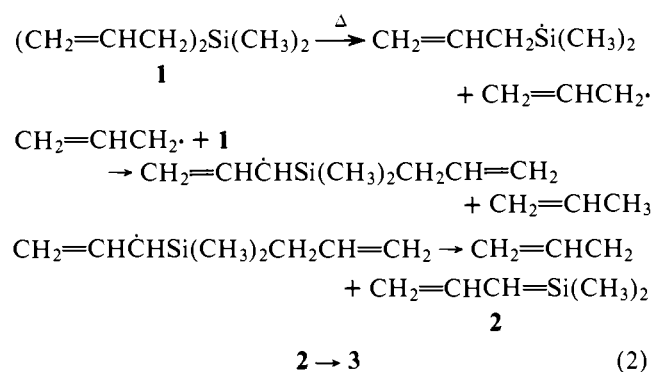


pyrolysis of diverse diallylic silanes are indicated in Table I along with selected spectral data for these new compounds.¹³ The requisite precursors¹³ are readily prepared through reaction of appropriate Grignard reagents and dichlorosilanes.¹⁴ The synthesis can be applied to spirocyclobutenes as well as silacyclobutenes variously substituted on the ring or at silicon, with some limitations. Thus, pyrolysis of diallylsilane itself at 710 °C led to a very complex mixture of products while diallyldimethoxysilane passed through the pyrolysis system unscathed at 710 °C.

A careful GC-MS study of the products from pyrolysis of **1** indicated the absence of 1,5-hexadiene (derived from combination of allyl radicals). This observation argues against significant contributions from a free radical mechanism¹⁵ such as that depicted in eq 2. However, additional kinetic studies would be desirable to confirm the mechanism proposed in eq 1 and provide activation parameters for comparison with retro-ene reactions involving first-row elements. In light of our findings it would also be desirable to reexamine the pyrolysis of allyltrimethylsilane, a reaction whose mechanism has been the subject of controversy.¹⁶ It is clear that the FVP of diallylsilanes represents a valuable new route to silacyclobutenes while the retro-ene reaction of suitably functionalized allylsilanes merits further study as a pyrolytic route to other silaolefinic compounds. We shall report separately on the chemistry of silacyclobutenes and on our efforts to obtain direct spectroscopic evidence for silabutadienes.



Acknowledgment. We gratefully acknowledge support from the donors of the Petroleum Research Fund, administered by the American Chemical Society, and the University of Missouri—St. Louis. We thank Professor William Sherman for the GC-MS studies, Professor Charles Kingsbury for the high resolution MS data, Professor Joyce Corey for helpful discussions, and Professor David Larson for obtaining some of the NMR data.

