

Figure 1. ORTEP drawing of $[(\text{C}_6\text{H}_5)(\text{PPh}_3)\text{Ir}(\eta^2\text{-NCC}_6\text{H}_4\text{Cl})]$ (**2a**). Hydrogen atoms have been omitted for clarity, and phenyl groups are depicted schematically.

The stability of **2a** and **2b** and their mode of formation strongly support a product which would result from formal oxidative addition of an Ir(I) 16-electron fragment to the CN triple bond, thereby generating an Ir(III) $\text{Ir}-\text{C}\equiv\text{N}$ metallacycle, rather than simple π -complexation of ϵ nitrile to a metal center. The nitrile ligands of **2a** and **2b** are not easily displaced. In contrast, the nitrile ligand of the side-bonded nitrile complex $(\text{PPh}_3)_2\text{Pt}(\pi\text{-CF}_3\text{CN})^8$ is readily displaced by CO and diphenylacetylene at room temperature. The only side-bonded nitrile complexes comparable to **2a** and **2b** are molybdenocene nitrile complexes⁴ for which no crystallographic study is available to confirm their structure.

An X-ray diffraction study was undertaken of compound **2a**,¹⁰ which established the nitrile ligand to be side-bonded to the Ir (Figure 1). The Ir-C(6) bond length is 2.11 (2) Å, which is the expected length for an Ir(III)-C bond;^{11,12} the Ir-N bond distance is 2.17 (2) Å which represents a long Ir-N single bond.^{13,14} The C(6)-N bond distance is 1.23 (3) Å, which represents a lengthening of 0.08 Å relative to that of the free nitrile. No structural information is available to compare this C-N bond distance with other side-bonded nitrile complexes; a number of acetylene η^2 -complexes have been structurally characterized and are observed to undergo large reductions in the C-C stretching frequencies and accompanying lengthening of the C-C bond.^{14,15} The average increase in the C-C bond length on coordination is 0.08 Å. The lengthening observed for the C-N distance of **2a** is of the same magnitude, suggesting a similar reduction in the bond order.

(10) Crystal data for **2a**: $\text{C}_{30}\text{H}_{24}\text{ClIrNP}$; M_r , 657.1; yellow-brown parallelepiped; orthorhombic; space group $Pbcn$ (standard setting, $Pbcn$); $a = 10.638$ (2) Å, $b = 14.298$ (3) Å, $c = 33.310$ (5) Å; $V = 5066$ Å³; $Z = 8$; $D(\text{calcd}) = 1.72$ g cm⁻³. A total of 4254 unique reflections were collected of which 2495 were considered observed ($I > 3\sigma(I)$) and were used in subsequent calculations (Hübler diffractometer built by Professor C. E. Strouse of this department; Mo K α radiation; graphite monochromator; $\lambda = 0.7107$ Å; θ - 2θ scan; $0 < 2\theta < 54^\circ$; $\mu = 5.733$ cm⁻¹). The structure was solved by the heavy-atom method using SHELX 76. In the final least-squares cycle, based on F , 307 parameters were refined including positional and anisotropic thermal parameters for one Ir, 30 C, one Cl, one N, and one P. Refinement is currently at $R = 0.077$ and $R_w = 0.086$. The goodness of fit is 2.26.

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From the intramolecular mode of formation of the nitrile complexes **2a** and **2b**, and their great chemical stability when compared to other side-bonded nitrile complexes, it appears that **2a** and **2b** are best described as formal Ir(III) metallacycles.

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Supplementary Material Available: Figure 1s, packing diagram, Figure 2s, atom numbering scheme, and tables of positional and thermal parameters and interatomic distances and angles (9 pages); a listing of observed and calculated structure factors (11 pages). Ordering information is given on any current masthead page.

