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Dialkylaminophosphorus metal carbonyls

VII *. Trinuclear iron carbonyl derivatives from reactions of disodium octacarbonyldiferrate with (dialkylamino)dichlorophosphines **

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Abstract

Reactions of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2 (R = Me, Et, $CHMe_2$ or cyclohexyl (Cx); R_2N = piperidino, 2,6-dimethylpiperidino or 2,2,6,6-tetramethylpiperidino) give orange $[(R_2NP)_2Fe_3(CO)_9]$ and dark purple $[(R_2NP)Fe_3(CO)_{10}]$ as the only hexane-soluble iron carbonyl derivatives. No evidence was obtained for the formation of any $[\{(R_2NP)_2CO\}Fe_2(CO)_6]$ or $[(R_2NP)_3Fe_2(CO)_6]$ derivatives in significant quantities.

Introduction

Reactions of $Na_2[Fe(CO)_4]$ with R_2NPCl_2 have been shown [2–5] to be a useful source of interesting (dialkylamino)phosphorus iron carbonyl derivatives. Thus reactions of $Na_2[Fe(CO)_4]$ with $(CHMe_2)_2NPCl_2$ in diethyl ether, and in tetrahydrofuran provide efficient syntheses of the phosphorus-bridging carbonyl derivative $[\{(R_2NP)_2CO\}Fe_2(CO)_6]$ (**1**; R = $CHMe_2$) and the triphosphine derivative $[(R_2NP)_3Fe_2(CO)_6]$ (**2**; R = $CHMe_2$), respectively [2,3,5,6]. Furthermore, the courses of reactions of $Na_2[Fe(CO)_4]$ with R_2NPCl_2 derivatives having a phosphorus–nitrogen bond were found to be significantly different from reactions of $Na_2[Fe(CO)_4]$ with $RPCl_2$ derivatives having a phosphorus–carbon bond, which in many cases give diphospheneiron carbonyl complexes [7,8].

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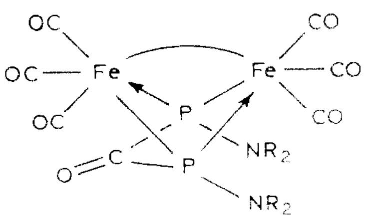
* Part VI of this series is given in ref. 1

** This paper is dedicated to Prof. Colin Eaborn in recognition of his important contributions in the field of organometallic chemistry.



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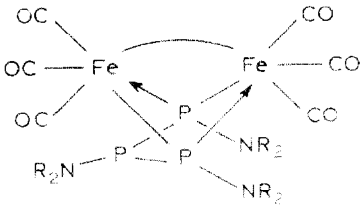

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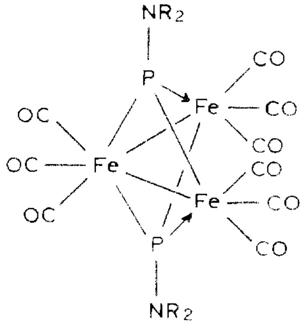

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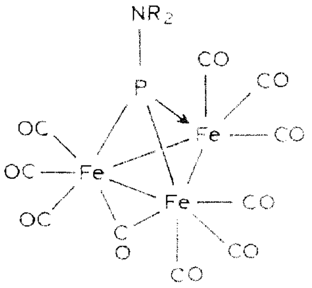

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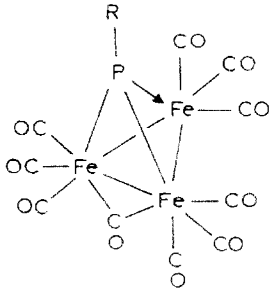

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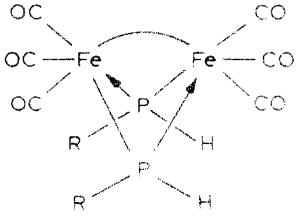

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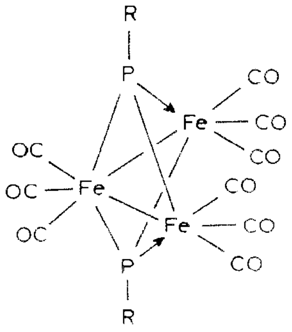

