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Electrophilic insertion of terminal phosphinidene complexes into the carbon-hydrogen bonds of ferrocene

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metal complex. Related examples of addition of I^+ to polynuclear anions have been reported.[30–33]

Regarding the formation of **6** as the initial step of the reaction of iodine, we expected subsequent nucleophilic attack of the halide anion, with the following alternate possibilities, depending on the site of attack: (i) formation of a neutral dihalogen species similar to $Os_3(CO)_{12}I_2$,[34,35] (ii) abstraction of H^+ as HI,[33] and (iii) substitution of CO by I^- (reaction of I_2 with **3** might end up as the reaction of $SnCl_4$ with $Ru_3(\mu_3\text{-}S)(\mu\text{-}H)_2(CO)_9$, which gave $Ru_3(\mu_3\text{-}S)(\mu\text{-}H)_2(\mu\text{-}Cl)(CO)_8SnCl_3$[36]).

Actually, nucleophilic attack of the halide anion was not observed, even in refluxing tetrahydrofuran,[37] which accounts for a particular stability of the cationic complex.

Following this study, we attempted the reaction of **3** with a variety of electrophiles (E^+) as H^+, $AuPPh_3^+$, or Ag^+. Cationic adducts of general formula $[Ru_3(\mu\text{-}E)(\mu_3\text{-}O)(\mu\text{-}H)_2(CO)_5(\mu\text{-}\eta^2\text{-dppm})_2]^+$ were isolated in all cases. This parallels recent observations of Lewis acid bonding to $Ru_3(CO)_8(dppm)_2$.[38]

While the bridging oxygen atom might be expected to have some nucleophilic character, no such evidence could be obtained. The presence of four donor phosphorus atoms increases the basicity at metal centers, in such a way that the privileged site for Lewis acid bonding is the metal–metal bond.

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Registry No. 1, 77611-27-9; 2, 79826-37-2; 3, 101835-50-1; 4, 101835-43-2; 5, 101835-45-4; 6, 101835-46-5; 7, 101835-48-7; $Ru_3(CO)_{10}(dppm)_2$, 64364-79-0; $AuPPh_3Cl$, 14243-64-2.

Supplementary Material Available: Table S1 (complete listing of atomic coordinates and thermal parameters for **3**), Table S2 (structure factors for **3**), Table S3 (atomic coordinated for **6**), Table S4 (anisotropic thermal parameters for **6**), Table S5 (rigid-group parameters for **6**), Table S6 (structure factors for **6**), Table S7 (selected interatomic distances for **6**), and Table S8 (selected bond angles for **8**) (129 pages). Ordering information is given on any masthead page.

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Electrophilic Insertion of Terminal Phosphinidene Complexes into the C–H Bonds of Ferrocene

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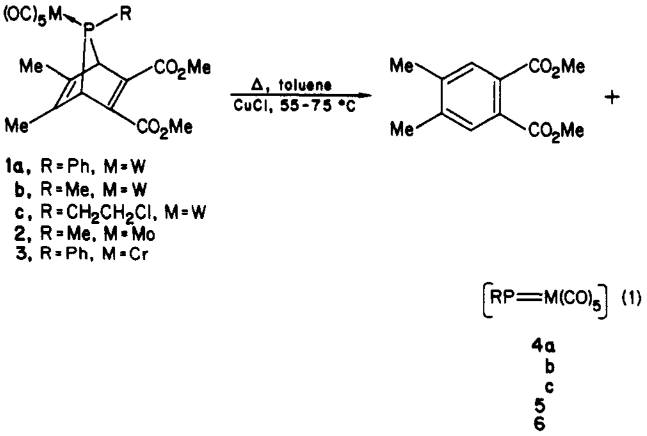
Terminal phosphinidene complexes, $[RP{=}M(CO)_5]$, as generated by catalytic decomposition of the corresponding 7-phosphanorbornadiene complexes at ca. 55–75 °C, insert into the C–H bonds of ferrocene to give secondary ferrocenylphosphine complexes $[FcP(H)R]M(CO)_5$ when M = Mo and W. The reaction fails when M = Cr. Metalation of the P–H bond of these ferrocenylphosphine complexes is easily achieved by *n*-butyllithium in THF at −78 °C. The anion thus formed can be alkylated or acylated. When R = CH_2CH_2Cl, the metalation affords a novel ferrocenylphosphirane complex.