HFe(CO)₄⁻, a Versatile Reagent toward Chlorophosphines. Facile Synthesis of New Phosphorus Transition-Metal Complexes

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Summary: Depending on the experimental conditions, reaction of $[\text{Ph}_4\text{P}][\text{HFe}(\text{CO})_4]$ (**1**) with phenyldichlorophosphine leads quantitatively either to the stable secondary halophosphine complex $\text{PhP}(\text{H})\text{ClFe}(\text{CO})_4$ (**2b**), or to the first nonhindered side-on and end-on diphosphine complex $[\text{Fe}(\text{CO})_4[\mu\text{-Fe}(\text{CO})_4](\text{PPh}_2)_2]$ (**5**), or to the first trimetallic anionic diphosphane species $[\text{Ph}_4\text{P}][\text{Fe}(\text{CO})_4]_3\text{P}_2\text{Ph}_2\text{H}]$ (**7**). X-ray diffraction study confirms the structure of **5**. Addition of $[\text{Et}_4\text{N}][\text{HW}(\text{CO})_5]$ to the diphosphane complex **5** affords another mixed trimetallic anionic diphosphane compound $[\text{Et}_4\text{N}][[\text{Fe}(\text{CO})_4]_2\text{W}(\text{CO})_5\text{P}_2\text{Ph}_2\text{H}]$ (**8**).

Recently, a number of publications described the reactivity of carbonylmetalate dianions $\text{Na}_2[\text{M}_2(\text{CO})_{10}]$ ($\text{M} = \text{Cr}, \text{Mo}, \text{or W}$) or $\text{Na}_2\text{Fe}(\text{CO})_4$ with $\text{R}_2\text{P}_2\text{Cl}_2$ leading to a variety of phosphinidene, $[(\text{CO})_5\text{M}]_2\text{PR}$, or diphosphene complexes, $[(\text{CO})_5\text{M}]_n\text{RP}=\text{PR}$ ($n = 1$ or 2), depending on the experimental conditions and the steric hindrance of R.^{1,2}

To our knowledge, no similar work has been devoted to the reactivity of anionic hydridocarbonylmetalate $[\text{PPh}_3][\text{HFe}(\text{CO})_4]$ (**1**) with dichlorophosphines. We have since begun to investigate this reaction, and in this paper we report (i) a simple one-step quantitative synthesis of stable secondary halophosphine complexes $\text{RP}(\text{H})\text{ClFe}$ -

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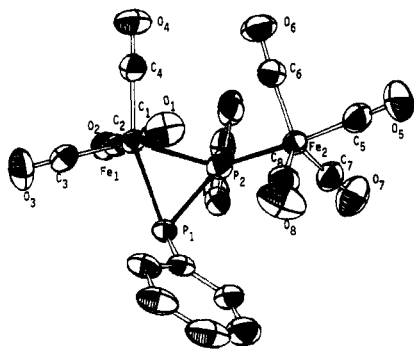
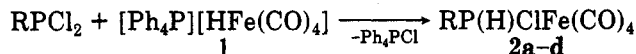


Figure 1. ORTEP view of $[\text{Fe}(\text{CO})_4(\mu\text{-Fe}(\text{CO})_4)(\text{PPh})_2]$ (5). Bond distances (Å) and angles (deg) of interest: Fe(1)–P(1), 2.389 (1); Fe(1)–P(2), 2.354 (1); Fe(2)–P(2), 2.267 (1); P(1)–P(2), 2.139 (2); P(1)–Fe(1)–P(2), 53.60 (4); Fe(1)–P(1)–P(2), 62.36 (4); Fe(1)–P(2)–Fe(2), 127.72 (5); Fe(1)–P(2)–P(1), 64.05 (5); Fe(2)–P(2)–P(1), 133.29 (7).

