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Transition Metal Chemistry of Main Group Hydrazides. 4.[†] Phosphorus Hydrazido Ferrocenes as Novel Synthons to New Iron(II)–Palladium(II) Heterotrimetallic Organometallic Compounds. Synthesis and Characterization of Palladium(II) Chloride Complexes of Ferrocene Functionalized Phosphorus Hydrazides. Single-Crystal X-ray Structures of $C_2H_5OP(S)(NCH_3NCHCp'FeCp)_2$ and $C_2H_5OP(S)(NCH_3NCHCp'FeCp)_2{\cdot}PdCl_2$ [Cp′ = C_5H_4, Cp = C_5H_5]

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The reactions of aryl-, alkyl-, and alkoxy-substituted phosphorus hydrazides, $RP(E)(NMeNH_2)_2$ (R = Ph, E = S, **1**; R = OEt, E = S, **2**; R = Ph, E = O, **3**; R = Et, E = O, **4**; R = Me, E = O, **5**) with ferrocenecarboxaldehyde gave metallocene-functionalized phosphorus hydrazides in good yields (~90–95%) as air-stable crystalline solids with the following chemical compositions: $RP(E)(NMeNCHCp'FeCp)_2$ (R = Ph, E = S, **6**; R = OEt, E = S, **7**; R = Ph, E = O, **8**; R = Et, E = O, **9**; R = Me, E = O, **10**). These ferrocenyl phosphorus hydrazides **6–10** can be used as versatile synthons to produce new heterometallic organometallic compounds. For example, **6–10** react with $PdCl_2(PhCN)_2$ at 25 °C to give Fe(II)–Pd(II) trimetallic compounds $RP(E)(NMeNCHCp'FeCp)_2{\cdot}PdCl_2$ (R = Ph, E = S, **11**; R = OEt, E = S, **12**; R = Ph, E = O, **13**; R = Et, E = O, **14**; R = Me, E = O, **15**) in good yields. The chemical constitutions of **6–10** and **11–15** were established by complete NMR (^1H and ^{31}P) spectroscopic and C, H, and N analysis. As representative examples, X-ray structures of **7** and **12** have been determined. Crystal data for $C_2H_5OP(S)(NMeNCHCp'FeCp)_2$, **7**: orthorhombic, space group $P2_12_12_1$, a = 7.5556(11) Å, b = 10.7652(12) Å, c = 33.186(4) Å, V = 2699.3(3) Å^3, Z = 4. The structure was solved by direct methods and was refined to R = 0.038. The structure of **12** comprises the neutral monomeric trimetallic complex $C_2H_5OP(S)(NMeNCHCp'FeCp)_2{\cdot}PdCl_2$ with the Pd(II) center in a square planar geometry. The structure of **12** is characterized by a six-membered ring with the Pd(II) bonded to two terminal hydrazine units of **7** in a *cis* disposition. Crystal data of **12**: monoclinic, space group $P2_1/c$, a = 15.731(3) Å, b = 18.695(3) Å, c = 11.918(2) Å, β = 98.180(7)°, V = 3469.3(10) Å^3, Z = 4. The structure was solved by direct methods and was refined to R = 0.033.

Introduction

The chemistry of organometallic compounds that contain heterobis- and heteropolymetallic units in their backbones continue to attract considerable attention in view of their relevance to catalysis.[2-7] Casey et al.,[8] Rausch et al.,[9] Cullen et al.,[10] Gervais et al.,[11] Stephan et al.,[2] and a host of other workers[12] have pioneered the development of a vast range of heterobimetallic compounds with finely tuned combinations of early and late transition metals. It is believed that the cooperative effects between the two/more metal units in heteropolymetallic compounds may

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have profound influence on the overall properties and reactivities of these complexes.[13] Some times the main group framework which links the dissimilar metals (similar metals also) may act as an innocent spectator, however, there are also reports wherein the bridging ligand is highly susceptible to further transformation resulting in useful metal–metal reactivity patterns.[13]

Of the numerous approaches that have been used to develop heterometallic compounds, the application of ferrocenes as ligating frameworks has received some prominence in recent years because the derivatives of ferrocenes represent a family of easily accessible, easily modifiable, and relatively air-stable ligands.[8,14] Therefore, the utility of ferrocene-based ligands to incorporate additional heterometal centers appears to be a viable pathway to produce multimetallic organometallic compounds.[8,14] Bimetallic compounds derived from ferrocenylbipyridyl ligands and tertiary phosphine-functionalized ferrocene systems are some of the examples of the aforementioned approach to heterobimetallic chemistry using ferrocene ligands.[8,14,15]

As part of our ongoing efforts on the development of new transition metal/organometallic chemistry using novel phosphorus hydrazido and related phosphorus ligands,[16] we are presently interested in the application of phosphorus hydrazido frameworks as ligating units for early–late transition metals with a view to understanding the fundamental coordination chemistry of this class of main group ligands. We report herein a general and versatile synthetic strategy to obtain a new class of bi- and triorganometallic compounds of Fe(II)–Pd(II) metal combinations. This paper will address the synthetic, spectroscopic, and X-ray structural aspects of the Pd(II) complexes of ferrocenyl phosphorus hydrazides.

Experimental Section

All reactions were carried out under anaerobic and anhydrous conditions using prepurified N_2 and conventional Schlenk techniques. Reagents such as ferrocenecarboxaldehyde, $PdCl_2$, $PhP(S)Cl_2$, etc. were purchased from Aldrich Chemical Co. and were used without further purification. $PhP(S)(NMeNH_2)_2$ (**1**) was prepared according to literature methods.[17] The alkyl- and alkoxy-substituted phosphorus hydrazides $RP(E)(NMeNH_2)_2$ (R = OEt, E = S, **2**; R = Ph, E = O, **3**; R = Et, E = O, **4**; and R = Me, E = O, **5**) were prepared by the reaction of $(R)P(E)Cl_2$ (R = OEt, Ph, Et, Me) with methylhydrazine.[18]

Infrared spectra were obtained using Nujol mulls and KBr cells on a Mattson Galaxy 3000 spectrometer. Nuclear magnetic resonance spectra were recorded on a Bruker AMX 500 spectrometer. The ^1H NMR chemical shifts are reported in parts per million (ppm) downfield from external standard $SiMe_4$. The ^{31}P NMR spectra were recorded with 85% H_3PO_4 as an external standard, and positive shifts (ppm) lie downfield of the standard.

