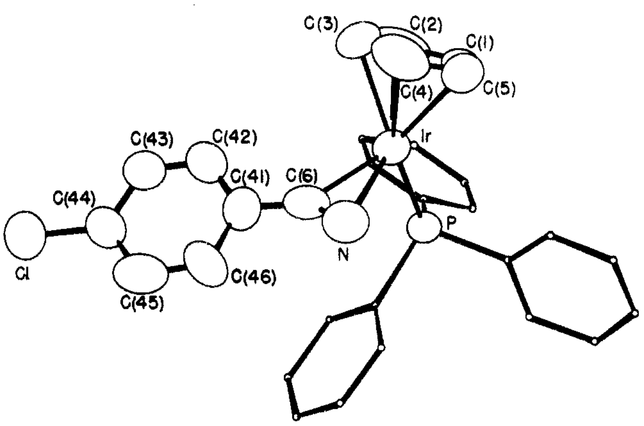

Figure 1. ORTEP drawing of [(C_5H_5)(PPh_3)Ir(η^2-NCC_6H_4Cl)] (**2a**). Hydrogen atoms have been omitted for clarity, and phenyl groups are depicted schematically.

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) $\overline{\mathrm{Ir{-}C{=}N}}$ metallacycle, rather than simple π-complexation of a 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 $(PPh_3)_2Pt(\pi\text{-}CF_3CN)$[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 complexes[4] 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.

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.

Acknowledgment. We are grateful to the Office of Naval Research for the support of this research (Contract No. N00014-76-C-0390). We also thank Johnson-Matthey Corp. for a generous gift of iridium chloride.

Registry No. 1a, 103731-57-3; 1b, 103731-58-4; 2a, 103731-59-5; 2b, 103751-00-4.

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.

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[10] Crystal data for **2a**: $C_{30}H_{24}ClIrNP$: M_r 657.1; yellow-brown parallelpiped; orthorhombic; space group *Pcan* (standard setting, *Pbcn*); $a = 10.638$ (2) Å, $b = 14.298$ (3) Å, $c = 33.310$ (5) Å; $V = 5066$ Å^3; $Z = 8$; D(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 (Hüber diffractometer built by Professor C. E. Strouse of this department; Mo Kα radiation; graphite monochromator; $\lambda = 0.7107$ Å; θ-2θ scan; $0 < 2\theta < 54°$; $\mu = 5.733$ 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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$HFe(CO)_4^-$, 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 $[Ph_4P][HFe(CO)_4]$ (**1**) with phenyldichlorophosphine leads quantitatively either to the stable secondary halophosphine complex $PhP(H)ClFe(CO)_4$ (**2b**), or to the first nonhindered side-on and end-on diphosphene complex $[Fe(CO)_4[\mu\text{-}Fe(CO)_4](PPh)_2]$ (**5**), or to the first trimetallic anionic diphosphane species $[Ph_4P][Fe(CO)_4]_3P_2Ph_2H]$ (**7**). X-ray diffraction study confirms the structure of **5**. Addition of $[Et_4N][HW(CO)_5]$ to the diphosphene complex **5** affords another mixed trimetallic anionic diphosphane compound $[Et_4N][[Fe(CO)_4]_2W(CO)_5P_2Ph_2H]$ (**8**).

Recently, a number of publications described the reactivity of carbonylmetalate dianions $Na_2[M_2(CO)_{10}]$ (M = Cr, Mo, or W) or $Na_2Fe(CO)_4$ with $RPCl_2$ leading to a variety of phosphinidene, $[(CO)_5M]_2PR$, or diphosphene complexes, $[(CO)_5M]_nRP{=}PR$ (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 $[PPh_4][HFe(CO)_4]$ (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 RP(H)ClFe-

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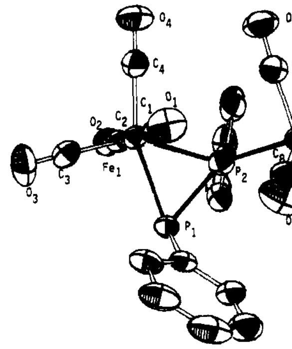


