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The chemical reactivity of phosphorus-bridging carbonyl compounds: a novel approach to homogeneous catalysis?

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Abstract

The use of a bicyclic metal carbonyl matrix allows the stabilization of phosphorus-bridging carbonyl groups in air-stable, readily synthesized iron carbonyl derivatives of the general type $(R_2NP)_2COFe_2(CO)_6$ (R_2N = *bulky* dialkylamino group, particularly iPr_2N). Such phosphorus-bridging carbonyl derivatives readily undergo extrusion of the phosphorus-bridging carbonyl group at ~100°C to give a $(R_2NP)_2Fe_2(CO)_6$ unit with reactive sites on the phosphorus atoms which can be trapped by a variety of reagents. For example, alcohols trap $(iPr_2NP)_2Fe_2(CO)_6$ as $(ROPN\text{-}iPr_2)(iPr_2NPH)Fe_2(CO)_6$ whereas the $(iPr_2NP)_2Fe_2(CO)_6$ unit can also add to the C≡N of acetonitrile or benzonitrile, the C=O of aldehydes, ketones, or even maleic anhydride, and the C=C of acrylonitrile. The phosphorus-bridging carbonyl extrusion chemistry of $(iPr_2NP)_2COFe_2(CO)_6$ suggests the possibility of designing novel synergistic catalyst systems in which reactive vacant sites are generated at phosphorus atoms adjacent to vacant sites on transition metal atoms. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Phosphorus-bridging; Carbonyl; Transition metal

1. Introduction

Many homogeneous catalyst systems use metal carbonyl precursors in which vacant transition metal sites are generated by loss of one of more carbonyl groups by processes schematically represented by the following equation where [M] represents the central metal with bonded ‘spectator ligands’ such as phosphines, cyclopentadienyl, or other carbonyl groups:

$$[M]{-}CO \rightarrow [M] + CO \quad (1)$$

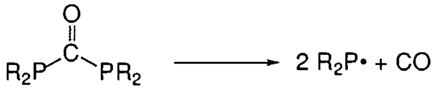
Well-established examples of homogeneous catalytic reactions using catalytic cycles generated from metal carbonyl derivatives include the hydroformylation of olefins [1], the water-gas shift reaction [2] and the carbonylation of methanol to acetic acid [3], summarized by Eqs. (2)–(4), respectively.

$$RCH{=}CH_2 + CO + H_2 \rightarrow RCH_2CH_2CHO \quad (2)$$

$$CO + H_2O \rightarrow CO_2 + H_2 \quad (3)$$

$$CH_3OH + CO \rightarrow CH_3CO_2H \quad (4)$$

A question of general as well as potential practical interest is whether similar reactive vacant sites can be generated by loss of carbonyl groups bonded to main group elements. For example, a carbonyl group bridging two phosphorus atoms can generate a reactive vacant site on each phosphorus atom according to the following general scheme:



Of particular interest are synergistic catalysts arising from simultaneous generation of vacant sites on adjacent transition metals and main group elements. The design of such synergistic catalysts requires a fundamental understanding of the chemistry of carbonyl

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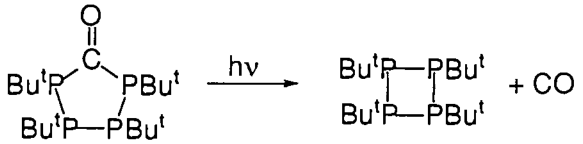
groups bonded to relevant main group elements such as phosphorus. This article summarizes such aspects of the chemistry of compounds containing phosphorus-bridging carbonyl groups developed in the author's laboratory at the University of Georgia during the past dozen years. This article supplements and extends several earlier more general review articles of the author's work in this area published elsewhere [4–6].