Synthesis of Compounds 6–10, RP(E)(NMeNCHCp′FeCp)$_2$ (R = Ph, E = S, 6; R = OEt, E = S, 7; R = Ph, E = O, 8; R = Et, E = O, 9; R = Me, E = O, 10). To a solution of the respective phosphodihydrazide (**1**–**5**) in 50 mL of absolute ethanol was added dropwise (15 min) at 25 °C with stirring a solution of $CpFeC_5H_4CHO$ also in absolute ethanol (50 mL) in a 1:2 stoichiometry (e.g., 2.12 mmol of **1** and 4.30 mmol of $CpFeC_5H_4CHO$). The mixture was stirred for 3 h, during which an orange precipitate is formed. The solution was filtered and the precipitate washed with 3 × 10 mL of absolute ethanol and 2 × 20 mL of diethyl ether. Recrystallization was done from a solution of boiling acetonitrile.

$C_6H_5P(S)(NMeNCHCp'FeCp)_2$ (6): orange powder; yield 89%; mp 168 °C. ^{31}P NMR ($CDCl_3$): 77.84. ^1H NMR ($CDCl_3$): 3.21 (d, $|^3J_{P-H}|$ = 9.8 Hz, 6H, NCH_3), 4.05 (s, 10H, Cp), 4.4–4.2 (m, 8H, Cp′), 7.4 (s, 2H, N=CH), 7.5–8.2 (m, 5H, C_6H_5). Anal. Calcd for $C_{30}H_{31}N_4SPFe_2$: C, 57.9; H, 5.0; N, 9.0. Found: C, 57.8; H, 4.9; N, 8.9. IR (KBr): ν (P=S) 697 cm^{-1}.

$C_2H_5OP(S)(NMeNCHCp'FeCp)_2$ (7): orange powder; yield 82%; mp 138 °C. ^{31}P NMR ($CDCl_3$): 71.80. ^1H NMR ($CDCl_3$): 1.44 (t, $|^3J_{P-H}|$ = 6 Hz, 3H, OCH_2CH_3), 3.21 (d, $|^3J_{P-H}|$ = 10 Hz, 6H, NCH_3), 4.08 (s, 10H, Cp), 4.25–4.53 (m, 8H, Cp′), 4.29 (m, 2H, OCH_2CH_3), 7.40 (s, 2H, N=CH). Anal. Calcd for $C_{26}H_{31}N_4OSPFe_2$: C, 52.9; H, 5.3; N, 9.5. Found: C, 52.7; H, 5.1; N, 9.4. IR (KBr): ν (P=S) 701 cm^{-1}.

$C_6H_5P(O)(NMeNCHCp'FeCp)_2$ (8): orange powder; yield 85%; mp 147 °C. ^{31}P NMR ($CDCl_3$): 24.41. ^1H NMR ($CDCl_3$): 3.2 (d, $|^3J_{P-H}|$ = 12.8 Hz, 6H, NCH_3), 4.14 (s, 10H, Cp), 4.2–4.5 (m, 8H, Cp′), 7.4 (s, 2H, N=CH), 7.4–8.2 (m, 5H, C_6H_5). Anal. Calcd for $C_{30}H_{31}N_4OPFe_2$: C, 59.4; H, 5.2; N, 9.2. Found: C, 59.3; H, 5.0; N, 9.1.

$C_2H_5P(O)(NMeNCHCp'FeCp)_2$ (9): orange powder; yield 89%; mp 68 °C. ^{31}P NMR ($CDCl_3$): 41.73. ^1H NMR ($CDCl_3$): 1.18 (m, 3H, PCH_2CH_3), 2.4 (m, 2H, PCH_2CH_3), 3.18 (d, $|^3J_{P-H}|$ = 7.1 Hz, 6H, NCH_3), 4.1 (s, 10H, Cp), 4.3–4.8 (m, 8H, Cp′), 7.4 (s, 2H, N=CH). Anal. Calcd for $C_{26}H_{31}N_4OPFe_2$: C, 55.9; H, 5.6; N, 10.0. Found: C, 55.7; H, 5.3; N, 9.9.

$CH_3P(O)(NMeNCHCp'FeCP)_2$ (10): orange powder; yield 91%; mp 128 °C. ^{31}P NMR ($CDCl_3$): 38.34. ^1H NMR ($CDCl_3$): 1.98 (d, $|^3J_{P-H}|$ = 16.78 Hz, 3H, PCH_3), 3.16 (d, $|^3J_{P-H}|$ = 6.6 Hz, 6H, NCH_3), 4.1 (s, 10H, Cp), 4.2–4.5 (m, 8H, Cp′), 7.46 (s, 2H, N=CH). Anal. Calcd for $C_{25}H_{29}N_4OPFe_2$: C, 55.2; H, 5.4; N, 10.3. Found: C, 55.0; H, 5.2; N, 10.1.

Synthesis of Trimetallic Fe(II)–Pd(II) Compounds 11–15, RP(E)(NMeNCHCp′FeCp)$_2$·PdCl$_2$ (R = Ph, E = S, 11; R = OEt, E = S, 12; R = Ph, E = O, 13; R = Et, E = O, 14; R = Me, E = O, 15). To a sample of the Schiff base (**6**–**10**) (1.0 mmol) in dichloromethane (25 mL) was added dropwise with stirring at 25 °C a solution of $PdCl_2(PhCN)_2$ (1.0 mmol), also in dichloromethane (25 mL). The mixture was stirred for 2 h before the solvent was removed *in vacuo* to obtain colored microcrystalline solids. This solid was washed first with hexane (3 × 10 mL) and then with diethyl ether (3 × 10 mL). The complexes (**11**–**15**)

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Table 1. Summary of Crystallographic Data of 7 and 12

