pattern of eight lines and a secondary pattern of two distinct multiplets. The former could be obtained free of the second isomer on crystallization using heptane. The pure isomer of $Fe_2(CO)_6(CH_3PH)_2$ has no P–H resonance in the nmr, and it has one type of methyl group; hence it must be one of the C_{2v} isomers. The explanation of the eight-line pattern from this latter resonance is unclear.

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Deprotonation and Subsequent Alkylation of Phosphine–Metal Carbonyl Complexes

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Deprotonations of several (secondary phosphine)metal carbonyls ($CH_3C_5H_4Mn(CO)_2L$, $Cr(CO)_5L$, $Mo(CO)_5L$, $W(CO)_5L$, $Mo(CO)_4L_2$, $Fe(CO)_4L$ ($L = P(C_6H_5)_2H$); $Fe(CO)_4P(C_6H_5)(CH_3)H$) with *n*-butyllithium have been carried out; the resulting lithio compounds react *in situ* with methyl iodide to yield appropriate methylphenylphosphinemetal complexes. The complex $Fe_2(CO)_6(C_6H_5PH)_2$ also deprotonates under the same conditions to give a dilithio species, which reacts as expected with methyl iodide and 1,3-dibromopropane.

Introduction

We have described reactions of several primary and secondary phosphines with carbonyls of iron[2] and of other metals.[3] A number of complexes were prepared in these studies, including simple products of substitution of a phosphine for a carbonyl. As a major part of this study we hoped to investigate the reactivity accorded such species through the presence of the P–H bond in the complex.

One particular reaction appeared rather promising. We reasoned that since hydrogen in the free ligand was reasonably acidic, it should retain this acid character when complexed to a metal; presumably with an appropriate base the proton could be removed. The data presented here indicate that this is so.

In one previous instance, an analogous deprotonation reaction sequence was carried out. Stone and coworkers,[4] investigating various pentafluorophenylphosphinemetal complexes, prepared $Cr(CO)_5[P(C_6F_5)_2H]$ and the molybdenum analog of this complex. Addition of *n*-butyllithium gave the isolable species $M(CO)_5[P(C_6F_5)_2]Li$ (M = Cr, Mo). Reactions with hydrogen chloride in dichloromethane, with methyl iodide, and with $(C_6F_5)_2PCl$ gave only small quantities of the expected products, however, and these authors felt that this was not a synthetic method of any particular promise so the studies were not extended further.

Experimental Section

The preparations of $Cr(CO)_5[P(C_6H_5)_2H]$, $Mo(CO)_5[P(C_6H_5)_2H]$, $W(CO)_5[P(C_6H_5)_2H]$, and $Mo(CO)_4[P(C_6H_5)_2H]_2$ are described in the literature.[5] We described the preparations of $Fe(CO)_4[P(C_6H_5)_2H]$,[2] $Fe(CO)_4[P(C_6H_5)(CH_3)H]$,[2] $Fe_2(CO)_6(C_6H_5PH)_2$,[2] and $CH_3C_5H_4Mn(CO)_2[P(C_6H_5)_2H]$[2] earlier. *n*-Butyllithium was a commercial sample and was standardized by established methods.[6] Other reagents were commercial samples.

All reactions were carried out under a nitrogen atmosphere.

Infrared spectra were obtained on a Beckman IR-10 spectrometer; nmr data were obtained on Varian A-60A and T-60 instruments. These data are presented in Table III.

Deprotonation and Alkylation of Secondary Phosphine–Metal Carbonyl Complexes. General Procedure.—The indicated quantity of the phosphine–metal complex was dissolved in 50 ml of tetrahydrofuran at room temperature and deprotonated using an equimolar quantity of standardized *n*-butyllithium in hexane. After *ca.* 10 min, a large excess (5 ml) of methyl iodide was added. Stirring was continued for 30 min; then the solvent was removed and the residue extracted with 1:1 hexane–chloroform. The resulting solution was chromatographed on alumina using the same solvent mixture. In each case one band eluted. The residue left on evaporation of the solvent was recrystallized from heptane.