2. Synthesis of phosphorus-bridging carbonyl derivatives

The first phosphorus-bridging carbonyl derivative was $O(C(PPh_2)_2$, reported by Becher and Langer [7] to be formed by the low-temperature phosphination of phosgene according to the following reaction:

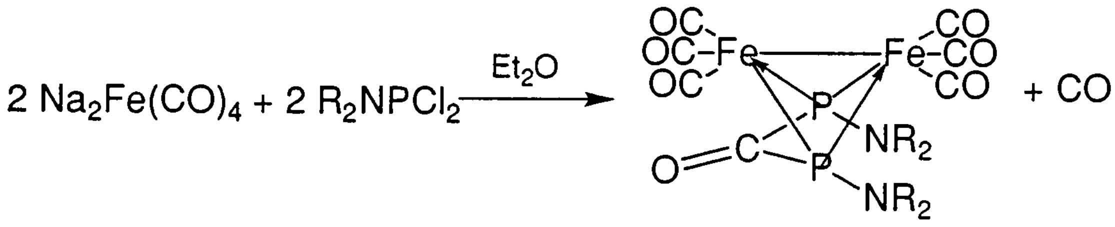
$$O{=}CCl_2 + 2Me_3SiPPh_2 \xrightarrow{Et_2O/-110°C} O{=}C(PPh_2)_2 + 2Me_3SiCl \quad (6)$$

The product, $O{=}C(PPh_2)_2$, is a yellow air-sensitive solid which readily decomposes with evolution of carbon monoxide according to Eq. 5 ($R = C_6H_5$). The phosphorus-bridging carbonyl functionality was subsequently shown by Appel and Paulen [8] to be stabilized by incorporation into a ring system as exemplified by the preparation of the cyclic tert-butyl derivative tBu_4P_4CO (**I**) from the rather complicated reaction of $O{=}CCl_2$ with $tBuP(SiMe_3)_2$. This product (**I**) is a lemon-yellow crystalline solid which is stable to CO extrusion at temperatures up to 180°C in the absence of light but readily undergoes photolytic CO extrusion to give the corresponding cyclotetraphosphine **II** by the following reaction:



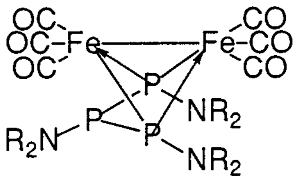
I **II** (7)

The chemistry of compounds containing phosphorus-bridging carbonyl groups received a major impetus with the discovery that the phosphorus-bridging carbonyl group could be greatly stabilized in a suitable bicyclic metal carbonyl matrix. The critical observation was our accidental discovery [9,10] that the reaction of $Na_2Fe(CO)_4$ ('Collman's reagent') with dialkylaminodichlorophosphines, R_2NPCl_2, containing bulky R_2N groups in diethyl ether as a solvent gives phosphorus-bridging carbonyl derivatives of the general type $(R_2NP)_2COFe_2(CO)_6$ (**III**) in 16–35% yields by the following reaction:



III (8)

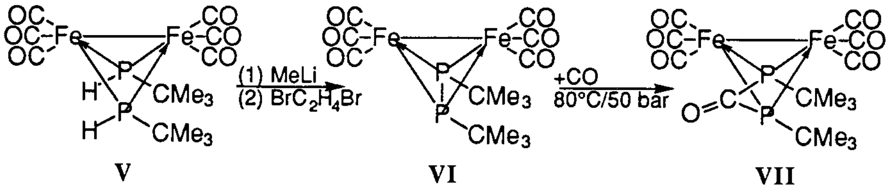
Since the only source of carbonyl groups in this synthesis is the $Na_2Fe(CO)_4$ reagent, this reaction necessarily involves a migration of a carbonyl group from iron to phosphorus. Bulky dialkylamino groups such as R = isopropyl or cyclohexyl or R_2N = 2,2,6,6-tetramethylpiperidinyl are essential for the success of this synthesis since the use of smaller dialkylamino groups such as Et_2N in the R_2NPCl_2 reagent leads to dialkylamino migration to give trinuclear products of the stoichiometries $(R_2NP)_3Fe_3(CO)_{12}$ and $(R_2NP)_3Fe_3(CO)_{11}$ with fairly complicated structures containing $(R_2N)_2P$ structural units [10,11]. In addition, the use of diethyl ether, in which $Na_2Fe(CO)_4$ has limited solubility, is a critical choice of solvent since the corresponding reactions of $Na_2Fe(CO)_4$ with R_2NPCl_2 (R = isopropyl and cyclohexyl) in tetrahydrofuran, in which $Na_2Fe(CO)_4$ is more soluble, give the triphosphine complexes $(R_2NP)_3Fe_2(CO)_6$ (**IV**) rather than the phosphorus-bridging carbonyl derivative **III** as the major product (~ 30% yields).