	7	12
formula	$C_{26}H_{31}N_4OSPFe_2$	$C_{26}H_{31}N_4OSPCl_2Fe_2Pd$
fw	590.28	886.96
space group	orthorhombic, $P2_12_12_1$, (No. 19)	monoclinic, $P2_1/c$ (No. 14)
a, Å	7.5556(11)	15.731(3)
b, Å	10.7652(12)	18.695(3)
c, Å	33.186(4)	11.918(2)
β, deg	–	98.180(7)
V, Å^3	2699.3(3)	3469.3(10)
Z	4	4
λ(Mo Kα), Å	0.70930	0.70930
temp, °C	22(1)	22(1)
d(calcd), g/cm^3	1.453	1.698
crystal size, mm	0.1 × 0.2 × 0.4	0.1 × 0.33 × 0.4
μ, cm^{-1}	12.3	16.1
total no. of data	2197	5095
no. of unique data	2197	4821
no. of obsd data	1759 [$I > 2.0\sigma(I)$]	4042 [$I > 2.5\sigma(I)$]
no. of variables	317	379
$R(F)^aR_1^a$	0.038	0.033
$R_w(F)^bR_2^b$	0.051	0.052

[a] $R = \sum[|F_o| - |F_c|]/\sum|F_o|$. [b] $R_w = [\sum w(|F_o| - |F_c|)^2/\sum|F_o|^2]^{1/2}$, where $w = 1/[\sigma^2|F_o| + 0.0008(F_o)^2]$.

Table 2. Positional Parameters and Their Estimated Standard Deviations for 7

	x	y	z	B_{iso}[a]
Fe(1)	−0.74641(14)	−0.67433(9)	−0.23769(3)	3.32(4)
Fe(2)	−0.72178(14)	−0.27795(12)	−0.42276(3)	3.97(5)
P	−1.0131(3)	−0.77955(23)	−0.39623(7)	4.34(10)
S	−1.1388(4)	−0.90630(24)	−0.36640(10)	6.86(15)
N(1)	−0.7979(9)	−0.7602(6)	−0.38520(21)	4.7(3)
C(1)	−0.6737(14)	−0.8624(10)	−0.3933(3)	6.4(5)
N(2)	−0.7802(8)	−0.6894(5)	−0.35030(19)	3.9(3)
C(2)	−0.6479(9)	−0.7059(7)	−0.3274(3)	3.6(3)
N(3)	−1.1071(8)	−0.6409(6)	−0.39227(20)	3.9(3)
C(3)	−1.1928(11)	−0.5997(8)	−0.35464(25)	4.7(4)
N(4)	−1.0292(8)	−0.5549(6)	−0.41794(19)	3.8(3)
C(4)	−1.0465(10)	−0.4391(9)	−0.40887(25)	4.0(4)
O	−0.9903(8)	−0.8042(5)	−0.44235(18)	5.1(3)
C5	−1.1463(14)	−0.8321(12)	−0.4669(4)	7.4(6)
C6	−1.1022(21)	−0.8219(4)	−0.5087(4)	10.3(8)
C(1′)	−0.6298(10)	−0.6302(8)	−0.29061(23)	3.7(3)
C(2′)	−0.5003(10)	−0.6510(8)	−0.2604(3)	4.3(4)
C(3′)	−0.5272(11)	−0.5654(8)	−0.2288(3)	4.3(4)
C(4′)	−0.6755(12)	−0.4906(7)	−0.2405(3)	4.8(4)
C(5′)	−0.7409(11)	−0.5315(6)	−0.27783(23)	3.9(3)
C(6′)	−0.8800(21)	−0.8345(13)	−0.2454(4)	8.0(7)
C(7′)	−0.7630(18)	−0.8466(9)	−0.2134(5)	7.9(7)
C(8′)	−0.8099(15)	−0.7587(12)	−0.1848(3)	6.4(5)
C(9′)	−0.9553(15)	−0.6930(9)	−0.1987(4)	6.1(5)
C(10′)	−0.9974(14)	−0.7397(13)	−0.2368(4)	7.2(6)
C(1″)	−0.9679(10)	−0.3433(8)	−0.43504(24)	3.7(4)
C(2″)	−0.8536(12)	−0.3648(9)	−0.4681(3)	5.0(4)
C(3″)	−0.7884(12)	−0.2469(10)	−0.4818(3)	5.5(4)
C(4″)	−0.8657(12)	−0.1548(9)	−0.4560(3)	5.2(4)
C(5″)	−0.9721(11)	−0.2143(8)	−0.4282(3)	4.3(4)
C(6″)	−0.5817(22)	−0.3997(13)	−0.3901(7)	8.8(9)
C(7″)	−0.4791(22)	−0.348(3)	−0.4184(5)	9.6(11)
C(8″)	−0.4715(24)	−0.233(3)	−0.4126(7)	11.6(14)
C(9″)	−0.567(4)	−0.1962(17)	−0.3816(8)	12.2(13)
C(10″)	−0.6452(17)	−0.309(3)	−0.3665(4)	12.1(15)

[a] B_{iso} is the mean of the principal axes of the thermal ellipsoid.

were isolated as pure crystalline solids by recrystallizing from boiling acetonitrile.

$C_6H_5P(S)(NMeNCHCp'FeCp)_2{\cdot}PdCl_2$ (11): brown powder; yield 87%; mp < 187 °C dec. ^{31}P NMR (DMSO): 78.55. ^1H NMR (DMSO): 3.45 (d, $|^3J_{P-H}|$ = 9.8 Hz, 3H, NCH_3), 3.92 (d, $|^3J_{P-H}|$ = 12.8 Hz, 3H, NCH_3), 4.18 (s, 10H, Cp), 4.3–5.2 (m, 8H, Cp′), 7.2–8.1 (m, 5H, PC_6H_5), 8.23 (s, 2H, N═CH). Anal. Calcd for $C_{30}H_{31}N_4SPCl_2Fe_2Pd$: C, 45.1; H, 3.9; N, 7.0; Cl, 8.9. Found: C, 44.9; H, 3.8; N, 6.9; Cl, 8.8. IR (KBr): ν (P═S) 647 cm^{-1}.