According to experimental and theoretical data, terminal phosphinidene complexes, $RP{=}M(CO)_5$ (M = Cr, Mo, W), are highly electrophilic. For example, SCF calculations on HP and $HP{=}Cr(CO)_5$ gave a net charge on phosphorus of −0.05 e in the first case and +0.41 e in the second case.[1] On this basis, we thought that it would be perhaps possible to observe a reaction between these terminal phosphinidene complexes and electron-rich aromatic rings. This led us to investigate their reaction with ferrocene selected as a prototype of such rings. The results of this study are reported here.

Results and Discussion

As described previously,[2] the terminal phosphinidene complexes were generated by catalytic decomposition of the appropriate 7-phosphanorbornadiene complexes (eq 1). The reaction between these transient phosphinidene complexes and ferrocene was carried out by performing the decomposition of the 7-phosphanorbornadiene com-



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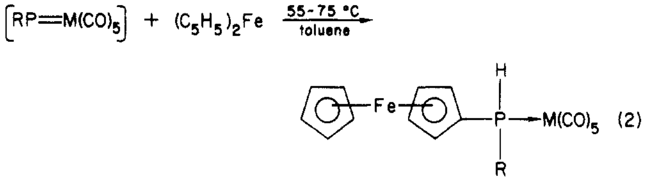
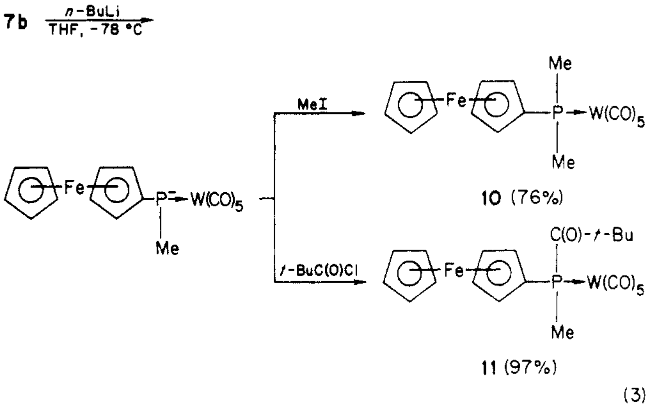
plexes in the presence of a three-fold excess of ferrocene. In such a way, we observed the expected insertion (eq 2).



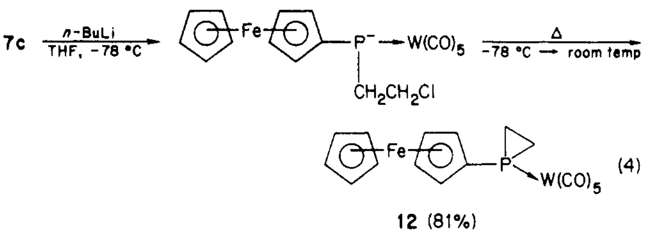
7a, R = Ph, M = W, yield 31%
b, R = Me, M = W, 41%
c, R = CH$_2$CH$_2$Cl, M = W, 31%
8, R = Me, M = Mo, yield 25%
9, R = Ph, M = Cr, 0%

The temperature was optimized for each experiment. According to ^{31}P NMR analysis of the crude reaction mixture, the reaction appears rather clean in the case of the tungsten complexes; however, the final yields after purification by chromatography on silica gel are modest, probably due to the low stability of the products (oxidation, polymerization). Apparently, the reaction does not work at all in the case of chromium. At the present time, we do not know whether this failure reflects a lower electrophilicity of the phosphinidene chromium complexes by comparison with their molybdenum or tungsten analogues or if it reflects a lower stability of the final products (lower steric protection in the case of Cr?). Anyhow, these result are interesting for two reasons. First, they confirm the high electrophilicity of terminal phosphinidene complexes; secondly, they provide an access to some novel secondary ferrocenylphosphine complexes. As far as we know, no such secondary ferrocenylphosphines nor their complexes have ever been described in the literature until now. This prompted us to carry out a preliminary study of their reactivity. The metalation of the P–H bond of **7b** was easily effected by reaction with *n*-BuLi in THF at low temperature. The anion thus formed reacted cleanly with methyl iodide and pivaloyl chloride to give the expected alkyl- and acylferrocenylphosphine complexes **10** and **11** (eq 3). It is interesting to note here that dimethylferrocenylphosphine has been prepared in only 17% yield through metalation of ferrocene by *n*-BuLi followed by reaction with Me$_2$PCl.[3] We also attempted the lithiation of **7c**. This reaction led to an interesting ferrocenylphosphirane complex, **12** (eq 4). The cyclization is accompanied by the classical upfield shift of the ^{31}P resonance observed in all three-membered rings containing phosphorus.