(CO)₄ (2), (ii) an original mode of formation of a new side-on and end-on nonhindered diphosphene complex 5 which is fully characterized by spectroscopic and X-ray crystallographic studies, and (iii) the preparation of the first trimetallic anionic diphosphane species $[\text{Ph}_4\text{P}][[(\text{CO})_4\text{Fe}]_3\text{P}_2\text{Ph}_2\text{H}]$ (7) and $[\text{Et}_4\text{N}][[(\text{CO})_4\text{Fe}]_2\text{W}(\text{CO})_5\text{P}_2\text{Ph}_2\text{H}]$ (8).

When a solution of the hydride $[\text{Ph}_4\text{P}][\text{HFe}(\text{CO})_4]$ (1) in dichloromethane and a solution of dichlorophosphine in dichloromethane are added simultaneously, drop by drop, at room temperature, stable complexes of secondary halophosphines 2³ are formed quantitatively.



a, R = N(*i*-Pr)₂; b, R = Ph; c, R = *t*-Bu; d, R = Me

The facile synthesis of these new complexes is in marked contrast to the few reactions previously described in the literature: formation of MeP(H)ClFe(CO)₄ (2d) from MePCL₂⁴ and of PhP(H)ClW(CO)₅ (3) from PhPCL₂,⁵ both involve five steps, while that of 2a necessitates three steps.⁶

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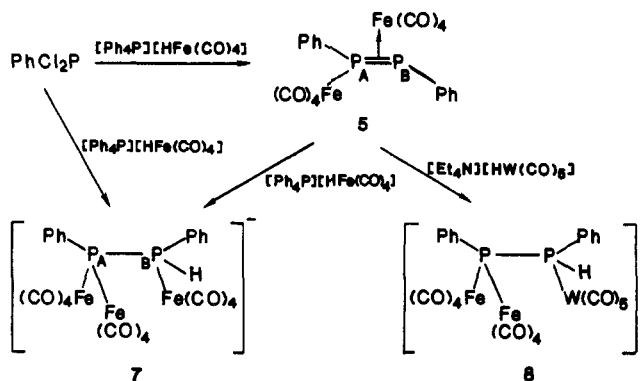
(3) 2a: oil; ³¹P NMR (CH₂Cl₂) δ 112.9 (dt, ¹J_{PH} = 448 Hz, ³J_{PH} = 14.8 Hz); ¹H NMR δ 0.90 (d, *J* = 14 Hz, CH₃), 3.50 (mult, CH), 7.97 (d, *J* = 44.7 Hz, PH); IR ν(CO) (hexane) 2073 (m), 1993 (m), 1965 (s), 1954 (s) cm⁻¹. 2b: oil; ³¹P NMR (C₆D₆) δ 154.7 (d, ¹J_{PH} = 391 Hz); ¹H NMR (C₆D₆) δ 6.63 and 7.07 (ph), 7.00 (d, *J*_{PH} = 391 Hz); IR ν(CO) (hexane) 2063 (m), 1986 (m), 1952 (s), 1946 (s) cm⁻¹. 2c: oil; ³¹P NMR (CH₂Cl₂) δ 156.3 (d of dec, ¹J_{PH} = 368 Hz, ³J_{PH} = 20 Hz); ¹H NMR (C₆D₆) δ 0.92 (d, ³J_{HP} = 20 Hz, *t*-Bu), 6.12 (d, ¹J_{HP} = 368 Hz, H); IR ν(CO) (hexane) 2065 (m), 1997 (m), 1968 (s), 1955 (s) cm⁻¹. 2d: oil; ³¹P NMR (CH₂Cl₂) δ 114.2 (d of q, ¹J_{PH} = 395 Hz, ²J_{PH} = 8.8 Hz); ¹H NMR (C₆D₆) δ 2.28 (dd, ²J_{PH} = 8.8 Hz, ³J_{HH} = 5.7 Hz, Me), 7.43 (d of q, ¹J_{PH} = 395 Hz, ³J_{HH} = 5.7 Hz, H); IR ν(CO) (hexane) 2068 (m), 2000 (m), 1968 (s), 1958 (s) cm⁻¹.