$C_2H_5OP(S)(NMeNCHCp'FeCp)_2{\cdot}PdCl_2$ (12): brown powder; yield 83%; mp < 161 °C dec. ^{31}P NMR (DMSO): 71.1. ^1H NMR (DMSO): 1.31 (t, $|^3J_{P-H}|$ = 6 Hz, 3H, OCH_2CH_3), 3.69 (d, $|^3J_{P-H}|$ = 14 Hz, 6H, NCH_3), 4.18 (m, 2H, OCH_2CH_3), 4.23 (s, 10H, Cp), 5.2–4.3 (m, 8H, Cp′), 8.28 (s, 2H, N═CH). Anal. Calcd for $C_{26}H_{31}N_4OSPCl_2Fe_2Pd$: C, 40.7; H, 4.1; N, 7.3; Cl, 9.2. Found: C, 40.5; H, 4.0; N, 7.2; Cl, 9.1. IR (KBr): ν (P═S) 698 cm^{-1}.

$C_6H_5P(O)(NMeNCHCp'FeCp)_2{\cdot}PdCl_2$ (13): brown powder; yield 91%; mp < 169 °C dec. ^{31}P NMR ($CDCl_3$): 22.3. ^1H NMR ($CDCl_3$): 3.78 (d, $|^3J_{P-H}|$ = 13.1 Hz, 6H, NCH_3), 4.15 (s, 10H, Cp), 5.4–4.2 (m, 8H, Cp′), 7.4–8.1 (m, 5H, PC_6H_5), 8.3 (s, 2H, N═CH). Anal. Calcd for $C_{30}H_{31}N_4POPdFe_2$: C, 46.0; H, 4.0; N, 7.1; Cl, 9.0. Found: C, 45.8; H, 3.8; N, 7.0; Cl, 8.9.

$C_2H_5P(O)(NMeNCHCp'FeCp)_2{\cdot}PdCl_2$ (14): brown powder; yield 85%; mp < 110 °C dec. ^{31}P NMR ($CDCl_3$): 36.53. ^1H NMR ($CDCl_3$): 1.2 (m, 3H, PCH_2CH_3), 1.6 (m, 2H, PCH_2CH_3), 3.98 (d, $|^3j_{P-H}|$ = 10.2, 6H, NCH_3), 4.16 (s, 10H, Cp), 4.2–5.4 (m, 8H, Cp′), 8.2 (s, 2H, N═CH). Anal. Calcd for $C_{26}H_{31}N_4Cl_2OPFe_2Pd$: C, 42.4; H, 4.2; N, 7.6; Cl, 9.6. Found: C, 42.2; H, 4.2; N, 7.6; Cl, 9.6.

$CH_3P(O)(NMeNCHCp'FeCp)_2{\cdot}PdCl_2$ (15): brown powder; yield 89%; mp < 140 °C dec. ^{31}P NMR ($CDCl_3$): 32.62. ^1H NMR ($CDCl_3$): 1.34 (d, $|^3J_{P-H}|$ = 15.9 Hz, PCH_3), 3.58 (d, $|^3J_{P-H}|$ = 9.8 Hz, 6H, NCH_3), 4.18 (s, 10H, Cp), 4.5–5.6 (m, 8H, Cp′), 8.25 (s, 2H, N═CH). Anal. Calcd for $C_{25}H_{29}N_4OPCl_2PdFe_2$: C, 41.6; H, 4.1; N, 7.8; Cl, 9.8. Found: C, 41.4; H, 4.0; N, 7.7; Cl, 9.8.

X-ray Data Collection and Processing. Orange-colored orthorhombic-shaped crystals of $C_2H_5OP(S)(NMeNCHCp'FeCp)_2$ (**7**) and brown-colored cubic-shaped crystals of $C_2H_5OP(S)(NMeNCHCp'FeCp)_2{\cdot}PdCl_2$ (**12**) were isolated from slow evaporations of their acetonitrile solutions, respectively. All X-ray data were collected on an Enraf-Nonius CAD4 diffractometer with Mo Kα radiation and a graphite monochromator at 22 ± 1 °C. Crystal data and details of data collection are given in Table 1. The positional parameters of compounds **7** and **12** are summarized in Tables 2 and 3, respectively. Selected bond distances and angles of compounds **7** and **12** are summarized in Tables 5 and 6, respectively. All the hydrogen atom parameters were calculated from the idealized geometry. The unit cell dimensions were obtained from a least squares fit to setting angles of 25 reflections. The crystals of **7** and **12** exhibited no significant decay under X-ray irradiation.

The structures were solved by Patterson and Fourier methods and refined by full matrix least squares methods, which minimized $\sum w(|F_o|-|F_c|)$ where $w^{-1} = \sigma^2(\text{counting}) + 0.008(F_o^2)^2/4F_o^2$. Atomic scattering factors which included anomalous scattering contributions were from ref 19. All the hydrogen atoms were introduced in the last step of the refinement procedure in calculated positions. The programs used for crystallographic computations are reported in ref 20. Listings of full experimental details, coordinates, temperature factors, and anisotropic temperature factors are deposited as supplementary material.

Results and Discussion

We have recently described the Schiff base coupling reactions of $PhP(S)(NMeNH_2)_2$ (**1**) with functionalized aldehydes.[16] These reactions have become viable pathways to produce a wide spectrum of ligands with finely tuned functionalities for various reactions with main group and transition metal precursors. In an effort to produce metallated phosphorus hydrazides which can be used as synthons to produce homo- and heteropolymetallic-organometallic compounds, we have discovered a general and straightforward reaction of **1** and similar alkyl- or alkoxy-substituted phosphorus hydrazides [$RP(E)(NMeNH_2)_2$ (R = OEt, E = S, **2**; R = Ph, E = O, **3**, R = Et,

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(19) *International Tables for Crystallography*: Kynoch Press: Birmingham, England, 1974; Vol. IV.

(20) Gabe, E. J.; Lepage, Y.; Charland, J-P; Lee, F. E. J. *Appl. Crystallogr.* **1989**, *22*, 384–387.