References and Notes

- (1) Flash Vacuum Pyrolysis Studies. 4. For previous paper in this series, see (a) E. Block, R. E. Penn, R. J. Olson, and P. F. Sherwin, *J. Am. Chem. Soc.*, **98**, 1264 (1976) (part 1); (b) E. Block, H. Bock, S. Mohmand, P. Rosmus, and B. Solouki, *Angew. Chem., Int. Ed. Engl.*, **15**, 383 (1976) (part 2); (c) H. Bock, B. Solouki, S. Mohmand, E. Block, and L. K. Revelle, *J. Chem. Soc., Chem. Commun.*, 287 (1977) (part 3).
- (2) (a) Silacyclopropanes: D. Seyferth, *J. Organomet. Chem.*, **100**, 237 (1975); D. Seyferth, D. P. Duncan, and S. C. Vick, *ibid.*, **125**, C5 (1977), and references therein; (b) silacyclopropenes: R. T. Conlin and P. P. Gaspar, *J. Am. Chem. Soc.*, **98**, 3715 (1976); D. Seyferth, D. C. Annarelli, and S. C. Vick, *ibid.*, **98**, 6382 (1976); M. Ishikawa, T. Fuchikami, and M. Kumada, *ibid.*, **99**, 245 (1977); H. Sakurai, Y. Kamiyama, and Y. Nakadaira, *ibid.*, **99**, 3879 (1977); M. Ishikawa, T. Fuchikami, and M. Kumada, *J. Chem. Soc., Chem. Commun.*, 352 (1977); (c) silacyclobutenes: R. Damrauer, *Organomet. Chem. Rev. A*, **8**, 67 (1972); P. B. Valkovich, T. I. Ito, and W. P. Weber, *J. Org. Chem.*, **39**, 3543 (1974); L. E. Gusel'nikov, N. S. Nametkin, and V. M. Ndovin, *Acc. Chem. Res.*, **8**, 18 (1975); T. J. Barton, G. Marquardt, and J. A. Killgour, *J. Organomet. Chem.*, **85**, 317 (1975); L. H. Sommer and J. McLick, *ibid.*, **101**, 171 (1975); C. M. Golino, R. D. Bush, and L. H. Sommer, *J. Am. Chem. Soc.*, **97**, 7371 (1975), and references therein; (d) silacyclobutenes: H. Gilman and W. H. Atwell, *J. Am. Chem. Soc.*, **87**, 2678 (1965); P. B. Valkovich and W. P. Weber, *Tetrahedron Lett.*, 2153 (1975); M. V. George and R. Balasubramanian, *J. Organomet. Chem. Libr.*, **2**, 103 (1976) (review: "organosilacyclenes"); (e) 1,2-disilacyclobut-3-enes: D. Seyferth and S. C. Vick, *J. Organomet. Chem.*, **125**, C11 (1977), and references therein.
- (3) For an excellent review, see D. Seebach in "Methoden der Organischen Chemie" (Houben-Weyl), Vol. IV/4, E. Müller, Ed., Georg Thieme, Stuttgart, 1971.
- (4) Retro-ene reactions of a variety of molecules of type $(\text{CH}_2=\text{CHCH}_2)_2\text{X}$ have been reported: (a) hepta-1,6-diene, K. W. Egger and P. Vitins, *J. Am. Chem. Soc.*, **96**, 2714 (1974); (b) diallyl sulfide, W. J. Bailey and M. Isogawa, *Am. Chem. Soc., Div. Polym. Chem., Prep.*, **14**, 300 (1963); H. G. Gilles, R. A. Marty, and P. de Mayo, *Can. J. Chem.*, **54**, 537 (1976); (c) diallyl ether, W. J. Bailey and R. A. Malzahn, Abstracts of the 136th National Meeting of the American Chemical Society, Atlantic City, N. J., Sept 1959 p 69-P; P. Vitins and K. W. Egger, *J. Chem. Soc., Perkin Trans. 2*, 1292 (1974); (d) diallyl amine, P. Vitins and K. W. Egger, *Helv. Chim. Acta*, **57**, 17 (1974); K. W. Egger and P. Vitins, *Int. J. Chem. Kinet.*, **6** 371 (1974); R. E. Penn, *J. Mol. Spectrosc.*, in press; (e) a retro-ene process is also suggested for the pyrolysis of allyltrimethylsilane; W. J. Bailey and M. S. Kaufman, 157th National Meeting of the American Chemical Society, Minneapolis, Minn., April 1969, Abstract ORGN-55.
- (5) Fisher Scientific Co.
- (6) A satisfactory flow rate for **1** was achieved by maintaining **1** at -5 °C using a refrigerated cold bath; the pyrolysis products were collected in a series of cold traps at -78 and -196 °C.
- (7) We thank Professor Peter Gaspar for a sample of 1,1-dimethylsilacyclobutane.
- (8) 1,1-Dimethylsilacyclobutane is reported⁹ to have a boiling point of 81 °C at 730 Torr.
- (9) L. H. Sommer and G. A. Baum, *J. Am. Chem. Soc.*, **76**, 5002 (1954).
- (10) L. J. Bellamy, "Advances in Infrared Group Frequencies", Methuen, London, 1958.
- (11) The coupling constant between the olefinic protons in **3** and other silacyclobutenes listed in Table I (7.1–7.3 Hz) is substantially larger than the analogous values for cyclobutenes (2.5–4.0 Hz)⁹ in accord with the trend seen with other silacycloalkenes (the olefinic proton coupling constant in 1-silacyclopent-2-enes is 10 Hz¹² while the analogous value for cyclopentene is 5 Hz⁹).
- (12) M. L. Filleux-Blanchard, N.-D. An, and G. Manuel, *J. Organomet. Chem.*, **137**, 11 (1977).
- (13) All new compounds were fully characterized by an exact mass determination and their IR, NMR, and mass spectra. This information will be reported in detail in the full paper.
- (14) For typical conditions, see E. W. Abel and R. J. Rowley, *J. Organomet. Chem.*, **84**, 199 (1975). It is desirable to use a substantial excess of Grignard reagent to maximize yields.
- (15) See the paper by de Mayo et al., ref 4b.
- (16) See the paper by Gusel'nikov et al., ref 2c.