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Scheme I



Furthermore, the principle of the reaction seems to be extended to other hydride anion complexes since first experiments show that 3 is obtained when PhPCL₂ is reacted with $[\text{Et}_4\text{N}][\text{HW}(\text{CO})_5]$ (4) in the same experiment conditions.⁷

A dramatic change occurs when a solution of hydride 1 in dichloromethane is slowly added to a solution of phenyldichlorophosphine (stoichiometry, 1:1) in dichloromethane at room temperature. After evaporation of the solvent, extraction with pentane affords stable red crystals of $[\text{Fe}(\text{CO})_4(\mu\text{-Fe}(\text{CO})_4)(\text{PPh})_2]$ (5; mp 113 °C dec; yield 65%).⁸

The ³¹P NMR values (δ 52.1, –34.5 (AB system, ¹J_{PP} = 415 Hz)) preclude an end-on bonded complex where each phosphorus atom behaves as a simple two-electron donor^{1d–f} and yet does not seem in agreement with those found for $[\text{Fe}(\text{CO})_4(\mu\text{-Fe}(\text{CO})_4)(\text{P-2,4,6-}t\text{-Bu}_3\text{C}_6\text{H}_2\text{O})_2]$ (6; δ(³¹P) 233.7, 193.4 (AB system, ¹J_{PP} = 532 Hz)), the first side-on and end-on diphosphene complex previously obtained by reacting the carbonylmetalate dianion Na₂Fe(CO)₄ with the sterically hindered dichlorophosphine Cl₂P(2,4,6-*t*-Bu₃C₆H₂O).^{1b}

Therefore, the structure of 5 was determined by single-crystal X-ray diffraction⁹ and is illustrated in Figure 1. Surprisingly, all the bond lengths and angle values are close to those obtained for 6 and suggest a side-on and end-on bonded complex structure. 5 is the first example of such a species stabilized without bulky substituents.

The fascinating versatility of the reaction of 1 with dichlorophosphines is illustrated once more when 1 equiv of phenyldichlorophosphine is added to 2 equiv of 1 in dichloromethane at room temperature: the unexpected first anionic diphosphane complex 7 is formed as the unique product of the reaction. 7 is isolated as red-orange crystals (mp 92 °C dec; yield 55% after recrystallization).¹⁰

(7) 3: mp 55 °C; yield 30%. 3 was previously prepared by a five-step synthesis.⁵

(8) 5: ¹H NMR (CD₂Cl₂) δ 5.83 and 6.09 (m, Ph); IR ν(CO) (hexane) 2108 (m), 2055 (sh), 2052 (s), 2047 (s), 2024 (m), 1982 (m), 1970 (sh), 1962 (m), 1960 (sh), 1940 (m) cm⁻¹; mass spectrum, *m/e* 522 with successive loss of 8 COs. A single crystal of 5·0.5CH₂Cl₂ was grown from dichloromethane/hexane at –20 °C.

(9) A yellow crystal of 5·0.5CH₂Cl₂ (dimensions 0.3 × 0.3 × 0.12 mm) was selected: Mo Kα radiation (λ = 0.71069 Å); *T* = 293 K; triclinic *P*1, *a* = 9.919 (1) Å, *b* = 10.916 (2) Å, *c* = 11.563 (2) Å, α = 81.81 (1)°, β = 88.66 (1)°, γ = 71.18 (1)°; *Z* = 2; μ = 15.3 cm⁻¹; 2069 unique data, 307 parameters. The structure was solved by a combination of Multan, Patterson, and heavy-atom methods. No absorption correction was made. The final full-matrix least-squares refinement converged to *R* = 0.032 and *R*_w = 0.043.