IV

Most of our studies at the University of Georgia on the chemical reactivity of phosphorus-bridging carbonyl derivatives have been done with the diisopropylamino derivative $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = isopropyl), whose synthesis can be scaled up to give 40 g of product in a single run using the inexpensive iPr_2NH, PCl_3, $Fe(CO)_5$ and sodium as ultimate raw materials [12]. The presence of the phosphorus-bridging carbonyl group in $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = isopropyl) was confirmed directly by an X-ray diffraction study [9,10]. The IR spectra of the phosphorus-bridging carbonyl derivatives $(R_2NP)_2COFe_2(CO)_6$ (**III**) are characterized by five terminal ν(CO) frequencies in the range 2060–1960 cm^{-1} from the $Fe_2(CO)_6$ group and a single bridging ν(CO) bridging carbonyl frequency at 1720 ± 5 cm^{-1} from the PCOP unit.

Shortly after our initial report on $(R_2NP)_2COFe_2(CO)_6$ (**III**) the related phosphorus-bridging carbonyl derivative $(tBuP)_2COFe_2(CO)_6$ (**VII**) was synthesized by De, Wolters and Vahrenkamp [13] by the following sequence of reactions:



(9)

In this synthesis the bis(P–H) compound **V**, readily obtained from $tBuPH_2$ and $Fe_2(CO)_9$ [14], was first metallated with MeLi. The resulting dilithium derivative was then coupled with 1,2-dibromoethane to give the Fe_2P_2 tetrahedrane **VI** as a red solid, which is relatively stable towards air. A carbonyl group can subsequently be inserted into the P–P bond of **VI** by reaction with CO at 80°C/50 bar in an autoclave to give the stable yellow phosphorus-bridging carbonyl derivative $(tBuP)_2COFe_2(CO)_6$ (**VII**), shown by X-ray diffraction to have a structure analogous to **III**.

The synthesis of $(tBuP)_2COFe_2(CO)_6$ (**VII**) on a large scale is considerably more difficult than our synthesis of $(R_2NP)_2COFe_2(CO)_6$ (**III**) [12]. However the potential availability of both **III** and **VII** provides a wider range of metal carbonyl matrices in which to compare the chemical reactivity of the phosphorus-bridging carbonyl group. Interestingly enough, the Fe_2P_2 tetrahedrane $(iPr_2NP)_2Fe_2(CO)_6$ in the diisopropylamino system analogous to $(tBuP)_2COFe_2(CO)_6$ (**VI**) has never been isolated as a stable compound despite a variety of attempts to obtain it by decarbonylation of $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr). This already suggests a major difference between the tert-butyl and diisopropylamino Fe_2P_2 systems.

3. Phosphorus-bridging carbonyl extrusion reactions

The phosphorus-bridging carbonyl derivative $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) is an interesting substrate for a variety of chemical reactions. For example, the phosphorus-bridging carbonyl group in $(iPr_2NP)_2COFe_2(CO)_6$ has ketone-like reactivity towards organolithium reagents to give the corresponding tertiary alcohols $(iPr_2NP)_2C(OH)(R)Fe_2(CO)_6$ (R = Me, nBu, $PhCH_2$) [15]. However, the aspect of the chemistry of $(iPr_2NP)_2COFe_2(CO)_6$ potentially relevant to the design of novel catalyst systems is the loss of its phosphorus bridging carbonyl group upon heating, typically to ~ 100°C, to give a reactive $(iPr_2NP)_2Fe_2(CO)_6$ unit, which can be trapped by a variety of substrates (Fig. 1). Loss of carbonyl groups bonded to iron to give tractable products is never observed under these conditions.

The chemical reactivity of the $(iPr_2NP)_2Fe_2(CO)_6$ unit can be rationalized by its formulation as a phosphorus diradical with an unpaired electron on each phosphorus atom (Fig. 2). This unit can be trapped either by active hydrogen compounds H–X (e.g. alcohols, aniline, or hydrogen chloride) or by addition across multiple bonds Y = Z (e.g. the C=O bonds in aldehydes, ketones, or anhydrides, the C≡N bonds in saturated nitriles, or the