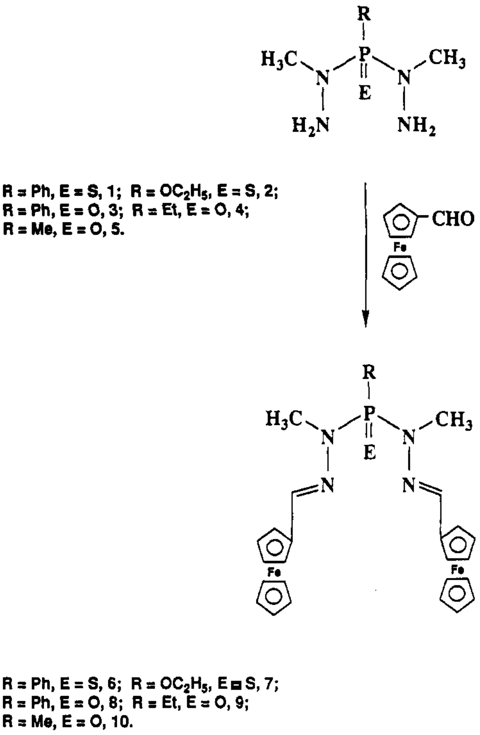
Table 3. Positional Parameters and Their Estimated Standard Deviations for 12

	x	*y*	*z*	B_{iso}[a]
Pd	0.172387(21)	0.617043(17)	0.11312(3)	2.364(16)
Cl(1)	0.12346(8)	0.57248(7)	-0.06414(9)	3.71(5)
Cl(2)	0.25904(9)	0.69929(8)	0.04097(11)	4.65(6)
Fe(1)	0.22714(4)	0.37743(3)	0.26641(6)	2.96(3)
Fe(2)	0.46236(5)	0.66377(4)	0.31567(6)	4.53(4)
P	0.05325(8)	0.63763(6)	0.33723(10)	2.98(5)
S	-0.03811(10)	0.68726(8)	0.39285(14)	5.23(7)
O	0.10191(21)	0.57768(15)	0.41387(24)	3.22(14)
C(1)	0.1379(4)	0.5932(3)	0.5310(4)	4.4(3)
C(2)	0.0852(4)	0.5578(4)	0.6091(4)	5.8(3)
N(1)	0.01972(23)	0.58736(20)	0.2228(3)	3.01(16)
C(3)	0.1152(4)	0.76371(25)	0.2589(5)	4.5(3)
N(2)	0.08987(21)	0.55161(18)	0.1790(3)	2.42(15)
C(10)	0.0853(3)	0.48334(23)	0.1851(3)	2.55(17)
C(11)	0.1464(3)	0.43346(23)	0.1508(4)	2.78(19)
C(12)	0.2328(3)	0.44388(25)	0.1320(4)	3.47(20)
C(13)	0.2672(4)	0.3749(3)	0.1100(5)	4.31(24)
C914)	0.2028(4)	0.3235(3)	0.1158(4)	4.21(25)
C(15)	0.1284(3)	0.35883(25)	0.1417(4)	3.56(20)
C(21)	0.3338(4)	0.3583(5)	0.3798(6)	6.3(3)
C(22)	0.2917(6)	0.4237(4)	0.4083(5)	6.8(4)
C(23)	0.2081(6)	0.4040(4)	0.4266(5)	6.4(4)
C(24)	0.1990(5)	0.3317(4)	0.4120(5)	6.1(3)
C(25)	0.2749(5)	0.3036(4)	0.3840(5)	5.8(3)
N(3)	0.1328(3)	0.69298(19)	0.3126(3)	3.21(16)
C(4)	-0.0413(3)	0.6179(3)	0.1286(5)	4.17(23)
N(4)	0.20262(23)	0.65671(18)	0.2722(3)	2.69915)
C(30)	0.2713(3)	0.65410(24)	0.3462(4)	3.11(20)
C(31)	0.3487(3)	0.61696(25)	0.3355(4)	3.35(20)
C(32)	0.3811(3)	0.5882(3)	0.2392(4)	3.67(22)
C(33)	0.4631(4)	0.5576(3)	0.2765(6)	5.2(3)
C(34)	0.4822(4)	0.5676(4)	0.3949(6)	5.7(3)
C(35)	0.4142(4)	0.6045(3)	0.4330(5)	4.8(3)
C(41)	0.4653(7)	0.7456(6)	0.2077(8)	9.4(5)
C(42)	0.5452(6)	0.7087(5)	0.2199(7)	8.8(5)
C(43)	0.5798(6)	0.7113(6)	0.3258(9)	9.0(5)
C(44)	0.5339(8)	0.7472(5)	0.3876(8)	8.3(5)
C(45)	0.4547(8)	0.7721(4)	0.3222(16)	11.8(9)

[a] B_{iso} is the mean of the principal axes of the thermal ellipsoid.

E = O, 4; R = Me, E = O, 5)] with ferrocenecarboxaldehyde. The reactions illustrated in Scheme 1 present a facile pathway to produce bis-ferrocene phosphorus hydrazides: $(R)P(E)(NMeNCHCp'FeCp)_2$ (where R = Ph, E = S, **6**; R = OEt, E = S, **7**; R = Ph; E = O, **8**; R = Et, E = O, **9**; R = Me, E = O, **10**) in excellent yields (~90–95%) as air-stable and hydrolytically robust organometallic compounds. The ferrocene-appended main group compounds **6**–**10** belong to a new class of multiferrocene organometallic frameworks. Their chemical composition was established by C, H, and N analysis. Complete NMR (^1H and ^{31}P (see Table 4)) spectroscopic analysis of **6**–**10** gave structural insights into the electronic features of this class of phosphorus hydrazido metallocenes.