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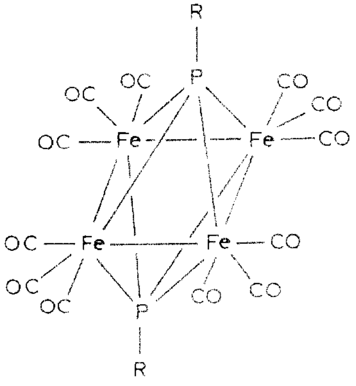

(**8**)

This paper summarizes our attempts to extend such studies to reactions of the binuclear iron carbonyl anion, $[Fe_2(CO)_8]^{2-}$ [9,10], with R_2NPCl_2. Such reactions have been found to provide an alternative synthesis of $[(R_2NP)_2Fe_3(CO)_9]$ (**3**), as well as a source of $[(R_2NP)Fe_3(CO)_{10}]$ (**4**), which cannot be obtained from reactions of $Na_2[Fe(CO)_4]$ with R_2NPCl_2 [5].

A number of observations related to those in this paper have been made by other workers. Thus Lang, Zsolnai, and Huttner [11] report the reaction of either $Na_2[Fe(CO)_4]$ or $Na_2[Fe_2(CO)_8]$ with 2,4,6-$Me_3C_6H_2PCl_2$ in tetrahydrofuran to give $[(RP)Fe_3(CO)_{10}]$ (**5**; R = 2,4,6-$Me_3C_6H_2$), with significantly higher yields being obtained with the binuclear iron carbonyl anion. In addition De and Vahrenkamp [12] have reported that reactions of $[Et_4N]_2[Fe_2(CO)_8]$ with $RPCl_2$ (R = Me, Ph or 4-MeC_6H_4) give $[(RPH)_2Fe_2(CO)_6]$ (**6**), $[(RP)_2Fe_3(CO)_9]$ (**7**), and $[(RP)_2Fe_4(CO)_{12}]$ (**8**). A relatively early report [13] of the reaction of $Na_2[Fe(CO)_4]$ with Et_2NPCl_2 in tetrahydrofuran to give the trinuclear complex $[(Et_2NP)_2Fe_3(CO)_9]$ (**3**; R = Et) may actually involve the salt $Na_2[Fe_2(CO)_8]$, since the $Na_2[Fe(CO)_4]$ actually used for that work was obtained by reaction of sodium metal with an excess of $[Fe(CO)_5]$ under conditions similar to those subsequently [9] found to be effective for the preparation of $Na_2[Fe_2(CO)_8]$.

Experimental

The general experimental procedure is similar to that described in previous papers of this series [5]. The reagents $Na_2[Fe_2(CO)_8]$ [10] and R_2NPCl_2 [14] were prepared by the cited procedures.

General procedure for the reactions of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2 derivatives

An orange slurry of freshly prepared $Na_2[Fe_2(CO)_8]$ (12–72 g) in dry ethereal solvent (300–450 ml diethyl ether or tetrahydrofuran) was treated at −78 °C, with vigorous stirring, with an equimolar amount of R_2NPCl_2 dissolved in the same solvent (100–150 ml). The resulting mixture was allowed to warm overnight to room temperature with vigorous stirring. Solvent was then removed from the dark brown reaction mixture at 25 °C/25 Torr leaving a black solid. ^{31}P NMR spectra of these black solids in $CDCl_3$ generally indicated than to be a mixture of several components. The trinuclear derivatives $[(R_2NP)_2Fe_3(CO)_9]$ (**4**) and $[(R_2NP)Fe_3(CO)_{10}]$ (**3**) were extracted from this black solid with hexane, until the hexane extracts were colourless. Concentration of these hexane extracts, followed by chromatography on silica gel, first gave the orange $[(R_2NP)_2Fe_3(CO)_9]$ derivatives followed by the dark purple $[(R_2NP)Fe_3(CO)_{10}]$ derivatives. The progress of the separation was monitored by ^{31}P NMR spectroscopy. Further purification could be effected by crystallization from hexane, after subliming away in vacuum any naphthalene impurity carried over from the original $Na_2[Fe_2(CO)_8]$ (prepared from sodium naphthalide). The yields of $[(R_2NP)_2Fe_3(CO)_9]$ and $[R_2NPFe_3(CO)_{10}]$ from these reactions are listed in Table 1. The residue from the original hexane extraction was extracted with dichloromethane; ^{31}P NMR spectra of these dark coloured dichloromethane extracts generally indicated the presence of a complex mixture of products. Attempts to separate pure products from the dichloromethane extracts by chromatography on silica gel led to decomposition, and crystalline products could not be isolated.