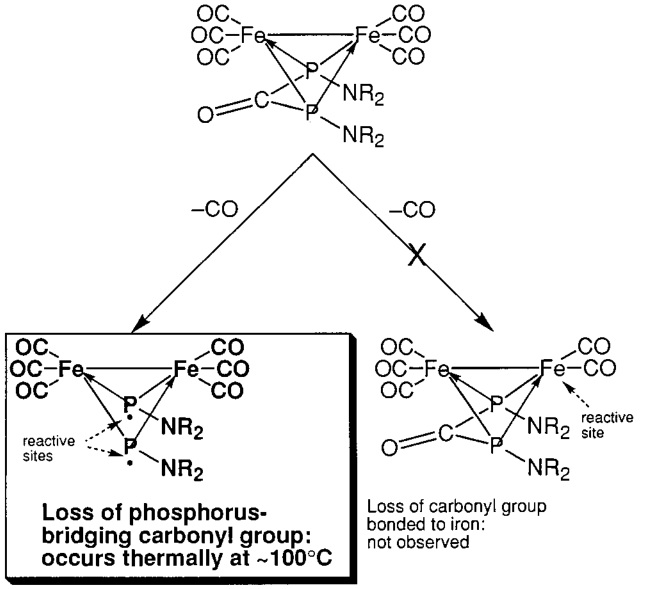


Fig. 1. Extrusion of a carbonyl group from $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr): preference of loss of the PCOP carbonyl group over an FeCO group.

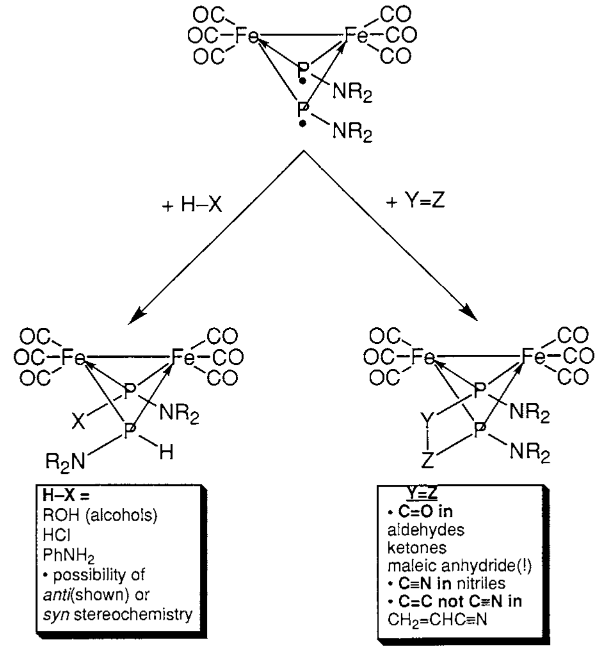


Fig. 2. Chemical reactivity of the $(iPr_2NP)_2Fe_2(CO)_6$ unit towards protic reagents (H–X) and unsaturated compounds (Y = Z).