Figure 1. ORTEP view of $[Fe(CO)_4[\mu\text{-}Fe(CO)_4](PPh)_2$ (**5**). Bond distances (Å) and angles (deg) of interest: Fe(1)–P(1), 2.389 (1); Fe(1)–P(2), 2.354 (1); Fe(2)–P(2), 2.267 (1); P(1)–P(2), 2.139 (2); P(1)–Fe(1)–P(2), 53.60 (4); Fe(1)–P(1)–P(2), 62.36 (4), Fe(1)–P-(2)–Fe(2), 127.72 (5); Fe(1)–P(2)–P(1), 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 $[Ph_4P][[(CO)_4Fe]_3P_2Ph_2H]$ (**7**) and $[Et_4N][[(CO)_4Fe]_2W(CO)_5P_2Ph_2H]$ (**8**).

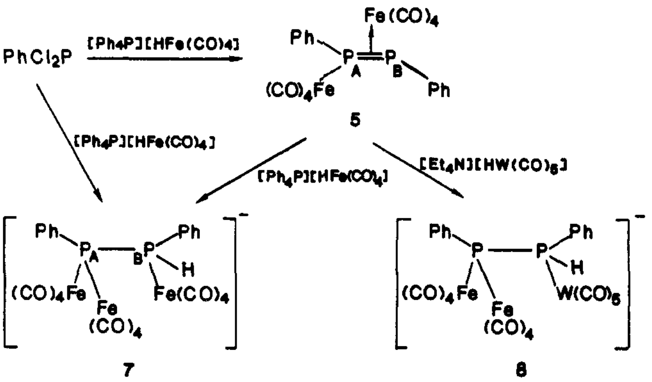
When a solution of the hydride $[Ph_4P][HFe(CO)_4]$ (**1**) in dichloromethane and a solution of dichlorophosphine in dichloromethane are added simultaneously, drop by drop, at room temperature, stable complexes of secondary halophosphines **2**[3] are formed quantitatively.

$$RPCl_2 + \underset{\mathbf{1}}{[Ph_4P][HFe(CO)_4]} \xrightarrow[-Ph_4PCl]{} \underset{\mathbf{2a\text{–}d}}{RP(H)ClFe(CO)_4}$$

a, R = $N(i\text{-}Pr)_2$; **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 $MePCl_2$[4] and of $PhP(H)ClW(CO)_5$ (**3**) from $PhPCl_2$,[5] both involve five steps, while that of **2a** necessitates three steps.[6]

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 $PhPCl_2$ is reacted with $[Et_4N][HW(CO)_5]$ (**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 $[Fe(CO)_4[\mu\text{-}Fe(CO)_4](PPh)_2]$ (**5**; mp 113 °C dec; yield 65%).[8]

The ^{31}P 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–g] and yet does not seem in agreement with those found for $[Fe(CO)_4[\mu\text{-}Fe(CO)_4](P\text{-}2,4,6\text{-}t\text{-}Bu_3C_6H_2O)_2]$ (**6**; $\delta(^{31}P)$ 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 $Na_2Fe(CO)_4$ with the sterically hindered dichlorophosphine $Cl_2P(2,4,6\text{-}t\text{-}Bu_3C_6H_2O)$.[1b]

Therefore, the structure of **5** was determined by single-crystal X-ray diffraction[9] 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]