(10) 7: ³¹P{¹H} NMR (CD₂Cl₂) δ(P_A) 91.15 (d), δ(P_B) 42.71 (d, ¹J_{PP} = 234.8 Hz, AB system), δ(P_C) 22.01; ³¹P NMR (CD₂Cl₂) δ(P_A) 91.15 (d), δ(P_B) 46.32 and 39.10 (d of d, ¹J_{PH} = 342 Hz, ¹J_{PP} = 234.8 Hz), δ(P_C) 22.01 (m); ¹H NMR (CD₂Cl₂) δ 4.38 (d, ¹J_{PH} = 342 Hz, H), 5.94, 6.34 (m, Ph); IR ν(CO) (CH₂Cl₂) 2050 (w), 2033 (m), 2020 (m), 1937 (s, br) cm⁻¹. Products 5, 7, and 8 gave satisfactory elemental analysis data. 2a–d were characterized by mass spectroscopy.

Compound 7 is also quantitatively obtained when the hydride 1 is reacted with the side-on and end-on bonded complex 5. The formation of 7 could be explained by a 1:1 addition of 1 on phosphorus P_B of 5. This is corroborated by the following experiment: reaction of the hydride 4 $[Et_4N][HW(CO)_5]$ with 5 leads to the formation of another anionic diphosphane complex, 8 (mp 116 °C dec; yield 62%). ^{31}P and 1H NMR spectra clearly show that the phosphorus atom P_B is bonded to a proton and to $W(CO)_5$: $^{31}P\{^1H\}$ NMR (CD_3CN) $\delta(P_A)$ 74.85 (d), $\delta(P_B)$ -6.64 (d), $J_{P-P_B} = 220$ Hz ($J_{^{31}P-^{183}W} = 208.5$ Hz); ^{31}P NMR (CD_3CN) $\delta(P_A)$ 74.85 (d), $\delta(P_B)$ -4.49, -8.42 (dd, $^1J_{PH} = 324.4$ Hz, $J_{P-P_B} = 220$ Hz, $J_{^{31}P-^{183}W} = 208.5$ Hz); 1H NMR (CD_3CN ; except $N(C_2H_5)_4$ resonances) δ 6.75 (dd, $^1J_{PH} = 324.4$ Hz, $^2J_{PH} = 7.92$ Hz, $J_{H-^{183}W} = 22.7$ Hz), 7.35 and 7.66 (m, Ph); IR $\nu(CO)$ (CH_2Cl_2) 2072 (w), 2040 (m), 2022 (m), 1940 (s), 1915 (sh) cm^{-1} .

These results summarized in Scheme I provide a new field of investigations for the synthesis of other unknown mono- or polymetallic complexes of phosphorus derivatives and demonstrated the potential utility of the $HFe(CO)_4$ anion. The extension of this work to other anionic hydridocarbonylmetalates and a variety of group 15 chlorides is in progress.

Acknowledgment. Thanks are due to Dr. Pierrot (Faculté des Sciences de Saint-Jérôme, Marseille, France) for the X-ray crystallographic determination.

Supplementary Material Available: Tables of bond lengths, bond angles, atomic coordinates, and thermal parameters (3 pages); a table of structure factors (8 pages). Ordering information is given on any current masthead page.

X-ray Crystal Structure of the Boron-Stabilized Carbanion

$[Li(12\text{-crown-4})_2][CH_2C_6H_2(3,5\text{-Me}_2)(4\text{-B}\{2,4,6\text{-Me}_3C_6H_2\}_2)] \cdot Et_2O$: Evidence for "Boron Ylide" Character

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Summary: The first X-ray crystal structure of a boron-stabilized carbanion is described; the title compound, made by treatment of $B(2,4,6\text{-Me}_3C_6H_2)_3$ with $n\text{-BuLi}$ in THF, was crystallized as a crown ether salt. The major structural features of the carbanion suggest that stabilization is achieved by extensive delocalization rather than steric effects. A significantly shortened B-C bond, 1.522 (10) Å, was also observed, suggesting substantial "boron ylide" character.

