[8]



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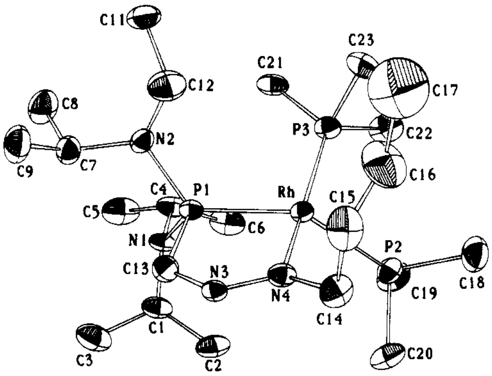

Figure 1. ORTEP drawing of the $(PMe_3)_2\overline{RhNBuNCHP}(N\text{-}i\text{-}Pr_2)_2$ molecule and atom-labeling scheme. Hydrogen atoms are omitted for clarity. Main distances (Å): Rh–P1, 2.261 (1); Rh–P2, 2.296 (1); Rh–P3, 2.239 (3); Rh–N4, 2.075 (4); N4–N3, 1.330 (6); N3–C13, 1.293 (6); P1–C13, 1.807 (5); N4–C14, 1.460 (7). Main angles (deg): P1–Rh–P2, 144.2 (1); P1–Rh–P3, 103.1 (1); P2–Rh–P3, 96.4 (1); P1–Rh–N4, 80.3 (1); P2–Rh–N4, 92.3 (1); P3–Rh–N4, 158.6 (2); Rh–P1–C13, 99.9 (2); P1–C13–N3, 117.8 (4); N4–N3–C13, 117.4 (4); Rh–N4–N3, 123.3 (3); Rh–N4–C14, 122.7 (4); N3–N4–C14, 110.3 (4).

Extension of this reaction to the closely related diazo compound $LiC(N_2)P(N\text{-}i\text{-}Pr_2)_2$ (with a reactive phosphorus lone pair) gave the 5-bis(trimethylphosphine)rhoda-4-bis(diisopropylamino)phospha-1-*n*-butyl-Δ^2-pyrazoline complex $[(PMe_3)_2\overline{RhNBuNCHP}(N\text{-}i\text{-}Pr_2)_2$ (2)] in 20% yield.[6] The use of an excess of *n*-BuLi (molar ratio of *n*-BuLi/$HC(N_2)P(N\text{-}i\text{-}Pr_2)_2$ = 2/1) increased this yield to 90%.

The IR spectrum of 2 (KBr) showed a medium absorption at 1660 cm^{-1}, corresponding to a >C=N– moiety. This is in the range of Ir–N bonded diazo compounds[9] and excluded the presence of a C-bonded diazo species. The $^{31}P\{^1H\}$ NMR spectrum (in benzene) presented an ABMX pattern consistent with two inequivalent PMe_3 ligands (P2 and P3) and a $P(N\text{-}i\text{-}Pr_2)_2$ group (P1) directly bonded to the metal center and trans to (P2) [−20 (P2), −10 (P3), +157 ppm (P1); $^1J_{P1Rh}$ = 199 Hz, $^1J_{P2Rh}$ = 156 Hz, $^1J_{P3Rh}$ = 147 Hz, $^2J_{P1P2}$ = 383 Hz, $^2J_{P1P3}$ = 39 Hz, $^2J_{P2P3}$ = 43 Hz]. The ^1H NMR spectrum (C_6D_6) consisted of a complex pattern of multiplets. Phosphorus decoupling and selective irradiation indicated two different PMe_3 [δ 1.27 and 1.30 (d, J_{HP} = 7 Hz, 9 H)], two isopropylamino groups [δ 1.42 and 1.44 (d, J_{HH} = 7 Hz, 12 H), 3.9 (d sept, J_{HH} = 7 Hz, J_{PH} = 11 Hz, 4 H)], a butyl group [δ 1.5 (m, 3 H), 1.9 (m, 2 H), 4.1 (m, 2 H)], and an imine proton [δ 6.0 (dd, J_{HP1} = 23.5 Hz, J_{HP3} = 3.5 Hz, 1 H)]. The crystal structure[10] of 2 is shown in Figure 1. It consists of monomeric molecules. The rhodium is in a distorted square-planar environment, bonded to two cis PMe_3 ligands, $P(N\text{-}i\text{-}Pr_2)_2$, and the N4 atom of the hydrazono group resulting from addition of Bu^- to the terminal nitrogen atom of the diazo ligand. Angle values at the rhodium center [P1RhP2 = 144.2 (1)° and P3RhN4 = 158.6 (2)°] are in the range of observed angles in rhodium complexes with sterically hindered ligands.[4] Thus, the rhodium atom belongs to the five-membered metalla heterocycle defined by Rh,-

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(6) The Rh(I) complexes are sensitive to oxygen. Solvents were dried and degassed prior to use. Reactions were performed by using standard inert-atmosphere techniques. Elementary analysis were obtained for both compounds. Anal. Calcd (Found) for $C_{19}H_{46}N_4P_3RhS$ (1): C, 40.86 (40.78); H, 8.24 (8.49); N, 10.03 (9.73). Calcd (Found) for $C_{23}H_{56}N_4P_3Rh$ (2): C, 47.26 (46.98); H, 9.65 (9.35); N, 9.58 (9.45).