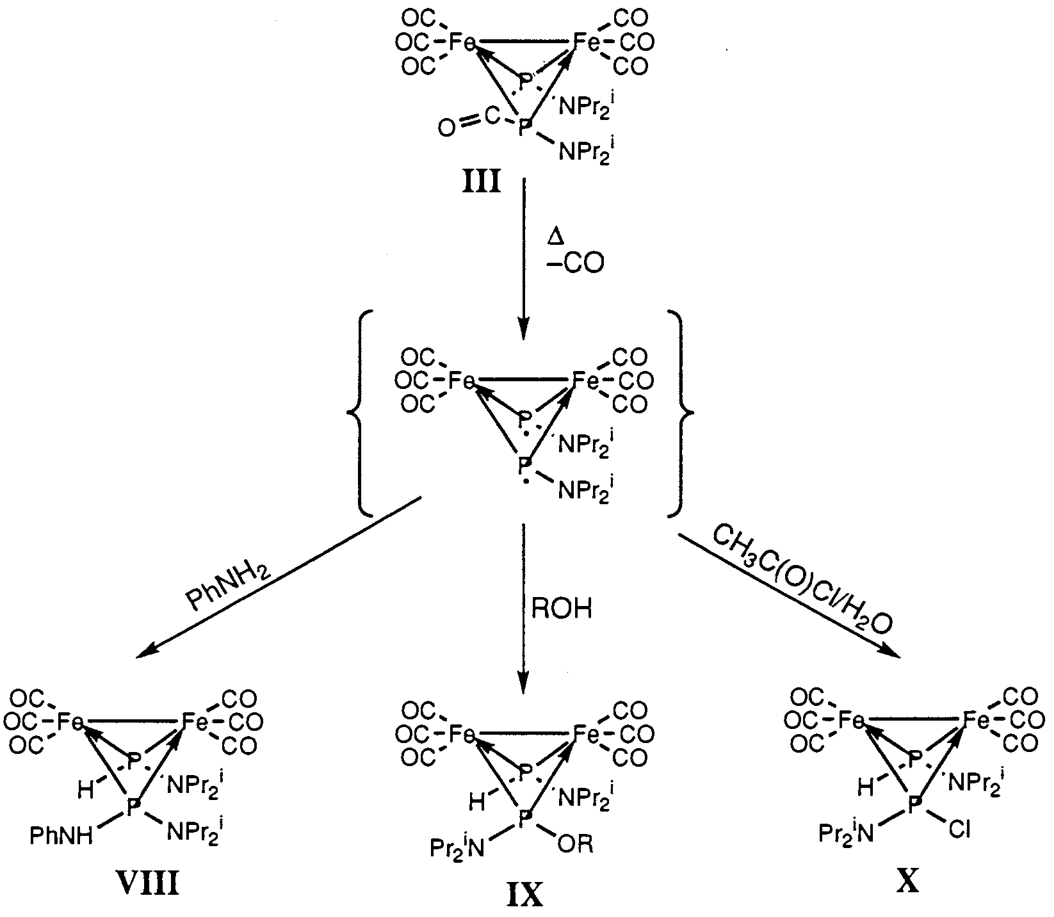
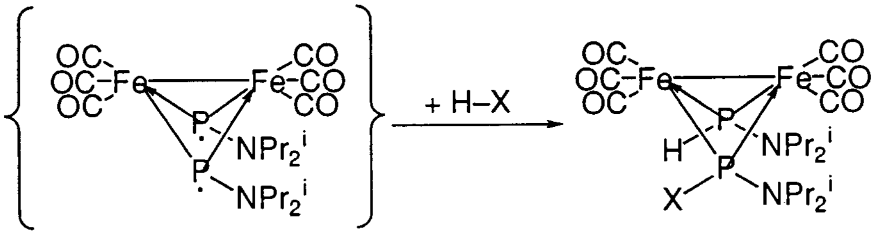


Fig. 3. Reactions of $(iPr_2NP)_2Fe_2(CO)_6$ generated from $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) with the protic reagents $PhNH_2$, ROH (R = Et, Me_3SiCH_2), and HCl (generated from CH_3COCl/H_2O).

C=C bond in acrylonitrile but not maleic anhydride). A stable $(iPr_2NP)_2Fe_2(CO)_6$ tetrahedrane analogous to the tert-butyl derivative **VI** has never been isolated from the pyrolysis or photolysis of $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) in the absence of a trapping agent.

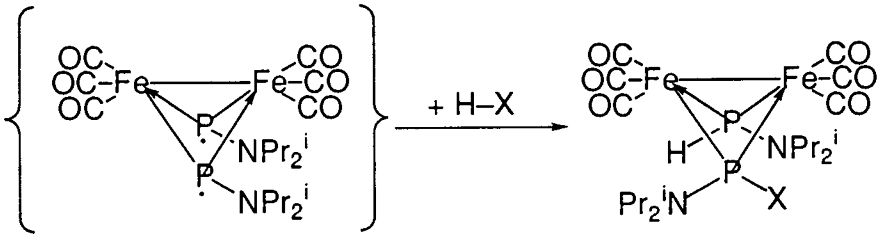
The addition reactions of various H–X derivatives to the $(iPr_2NP)_2Fe_2(CO)_6$ unit generated by carbonyl extrusion from $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) are summarized in Fig. 3. Such addition reactions can proceed according to either *syn* or *anti* stereochemistry as follows (X = NHPh, OR, Cl in the known examples):

***Syn* stereochemistry:**



(10s)

***Anti* stereochemistry:**



(10a)