Scheme 1



Molecular Structure of $C_2H_5OP(S)(NMeNCHCp'FeCp)_2$ (7). As a representative example, we have undertaken the X-ray structural analysis of $EtOP(S)[NMeNCHCp'FeCp]_2$ (**7**). The ORTEP plot is shown in Figure 1 and the selected bonding parameters are shown in Table 5. The structure of **7** comprises two ferrocenyl units each attached to the terminal nitrogens of the phosphorus hydrazide through the C=N bonds (Figure 1). The parent phosphorus hydrazido precursor $EtOP(S)(NMeNH_2)_2$ (**2**) is an oil and, therefore, precludes bonding comparisons to be made between **2** and its ferrocene adduct **7**. Assuming that the substituents on the phosphorus will have little or no impact on the N–N bond lengths in these type of phosphorus hydrazido compounds,[16] comparisons of bonding features of a phenyl analogue of **2** (e.g., $PhP(S)(NMeNH_2)_2$ (**1**)) and the ferrocene adduct **7** were made. For example the P–S bond in **7** (P = S 1.935(4) Å) is slightly shorter compared to its length in **1** (P=S 1.947(7) Å). The differences in the P–N bond lengths in **7** (N3–N4 = 1.389(9) Å; N1–N2 = 1.393(9) Å) are noticeably shorter than those found in the free phosphorus hydrazide **1** (N3–N4 = 1.446(4) Å; N1–N2 = 1.478(7) Å). The NPN angle in **7** is ~4° wider compared

Table 4. ^1H[a] and ^{31}P[b] NMR Spectroscopic Data[c] of Ferrocenyl Phosphorus Hydrazides (6–10) and Their Pd(II) Complexes (11–15)

compounds	^1H NMR: δ (^1H) ($^3J_{(P-H)}$, (Hz)) PNCH$_3$ region	^{31}P NMR: δ ^{31}P
$PhP(S)(NMeNCHCp'FeCp)_2$ (**6**)	3.21(9.8)	77.84
$EtOP(S)(NMeNCHCp'FeCp)_2$ (**7**)	3.21(10.0)	71.8
$PhP(O)(NMeNCHCp'FeCp)_2$ (**8**)	3.2(12.8)	24.41
$EtP(O)(NMeNCHCp'FeCp)_2$ (**9**)	3.18(7.1)	41.73
$MeP(O)(NMeNCHCp'FeCp)_2$ (**10**)	3.16(6.6)	38.34
$PhP(S)(NMeNCHCp'FeCp)_2 \cdot PdCl_2$ (**11**)	3.45(9.8); 3.92(12.8)	78.55
$EtOP(S)(NMeNCHCp'FeCp)_2 \cdot PdCl_2$ (**12**)	3.69(14)	70.1
$PhP(O)(NMeNCHCp'FeCp)_2 \cdot PdCl_2$ (**13**)	3.78(13.1)	22.31
$EtP(O)(NMeNCHCp'FeCp)_2 \cdot PdCl_2$ (**14**)	3.98(10.2)	36.53
$MeP(O)(NMeNCHCp'FeCp)_2 \cdot PdCl_2$ (**15**)	3.58(9.8)	32.62

[a] Spectra either in $CDCl_3$ or DMSO; ppm vs $SiMe_4$. [b] Spectra either in $CDCl_3$ or DMSO; ppm vs 85% H_3PO_4. [c] Values quoted are those determined at normal probe temperatures.

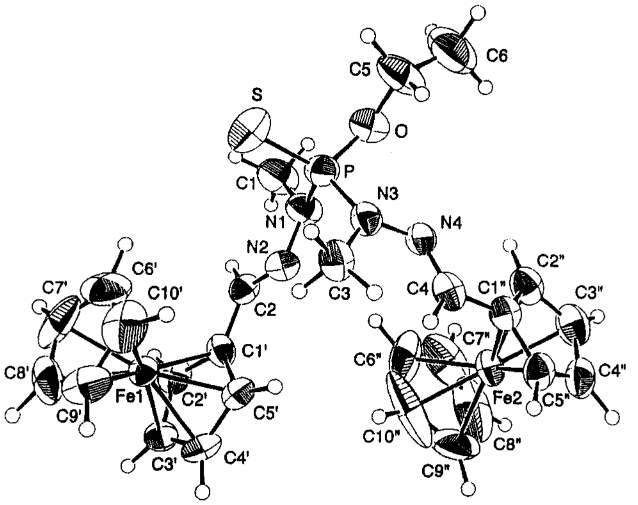


Figure 1. ORTEP drawing of the molecular structure of compound 7 with 50% thermal ellipsoids.

Table 5. Selected Bond Length and Bond Angle Data for 7

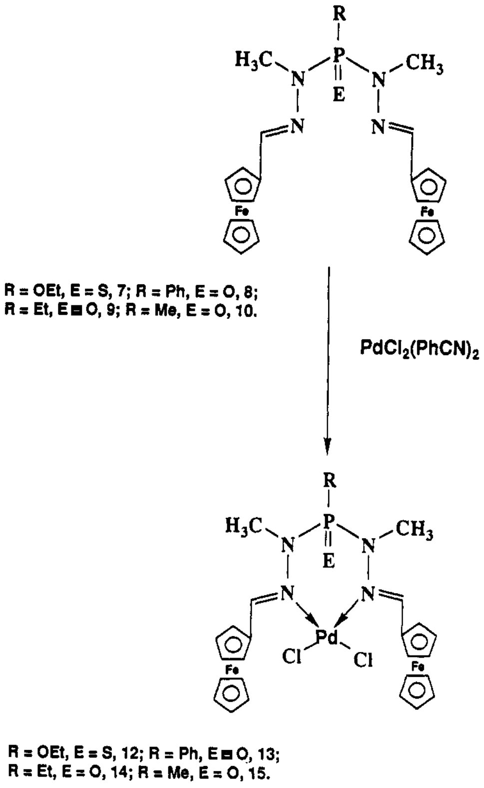
	Bond Lengths (Å)		
P–S	1.935(4)	C(2)–C(1′)	1.474(12)
P–N(1)	1.679(7)	N(3)–C(3)	1.475(11)
P–N(3)	1.658(7)	N(3)–N(4)	1.389(9)
P–O	1.563(6)	N(4)–C(4)	1.290(12)
N(1)–C(1)	1.471(12)	C(4)–C(1″)	1.473(12)
N(1)–N(2)	1.393(9)	O–C(5)	1.463(11)
N(2)–C(2)	1.268(10)	C(5)–C(6)	1.432(19)
	Bond Angles (deg)		
S–P–N(1)	116.8(3)	C(1)–N(1)–N(2)	120.0(7)
S–P–N(3)	112.6(3)	N(1)–N(2)–C(2)	119.9(6)
S–P–O	115.8(3)	N(2)–C(2)–C(1′)	119.5(7)
N(1)–P–N(3)	106.6(3)	P–N(3)–C(3)	121.7(5)
N(1)–P–O	97.3(3)	P–N(3)–N(4)	111.7(5)
N(3)–P–O	106.1(3)	C93)–N(3)–N(4)	120.4(6)
P–N(1)–C(1)	119.0(6)	N(3)–N(4)–C(4)	117.3(7)
P–N(1)–N(2)	110.0(5)	N(4)–C(4)–C(1″)	119.9(7)