Eric Block,* Larry K. Revelle

Department of Chemistry, University of Missouri—St. Louis
St. Louis, Missouri 63121

Received September 12, 1977

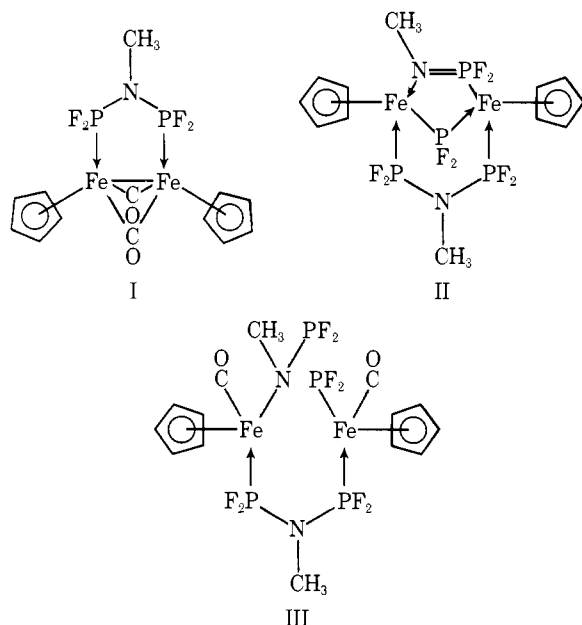
Complete Substitution of Carbonyl Groups in Cyclopentadienyliron Dicarbonyl Dimer by Methylaminobis(difluorophosphine). A Novel Bridging CH_3NPF_2 Ligand Bonded to Metals through Both Phosphorus and Nitrogen

Sir:

Recently we have reported unusual products obtained by reactions of the bidentate ligand $\text{CH}_3\text{N}(\text{PF}_2)_2^1$ with a variety of metal carbonyls and their derivatives including $\text{M}(\text{CO})_6$ ($\text{M} = \text{Cr}, \text{Mo},$ and W),² $\text{Fe}(\text{CO})_5$,³ $\text{Fe}_2(\text{CO})_9$,³ $\text{Fe}_3(\text{CO})_{12}$,^{3,4} $\text{Co}_2(\text{CO})_8$,⁵ and $\text{C}_5\text{H}_5\text{V}(\text{CO})_4$.⁶ This communication reports the pairwise substitution of the four carbonyl groups in $[\text{C}_5\text{H}_5\text{Fe}(\text{CO})_2]_2$ to give successively the dicarbonyl $[\text{C}_5\text{H}_5\text{FeCO}]_2(\text{PF}_2)_2\text{NCH}_3$ and the carbonyl-free complex $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$. The carbonyl-free complex is of more than routine interest since x-ray crystallography indicates an unexpected structure containing a bridging CH_3NPF_2 ligand bonded to one iron atom through nitrogen and to the other iron atom through phosphorus. The complex $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ is thus the first example of a transition metal complex in which an aminodifluorophosphine-derived ligand⁷ is bonded to a transition metal through nitrogen as well as phosphorus.