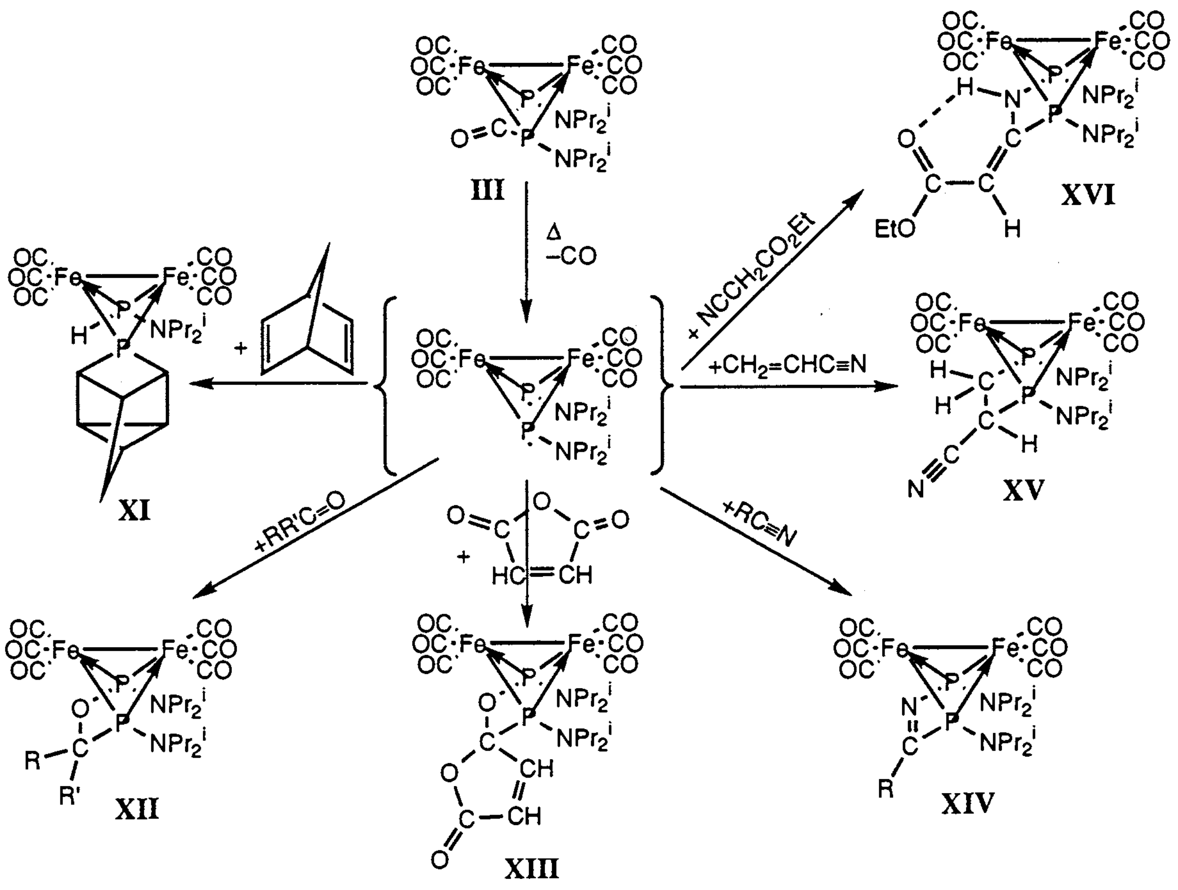


Fig. 4. Reactions of $(iPr_2NP)_2Fe_2(CO)_6$ generated from $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) with diverse unsaturated compounds.