(7) NMR spectra were recorded at 250 MHz for ^1H and at 101.27 MHz for ^{31}P with a Bruker WM-250.

(8) A strong absorption, which can be associated with the P=S stretch, is observed at 645 cm^{-1} in Nujol for 1, whereas the P=S stretch of the free ligand occurs at 672 cm^{-1}. A decrease in the frequency of the $\bar{\nu}$(P=S) stretch is to be expected because of the unsaturated rhodium center. This assignment agrees well with the data observed in $[Rh(CO)_2PF_2S_2]_2$ (735 and 716 cm^{-1} in the free ligand and 692 and 670 cm^{-1} in the complex) (Hartman, F. A.; Lustig, M. *Inorg. Chem.* **1968**, *7*, 2669–2670).

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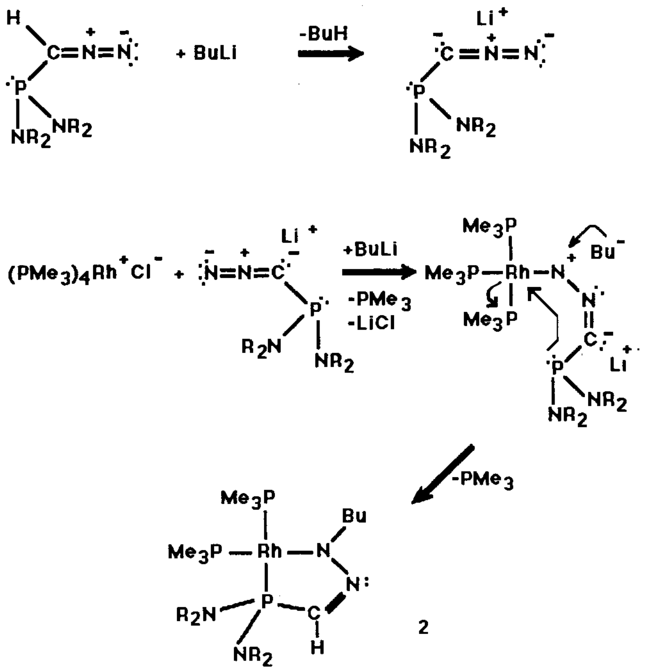
(10) $(PMe_3)_2\overline{RhN(Bu)NCHP}(N\text{-}i\text{-}Pr_2)_2$ (2), crystallized in the centrosymmetric triclinic space group $P\bar{1}$ with a = 11.237 (1) Å, b = 16.148 (4) Å, c = 9.238 (1) Å, α = 103.37 (2)°, β = 101.00 (1)°, γ = 75.79 (1)°, and V = 1565 (1) Å^3. R = 0.0404 (R_w = 0.0420) for 3714 reflections with $I > 3\sigma(I)$.

Organometallics 1988, *7*, 2233–2234

Scheme I



P1,C13,N3,N4. This ring is planar[11] with bond distances intermediate between single and double bonds, which confirms electron delocalization.

The mechanism of the reaction leading to **2** is of obvious interest since it is unprecedented. A possibility is that the diazo precursor reacts through the N terminus, affording a transient nitrile imine intermediate which is stabilized as an pyrazolyne heterocycle by reaction with butyllithium and by ring formation through the phosphorus lone pair. (This is in good agreement with the increase of the yield when a twofold excess of BuLi is used (Scheme I).) Such a reactivity is also observed when $LiC(N_2)P(N\text{-}i\text{-}Pr_2)_2$ reacts with organic electrophiles, the reaction occurring either at carbon, leading to diazo derivatives, or at the terminal nitrogen, affording nitrile imine derivatives.[12]

In conclusion, the difference in reactivity observed between these two diazo anions, i.e. $[C(N_2)[(S{=})P(N\text{-}i\text{-}Pr_2)_2]^-$ and $[C(N_2)[P(N\text{-}i\text{-}Pr_2)_2]^-$, illustrates the influence of the diazo substituent on the bonding. The electron-attracting $(S{=})P(N\text{-}i\text{-}Pr_2)_2$ group increases the negative charge at the α-carbon, thus giving rise to a C-bonded diazo derivative, while $[P(N\text{-}i\text{-}Pr_2)_2]$ bearing an electron pair on the phosphorus atom leaves more negative charge at the γ-nitrogen, thus giving rise to the N-coordinated compound. Further experiments are under active investigation in order to confirm the mechanism of these reactions and to study the reactivity of these new carbyne or carbene precursors.