Table 1

Products from the reactions of disodium octacarbonyldiferrate with dialkylaminodichlorophosphines

Compound [a]	Color	m.p. (°C)	Yield (%) [b]	^{31}P NMR δ(ppm) [c]	IR ν(CO) (cm^{-1}) in hexane
(A) $[(R_2NP)_2Fe_3(CO)_9]$					
$[(Me_2NP)_2Fe_3(CO)_9]$	orange	190–192	0.4 (E)	406.1	2069w, 2034s, 2014s, 2000s, 1985s, 1977m
$[(C_5H_{10}NP)_2Fe_3(CO)_9]$	orange	168–170	4.0 (E)	402.5	2069w, 2033s, 2014s, 1998s, 1984s, 1975m
$[(Et_2NP)_2Fe_3(CO)_9]$	orange	108–110	0.5 (E)	401.5	2067w, 2035s, 2015s, 1997s, 1982s, 1973m
$[(C_5H_8Me_2NP)_2Fe_3(CO)_9]$	orange	127–128	6.0 (E), 1.4 (T)	402.2	2065w, 2030s, 2010s, 1995s, 1980s, 1975m
$[\{(CHMe_2)_2NP\}_2Fe_3(CO)_9]$	orange	128–129	15.8 (E), 9.3 (T)	399.8	2067w, 2030s, 2013s, 1997s, 1986m, 1978s
$[(Cx_2NP)_2Fe_3(CO)_9]$	orange	141–142	7.6 (T)	403.9	2065w, 2025s, 2008s, 1997s, 1983m, 1975s
$[(C_5H_6Me_4NP)_2Fe_3(CO)_9]$	orange	72–73	24.6 (E), 5.0 (T)	390.6	2065w, 2030s, 2010s, 1993s, 1975s, 1969m
(B) $[(R_2NP)Fe_3(CO)_{10}]$					
$[(C_5H_8Me_2NP)Fe_3(CO)_{10}]$	purple	94–95	8.6 (E), < 1 (T)	500.3	2070m, 2025s, 2015s, 2000s, 1992s, 1983w, 1965m, 1847 m [d]
$[\{(CHMe_2)_2NP\}Fe_3(CO)_{10}]$	purple	> 300	4.8 (E), 12.4 (T)	471.7	2078m, 2036s, 2025s, 2012s, 1993m, 1982w, 1966m, 1844m [d]
$[(Cx_2NP)Fe_3(CO)_{10}]$	purple	170–172	7.0 (T)	474.4	2084m, 2037s, 2025s, 2013s, 1994m, 1981w, 1965w, 1845m [d]

[a] Cx = cyclohexyl; $C_5H_{10}N$ = piperidino; $C_5H_8Me_2N$ = 2,6-dimethylpiperidino; $C_5H_6Me_4N$ = 2,2,6,6-tetramethylpiperidino. [b] E = Et_2O solvent; T = tetrahydrofuran solvent. [c] These spectra were recorded in $CDCl_3$ solutions with proton decoupling. Chemical shifts (δ) are given in ppm downfield from external 85% H_3PO_4. [d] Bridging ν(CO) frequency.

Analytical data

$[(Et_2NP)_2Fe_3(CO)_9]$, $[\{(CHMe_2)_2NP\}_2Fe_3(CO)_9]$ and $[(Cx_2NP)_2Fe_3(CO)_9]$. These known compounds [5,13] were identified by comparison of their ^{31}P NMR and IR spectra {especially $\nu(CO)$}, as well as their melting points, with those reported in the literature.

$[(C_5H_{10}NP)_2Fe_3(CO)_9]$. Found: C, 53.3; H, 3.1; N, 4.3. $C_{19}H_{20}Fe_3N_2O_9P_2$ calc: C, 35.1; H, 3.1; N, 4.3%.