Data for these reactions and for the products obtained are given in Table I. Routine infrared and nmr data on the products of these reactions are recorded in Table III.

Infrared Study on the Deprotonation Reactions.—A sample of the starting material was dissolved in tetrahydrofuran and the infrared spectrum (ν_{CO}) recorded under high resolution. The equivalent amount of standard butyllithium was added and the spectrum of the lithium complex recorded. Data are given below (Table II).

It might be noted that addition of chloroform to this solution apparently destroyed the lithio complex almost instantly. The bands appropriate to this complex were lost, and new ν_{CO} bands were observed. The latter did not appear to correspond with the starting material, $M(CO)_x(PR_2H)$. Extensive decomposition was noted visually. This reaction is under study at this time.

Proton-Exchange Reactions as an Indication of Acid Strengths.—(1) A sample of $P(C_6H_5)_2H$ (0.383 g, 206 mmol) in 100 ml of tetrahydrofuran was deprotonated with 1.53 ml of 1.355 *M* *n*-butyllithium. Then 0.7265 g of $Fe(CO)_4[P(C_6H_5)_2H]$ (2.06 mmol) was added. The infrared spectrum showed peaks at 2000 and 1893 cm^{-1}, indicating that only $Fe(CO)_4[P(C_6H_5)_2]Li$ was present.

(2) A 0.7309-g sample of $Fe(CO)_4[P(C_6H_5)_2H]$ (2.06 mmol) in 100 ml of tetrahydrofuran was deprotonated with 1.52 ml of 1.355 *M* *n*-butyllithium. Then 0.383 g (2.06 mmol) of $P(C_6H_5)_2H$ was added. An infrared spectrum showed peaks at 2000 and 1893 cm^{-1}, characteristic of $Fe(CO)_4[P(C_6H_5)_2]Li$.

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[1] National Science Foundation Predoctoral Fellow.

[2] P. M. Treichel, W. K. Dean, and W. M. Douglas, *Inorg. Chem.*, **11**, 1609 (1972).

[3] P. M. Treichel, W. K. Dean, and W. M. Douglas, *J. Organometal. Chem.*, in press.

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[5] J. G. Smith and D. T. Thompson, *ibid.*, 1694 (1967).

[6] H. Gilman and F. K. Cartledge, *J. Organometal. Chem.*, **2**, 447 (1964).

TABLE I

DATA ON DEPROTONATION METHYLATIONS OF (SECONDARY PHOSPHINE)METAL CARBONYLS

Reactants	Products	Yield, g (%)	Other product data[a]
$\mathrm{Cr(CO)_5[P(C_6H_5)_2H]}$ (1.15 g, 3.02 mmol)	$\mathrm{Cr(CO)_5[P(C_6H_5)_2CH_3]}$	0.79 (67)	White crystals, mp 89–90°, lit.[b] mp 90–92°
$\mathrm{Mo(CO)_5[P(C_6H_5)_2H]}$ (1.34 g, 3.16 mmol)	$\mathrm{Mo(CO)_5[P(C_6H_5)_2CH_3]}$	0.72 (52)	White crystals, mp 78–80°, lit.[b] mp 87–88°
$\mathrm{W(CO)_5[P(C_6H_5)_2H]}$ (1.44 g, 2.88 mmol)	$\mathrm{W(CO)_5[P(C_6H_5)_2CH_3]}$	0.98 (67)	White crystals, mp 100–101°, lit.[b] mp 98–99°
cis-$\mathrm{Mo(CO)_4[P(C_6H_5)_2H]_2}$ (1.92 g, 3.33 mmol)	*cis*-$\mathrm{Mo(CO)_4[P(C_6H_5)_2CH_3]_2}$	1.73 (85)	Light tan crystals, mp 124–125°. *Anal.* Calcd: C, 41.2; H, 2.48; mol wt 524. Found: C, 41.5; H, 2.38; mol wt 514
$\mathrm{Fe(CO)_4[P(C_6H_5)_2H]}$ (1.31 g, 3.70 mmol)	$\mathrm{Fe(CO)_4[P(C_6H_5)_2CH_3]}$	0.75 (55)	Yellow oil, vacuum distilled at 110° (0.05 mm). *Anal.* Calcd: C, 55.4; H, 3.53; mol wt 368. Found: C, 55.6; H, 3.39; mol wt 373
$\mathrm{Fe(CO)_4[P(C_6H_5)(CH_3)H]}$ (1.43 g, 4.42 mmol)	$\mathrm{Fe(CO)_4[PC_6H_5(CH_3)_2]}$	0.63 (40)	Yellow crystals, mp 48–50°, subl pt 80° (0.02 mm). *Anal.* Calcd: C, 47.0; H, 3.60; mol wt 306. Found: C, 46.4; H, 3.97; mol wt 306
$\mathrm{CH_3C_5H_4Mn(CO)_2[P(C_6H_5)_2H]}$ (1.01 g, 2.57 mmol)	$\mathrm{CH_3C_5H_4Mn(CO)_2[P(C_6H_5)_2CH_3]}$	0.30 (30)	Yellow crystals, mp 55–57°. *Anal.* Calcd: C, 64.4; H, 5.13; mol wt 390. Found: C, 64.8; H, 5.33; mol wt 382