Syn stereochemistry is preferred if loss of the phosphorus-bridging carbonyl and addition of the H–X unit are concerted processes since the pair of sites initially vacated by loss of the phosphorus-bridging carbonyl are necessarily in *syn* positions. However, if the $(iPr_2NP)_2Fe_2(CO)_6$ intermediate has a sufficiently long lifetime, the diisopropylamino groups on its three-coordinate phosphorus atoms can occupy intermediate positions so that either *syn* or *anti* addition of the H–X unit is possible. In fact, formation of the *anti* isomer reduces the steric hindrance between the bulky diisopropylamino groups. The only structurally characterized example of *syn* stereochemistry is found in the product $(iPr_2NPNHPh)(iPr_2NPH)Fe_2(CO)_6$ (Fig. 3: **VIII**) obtained from the thermal reaction of $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) with aniline [16]. Structurally characterized examples of *anti* stereochemistry are found in the products $(ROPN\text{-}iPr_2)(iPr_2NPH)Fe_2(CO)_6$ (Fig. 3: **IX**, R = Et, Me_3SiCH_2) obtained by heating $(iPr_2NP)_2COFe_2(CO)_6$ with alcohols [16]. Similar *anti* stereochemistry is found in the product $(ClPN\text{-}iPr_2)(iPr_2NPH)Fe_2(CO)_6$ (Fig. 3: **X**) obtained by reaction of $(iPr_2NP)_2COFe_2(CO)_6$ with the limited amount of HCl generated from acetyl chloride and adventitious water [16]. In this last reaction the amount of hydrogen halide must be strictly limited since reaction of $(iPr_2NP)_2COFe_2(CO)_6$ with excess hydrogen halide (e.g. HBr) even under ambient conditions results in cleavage of one of the two iPr_2N groups to give a mixture of both stereoisomers of $(iPr_2NPBr)(HBr)Fe_2(CO)_6$ as determined by ^{31}P-NMR. One of these stereoisomers, not yet characterized structurally, can be isolated from the mixture in 45% yield by fractional crystallization [17].

Fig. 4 summarizes reactions of the $(iPr_2NP)_2Fe_2(CO)_6$ unit generated from $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) with a variety of multiply bonded compounds, all of which give products that have been characterized structurally by X-ray diffraction methods. In general, such reactions lead to simple addition of the $(iPr_2NP)_2Fe_2(CO)_6$ unit across the multiple bond with the exception of the reaction with norbornadiene, which gives the nortricyclane derivative $(iPr_2NPH)($*nor*-$C_7H_8P)Fe_2(CO)_6$ (Fig. 4: **XI**) through elimination of a diisopropylamino group possibly involving a phosphinidene-iron carbonyl intermediate of the type $(iPr_2NPH)(P)Fe_2(CO)_6$ [18]. The first examples of additions of the $(iPr_2NP)_2Fe_2(CO)_6$ unit to multiple bonds were the reactions of $(iPr_2NP)_2COFe_2(CO)_6$ with the aldehydes and ketones RR′C=O (R = R′ = H, Ph; R =

Ph, $R' = H$, Me; $R + R' = -(CH_2)_5-$) to give the corresponding $[(iPr_2NP)_2OCRR']Fe_2(CO)_6$ derivatives [19]. X-ray diffraction on the benzaldehyde derivative ($R = Ph$, $R' = H$) indicates structure **XII** (Fig. 4) in which the $(iPr_2NP)_2Fe_2(CO)_6$ unit has added across the C=O of the aldehyde or ketone. The $(iPr_2NP)_2Fe_2(CO)_6$ unit also adds in a similar manner to one of the carbonyl groups in maleic and phthalic anhydrides [18]. The preference of the $(iPr_2NP)_2Fe_2(CO)_6$ unit to add to a C=O rather than the C=C double bond of maleic anhydride to give **XIII** (Fig. 4) is rather surprising since maleic anhydride is a well-known reactive dienophile for the Diels–Alder reaction using its C=C bond. In the structure of the maleic anhydride adduct **XIII**, the bond length of 1.43(3) Å for the C–O single bond formed by addition of the $(iPr_2NP)_2Fe_2(CO)_6$ unit to one of the maleic anhydride carbonyl groups can be compared with the shorter bond length of 1.30(4) Å for the unreacted maleic anhydride C=O double bond.

A variety of addition reactions occur when the $(iPr_2NP)_2Fe_2(CO)_6$ unit is generated in the presence of various nitriles (Fig. 4) [18]. The $(iPr_2NP)_2Fe_2(CO)_6$ unit adds across the C≡N triple bonds of acetonitrile and benzonitrile in a straightforward manner to give **XIV** (R = Me, Ph). X-ray diffraction of the acetonitrile derivative **XIV** (R = Me) indicates that the C≡N bond length of 1.16 Å in free acetonitrile has been increased to 1.279(9)Å as its formal bond order is reduced from three to two in **XIV** (R = Me). In the case of acrylonitrile, the $(iPr_2NP)_2Fe_2(CO)_6$ unit adds preferentially across the C=C rather than the C≡N triple bond to form **XV** in which the length of the C≡N bond, which is not involved in the reaction, remains at 1.14 Å close to the characteristic triple bond value. The preferential addition of the $(iPr_2NP)_2Fe_2(CO)_6$ unit to the C=C rather than the C≡N bond of acrylonitrile is interesting in view of its unusual preferential addition to the C=O rather than the C=C bond in maleic anhydride to form **XIII** discussed above. This information suggests that the reactivity of the $(iPr_2NP)_2Fe_2(CO)_6$ unit towards carbon multiple bonds bearing normal alkyl and aryl substituents decreases in the sequence C=O > C=C > C≡N.