The complexes⁸ $[\text{C}_5\text{H}_5\text{FeCO}]_2(\text{PF}_2)_2\text{NCH}_3$ (purple crys-

tals, mp 280–282 °C) and $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ (red crystals, mp 162–164 °C) were obtained in poor yields (<10%) by the ultraviolet irradiation of $[\text{C}_5\text{H}_5\text{Fe}(\text{CO})_2]_2$ with $\text{CH}_3\text{N}(\text{PF}_2)_2$ in tetrahydrofuran or pentane and were separated by column chromatography on Florisil, using mixtures of dichloromethane and hexane as eluents. The dicarbonyl $[\text{C}_5\text{H}_5\text{FeCO}]_2(\text{PF}_2)_2\text{NCH}_3$ exhibits a single infrared $\nu(\text{CO})$ frequency at 1735 cm^{-1} in dichloromethane and ^1H NMR resonances at τ 5.16 (s) and 6.53 (t, $J = 7\text{ Hz}$) assigned to the C_5H_5 and CH_3 protons, respectively. These data indicate equivalence of both $\text{C}_5\text{H}_5\text{Fe}$ units in this structure and are completely in accord with the expected structure I completely



analogous to numerous reported⁹ compounds of the general type $[\text{C}_5\text{H}_5\text{FeCO}]_2(\text{diphos})$ obtained from $[\text{C}_5\text{H}_5\text{Fe}(\text{CO})_2]_2$ and various ditertiary phosphines. The carbonyl-free complex $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ exhibits in its ^1H NMR spectrum C_5H_5 resonances of equal relative intensities at τ 5.17 (s) and 5.50 (s) and CH_3 resonances of equal relative intensities at τ 6.95 (t, $J = 7\text{ Hz}$) and 7.57 (dd, $J_1 = 18, J_2 = 4\text{ Hz}$) and in its ^{13}C NMR spectrum C_5H_5 resonances of equal relative intensities at δ 81.6 and 79.2 and weak broad CH_3 resonances of approximately equal relative intensities at δ 37.0 and 27.4. These NMR data indicate nonequivalence of the two $\text{C}_5\text{H}_5\text{Fe}$ units in $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ as well as asymmetry in the CH_3 groups and their couplings to phosphorus. Such asymmetry is unexpected if $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ is a simple substitution product of $[\text{C}_5\text{H}_5\text{Fe}(\text{CO})_2]_2$ in which the four CO groups are pairwise replaced by $\text{CH}_3\text{N}(\text{PF}_2)_2$ ligands. In order to resolve this question the structure of $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ was determined by x-ray crystallography.

The complex $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ forms red monoclinic crystals by slow evaporation at room temperature of a solution in a mixture of dichloromethane and hexane: space group $P2_1/c$; $a = 9.903(2)$, $b = 13.789(3)$, $c = 18.167(2)\text{ \AA}$; $\beta = 128.72(1)^\circ$; $Z = 4$. The structure was solved by direct methods (MULTAN). Least-squares anisotropic refinement using the 2253 observed reflections ($R = 0.0422$, $R_w = 0.0464$) indicates the structure II depicted in detail in Figure 1. The iron–iron distance is 3.646 Å, clearly a nonbonding distance. These two nonbonded iron atoms are bridged by three different ligands: (1) a bridging $\text{CH}_3\text{N}(\text{PF}_2)_2$ ligand, (2) a bridging PF_2 ligand, and (3) a bridging CH_3NPF_2 ligand. Thus one of the two $\text{CH}_3\text{N}(\text{PF}_2)_2$ ligands in $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ has undergone rupture of a phosphorus–nitrogen bond to give separate CH_3NPF_2 and PF_2 units similar to one of the four