$[(C_5H_8Me_2NP)_2Fe_3(CO)_9]$. Found: C, 39.2; H, 4.0; N, 4.0. $C_{23}H_{28}Fe_3N_2O_9P_2$ calc: C, 39.1; H, 4.0; N, 4.0%.

$[(C_5H_6Me_4NP)_2Fe_3(CO)_9]$. Found: C, 42.9; H, 4.8; N, 3.7. $C_{27}H_{36}Fe_3N_2O_9P_2$ calc.: C, 42.5; H, 4.7; N, 3.7%.

$[C_5H_8Me_2NPFe_3(CO)_{10}]$. Found: C, 34.4; H, 2.5; N, 2.3. $C_{17}H_{14}Fe_3NO_{10}P$ calc: C, 34.5; H, 2.4; N, 2.4%.

$[(CHMe_2)_2NPFe_3(CO)_{10}]$. Found: C, 32.6; H, 2.6; N, 2.4. $C_{16}H_{14}Fe_3NO_{10}P$ calc: C, 33.2; H, 2.4; N, 2.4%.

$[Cx_2NPF_3(CO)_{10}]$. Found: C, 39.9; H, 3.5; N, 2.2. $C_{22}H_{22}Fe_3NO_{10}P$ calc: C, 40.1; H, 3.3; N, 2.1%.

Results

The reactions of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2 in either tetrahydrofuran or diethyl ether were found to give dark brown solutions, shown by their ^{31}P NMR spectra to contain complex mixtures of products. Extractions of the crude mixtures with hexane removed two types of trinuclear iron carbonyl derivatives, which could be separated by column chromatography. The orange, less-strongly adsorbed, $[(R_2NP)_2Fe_3(CO)_9]$ complexes (3) are characterized by a ^{31}P resonance at δ 400 ± 10, six terminal $\nu(CO)$ frequencies, and no bridging $\nu(CO)$ frequencies (see Table 1). These derivatives have been previously [3,5,13] found as minor products from reactions of $Na_2[Fe(CO)_4]$ with R_2NPCl_2. Related alkylphosphorus clusters $[(RP)_2Fe_3(CO)_9]$ (7) have been obtained by a variety of methods [12,13,15–19]. The dark purple, more-strongly adsorbed, $[(R_2NP)Fe_3(CO)_{10}]$ complexes (4) are characterized by a single ^{31}P resonance at δ 485 ± 15, seven terminal $\nu(CO)$ frequencies, and a single bridging $\nu(CO)$ frequency around 1845 cm^{-1} (see Table 1). These clusters are <u>not</u> obtained from reactions of $Na_2[Fe(CO)_4]$ with R_2NPCl_2 derivatives [5]; the dinuclear anion $[Fe_2(CO)_8]^{2-}$ thus appears to be required for the preparation of the $[(R_2NP)Fe_3(CO)_{10}]$ derivatives. However, the analogous cluster $[(RP)Fe_3(CO)_{10}]$ (5; $R = 2,4,6\text{-}Me_3C_6H_2$) has been obtained from the reaction of either $Na_2[Fe(CO)_4]$ or $Na_2[Fe_2(CO)_8]$ with $(2,4,6\text{-}Me_3C_6H_2)PCl_2$ and a variety of other alkyl- and arylphosphorus derivatives $[(RP)Fe_3(CO)_{10}]$ (5) have been prepared by other methods [11,20]. The ^{13}C NMR spectra of both the $[(R_2NP)_2Fe_3(CO)_9]$ and the $[(R_2NP)Fe_3(CO)_{10}]$ complexes prepared from $Na_2[Fe_2(CO)_8]$ and R_2NPCl_2 (Table 2) exhibit appropriate resonances for the R_2N group, as well as one or two discernible carbonyl resonances (in some cases depending on whether the shiftless relaxation reagent [21] $[Cr(acac)_3]$ (acac H: pentane-2,4-dione) is added). Attempts to obtain positive-ion mass spectra of $[(C_5H_8Me_2NP)_2Fe_3(CO)_9]$, $[\{(CHMe_2)_2NP\}_2Fe_3(CO)_9]$, $[(Cx_2NP)_2Fe_3(CO)_9]$, $[\{(CHMe_2)_2NP\}Fe_3(CO)_{10}]$, and $[(Cx_2NP)Fe_3(CO)_{10}]$ under conventional electron-impact conditions failed to give any evi-