[a] Molecular weights were determined osmometrically. [b] S. O. Grim, D. A. Wheatland, and W. McFarlane, *J. Amer. Chem. Soc.*, **89,** 5573 (1967).

TABLE II

INFRARED DATA ON PROTONATED AND LITHIO COMPLEXES[a]

Compd	$L = P(C_6H_5)_2H$	$L = P(C_6H_5)_2Li$
$\mathrm{Cr(CO)_5L}$	2066 m, 2044 s, b	2023 m, 1925 s, b
$\mathrm{Mo(CO)_5L}$	2076 m, 1949 s, b	2040 m, 1914 s, b
$\mathrm{W(CO)_5L}$	2076 m, 1941 s, b	2042 m, 1907 s, b
$\mathrm{Fe(CO)_4L}$	2054, 1976, 1945 s, b	2000, 1907 sh, 1893 b

[a] Tetrahydrofuran solution; Perkin-Elmer 421 (see text).

Reaction of $\mathrm{Fe_2(CO)_6(C_6H_5PH)_2}$ and Methyllithium.—Solid $\mathrm{Fe_2(CO)_6(C_6H_5PH)_2}$ (1.5 g, 3.0 mmol) was slurried in 50 ml of dry tetrahydrofuran in a 100-ml round-bottom flask. Using a nitrogen atmosphere and rapid stirring, 4.0 ml of 1.5 *M* $\mathrm{CH_3Li}$ in ethyl ether (6 mmol) was added slowly by syringe. The stirring was continued several minutes until gas evolution ($\mathrm{CH_4}$) ceased. The solution of $\mathrm{Fe_2(CO)_6(C_6H_5PLi)_2}$ was used directly in the subsequent reactions and was not characterized. However, the infrared spectrum of this solution was recorded. $\mathrm{Fe_2(CO)_6(C_6H_5PLi)_2}$: ν_{CO} 2010 s, 1970 s, 1940 s, 1920 s, 1890 sh, 1850 sh cm^{-1}; no ν_{P-H} (THF solution). [*Cf.* $\mathrm{Fe_2(CO)_6(C_6H_5PH)_2}$: ν_{CO} 2074 s, 2014 s, 1980 s, b; ν_{P-H} 2320 cm^{-1} ($\mathrm{CH_2Cl_2}$ solution).] The infrared studies using a deficiency of methyllithium showed the solution contained only $\mathrm{Fe_2(CO)_6(C_6H_5PH)_2}$ and $\mathrm{Fe_2(CO)_6(C_6H_5PLi)_2}$.