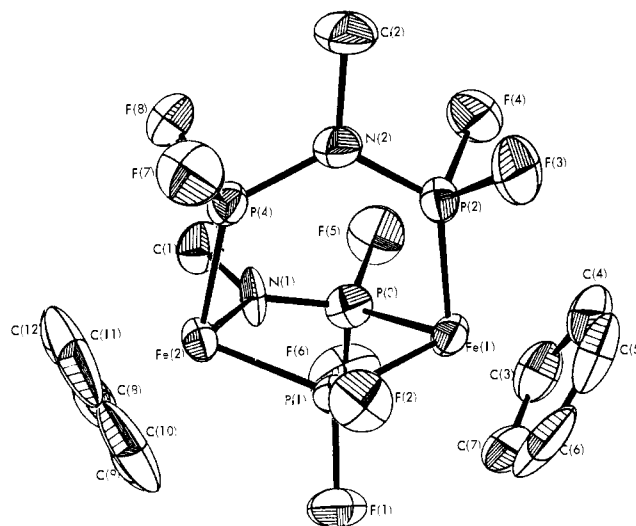


Figure 1. ORTEP drawing of the structure of $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$. Additional pertinent bond distances (ångstroms): Fe(1)–P(1), 2.138 (2); Fe(1)–P(2), 2.066 (2); Fe(1)–P(3), 2.112 (2); Fe(2)–P(1), 2.129 (1); Fe(2)–P(4), 2.061 (2); Fe(2)–N(1), 2.030 (5). Bond angles (degrees): P(1)–Fe(1)–P(2), 91.92 (7); P(1)–Fe(1)–P(3), 85.75 (8); P(2)–Fe(1)–P(3), 92.50 (8); P(1)–Fe(2)–P(4), 92.90 (7); P(1)–Fe(2)–N(1), 88.8 (1); P(4)–Fe(2)–N(1), 89.0 (2).

$\text{CH}_3\text{N}(\text{PF}_2)_2$ ligands in the previously reported⁴ complex $[\text{CH}_3\text{N}(\text{PF}_2)_2]_4\text{Fe}_2\text{CO}$. However, in $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$, unlike $[\text{CH}_3\text{N}(\text{PF}_2)_2]_4\text{Fe}_2\text{CO}$, the CH_3NPF_2 unit formed by phosphorus–nitrogen cleavage of $\text{CH}_3\text{N}(\text{PF}_2)_2$ functions as a bridging ligand bonding to one iron through its nitrogen and to the other iron through its phosphorus. The short P(3)–N(1) distance of 1.521 (3) Å in this bridging CH_3NPF_2 ligand suggests a phosphorus–nitrogen double bond as indicated in II, particularly since the phosphorus–nitrogen distances in the intact $\text{CH}_3\text{N}(\text{PF}_2)_2$ ligand in II are 1.664 (3) and 1.674 (6) Å.

In $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ the cleaved $\text{CH}_3\text{N}(\text{PF}_2)_2$ ligand effectively functions as a six- rather than a four-electron donor since the PF_2 and CH_3NPF_2 fragments in bridging the two iron atoms each donate two electrons to one iron atom and one electron to the another iron atom. An iron–iron bond is therefore no longer necessary for each iron atom to attain the favored rare gas electronic configuration in $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$. Mechanistically, the carbonyl-free complex $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ (II) can form from the dicarbonyl $[\text{C}_5\text{H}_5\text{FeCO}]_2(\text{PF}_2)_2\text{NCH}_3$ (I) through oxidative cleavage of the iron–iron bond in I by a phosphorus–nitrogen bond in the second $\text{CH}_3\text{N}(\text{PF}_2)_2$ ligand to give III followed by displacement of the two remaining carbonyl groups by the distal ends of the terminal PF_2 and CH_3NPF_2 fragments in III. The scope of reactions of the phosphorus–nitrogen bonds in $\text{CH}_3\text{N}(\text{PF}_2)_2$ involving oxidative additions to low-valent transition metal systems similar to the formation of $[\text{C}_5\text{H}_5\text{Fe}(\text{PF}_2)_2\text{NCH}_3]_2$ (II) from $[\text{C}_5\text{H}_5\text{FeCO}]_2(\text{PF}_2)_2\text{NCH}_3$ and excess $\text{CH}_3\text{N}(\text{PF}_2)_2$ (I) remains largely unexplored despite the rich coordination chemistry of $\text{CH}_3\text{N}(\text{PF}_2)_2$ cited above.

Acknowledgment. We are indebted to the Air Force Office of Scientific Research for partial support of this work under Grant AFOSR-75-2869. One of us (J.G.) is indebted to the Program of Cultural Cooperation between the U.S.A. and Spain administered by the Fulbright Commission of Spain for a fellowship.