Table 6. Selected Bond Length and Bond Angle Data for 12

	Bond Lengths (Å)		
Pd–Cl(1)	2.299(1)	O–C(1)	1.459(6)
Pd–Cl(2)	2.301(1)	C(1)–C(2)	1.486(8)
Pd–N(2)	2.022(3)	N(1)–N(2)	1.449(5)
Pd–N(4)	2.028(3)	N(1)–C(4)	1.484(6)
P–S	1.907(2)	C(3)–N(3)	1.478(6)
P–O	1.574(3)	N(2)–C(10)	1.281(5)
P–N(1)	1.678(4)	N(3)–N(4)	1.432(5)
P–N(3)	1.681(4)	N(4)–C(30)	1.295(6)
	Bond Angles (deg)		
Cl(1)–Pd–Cl(2)	92.14(5)	P–O–C(1)	120.3(3)
Cl(1)–Pd–N(2)	89.31(10)	O–C(1)–C(2)	109.6(4)
Cl(1)–Pd–N(4)	174.05(11)	P–N(1)–N(2)	112.6(3)
CL(2)–Pd–N(2)	175.25(10)	P–N(1)–C(4)	119.6(3)
Cl(2)–Pd–N(4)	91.85(10)	N(2)–N(1)–C(4)	110.4(3)
N(2)–Pd–N(4)	86.36(13)	Pd–N(2)–N(1)	115.13(25)
S–P–N(3)	112.18(14)	Pd–N(2)–C(10)	132.0(3)
O–P–N(1)	98.25(17)	N(1)–N(2)–C(10)	112.8(3)
O–P–N(3)	103.11(19)	N(2)–C(10)–C(11)	125.8(4)
N(1)–P–N(3)	111.01(19)	S–P–O	118.24(13)
		S–P–N(1)	112.94(15)

to this angle in the non-ferrocene phosphorus hydrazide analogue 1, presumably a consequence of the interelectronic repulsions and the steric effects of the ferrocenyl substituents in 7. The angles of ~119° around the imine carbons in 7 (119.5(7)° for N2–C2–C1′ and 119.9(7)° for N4–C4–C1″) appear to indicate limited or no steric crowding around the imine nitrogen N2 or N4 due to the bulky ferrocenyl groups. This observation may present a potential of using these imine nitrogens for further

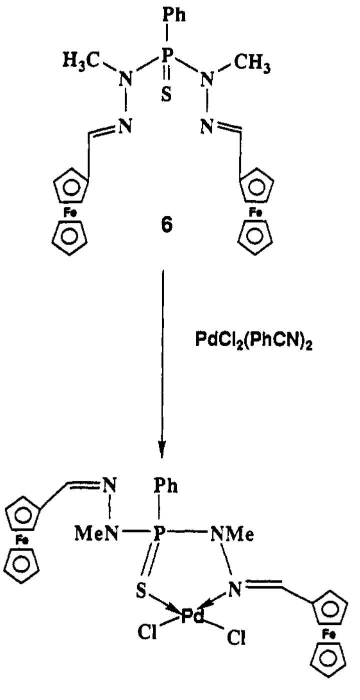
Scheme 2



R = OEt, E = S, 7; R = Ph, E = O, 8;
R = Et, E = O, 9; R = Me, E = O, 10.

R = OEt, E = S, 12; R = Ph, E = O, 13;
R = Et, E = O, 14; R = Me, E = O, 15.

Scheme 3

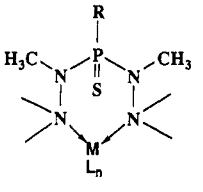


6

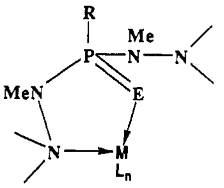
11

coordination with metal centers to produce new heterometallic compounds.

Ligating Properties of Phosphorus Hydrazido Ferrocenes 6–10: Formation of New Heterometallic Compounds. The reactions of phosphorus hydrazido ferrocenes 6–10 with $PdCl_2(PhCN)_2$ were carred out in CH_2Cl_2 at 25 °C to produce a new class of trimetallic organometallic compounds in good yields as outlined in Schemes 2 and 3. The bis-ferrocene palladates 11–15 have a dark-brown metal-like, shiny appearance and give intensely colored orange-brown solutions in CH_2Cl_2/$CHCl_3$. These trimetallic compounds are air-stable with little/no hydrolysis occurring in solid/liquid samples when exposed to atmospheric moisture for 24 h. The chemical constitutions of all the new compounds **11**–**15** were established by C, H, N, and Cl analysis. The spectroscopic (^1H and ^{31}P NMR) data described in the following sections (Table 4) clearly distinguishes the NN′ coordination with Pd(II) (as shown for **12**–**15** in Scheme 2) versus the NS coordination observed in **11** (Scheme 3).