10 (76%)

11 (97%)



12 (81%)

All the attempted generalizations of this type of electrophilic insertions of terminal phosphinidene complexes into the C–H bonds of other electron-rich aromatic rings have failed so far. It seems that a further increase of the electrophilicity of these phosphinidene complexes is necessary in order to achieve some success in that direction.

Experimental Section

NMR spectra were recorded on a Bruker WP 80 instrument at 80.13 MHz for ^{1}H, 32.435 MHz for ^{31}P, and 20.15 MHz for ^{13}C. Chemical shifts are reported in parts per million from internal Me$_4$Si for ^{1}H and ^{13}C and from external 85% H$_3$PO$_4$ for ^{31}P. Downfield shifts are noted positive in all cases. IR spectra were recorded on a Perkin-Elmer Model 297 spectrometer. Mass spectra were recorded on a VG 70-70F spectrometer by Service Central d'Analyse du CNRS (Lyon). All reactions were carried out under argon. Chromatographic separations were performed on silica gel columns (70–230 mesh Riedel de Haën). The synthesis of the starting 7-phosphanorbornadiene complexes **1**–**3** is described in ref 4 and 5.

(Ferrocenylphenylphosphine)pentacarbonyltungsten (7a). A solution of **1a** (5.0 g, 7.64 mmol) and ferrocene (7.11 g, 38.2 mmol) in 10 mL of toluene was heated in the presence of CuCl (ca. 10 mg) at 55 °C for 4 h. Unreacted ferrocene was removed by chromatography with hexane. The product was eluted with hexane/methylene chloride (80:20) as a brown oil which solidified upon standing: yield 1.49 g (31.6%); mp 111 °C dec; ^{31}P NMR (pentane) δ −26.0 (^{1}J(^{1}H–^{31}P) = 341.8 Hz; ^{1}J(^{31}P–^{183}W) = 234.4 Hz); IR (decalin) ν(CO) 2064 (w), 1947 (sh), 1942 (s) cm^{-1}; ^{1}H NMR (C$_6$D$_6$) δ 3.9 (m), 4.45 (m, 4 H, C$_5$H$_4$), 4.10 (s, 5 H, C$_5$H$_5$), 6.47 (d, ^{1}J(P–H) = 351.8 Hz, 1 H, PH), 7.0–7.3 (m, 5 H, C$_6$H$_5$); mass spectrum (EI, 70 eV, ^{184}W), m/e (relative intensity) 618 (M$^+$, 48), 534 (M − 3CO, 26), 478 (M − 5CO, 81), 400 (M − 5CO − C$_6$H$_6$, 43), 294 (M − W(CO)$_5$, 100), 297 (ferrocenyl-PH, 25), 186 (ferrocene, 36). Anal. Calcd for C$_{21}$H$_{15}$FeO$_5$PW: C, 40.81; H, 2.45; P, 5.01; W, 29.75. Found: C, 40.05; H, 2.39; P, 5.01; W, 29.72.