References and Notes

- J. F. Nixon, *J. Chem. Soc. A*, 2689 (1968).
- R. B. King and J. Gimeno, *J. Chem. Soc., Chem. Commun.*, 142 (1977).
- M. G. Newton, R. B. King, M. Chang, and J. Gimeno, *J. Am. Chem. Soc.*,

99, 2802 (1977).

- (4) M. G. Newton, R. B. King, M. Chang, and J. Gimeno, *J. Am. Chem. Soc.*, in press.
 (5) M. G. Newton, R. B. King, M. Chang, N. S. Pantaleo, and J. Gimeno, *J. Chem. Soc., Chem. Commun.*, 531 (1977).
 (6) R. B. King and K.-N. Chen, *Inorg. Chim. Acta*, **23**, L19 (1977).
 (7) J. F. Nixon, *Adv. Inorg. Chem. Radiochem.*, **13**, 363 (1970).
 (8) The compounds $[C_5H_5FeCO]_2(PF_2)_2NCH_3$ and $[C_5H_5Fe(PF_2)_2NCH_3]_2$ were characterized by correct C, H, N, P, and F analyses and correct molecular weight determinations in benzene solution by vapor pressure osmometry.
 (9) R. J. Haines and A. L. Du Preez, *J. Organomet. Chem.*, **21**, 181 (1970).
 (10) University of Georgia Graduate Fellow, 1976–1978.
 (11) Permanent address: Departamento de Quimica Inorganica, Universidad de Zaragoza, Spain.

M. G. Newton, R. B. King, M. Chang,¹⁰ J. Gimeno¹¹Department of Chemistry, University of Georgia
Athens, Georgia 30602

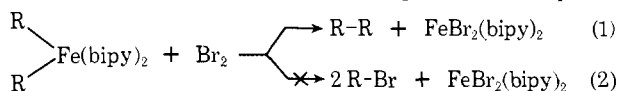
Received November 14, 1977

Reductive Coupling of Organometals Induced by Oxidation. Detection of Metastable Paramagnetic Intermediates

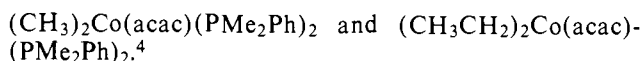
Sir:

The labilization of organometal intermediates is vital to the success of many catalytic organic processes. Indeed, a large number and variety of rather stable alkyl derivatives of transition metals are now known,¹ and it is necessary to find factors which determine how the alkyl-metal bond is selectively cleaved.

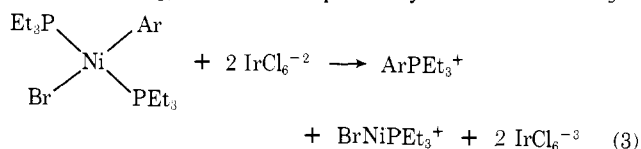
We wish to show that otherwise stable organometal complexes can be induced to undergo facile reductive coupling of a pair of alkyl ligands when subjected to oxidation. For example, dialkyl(bipyridyl)iron(II) complexes, $R_2Fe(bipy)_2$, usually afford a mixture of hydrocarbons consisting mostly of disproportionation products (RH and R(-H)) on thermolysis.² However, these six-coordinate complexes decompose immediately at room temperature when treated with a variety of reagents listed in Table I, especially the 1-equiv oxidants such as hexachloroiridate(IV) and cerium(IV). The most striking result is the high yields of coupled products, R-R, obtained with even halogens.³ Reductive coupling of alkyl groups in-



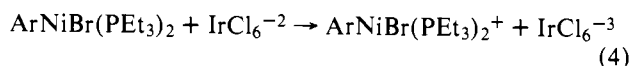
duced in this manner appears to be intramolecular, since no crossover product (propane) is obtained when mixtures of methyl- and ethyliron derivatives are treated. Similar results are obtained with the other transition metal analogues,