Reactions of $\mathrm{Fe_2(CO)_6(C_6H_5PLi)_2}$ and Alkyl Halides. (a) **With Methyl Iodide.**—To the solution prepared as above was added 10.8 g of $\mathrm{CH_3I}$ (7.6 mmol). The reaction mixture was stirred for 15 min and evaporated to dryness using a water aspirator, followed by pumping with a mechanical pump (~0.1 mm). The solid residue was extracted with dichloromethane and filtered. About 20 ml of sand was added to the filtrate and the solvent was removed by a water aspirator and finally by vacuum (27°, 0.1 mm). This mixture was then placed on an alumina column (15 × 50 cm) and eluted with 1:2 benzene–hexane. A single yellow band eluted. After evaporation the product $\mathrm{Fe_2(CO)_6(C_6H_5PCH_3)_2}$ could be purified by crystallization from acetone–water or by sublimation to give yellow crystals, 0.6 g (38%); mp 115–130° (mixture of isomers). *Anal.* Calcd for $\mathrm{C_{20}H_{16}P_2Fe_2O_6}$: C, 45.60; H, 3.09; P, 11.79; mol wt 526. Found: C, 45.74; H, 3.14; P, 11.61; mol wt 526 (mass spectrometric).

The product obtained was a mixture of two geometric isomers. Partial separation was accomplished by very slow sublimation at 80° (0.01 mm). The proton nmr was a key tool in this study (see Figure 1). The less symmetric isomer B was slightly more volatile than the symmetric isomer A. After about 90% of the solid had been sublimed, the remainder of the product was almost pure A.

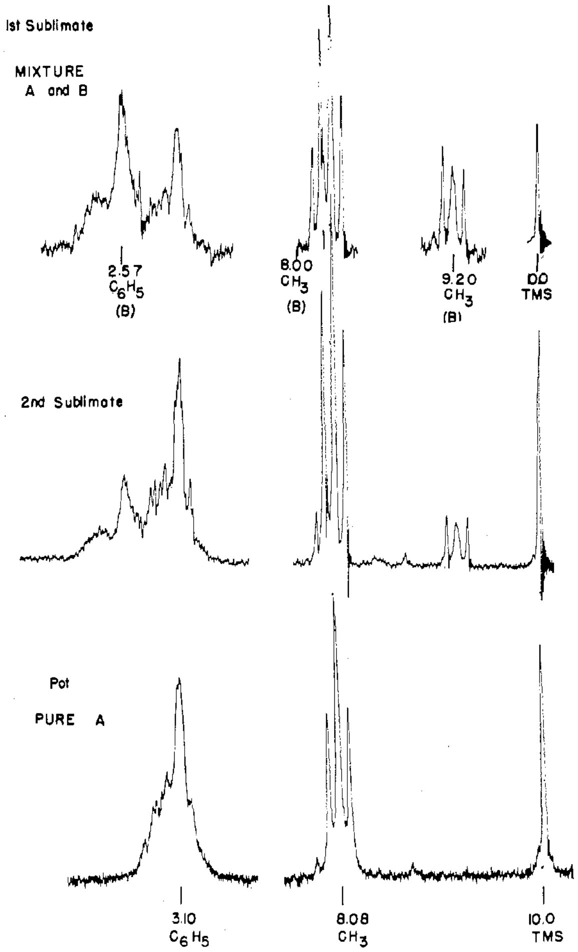

Figure 1.—Nmr spectra of fractions in fractional sublimation of $\mathrm{Fe_2(CO)_6(C_6H_5PCH_3)_2}$.

The same degree of separation could be accomplished from a careful fractional crystallization from heptane. The symmetric