Recently we showed that 12-crown-4 could be used to crystallize the lithium salts of the free carbanions $[CPh_3]^-$ and $[CHPh_2]^-$.¹ At the same time we reported that we could isolate a yellow microcrystalline substance which probably contained the next member of the series $[CH_2Ph]^-$ as a free ion. Unfortunately we were unable to grow crystals of this material, suitable for X-ray crystallography, due to its high reactivity toward ether solvents. In our search for ways of reducing the powerful nucleophilic character of the benzyl carbanion $[CH_2Ph]^-$, we were

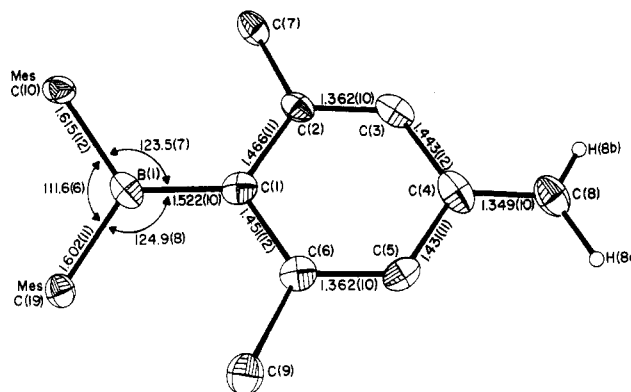


Figure 1. Important bond distances and angles for the anion of 1.

drawn to the report² of Ramsey and Isabelle, who showed (NMR) that by treating BMe_3 (Mes = mesityl, 2,4,6- $Me_3C_6H_2$) with $n\text{-BuLi}$, they could obtain the anion $[CH_2C_6H_2(3,5\text{-Me}_2)(4\text{-B}\{2,4,6\text{-Me}_3C_6H_2\}_2)]^-$ (1) in THF or Me_2SO solution. This anion arose by the deprotonation of one of the para methyl groups of BMe_3 and has a benzyl-like environment at the deprotonated carbon. As part of our interest in carbanion structures, we decided to investigate the structural effects of a boron-centered stabilizing group on a carbanion.

The anion 1 was crystallized as its $[Li(12\text{-crown-4})_2]^+$ salt by the addition of 2 equiv of 12-crown-4³ to a solution of a lithium salt of 1. The resultant red crystals were isolated in 35% yield. Important bond distances and angles of 1 arising from the X-ray data are illustrated in Figure 1. Crystal data at 140 K: $C_{47}H_{74}O_9BLi$, M , 800.86, monoclinic, space group $P2_1/n$, $a = 15.187$ (7) Å, $b = 11.791$ (4) Å, $c = 26.031$ (11) Å, $\beta = 105.12$ (3)°, $U = 4500$ (3) Å³, $D_{\text{calcd}} = 1.18$ g cm^{-3} for $Z = 4$, $\lambda = 0.71069$ Å, $\mu = 0.76$ cm^{-1} , 2580 data with $I > 4\sigma(I)$, $R = 0.069\%$. The structure confirms the removal of one of the para methyl protons by $n\text{-BuLi}$. The compound is clearly a boron-stabilized carbanion, and 1 is the first X-ray structural proof of this type of stabilization. The growing interest in this class of compound originated with the work of Rathke and Kow⁴ although boron-stabilized carbanions had been proposed as intermediates much earlier.⁵ The spectroscopically characterized boron-stabilized carbanions for the most part involve deprotonation of an α -carbon atom, for example, in B -methyl-9-boradicyclononane,⁴ 4- Et_2B -fluorene,⁶ or the versatile reagent Mes_2BMe developed by Wilson, Pelter, and co-workers.^{7,8} The latter have shown that anions produced by the deprotonation of organoboranes undergo many of the same reactions as ylides; hence their designation as Boron-Wittig reagents.

The most interesting aspect of 1 concerns the core B-

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