Table 2

^{13}C NMR spectra of the trinuclear iron carbonyl derivatives [a]

Compound	δ(CO) (ppm) [b]	δ(R_2N) (ppm)
(A) [($R_2NP)_2Fe_3(CO)_9$]		
[$(Me_2NP)_2Fe_3(CO)_9$]	213.4, 209.0	44.9(CH_3)
[$(C_5H_{10}NP)_2Fe_3(CO)_9$]	213.8, 209.2	54.4(CH_2), 26.2(CH_2), 23.8(CH_2)
[$(C_5H_8Me_2NP)_2Fe_3(CO)_9$]	214.3, 207.5	55.8(CH), 30.1(CH_3), 21.5(CH_2), 13.1(CH_2)
[$\{(CHMe_2)_2NP\}_2Fe_3(CO)_9$]	214.0, 206.5	55.5(CH), 23.1(CH_3)
[$(Cx_2NP)_2Fe_3(CO)_9$]	214.6, 206.8	65.2(CH), 33.9(CH_2), 26.7(CH_2), 25.4(CH_2)
[$(C_5H_6Me_4NP)_2Fe_3(CO)_9$]	216.1	59.3(C), 42.8(CH_2), 30.9(CH_3), 16.3(CH_2)
(B) [$(R_2NP)Fe_3(CO)_{10}$]		
[$(C_5H_8Me_2NP)Fe_3(CO)_{10}$]	212.7	58.1(CH), 30.3(CH_3), 21.5(CH_2), 14.0(CH_2)
[$\{(CHMe_2)_2NP\}Fe_3(CO)_{10}$]	212.9	55.4(CH), 22.3(CH_3), 22.1(CH_3)
[$(Cx_2NP)Fe_3(CO)_{10}$]	212.8	52.8(CH), 31.7(CH_2), 22.7(CH_2), 13.8(CH_2)

[a] These spectra were recorded in $CDCl_3$ solutions with proton decoupling. [b] In some cases addition of the shiftless relaxation reagent [Cr(acac)$_3$] (acac H = pentane-2,4-dione) was necessary to observe the higher field metal carbonyl resonance in the [$(R_2NP)_2Fe_3(CO)_9$] derivatives.

dence for the corresponding molecular ions; only apparent decomposition within the mass spectrometer was observed.

The [$(R_2NP)_2Fe_3(CO)_9$] and [$(R_2NP)Fe_3(CO)_{10}$] complexes in Table 1 are listed in order of increasing size of the dialkylamino group. When small dialkylamino groups such as Me_2N and Et_2N are used, only traces (< 1%) of the corresponding [$(R_2NP)_2Fe_3$-$(CO)_9$] derivatives (**3**) and none of the corresponding [$(R_2NP)Fe_3(CO)_{10}$] derivatives (**4**) were isolated (Table 1). A similar effect has been noted in the reactions of $Na_2[Fe(CO)_4]$ with R_2NPCl_2 [5], where no isolable products were obtained when Me_2NPCl_2 was used. The complicated structures of two trinuclear products obtained from the reaction of $Na_2[Fe(CO)_4]$ with Et_2NPCl_2 indicate dialkylamino transfer from phosphorus to phosphorus and from phosphorus to carbon [4,5]. This suggests that the facile mobility of small dialkylamino groups attached to phosphorus might also be responsible for the very low yields of [$(R_2NP)_2Fe_3(CO)_9$] and [$(R_2NP)Fe_3(CO)_{10}$] from the reactions of Me_2NPCl_2 or Et_2NPCl_2 with $Na_2[Fe_2(CO)_8]$. A comparison of diethyl ether and tetrahydrofuran as solvents for the reaction of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2 derivatives suggests higher overall yields in diethyl ether, rather than tetrahydrofuran, but a greater tendency for the formation of [$(R_2NP)Fe_3(CO)_{10}$] (**4**) relative to [$(R_2NP)_2Fe_3(CO)_9$] (**3**) in tetrahydrofuran rather than diethyl ether.