A



B

^1H, ^{31}P NMR and IR Spectroscopy of 11–15. The interaction of Pd(II) precursors with **6**–**10** can occur in two possible ways: (1) one that involves the coordination of the two hydrazinic nitrogens (structure A); and (ii) the formation of a five-membered metallacyclic ring through the coordination of P(S) or P(O) chalcogenides and one of the terminal hydrazido nitrogens with the Pd(II) center as shown in structure B. The ^1H NMR spectra of the ligands **6**–**10** showed doublet signals (Table 4) for the NCH_3 protons due to the coupling with the P(V) center across three bonds ($^3J_{P-H}$ ~6–12 Hz). The coordination of the two temrinal hydrazido nitrogens with Pd(II) results in a symmetric structure (structure A) and should give a single chemical environment for the NCH_3 protons in the ^1H NMR spectra. In fact, a close look at the proton NMR signals revealed that the compounds **12**–**15** showed a single doublet for the NCH_3 protons signifying that these complexes belong to structure A type. However, the ^1H NMR spectrum of the trimetallic complex **11** consisted of two doublets of equal intensities centered in the region δ 3.92 and 3.45 which may be attributed to the NCH_3 protons present in the metal-coordinated and -uncoordinated hydrazine arms, respectively, as in structure B. The P=S coordination in **11** with the Pd(II) center results in a modest deshielding effect on the ^{31}P chemical shifts as compared to the free phosphorus hydrazido ligand **6** (Table 4).[16] This effect is, presumably, a consequence of withdrawal of electronic charge from the electron-rich Pd(II) center. On the other hand, the NN′ coordination as observed in **12**–**15** causes a shielding in the chemical shifts of phosphorus in the trimetallic complexes **12**–**15** as compared to the corresponding free ligands **7**–**10**, respectively (Table 4). The lack of P–E–M (E = S or O) interaction in **12**–**15** may push the residual electron density from the electron-rich Pd(II) unit on the P^v center via the smooth $p\pi$–$p\pi'$ N–N hydrazido bond causing shielding of the ^{31}P nucleus. Comparison of the ν (P=S) stretching frequency of the free ligand **6** with that of its corresponding Pd(II) complex **11** proved to be diagnostic of the metal coordination with the P=S unit. The ν (P=S) in **6** consisted of a band centered at 697 cm^{-1} and this band moved to lower wavenumbers by 50 cm^{-1} in the metal complex **11**: ~647 cm^{-1} indicating the P=S → M coordination. However, the ν P=S did not change on going from the free ligand **7** to its Pd(II) complex **12**. This observation is consistent with the formulation for the complex **12** (Scheme 2) in which the P=S unit is not involved in coordination with the Pd(II) center.

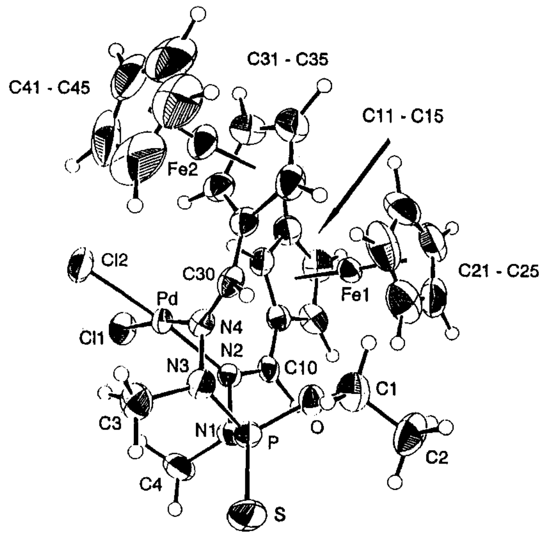


Figure 2. ORTEP drawing of the molecular structure of compound **12** with 50% thermal ellipsoids.

Molecular Structure of $C_2H_5OP(S)(NMeNCHCp'FeCP)_2{\cdot}PdCl_2$ (12). The NN′ vs the SN coordination as noted above prompted us to undertake the X-ray structural analysis of **12** which according to the description of the spectroscopic data belonged to class A structure. Crystals suitable for an X-ray structure determination of **12** were obtained by slow crystallization from acetonitrile at 25 °C. As expected, the structure of **12** shown in the form of an ORTEP plot (Figure 2) consists of a six-membered cyclopalladaphosphohydrazide with Pd(II) bonded to two terminal hydrazido nitrogens. The sulfur atom of the phosphorus chalcogenide (P=S) is not involved in the coordination indicating a contrasting bonding feature as compared to structures of the type B. The square planar geometry of Pd(II) in **12** is further characterized by a *cis* disposition of the hydrazido nitrogens bonded to it. The N–N bonds in **12** (N1–N2 = 1.449(5) Å; N3–N4 = 1.432(5) Å; Table 4) have suffered elongation compared to these distances in the free ligand **7** (e.g., N1–N2 = 1.393(9) Å; N3–N4 = 1.389(9) Å), as a result of coordination to the Pd(II) center.[21] The accommodation of the Pd(II) unit across the two terminal hydrazido units has, presumably, caused widening of the NPN angle by ~5° in **12** as compared to the free ligand **7**.

The presence of alkoxy groups on the phosphorus (e.g., in **7**) seems to decrease the nucleophilicity of the adjacent P=S group. Therefore, the alkoxy-substituted phosphorus hydrazides, as in **7** and in a number of similar phosphorus hydrazides, have shown NN′ coordination with the Pd(II) center. It is noteworthy that the phosphine oxide-containing phosphorus hydrazides, as demonstrated in the present investigation, have shown NN′ coordination with the Pd(II) metal center. This may also be rationalized in terms of the narrow NPN bond angles in compounds containing the P=O unit as compared to the similar angles in P=S bearing phosphorus hydrazides.

Conclusions

Functionalization of ferrocene on phosphorus hydrazides as demonstrated in the present investigation has opened

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(21) Katti, K. V.; Singh, P. R., unpublished results.

a new approach to the chemistry of organometallic-based main group frameworks (Scheme 1). The high kinetic stability of the phosphorus hydrazido ferrocenes **6–10** is an important feature in the context of the application of these compounds as a new class of chemical precursors to homo- and heteropolymetallic organometallic compounds. The incorporation of Pd(II) in **6–10** as presented in Schemes 2 and 3 to produce new Fe(II)–Pd(II) trimetallic compounds provide illustrative examples of the extensive organometallic chemistry that can be derived through these compounds. The utility of the ferroceno phosphorus hydrazides to produce a new range of trimetallic compounds akin to **11–15** with other electron-rich late transition metals (e.g.; Cu(I), Cu(II), Pt(II), and Rh(III)) are underway.

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Supplementary Material Available: Structure determination summary with tables of atomic coordinates, bond lengths, bond angles, anisotropic displacement coefficients, and H atom coordinates for $C_2H_5OP(S)(NMeNCHCp'FeCp)_2$ and $C_2H_5OP(S)(NMeNCHCp'FeCp)_2{\cdot}PdCl_2$ (8 pages). Ordering information is given on any current masthead page.

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