(Ferrocenylmethylphosphine)pentacarbonyltungsten (7b). **1b** (7.3 g, 12.33 mmol) and ferrocene (6.88 g, 36.98 mmol) were heated with a small amount of CuCl in toluene (5 mL) at 65 °C for 4 h. Unreacted ferrocene was removed by chromatography with pentane. **7b** was eluted with pentane/CH$_2$Cl$_2$ (95:5): red crystals from hexane; yield 2.81 g (41%); mp 91 °C; ^{31}P NMR (CH$_2$Cl$_2$) δ −59.0 (^{1}J(^{1}H–^{31}P) = 344.2 Hz, ^{1}J(^{31}P–^{183}W) = 229.5 Hz); IR (decalin) ν(CO) 2064 (w), 1940 (s) cm^{-1}; ^{1}H NMR (C$_6$D$_6$) δ 1.31 (dd, ^{2}J(P–H) = 5.13 Hz, ^{3}J(H–H) = 6.59 Hz, 3 H, CH$_3$), 3.94 (s, 5 H, C$_5$H$_5$), 3.99 (m, 4 H, C$_5$H$_4$), 5.14 (dq, ^{1}J(P–H) = 344.5 Hz, 1 H, PH); mass spectrum (EI, 70 eV, ^{184}W), m/e (relative intensity) 556 (M$^+$, 89), 472 (M − 3CO, 34), 416 (M − 5CO, 76), 400 (ferrocenyl-P-W, 75), 232 (ferrocenyl-PHMe, 100), 186 (ferrocene, 33), 121 (CpFe, 36). Anal. Calcd for C$_{16}$H$_{13}$FeO$_5$PW: C, 34.57; H, 2.36; P, 5.57; W, 33.07. Found: C, 34.73; H, 2.42; P, 5.97; W, 32.56.

[(2-Chloroethyl)ferrocenylphosphine]pentacarbonyltungsten (7c). **1c** (4.07 g, 6.35 mmol) and ferrocene (6.1 g, 32.8 mmol) were reacted in toluene (10 mL) in the presence of a catalytic quantity of CuCl at 65 °C for 3 h. After removal of ferrocene by chromatography with pentane, **7c** was eluted with pentane/CH$_2$Cl$_2$ (90:10). Light brown crystals from pentane: yield 1.19 g (31%); mp 64 °C; ^{31}P NMR (CH$_2$Cl$_2$) δ −52.1 (^{1}J(^{31}P–^{1}H) = 346.7 Hz, ^{1}J(^{31}P–^{183}W) = 234.4 Hz); IR (decalin) ν(CO) 2065 (w), 1948 (sh), 1940 (vs) cm^{-1}; ^{1}H NMR (C$_6$D$_6$) δ 1.97 (ddd, ^{3}J(HCPH) $\simeq$ 6.4 Hz, ^{2}J(P–H) $\simeq$ 6.5 Hz, ^{3}J(HCCH) = 6.96 Hz, 2 H, C$_\alpha$HH′), 3.11 (~td, ^{3}J(HCCH) = 6.84 Hz, C$_\beta$H), 3.13 (~td, ^{3}J(HCCH) = 7.33 Hz, ^{2}J(HCH) (geminal) $\simeq$ 0, C$_\beta$H′) (2 H), 3.8–4.1 (m, C$_5$H$_4$), 3.97 (s, C$_5$H$_5$) (9H), 5.37 (dt, ^{3}J(P–H) = 6.35 Hz,

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^{1}J(P–H) ≃ 350 Hz, 1 H, PH); mass spectrum (EI, 70 eV, ^{184}W), *m/e* (relative intensity) 604 (M^+, 38), 520 (M – 3CO, 12), 492 (M – 4CO, 15), 464 (M – 5CO, 8), 436 (M – 5CO – $H_2C{=}CH_2$, 97), 400 (M – 5CO – CH_2CH_2Cl, 30), 280 (ferrocenyl–PCH_2CH_2Cl, 86), 217 (ferrocenyl–P, 100), 186 (ferrocene, 50). Anal. Calcd for $C_{17}H_{14}ClFeO_5PW$: C, 33.78; H, 2.34; Cl, 5.87; P, 5.13; W, 30.42. Found: C, 33.39; H, 2.26; Cl, 5.55; P, 5.45; W, 30.82.