The reaction of the $(iPr_2NP)_2Fe_2(CO)_6$ unit generated from $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) with ethyl cyanoacetate was also investigated in order to provide additional information concerning the relative reactivity of different carbon multiple bonds in this system [18]. However, this reaction was found to follow a very different course with the $(iPr_2NP)_2Fe_2(CO)_6$ unit adding to the C=N double bond of the ketenimine tautomer of ethyl cyanoacetate, namely $HN{=}C{=}CHCO_2Et$, to give a product shown by X-ray diffraction to have the structure **XVI**. The geometry of the pendant $EtCO_2CH$ group and particularly the strongly prolate thermal ellipsoid of its carbonyl oxygen suggest a novel type of O···H–N hydrogen bonding involving the ester carbonyl oxygen and the imine nitrogen. The decreasing carbon–nitrogen bond lengths of 1.43(2) in **XVI**, 1.279(9) in **XIV** and 1.14 Å in **XV** provide an interesting progression as the bond order increases from single in **XVI** to double in **XIV** and triple in **XV**.

4. Towards metal-phosphorus synergism in homogeneous catalysis

The chemistry of $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) shows that reactive sites on its phosphorus atoms can be generated by thermal extrusion of the phosphorus-bridging carbonyl group, typically at temperatures around 100°C. The products isolated from such reactions suggest the intermediacy of $(iPr_2NP)_2Fe_2(CO)_6$ in which any P–P bond, if present at all, is so labile that this species reacts as a diradical (Fig. 2) with reactive sites on both phosphorus atoms. In particular, $(iPr_2NP)_2Fe_2(CO)_6$ does not appear to be a stable isolable species in contrast to the tert-butyl analogue $(tBuP)_2Fe_2(CO)_6$ (**VI**), reported by De, Wolters, and Vahrenkamp [13].

The ability to observe extensive chemistry of the $(iPr_2NP)_2Fe_2(CO)_6$ unit generated from $(iPr_2NP)_2CO$-$Fe_2(CO)_6$ (**III**: R = iPr) is related to the kinetic stability of the 'spectator' $Fe_2(CO)_6$ unit in this system, which apparently does not undergo decarbonylation under any conditions close to where the phosphorus-bridging carbonyl group is extruded. Thus reactive phosphorus sites can be generated in this system under conditions where the iron atoms remain coordinately saturated. A question of interest is whether a phosphorus-bridging carbonyl group with similar extrusion chemistry to that of $(iPr_2NP)_2COFe_2(CO)_6$ can be imbedded into a transition metal matrix containing ligands which are labile under the conditions of extrusion of the phosphorus-bridging carbonyl group. Such systems could provide the basis of synergistic metal–phosphorus catalysts using simultaneously generated vacant sites on both the transition metal and adjacent phosphorus atoms to effect novel catalytic transformations.

A key question in this connection is how the structure of $(iPr_2NP)_2COFe_2(CO)_6$ (**III**: R = iPr) might be modified to provide a potential synergistic metal-phosphorus catalyst system based on chemistry analogous to that reported for $(iPr_2NP)_2COFe_2(CO)_6$. The obvious possibility is the replacement of one or more of the carbonyl ligands in the $Fe_2(CO)_6$ group with more labile ligands such as nitriles, amines, or olefins so that a vacant site on iron can be generated concurrently under conditions where extrusion of the phosphorus-bridging carbonyl takes place. Also of interest is the extent to which the lability of the phosphorus-bridging carbonyl group is influenced by the other phosphorus substituents. The major role that can be played by the spectator groups on phosphorus is illustrated by the stability and isolability of $(tBuP)_2Fe_2(CO)_6$ (**VI**) contrasted with the inability to isolate the analogous $(iPr_2NP)_2Fe_2(CO)_6$ despite a number of attempts at the thermal or photochemical decarbonylation of $(iPr_2NP)_2COFe_2(CO)_6$ in the absence of trapping agents.

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