Cis and trans, square-planar organonickel(II) complexes also undergo reductive coupling under similar conditions. Thus, *n*-butane is the major product from *cis*-diethyl(bipyridine)-nickel(II), $(CH_3CH_2)_2Ni(bipy)$, when it is treated with either Na_2IrCl_6 , Br_2 , or even O_2 in THF solutions.⁵ Similarly, the thermally stable diarylnickel(II) complexes, *trans*- $Ar_2Ni(PET_3)_2$, afford excellent yields of biaryls when treated with either Na_2IrCl_6 , $CuBr_2$, Br_2 , I_2 , ICl , $Co(III)$, $Ce(IV)$, or $Tl(III)$ trifluoroacetates.⁶ Reductive coupling of ligands is not limited to only metal complexes containing two carbon-centered ligands. When a series of stable monoarylnickel(II) complexes, *trans*- $ArNi(X)(PET_3)_2$, were treated with these reagents, arylphosphonium salts containing $ArPET_3^+$ were formed in excellent yields, with $X = Br, Cl, I, NCS, NO_2$, and NCO .⁷ The arylphosphonium group is also readily eliminated from *trans*- $ArNi(X)(PET_3)_2$ with hexachloroiridate, when $X = CN$ and CH_3 , but it is accompanied by $ArCN$ and $ArCH_3$.^{7b}



The stoichiometry of arylphosphonium elimination induced by hexachloroiridate is represented by eq 3. If the reaction of *o*- $CH_3C_6H_4NiBr(PET_3)_2$ is carried out at $-50^\circ C$, an intense, stable electronic spectrum with λ_{max} 410 nm and ESR spectrum with $g = 2.196$ are observed immediately, independent of whether Na_2IrCl_6 , $Ce(TFA)_4$, or $CuBr_2$ is employed. The g value, however, varies with the aryl group, being shifted to 2.205 and 2.198 with $Ar = o-CH_3OC_6H_4$ and *o*- ClC_6H_4 , respectively. We ascribe these spectral changes to a paramagnetic nickel(III) species⁸ formed by 1-equiv oxidation.⁹



On warming this solution to $-18^\circ C$, the ESR spectrum disappeared irreversibly with a half-life of ~ 20 min, which we associate with reductive elimination from the paramagnetic arylnickel(III) species.¹⁰



Independent experiments show that similar nickel(I) complexes¹¹ are readily converted to nickel(II) derivatives under the conditions of these experiments.

The oxidation of $ArNiBr(PET_3)_2$ in eq 4 does not go to completion, since more than an equivalent amount of Na_2IrCl_6 is required to convert it completely to the paramagnetic nick-

Table I. Induced Decomposition of Dialkyliron(II) Complexes^a

Me ₂ Fe(bipy) ₂ , ^b mmol	Et ₂ Fe(bipy) ₂ , mmol	Reagent	Products, mmol				
			MeH	Me-Me	Me-Et	Et-Et	Other
0.05		Na ₂ IrCl ₆	0.007	0.034			<i>c, d</i>
0.05		Br ₂	0.002	0.030			<i>e</i>
	0.05	Na ₂ IrCl ₆		0.005		0.044	<i>c</i>
	0.05	Br ₂		0.002		0.048	
0.05	0.05	Na ₂ IrCl ₆	0.009	0.040	0	0.051	<i>c, f</i>
0.05	0.05	Br ₂	0.002	0.030	0	0.051	<i>g</i>
	0.05	Cl ₂		0.005		0.046	<i>c</i>
	0.05	I ₂		0.002		0.031	<i>c</i>
	0.05	ICl		0.002		0.043	
	0.05	Tl(TFA) ₃		0.002		0.052	<i>c</i>
	0.05	Ce(TFA) ₄		0.007		0.050	<i>c</i>
	0.05	Co(OAc) ₃		0.003		0.053	<i>c</i>

^a In 3 mL of THF at 25 °C with excess reagent under argon. All reactions occurred on mixing. ^b This compound is difficult to purify and may be slightly impure. ^c Traces (<0.001 mmol) of ethylene detected. ^d No methyl chloride. ^e Methyl bromide (0.011 mmol). ^f No methyl or ethyl chloride. ^g Methyl and ethyl bromides (0.014 and 0.006 mmol).