(Ferrocenylmethylphosphine)pentacarbonylmolybdenum (8). **2** (2.0 g, 3.97 mmol) was reacted with ferrocene (2.21 g, 11.9 mmol) in the presence of CuCl in toluene (4 mL) at 65 °C for 13 h. Ferrocene was removed by chromatography with hexane; **8** was isolated with hexane/CH_2Cl_2 (90:10) and recrystallized from hexane: yield 0.47 g (25.3%) of brown crystals; mp 68 °C; ^{31}P NMR (CH_2Cl_2) δ –38.3 (^{1}J(^{1}H–^{31}P) = 329.6 Hz); IR (decalin) ν(CO) 2064 (w), 1944 (s) cm^{-1}; ^{1}H NMR (C_6D_6) δ 1.17 (pseudo-t, ^{3}J(H–H) = 6.53 Hz, ^{1}J(P–H) ≃ 6.8 Hz, 3 H, CH_3), 3.89–4.06 (m, C_5H_4), 3.95 (s, C_5H_5) (9 H), 4.87 (dq, ^{1}J(P–H) = 331.05 Hz, 1 H, PH); mass spectrum (EI, 70 eV, ^{98}Mo), *m/e* (relative intensity) 470 (M^+, 20), 386 (M – 3CO, 11), 330 (M – 5CO, 21), 314 (M – 5CO – CH_4, 48), 232 (ferrocenyl–PHMe, 99), 217 (ferrocenyl–PH, 100), 186 (ferrocene, 41), 121 (CpFe, 55). Anal. Calcd for $C_{16}H_{13}FeMoO_5P$: C, 41.06; H, 2.80; Fe, 11.93; Mo, 20.50; P, 6.62. Found: C, 41.23; H, 2.78; Fe, 11.95; Mo, 20.62; P, 6.47.

(Dimethylferrocenylphosphine)pentacarbonyltungsten (10). **7b** (0.42 g, 0.76 mmol) was metalated with 1.55 M butyllithium (0.49 mL, 0.76 mmol) in 30 mL of THF at –78 °C. After 15 min, MeI (0.11 g, 0.76 mmol) was added, and the mixture was warmed up to room temperature. Chromatography with CH_2Cl_2 yielded a yellow oil which was crystallized from hexane: yield 0.33 g (76%); mp 56 °C; ^{31}P NMR (CH_2Cl_2) δ –30.8 (^{1}J(^{31}P–^{183}W) = 251.47 Hz); IR (Nujol) ν(CO) 2064 (w), 1943 (s) cm^{-1}; ^{1}H NMR (C_6D_6) δ 1.38 (d, ^{2}J(P–H) = 7.32 Hz, 6 H, CH_3), 3.94 (s, C_5H_5), 3.86–4.0 (m, C_5H_4) (9 H); mass spectrum (EI, 70 eV, ^{184}W), *m/e* (relative intensity) 570 (M^+, 90), 486 (M – 3CO, 66), 430 (M – 5CO, 100), 400 (M – 5CO – $2CH_3$, 51), 246 (ferrocenyl–PMe_2, 54), 231 (ferrocenyl–PMe, 36). Anal. Calcd for $C_{17}H_{15}FeO_5PW$: C, 35.82; H, 2.65; Fe, 9.80; P, 5.43. Found: C, 35.77; H, 2.52; Fe, 9.74; P, 5.38.

(Ferrocenylmethylpivaloylphosphine)pentacarbonyltungsten (11). **7b** (0.35 g, 0.63 mmol) was metalated with 1.5 M butyllithium (0.42 mL, 0.63 mmol) in 20 mL of THF at –78 °C. After 30 min, pivaloyl chloride (0.09 g, 0.76 mmol) was added, and the mixture was warmed up to room temperature. Chromatography with pentane/CH_2Cl_2 (70:30) yielded an orange solid which was washed with pentane: yield 0.39 g (97%); mp 148 °C; ^{31}P NMR (CH_2Cl_2) δ –0.12 (^{1}J(^{31}P–^{183}W) = 227.0 Hz); IR (decalin) ν(CO) 2065 (w), 1948 (sh), 1941 (s) cm^{-1}; ^{1}H NMR (C_6D_6) δ 0.97 (s, 9 H, C_4H_9), 1.90 (d, ^{2}J(P–H) = 6.84 Hz, 3 H, CH_3), 3.66 (m, 1 H, CH_{α}), 3.95 (s, 5 H, C_5H_5), 3.99 (m, 2 H, $CH_{\beta\beta'}$), 4.50 (m, 1 H, $CH_{\alpha'}$); mass spectrum (EI, 70 eV, ^{184}W), *m/e* (relative intensity) 640 (M^+, 12), 555 (M – 3CO – H, 32), 499 (M – 5CO, 41), 471 (M – 6CO – H, 22), 443 (M – 5CO – C_4H_9, 14), 415 (M – 6CO – C_4H_9, 48), 399 (M – 6CO – C_4H_9 – CH_4, 29), 231 (ferrocenyl–PMe, 100). Anal. Calcd for $C_{21}H_{21}FeO_6PW$: C, 39.41; H, 3.31; Fe, 8.73; P, 4.84. Found: C, 39.31; H, 3.32; Fe, 8.73; P, 4.92.