Supplementary Material Available: Crystal data and details of data collection and structure refinement (Table I), fractional atomic coordinates with estimated standard deviations in parentheses (Table II), final anisotropic thermal parameters ($Å^2 \times 100$) with estimated standard deviations in parentheses (Table III), and hydrogen atomic positional and thermal parameters (Table IV) (4 pages); observed and calculated structure factor amplitudes (Tables V) (18 pages). Ordering information is given on any current masthead page.

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(11) The equation of the least-squares plane through Rh,-P1,C13,N3,N4 is $3.0654X - 8.9240Y + 2.1705Z + 5.0778 = 0$. Distances of these atoms from the plane are 0.0138 (Rh), –0.0317 (P1), 0.0066 (N4), –0.0392 (N3), and 0.0506 (C13).

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Interaction between a Vinylcarbene Complex and a Phosphole Ring: Synthesis of a New *cis*-[(7-Phosphanorbornenyl)ethoxycarbene]tetracarbonylchromium Complex

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Summary: The one-step synthesis of a new (phosphinocarbene)tetracarbonylchromium chelate is performed via an intramolecular [4 + 2] cycloaddition between a phosphole and a vinylcarbene complex.

Recently, we published some applications of the Fischer carbene complexes in the synthesis of new low-coordinated phosphorus compounds via their coupling with phosphinidene complexes.[1]

In view of the synthetic potential of these carbene complexes in the phosphorus field, we decided to explore some other types of reactions. In this paper, we take advantage of the dienophilicity of vinylcarbene complexes to build some new types of phosphinocarbene chelates.

Since the $M(CO)_5$ groups (M = Cr, Mo, W) are isolobal with the oxygen atom,[2] vinylcarbene complexes may be regarded as analogues of acrylic esters and thus can react as the 2π component in [2 + 4] cycloadditions with some dienes.[3] However, as far as we know, no intramolecular Diels–Alder reaction of this type has been described probably because it is not so obvious to keep a free and reactive dienic system close to the coordination sphere of a metal. We report here the easy introduction of a phosphole ring into the coordination sphere of a vinylcarbene chromium complex. The cis disposition of the two ligands allows a [4 + 2] intramolecular cycloaddition to take place between the phosphole dienic system and the C=C double bond of the carbene. A new cis-[(7-phosphanorbornenyl)ethoxycarbene]tetracarbonylchromium complex is thus obtained in one step.

The reaction of the carbene complex **1**[4] with a slight excess of the 1-phenyl-3,4-dimethylphosphole (**2**)[5] in hexane at 60 °C for 6 h afforded directly complex **3**[6] in ca. 85% yield (Scheme I).

Compound **3** was fully characterized by elemental analysis and mass spectrometry (EI, 70 eV): *m/z* (relative intensity) 512 (M^+, 10), 484 ($M^+ - CO$), 456 ($M^+ - 2CO$),

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(6) Compound **3** was purified by chromatography on silica gel (eluent pentane/CH_2Cl_2, 3/1): salmon crystals; mp 128 °C (with decomposition); ^{31}P NMR ($CDCl_3$) δ_p + 168.1 (reference external 85% H_3PO_4, δ positive for downfield shifts); $^{13}C\{^1H\}$ NMR ($CDCl_3$) δ_C 14.99 (s, Me), 15.98 (s, Me), 51.58 (d, $^1J_{C-P}$ = 20.7 Hz, CH), 56.39 (d, J_{C-P} = 16.6 Hz, CH), 61.51 (d, J_{C-P} = 23.8 Hz, CH), 66.38 (d, J_{C-P} = 26.7 Hz, CH), 76.47 (s, OCH_2), 140.98 (d, $^2J_{C-P}$ = 9.6 Hz, C=C), 219.80 (d, $^2J_{C-P}$ = 12 Hz, CO), 221.48 (d, $^2J_{C-P}$ = 13.5 Hz, CO), 229.77 (s, CO), 231.93 (d, $^2J_{C-P}$ = 12 Hz, CO), 356.36 (d, $^2J_{C-P}$ = 7.8 Hz, C=Cr); ^1H NMR ($CDCl_3$) δ 0.94 (s, CH_3), 1.59 (t, CH_3), 1.68 (s, CH_3), 3.07 (m, 2 H, PC_1H), 3.63 (m, 1 H, C_6HPh), 3.97 (m, 1 H, $C_5HC{=}Cr$), 5.03 (q, 2 H, OCH_2); IR (hexane) ν_{CO} 2010 m, 1940 m, 1910 vs, 1895 s cm^{-1}. Anal. Calcd for $C_{27}H_{25}O_5PCr$: C, 63.28; H, 4.88. Found: C, 63.20; H, 5.19.

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