TABLE III

SPECTROSCOPIC DATA ON NEW COMPOUNDS

Compd	Infrared[a]	Nmr[b]
$Mo(CO)_4[P(C_6H_5)_2CH_3]_2$	ν_{CO} 2025 m, 1928 m, 1904 s	2.67, multiplet, C_6H_5; 8.23 AA′ pattern, unresolved $-CH_3$
$Fe(CO)_4[P(C_6H_5)_2CH_3]$	ν_{CO} 2055.5 s, 1980 m, 1940 vs, b	2.52, multiplet, C_6H_5; 7.85, doublet, J_{P-CH_3} = 9.8 Hz, CH_3
$Fe(CO)_4[P(C_6H_5)(CH_3)_2]$	ν_{CO} 2055 s, 2018 w, 1979 s, 1939 vs, b	2.52 multiplet, C_6H_5; 8.12, doublet, J_{P-CH_3} = 10.4, CH_3
$CH_3C_5H_4Mn(CO)_2[P(C_6H_5)_2CH_3]$	ν_{CO} 1937 s, 1876 s	2.62, multiplet, C_6H_5; 5.84, singlet, C_5H_4; 8.13, unresolved, both CH_3 groups
$Fe_2(CO)_6(C_6H_5PCH_3)_2$ (sym)	2055 m, 2017 s, 1996 m, 1968 m, 1960 w	3.10, multiplet, C_6H_5; 8.08, triplet, $J_{P_2-CH_3}$ = 6 Hz, CH_3
$Fe_2(CO)_6(C_6H_5PCH_3)_2$ (asym)	2056.5 m, 2017.5 s, 1998.5 m, 1967 m	2.57, 3.10, multiplets, C_6H_5; 8.00, triplet, $J_{P_2-CH_3}$ = 6 Hz, CH_3; 9.20, triplet, $J_{P_2-CH_3'}$ = 6.0, CH_3'
$Fe_2(CO)_6[C_6H_5P(CH_2)_3PC_6H_5]$	2070 m, 2019 s, 1996 m, 1967 m, 1958 w	3.00, multiplet, C_6H_5; 8.30, broad, singlet, $-CH_2-$

[a] ν_{CO}: heptane solutions; Perkin-Elmer 421. Values are accurate to ±1 cm^{-1}. [b] τ values *vs.* TMS internal standard (τ 10.00); $CDCl_3$ solutions.

isomer, A, was less soluble and could be obtained in almost pure form; mp 170.5–172°. It was not possible by either crystallization or sublimation to get a pure sample of the unsymmetric isomer.

(b) **Reaction with $Br(CH_2)_3Br$.**—This reaction was run as above. From 6.3 g of $Br(CH_2)_3Br$ (3.1 mmol) there was obtained $Fe_2(CO)_6[C_6H_5P(CH_2)_3PC_6H_5]$, 0.7 g (44%), as yellow crystals recrystallized from dichloromethane–hexane; mp 184–187°. *Anal.* Calcd for $C_{21}H_{16}P_2Fe_2O_6$: C, 46.7; H, 2.97; P, 11.5; mol wt 538. Found: C, 46.8; H, 3.06; P, 11.7; mol wt 538 (mass spectrometric).

Spectroscopic data on the new compounds reported here are summarized in Table III.

Results and Discussion

Phosphines are not particularly strong acids, but on treatment with suitable bases they can furnish one or more protons, forming phosphide ions. A rough estimate of the acidity of phosphine, PH_3, in water has been determined; the pK_a value was found to be about 28.[7] This value is in accord with the observation that the amide ion NH_2^- (NH_3 has a pK_a of 35) is a sufficiently strong base to react completely with phosphine giving PH_2^- and ammonia. Stronger bases than the amide ion, such as organolithium reagents and Grignard reagents, will deprotonate phosphines; this route is commonly used to generate phosphide anions as synthetic intermediates.[8]

Substitution of phenyl groups for hydrogen in phosphine will presumably cause the remaining protons to be more acidic, since the resultant negative charge can be somewhat delocalized. The same comparison can be made between ammonia and aniline (pK_a = 27).

The effect of coordination to a metal on the ability of a phosphine to donate a proton has not previously been evaluated. However, the possibility that the acidity would be diminished substantially seems unlikely. Thus the use of organolithium reagents, ordinarily used to deprotonate the phosphines, seems reasonable. Of course there is an added advantage of convenience with use of these reagents.