Some reactions of $Na_2[Fe_3(CO)_{11}]$ [10] with R_2NPCl_2 derivatives were also investigated, since in theory such reactions might be expected to provide a more direct route to the [$(R_2NP)Fe_3(CO)_{10}$] derivatives (**4**). However, attempted reactions of $Na_2[Fe_3(CO)_{11}]$ with Me_2NPCl_2 and with $(CHMe_2)_2NPCl_2$ in tetrahydrofuran solution, under conditions similar to those used for the corresponding reactions with $Na_2[Fe_2(CO)_8]$, gave no evidence for the formation of any [$(R_2NP)Fe_3(CO)_{10}$] or other hexane-soluble organophosphorus iron carbonyl derivatives. We suspect that the lower charge/metal ratio {(−2/3) in $[Fe_3(CO)_{11}]^{2-}$ relative to $[Fe_2(CO)_8]^{2-}$ (−1) and $[Fe(CO)_4]^{2-}$ (−2)} makes the $[Fe_3(CO)_{11}]^{2-}$ anion insufficiently nucleophilic for effective reaction with R_2NPCl_2 to occur.

Discussion

The low yields of $[(R_2NP)_2Fe_3(CO)_9]$ (**3**) and $[(R_2NP)Fe_3(CO)_{10}]$ (**4**) from the reactions of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2 preclude a detailed discussion of the mechanism of this reaction. However, a few general points can be made. In the case of the reactions of $Na_2[Fe(CO)_4]$ with R_2NPCl_2 discussed in detail in a previous paper of this series [5], the initial formation and further reaction of an unstable terminal dialkylaminophosphinidene intermediate $[(R_2NP)Fe(CO)_4]$, related to $[Fe(CO)_5]$, can account for the observed products. In a similar way, formation of an analogous binuclear dialkylaminophosphinidene intermediate $[(R_2NP)Fe_2(CO)_8]$, related to $[Fe_2(CO)_9]$, can be invoked in the reactions of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2. Dimerization of $[(R_2NP)Fe_2(CO)_8]$ followed by elimination of $[Fe(CO)_5]$ and decarbonylation could lead to the observed $[(R_2NP)_2Fe_3(CO)_9]$ products (**3**). The pathway for the conversion of $[(R_2NP)Fe_2(CO)_8]$ to $[(R_2NP)Fe_3(CO)_{10}]$ is more obscure. The failure to obtain any $[(R_2NP)Fe_3(CO)_{10}]$ derivatives (**4**) from the reactions of $Na_2[Fe(CO)_4]$ with R_2NPCl_2, and the failure to obtain $[\{(R_2NP)_2CO\}Fe_2(CO)_6]$ (**1**) or $[(R_2NP)_3Fe_2(CO)_6]$ (**2**) from the reactions of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2 indicates that the reactions of $Na_2[Fe(CO)_4]$ and $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2 do not involve any common intermediates, and in particular that $Na_2[Fe(CO)_4]$ is not generated as an intermediate in the reactions of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2. Further support, by analogy, for the intermediacy of a reactive $[(R_2NP)Fe_2(CO)_8]$ derivative in the reactions of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2 is provided by the observations of the tetranuclear derivatives $[(RP)_2Fe_4(CO)_{12}]$ (**8**) from reactions of $[Et_4N]_2[Fe_2(CO)_8]$ with $RPCl_2$ ($R = Me$, Ph or 4-MeC_6H_4) [12]. Such tetranuclear derivatives can arise from dimerization of an $[(RP)(Fe_2(CO)_8]$ intermediate, followed by loss of four carbonyl groups with the formation of additional iron–iron and iron–phosphorus bonds. The failure to observe any $[(R_2NP)_2Fe_4(CO)_{12}]$ derivatives analogous to **8** in the reactions of $Na_2[Fe_2(CO)_8]$ with R_2NPCl_2 is a further example of the major effect of the dialkylamino group in the reactivity of R_2NPCl_2 with metal carbonyl anions, which has been observed in our studies of the reactions of $Na_2[Fe(CO)_4]$ with R_2NPCl_2 [2–5].

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