(1-Ferrocenylphosphirane)pentacarbonyltungsten (12). To a solution of **7c** (0.36 g, 0.6 mmol) in 50 mL of THF at –78 °C was added 1.5 M butyllithium (0.4 mL, 0.6 mmol). After 30 min, the mixture was slowly warmed up to room temperature. **12** was isolated by chromatography with CH_2Cl_2 as an orange oil, which crystallized upon standing at –20 °C. Recrystallization from hexane yielded 0.28 g (81%): mp 85 °C; ^{31}P NMR (CH_2Cl_2) δ –195.8 ppm (^{1}J(^{31}P–^{183}W) = 261.2 Hz); IR (Nujol) ν(CO) 2066 (w), 1943 (s) cm^{-1}; ^{1}H NMR (C_6D_6) δ 0.95 (m, 4 H, CH_2CH_2), 3.75–3.95 (m, 4 H, C_5H_4), 3.99 (s, 5 H, C_5H_5); mass spectrum (EI, 70 eV, ^{184}W), *m/e* (relative intensity) 568 (M^+, 34), 540 (M – CO, 21), 484 (M – 3CO, 27), 456 (M – 4CO, 30), 428 (M – 5CO, 52), 400 (M – 5CO – C_2H_4, 93), 335 (M – 5CO – C_2H_4 – C_5H_5, 11), 244 (ferrocenyl–P(CH_2CH_2), 20), 216 (ferrocenyl–P, 100). Anal. Calcd for $C_{17}H_{13}FeO_5PW$: C, 35.95; H, 2.31; Fe, 9.83; P, 5.45; W, 32.37. Found: C, 36.25; H, 2.41; Fe, 9.84; P, 5.56; W, 32.19.

Registry No. **1a**, 83603-06-9; **1b**, 83603-07-0; **1c**, 101200-20-8; **2**, 101200-21-9; **7a**, 101200-13-9; **7b**, 101200-14-0; **7c**, 101200-15-1; **8**, 101200-16-2; **10**, 101200-17-3; **11**, 101200-18-4; **12**, 101200-19-5; CuCl, 7758-89-6; ferrocene, 102-54-5; pivaloyl chloride, 3282-30-2.

A Preliminary Chemical Study of Terminal Allyl-, 3-Butenyl-, and 4-Pentenylphosphinidene Complexes. Thermal Rearrangement of 1-Allylphosphirene Complexes by Insertion of the Allylic C=C Double Bond into the Phosphirene Ring

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Received July 11, 1985

The reaction of dimethyl acetylenedicarboxylate with the appropriate phosphole complexes leads to the 7-allyl-, 7-(3-butenyl)-, and 7-(4-pentenyl)-7-phosphanorbornadiene P–W(CO)$_5$ complexes. In the 3-butenyl case, it is necessary to use a large excess of acetylenedicarboxylate in order to avoid an intramolecular [4 + 2] cycloaddition between the phosphole dienic system and the double bond of the butenyl P substituent. The thermal decomposition of the 7-phosphanorbornadiene complexes leads to the corresponding terminal phosphinidene tungsten pentacarbonyl complexes. The pentenylphosphinidene complex undergoes an internal cycloaddition involving the phosphorus atom and the C=C double bond to give a bicyclic phosphirane complex. The terminal allylphosphinidene complex reacts with olefins and acetylenes to give the expected phosphirane and phosphirene complexes. In the latter case, however, additional heating induces a brand new insertion of the allylic double bond within the phosphirene cycle leading to a new type of bicyclic phosphirane complex. The postulated mechanism implies an equilibrium between phosphirene complexes and 1-metalla-2-phosphacyclobutenes at high temperature. No such insertion has been observed with 1-(3-butenyl)phosphirene complexes nor with an external olefin and a phosphirene complex.

In a series of recent papers,[1-4] we demonstrated that it was possible to generate transient terminal phenyl- and methylphosphinidene complexes by thermal or catalytic decomposition of the appropriate 7-phosphanorbornadiene

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