When various secondary phosphine metal complexes are treated with *n*-butyllithium or methyllithium, an immediate reaction occurs. The reaction can be followed by color change, by evolution of gas (CH_4 or C_4H_{10}), or by the infrared spectrum in the carbonyl region. The resulting solution of the lithio salt is thermally stable, but of course it is reactive toward protonic reagents such as water; the solution is also air sensitive. The lithio complexes react cleanly with methyl iodide to give appropriate methylphosphine-metal complexes in good yield. Reaction data are summarized in the equations

$$M(CO)_5[P(C_6H_5)_2H] \xrightarrow[THF]{BuLi} [M(CO)_5[P(C_6H_5)_2]Li] \xrightarrow{CH_3I} M(CO)_5[P(C_6H_5)_2CH_3] \quad (1)$$

$$M = Cr\ (67\%)$$
$$M = Mo\ (52\%)$$
$$M = W\ (67\%)$$

$$Mo(CO)_4[P(C_6H_5)_2H]_2 \xrightarrow[THF]{2BuLi} [Mo(CO)_4[P(C_6H_5)_2Li]_2] \xrightarrow{CH_3I} Mo(CO)_4[P(C_6H_5)_2CH_3]_2\ (85\%) \quad (2)$$

$$Fe(CO)_4[P(C_6H_5)(R)H] \xrightarrow[THF]{BuLi} [Fe(CO)_4[P(C_6H_5)R]Li] \xrightarrow{CH_3I} Fe(CO)_4[P(C_6H_5)(R)CH_3] \quad (3)$$

$$R = C_6H_5\ (55\%)$$
$$R = CH_3\ (40\%)$$

$$CH_3C_5H_4Mn(CO)_2[P(C_6H_5)_2H] \xrightarrow[THF]{BuLi} [CH_3C_5H_4Mn(CO)_2[P(C_6H_5)_2]Li] \xrightarrow{CH_3I} CH_3C_5H_4Mn(CO)_2[P(C_6H_5)_2CH_3]\ (30\%) \quad (4)$$

The yields observed in these reactions suggest that this route is a useful one, in contrast to the previous report.[4]

The deprotonation reaction could be followed by monitoring the infrared spectrum of the reaction mixture in tetrahydrofuran. For example, the infrared spectrum of $Cr(CO)_5[P(C_6H_5)_2H]$ in tetrahydrofuran shows peaks at 2066 m and 1944 s, b cm^{-1}.[9] On addition of 1 equiv of butyllithium, these bands are shifted to lower frequency, occurring at 2023 m and 1925 s, b cm^{-1}. The same shift, of about 30 cm^{-1}, was observed to occur with the other deprotonation studied (Table II). The lower ν_{CO} frequencies reaffirm the earlier suggestion that deprotonation will lead to a higher negative charge on the metal.

Stone and coworkers[4] reported that the ν_{CO} for $M(CO)_5[P(C_6F_5)_2H]$ and $Mo(CO)_5[P(C_6F_5)_2Li]$ are about

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[7] W. Jolly, "The Chemistry of the Non-Metals," Prentice-Hall, Englewood Cliffs, N. J., 1968.

[8] L. Maier, *Progr. Inorg. Chem.*, **5**, 27 (1963).

[9] In hexane there are three major bands[5] at 2067 m, 1953 s, and 1945 s cm^{-1}, appropriate to $LM(CO)_5$ complexes. The bands in tetrahydrofuran are broader, and the latter two do not resolve in this solvent.

the same. This seems rather unlikely in view of our results. Their products were isolated using chloroform for recrystallizations and as an infrared solvent. We found, however, that addition of chloroform to the lithio complexes in tetrahydrofuran resulted in their immediate destruction. In view of this we suspect that their infrared data and also their very low yield data may be regarded with some scepticism.