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[3] **2a**: oil; ^{31}P NMR (CH_2Cl_2) δ 112.9 (dt, $^1J_{PH}$ = 448 Hz, $^3J_{PH}$ = 14.8 Hz); ^1H NMR δ 0.90 (d, J = 14 Hz, CH_3), 3.50 (mult, CH), 7.97 (d, $J.$ = 447.7 Hz, PH); IR ν(CO) (hexane) 2073 (m), 1993 (m), 1965 (s), 1954 (s) cm^{-1}. **2b**: oil; ^{31}P NMR (C_6D_6) δ 154.7 (d, $^1J_{PH}$ = 391 Hz); ^1H NMR (C_6D_6) 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; ^{31}P NMR (CH_2Cl_2) δ 156.3 (d of dec, $^1J_{PH}$ = 368 Hz, $^3J_{PH}$ = 20 Hz); ^1H NMR (C_6D_6) 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; ^{31}P NMR (CH_2Cl_2) δ 114.2 (d of q, $^1J_{PH}$ = 395 Hz, $^2J_{PH}$ = 8.8 Hz); ^1H NMR (C_6D_6) 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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[7] **3**: mp 55 °C; yield 30%. **3** was previously prepared by a five-step synthesis.[5]

[8] **5**: ^1H NMR (CD_2Cl_2) 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 $\mathbf{5}\cdot 0.5CH_2Cl_2$ was grown from dichloromethane/hexane at −20 °C.

[9] A yellow crystal of $\mathbf{5}\cdot 0.5CH_2Cl_2$ (dimensions 0.3 × 0.3 × 0.12 mm) was selected: Mo Kα radiation (λ = 0.71069 Å); T = 293 K; triclinic $P\bar{1}$, 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**: $^{31}P\{^1H\}$ NMR (CD_2Cl_2) $\delta(P_A)$ 91.15 (d), $\delta(P_B)$ 42.71 (d, $^1J_{PP}$ = 234.8 Hz, AB system), $\delta(P_C)$ 22.01; ^{31}P NMR (CD_2Cl_2) $\delta(P_A)$ 91.15 (d), $\delta(P_B)$ 46.32 and 39.10 (d of d, $^1J_{PH}$ = 342 Hz, $^1J_{PP}$ = 234.8 Hz), $\delta(P_C)$ 22.01 (m); ^1H NMR (CD_2Cl_2) δ 4.38 (d, $^1J_{PH}$ = 342 Hz, H), 5.94, 6.34 (m, Ph); IR ν(CO) (CH_2Cl_2) 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 ^{1}H 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), $J_{P_AP_B} = 220$ 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, $J_{P_AP_B} = 220$ 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, $J_{^1H-^{183}W} = 22.7$ Hz), 7.35 and 7.66 (m, Ph); IR ν(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-Jérome, 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 $[Li(12\text{-}crown\text{-}4)_2][CH_2C_6H_2(3,5\text{-}Me_2)(4\text{-}B\{2,4,6\text{-}Me_3C_6H_2\}_2)]\cdot Et_2O$: Evidence for "Boron Ylide" Character

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Received April 23, 1986

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*-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 drawn to the report[2] of Ramsey and Isabelle, who showed (NMR) that by treating $BMes_3$ (Mes = mesityl, 2,4,6-$Me_3C_6H_2$) with *n*-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 $BMes_3$ 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.

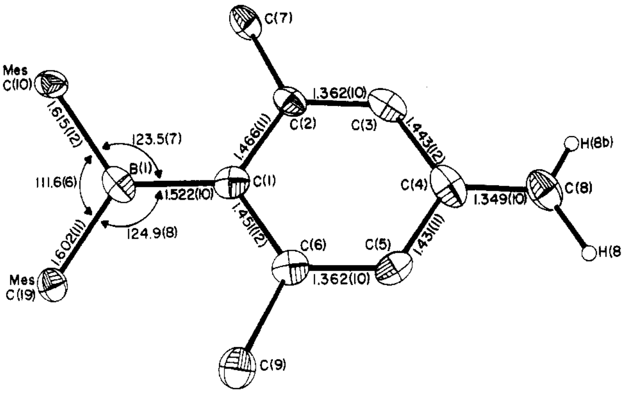

Figure 1. Important bond distances and angles for the anion of 1.

The anion **1** was crystallized as its $[Li(12\text{-}crown\text{-}4)_2]^+$ salt by the addition of 2 equiv of 12-crown-4[3] 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) Å^3, $D_{calcd} = 1.18$ 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*-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 Kow[4] 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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