In order qualitatively to assess the acidity of a phosphine complexed to a metal the competitive reactions (5) and (6) were run. These reactions could be monitored by infrared spectroscopy. The results, as indicated in (5) and (6), show that $P(C_6H_5)_2^-$ is a stronger base than $Fe(CO)_4P(C_6H_5)_2^-$ (by more than an order of magnitude) as no $Fe(CO)_4[P(C_6H_5)_2H]$ was detected in the solution. Hence we conclude that the acidity of a phosphine increases on coordination; $Fe(CO)_4[P(C_6H_5)_2H]$ is a stronger acid than $P(C_6H_5)_2H$. This result can be explained if it is assumed that in the complex the negative charge resulting upon deprotonation can be delocalized to the metal and other ligands. The same argument was applied in comparisons of ammonia and aniline and of phosphine and phenylphosphine.

$$Fe(CO)_4[P(C_6H_5)_2H] + P(C_6H_5)_2Li \longrightarrow Fe(CO)_4[P(C_6H_5)_2]Li + P(C_6H_5)_2H \quad (5)$$

$$Fe(CO)_4[P(C_6H_5)_2]Li + P(C_6H_5)_2H \longrightarrow \text{no reaction} \quad (6)$$

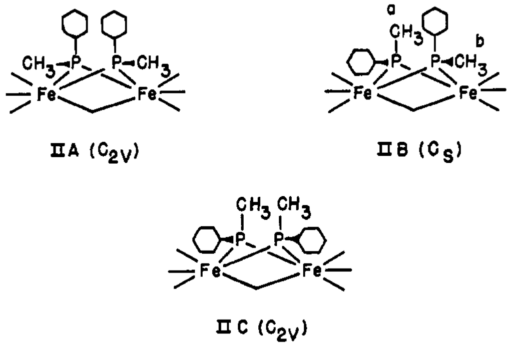

Figure 2.—The three possible isomers of $Fe_2(CO)_6(C_6H_5PCH_3)_2$.

We also observed deprotonation of the complex $Fe_2(CO)_6(C_6H_5PH)_2$ by methyllithium. The lithio species in solution shows the analogous ν_{CO} shift to lower frequencies. This reagent was used *in situ* for reactions with 1,3-dibromopropane and methyl iodide. In the former reaction the product $Fe_2(CO)_6[C_6H_5P(CH_2)_3PC_6H_5]$ forms in good yield. Models show that the trimethylene group $-CH_2CH_2CH_2-$ is of ideal size to bridge the two phosphorus atoms. The methyl iodide reaction gives two isomeric products. One of the two isomers could be obtained pure, either by careful sublimation or by fractional crystallization from heptane. The isomer so obtained is one of the symmetric (C_{2v}) isomers; thus in its nmr spectrum there is only one methyl resonance which appears as a triplet, due to equivalent coupling of methyl protons to both phosphorus atoms. The lower volatility and solubility of this isomer, when compared to the second isomer, are also in accord with this formulation. Subsequent to our work, the isomer was identified by X-ray crystallography as IIA.[10]

The second isomer, a less symmetric (C_s symmetry) compound, IIB, could not be completely separated from this symmetric isomer. However, the nmr spectral parameters for this isomer could be ascertained from a spectrum of a mixture of the isomers. Two methyl resonances were detected in the nmr, the lower field resonance overlapping the triplet from the symmetric complex. Both methyl resonances were triplets. The similarity in position of the low-field resonances suggests that this is probably the CH_3^a group, and the higher field absorption would then be due to CH_3^b (see IIB). Although these isomers differ in overall symmetry, there is very little, if any, difference in site symmetry at the metal, and moreover there should be no important electronic differences between the possible isomers. Hence it is not surprising to find that differentiation of isomers by carbonyl stretching frequencies is difficult or impossible. A comparison of the patterns of ν_{CO} for the two isomers of $Fe_2(CO)_6(C_6H_5PCH_3)_2$ showed positional differences of only 1–2 cm^{-1}, and no differences in intensity were obvious from the high-resolution spectra.

The stereochemistry of the starting material $Fe_2(CO)_6(C_6H_5PH)_2$ is not known,[2] nor is it apparent whether there is more than one isomer present. Thus, we do not know anything about the stereochemistry of the lithiation–alkylation sequence. It is appropriate to observe that the three products (the two products from methyl iodide and the product from 1,3-dibromopropane) have the alkyl groups substituted in different positions.

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(10) L. F. Dahl and J. J. Huntsman, submitted for publication.